

Synthesis, Characterization, and Antibacterial effect of Complexes lead(II) and Tin (II) With (L-leucin and 8-hydroxyquinoline)

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Abstract:

The research includes the synthesis and identification of two types complexes of M^{+2} Ions using amino acid (L-leucin) and 8-hydroxyquinoline as ligands in general compositions $[M(\text{Leu})_2]$ and $[M(\text{Q})_2]$ were L-leucin ($\text{C}_6\text{H}_{13}\text{NO}_2$) symbolized as (LeuH), and 8-hydroxyquinoline, ($\text{C}_9\text{H}_7\text{NO}_2$) symbolized as (8-HQ) $\text{M}(\text{II}) = \text{Sn}(\text{II})$ and $\text{Pb}(\text{II})$, The ligand and metal ions were brought into reaction in ethanolic medium using (1:2) metal : ligand molar ratios as required.

Products were found to be solid crystalline complexes, which have been characterized through the following techniques: Melting points, Solubility, Molar conductivity. Determination the percentage of the metal in the complexes by (Atomic Absorption Spectrometry). chloride ion content were also evolution by (mohr method) . Spectroscopic Methods [FT-IR and UV-Vis]. The proposed structure of the complexes using program , chem office 3D(2006) .

INTRODUCTION :

8-Hydroxyquinoline is an organic compound with the formula $(\text{C}_9\text{H}_7\text{NO})$. It is a derivative of the heterocyclic by placement of an OH group on carbon number 8. [1-2]. The amino acid L-leucin and various transition metals are important in the biological functions of humans, animals, and plants. L- Leucin is one of the twenty major amino acids and is considered an essential and aliphatic amino acid.[3-4]

Ganesh and Co-worker[5] were prepared and characterized of mixed ligand di-oxuranium(VI) and thorium(IV) complexes of 8-hydroxyquinoline as a primary ligand and amino acids such as L-proline (ProH) and 4-hydroxy-L-proline (Hyp) as secondary ligands. These complexes have been screened for their antibacterial and cyto-toxic (IC_{50}) characteristic properties .

Antibacterial activity has been studied more than antifungal activity. Because bacteria can achieve resistance to antibiotics through biochemical and morphological modifications [6-7] .

The N,N'-ethylenedi -L- Systine multi dentate ligand is a very effective chelator as was concluded from the crystal structures of its gallium(III) and indium(III) complexes and also from the stability constants of its complexes with cobalt(II), lead(II), cadmium(II), zinc(II) and nickel(II) ions. [8] The stability constants of these complexes are significantly higher than the overall constants of bis-complexes containing Cys.

Stability constants for complexes formed in the tin(II)-Glycine system were determined by potentiometry, and evidence for bidentate coordination of Glycine was obtained. Characterized. [9]

Quinoline and its derivatives have been widely used as metal ion chelating agents, metal extracting agents, corrosive inhibitors and they often show biological activity. They can be used as building blocks in medicine synthesis when the compounds possess other functional group (1). The chloromethylation of 8-hydroxyquinoline was reported [10].

In this paper reports the synthesis and identification of new complexes of Sn(II) and Pb (II) with L-leucin and 8-hydroxyquinoline respectively.

Experimental:

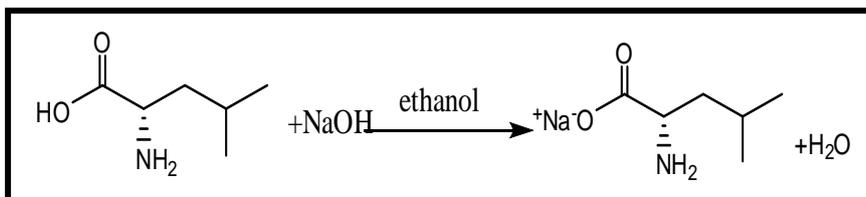
a- Materials:- All chemicals used were of reagent grade and were used without further purification SnCl_2 , PbCl_2 (Fluka) L-leucin was purchased from (Merck), 8-hydroxyquinoline a Fluka Chemie AG, .

b- Instruments: FTIR spectra were recorded as KBr discs using Fourier transform Infrared Spectrophotometer Shimadzu FT-I.R 8400s. Electronic spectra of the prepared complexes were measured in the region (200- 1100) nm for 10^{-3}M solutions in ethanol at 25°C using shimadzu-U.V-160. A Ultra Violet Visible- Spectrophotometer with 1.000 ± 0.001 cm matched quartz cell. While metal contents of the complexes were determined by Atomic Absorption (A.A) Technique using Japan A.A-67G Shimadzu. Electrical conductivity measurements of the complexes were recorded at 25°C for 10^{-3} M solutions of the

samples in ethanol using pw 9527 Digital conductivity meter (Philips). Melting points were recorded by using Stuart melting point apparatus. The proposed molecular structure of the complexes were determined by using chem. office program, 3DX (2006).

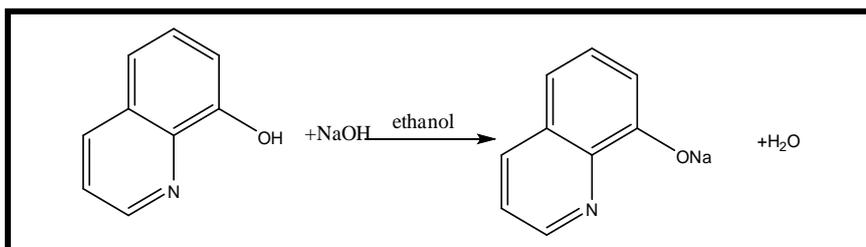
C- General Method for the Synthesis [11]

a) Sodium leucinate(Na^+Leu^-) : L-leucin [0.262 gm, 2 m mol] was dissolved in 10 ml ethanol and added to 10 ml of ethanolic solution containing [0.08 gm (2mmol)] of the sodium hydroxide , the solution was deprotonated according to the following reaction scheme (1)



scheme (1) : Preparation of Sodium leucinate

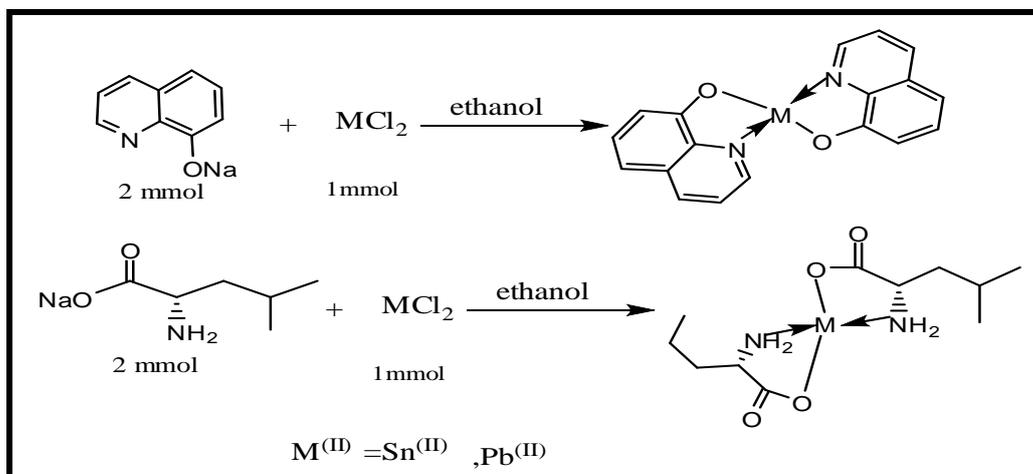
b)Sodium-8-oxyquinolate: [0.145gm ,1 m mol] 8-hydroxyquinoline (8-QH) with [0.04 gm (1mmol)] sodium hydroxide in ethanol was deprotonated according to the following reaction scheme (2)



scheme (2) : Preparation of sodium-8-oxyquinolate

C- General Method for the Synthesis of complexes: [11]

The mono ligand complexes were synthesized by the reactions of ethanolic solutions of metal chlorides with two different ligand in a 1 : 2 (M:L) mole ratio by raising the pH to 6–6.5 with a 5 % aqueous sodium hydroxide solution and stirring the solution on a magnetic stirrer at 20–60 °C. The pH was measured with the aid of pH paper and stirring was continued for ninety minutes. The solid that separated was filtered and dried at room temperature.



scheme (3) : The Synthesis of complexes

Results and Discussion

The complexes have been prepared by direct reaction of ethanol solution of the ligand with aqueous solution of metal ions at (1:2) metal to ligand molar ratio.

The complex combinations of Sn(II) and Pb (II) with L-leucin and 8-hydroxyquinoline respectively appeared as powders with high melting points.

All the complexes are colored, non-hygroscopic, indicating a strong metal-ligand bond. The complexes are insoluble in water but soluble in common organic solvents such as ethanol, ethyl alcohol, acetone, chloroform, DMF and DMSO. The observed molar conductance (Table 1) values measured in ethanol solution fall in the range (6.58-12.34) ohm⁻¹ cm² mol⁻¹. These observed values of the molar conductance are well within the expected range for non-electrolyses [12]

Atomic Absorption measurements :

The atomic absorption measurements (Table-1) for all complexes gave approximated values for theoretical values.

Infrared spectra and mode of coordination :

The most important infrared spectral bands that provide conclusive structural evidence for the coordination of the ligands to the central metal ions .

The characteristic vibrations and assignments of ligands (QH) and (LeuH) and their complexes as KBr spectrum discs are described (Tables 2-3) , Figure (1-6).

In conclusion, our investigation this suggest that the ligands L-leucin and Sodium-8-oxyqinolate coordinate with M (II) forming tetrahedral geometry Table (2), displays the (FT-IR) spectrum for the (L-leucin) exhibited a band around ν (3417) cm⁻¹ that corresponds to the stretching vibration of ν (N-H) + ν (O-H), while another strong absorption band at ν (3070) cm⁻¹ is due to the ν (NH₂)_{sym} while the bands at (1585) cm⁻¹ and (1415) cm⁻¹ were assigned to the ν (-COO⁻)_{asy} and ν (-COO⁻)_{sym} respectively.

$\nu\Delta$ (-COO)_{asy-sym} = 170 cm⁻¹. [13-15]

An important feature of infra-red spectra of metal complexes with 8-HQ Table(3), is the absence of the band at (3240-3047) cm⁻¹ due to the O-H stretching vibration of the OH group of HQ [16]. This observation leads to the conclusion that the complex formation takes place by deprotonation of the hydroxyl group of HQ moiety. A strong ν (C-O) band observed at ~1104 cm⁻¹ indicates the presence of oxine moiety in the complexes coordinated through its nitrogen and oxygen atoms as uninegative bidentate ligand. [14]

The complexes show band at (536-555) and (424-478) cm⁻¹ rang, due to the ν (M-N) and ν (M-O) vibrations respectively. [15-16]

The electronic spectra:

The electronic spectral data of the free ligands 8-hydroxyquinoline, L-leucin and their complexes are summarized in Table -4 together with the proposed assignments and suggested geometries. The results obtained are in good agreement with other spectra and the literature. [17-18]

The electronic spectrum of leucin show an absorption band at 305 nm (32786 cm⁻¹) in ethanol this band is attributed to ($\pi \rightarrow \pi^*$). The uv - vis spectrum of the free ligand (8-hydroxyquinoline) in ethanol solvent appeared a high, intense absorption bands at 280 nm (35714 cm⁻¹) (ϵ_{\max} = 1880 molar⁻¹.cm⁻¹) and at 301 nm (33222 cm⁻¹) (ϵ_{\max} = 2407 molar⁻¹.cm⁻¹) These bands are attributed to ($\pi \rightarrow \pi^*$) and ($n \rightarrow \pi^*$) transitions respectively. [18]

The uv - vis spectra of the prepared complexes dissolved in ethanol (10⁻³ M) have been measured and the data obtained were included in (Table- 4) shows absorption bands at 291, 306, 389, 331 and 379 nm. The bands are indicative of benzene and other chromofore moieties present in the complex [19].

A moderately intensive band observed in the range of (320-380)nm is attributed to the $n \rightarrow \pi^*$ transition of [20]coordinated and the strong band observed in range of (270-280) nm is due to the $\pi \rightarrow \pi^*$ transition of these complexes [21-22].

Nomenclature of prepared complexes:

Table (5) shows empirical formula and nomenclature (IUPAC) with abbreviated.

Table 1-The physical properties of the Compounds

Compounds	M .wt	Color	M. p °c (de)°c	ΔM Molar conductivity $\Omega^{-1}cm^2mol^{-1}$	Metal % theory	Metal % experiment	Cl %
$C_6H_{13}NO_2$ (Leu)	131.17	white	299(dec)	1.24			-
(C_9H_6NO) (8-HQ)	145.15	Pale brown	277(dec)	1.77	-	-	-
$[Pb(Q)_2]$	495.50	white - yellow	>300(dec)	6.58	41.82	43	Nil
$[Sn(Q)_2]$	407.01	white - yellow	>300(dec)	8.68	29.17	28	Nil
$[Pb(Leu)_2]$	467.53	white - yellow	210	9.62	44.32	47	Nil
$[Sn(Leu)_2]$	397.3	white - yellow	230	12.34	31.32	33	Nil

Q = C_9H_5NO , Leu $C_6H_{12}NO_2$, **Dec. = Decomposition temperature

Compound	$\nu(\text{N-H})_{\text{a sym}} + \nu(\text{O-H})$	$\nu(\text{N-H}_2)_{\text{sym}}$	$\nu(\text{C-H})_{\text{aliphatic}}$	$\nu(\text{-COO})_{\text{asy}}$	$\nu(\text{-COO})_{\text{sym}}$	$\nu\Delta(\text{-COO})_{\text{asy-sym}}$	M-N	M-O
leucin ($\text{C}_6\text{H}_{13}\text{NO}_2$)	3417m	3070s $\Delta\nu = 347$	1580s	1585vs	$\nu(\text{-COO})_{\text{sym}}$	170	-	
[Pb($\text{C}_6\text{H}_{12}\text{NO}_2$) ₂]	3448vs	3097s	1620vs	1589vs	1415vs	174	536m	474m
[Sn ($\text{C}_6\text{H}_{12}\text{NO}_2$) ₂]	3448vs	3105m	1620vs	1585vs	1416vs	106	536m	478m

Table 2-FT-R spectral data of leucin and its complexes

Compound	$\nu(\text{O-H})_{\text{aromatic}}$	$\nu(\text{C-H})_{\text{aromatic}}$	$\nu(\text{C=N})$	$\nu(\text{C=C})$	$\nu(\text{C-O})$	$\delta(\text{C-O})$	$\delta(\text{O-H})_{\text{in plane}}$ $\delta(\text{O-H})_{\text{out plane}}$	M-N	M-O
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Table 3-FT-R spectral data of 8-hydroxyquinoline and its complexes

8-ydroxyquinoline	3240-3047br	3040 -2854s	1577vs	1508vs	1276vs	470s	1381vs 779s	-	-
[Pb(C ₉ H ₅ NO) ₂]	3448vs	3043s	1635m	1492m	1280s	486m	1377 vs 732s	555m	424w
[Sn (C ₉ H ₅ NO) ₂]	3448vs	2987w	1635s	1496m	1273m	488m	1381m 737m	536m	428w

str: stretching, v.s: very strong, s: strong, m: medium, w: week,

o.p: out of plane,

Sym: symmetric, asy: asymmetric, am: amide, py: pyridine,

Table 4- Electronic Spectral data of the studied complexes

Complexes	λ_{nm}	$\nu^{-1} (Cm)^{-1}$	ξ_{max} L. mol ⁻¹ .cm ⁻¹	Assignment	Probable Figure
8-ydroxyquinoline	314	31847	2352	n→π*	-
leucin	294	34013	79	π→π*	-
[Pb(C ₆ H ₁₂ NO ₂) ₂]	291	34364	45	n→π*(blue shift)	Th
[Sn (C ₆ H ₁₂ NO ₂) ₂]	306	32679	451	n→π*(red shift)	Th
[Pb(C ₉ H ₅ NO) ₂]	389 331	25706 30211	1412 1148	n→π*(red shift) n→π*(red shift)	Th
[Sn (C ₉ H ₅ NO) ₂]	379	26385	1161	n→π*(red shift)	Th

Table (5) Nomenclature of prepared complexes

Empirical formula	Nomenclature	Abbreviation
[Pb(C ₉ H ₅ NO) ₂]	Bis- (8-oxyquinolate) lead (II)	Pb(Q) ₂
[Sn (C ₉ H ₅ NO) ₂]	Bis- (8-oxyquinolate) tin(II)	Sn (Q) ₂
Pb(C ₆ H ₁₂ NO ₂) ₂]	di- (leucinate) lead (II)	Pb(leu) ₂
[Sn(C ₆ H ₁₂ NO ₂) ₂]	di- (leucinate) tin(II)	Sn (leu) ₂

Table (6) Represent the antimicrobial activity of chemical compound against some pathogenic bacteria

compounds	<i>Escherichia coli</i>	<i>Candida albicans</i>	<i>Pseudomonas</i>	Bacillus	+ <i>Staphylococcus S.P</i>
Control (DM F)	10	10	9	10	10
Q	20	25	12	22	20
L- Leu	15	18	11	14	15
[Sn(Leu) ₂] (No 4)	14	13	10	16	10
[Sn (Q) ₂] (No 5)	27	35	15	35	31

Antibacterial Activities:

The antibacterial activity of the ligands and some there complex were tested on Gram positive bacteria, *Staphylococcus aureus* and Gram negative, *Bacillus*, *Pseudomonas*, and *Candida albicans*. The solvent used was dimethylformamid(DMF) and sample from 1 to 200 $\mu\text{g/ml}$ were used. Anti bactericidal activities of each compound were evaluated by the well-diffusion method. 1 cm^3 of a 24 h broth culture containing 10^6 CFU/ cm^3 was placed in sterile Petri-dishes. Molten nutrient agar (15 cm^3) kept at ca. 45°C was then poured in the Petri-dishes and allowed to solidify. Then holes of 6 mm diameter were punched carefully using a sterile cork borer and these were completely filled with the test solutions. The plates were incubated for 24 h at 37°C . The zone inhibition of bacterial growth were measured in mm depending upon the diameter as shown in Table (6) Figure. (13) [23].

The antibacterial activity results revealed that the ligands and there complexes shown weak to good activity when compared to the Control (DMF). The results of antimicrobial screening, indicate that thre complexes shows more activity against *Staphylococcus aureus*, *Escherichia coli*, *Pseudomona* and *Bacillus than Pseudomonas*.

The results of antimicrobial screening, indicate that complexes shows activity against microorganisms. The activity of these substance may be due to carboxyl group [24-25].

Determination of the Stoichiometry of Complex Formation Between lead with (L-leucin) and (8-hydroxyquinoline) :

The composition of the complexes formed in solution has been established by job method.

In this study, ultraviolet-visible spectrophotometry is used to determine the stoichiometry of complex formation between [8-HQ], [leu] and metal ions pb (II) respectively. There are four types of transitions between quantized energy levels that are responsible for the UV-Vis spectra: $\sigma \rightarrow \sigma^*$, $n \rightarrow \sigma^*$, $\pi \rightarrow \pi^*$, and $n \rightarrow \pi^*$. The two most important transitions are $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ because they involve functional groups that are characteristic of the analyte. 8-ydroxyquinoline's aromatic group contains mobile π electrons which correspond to the $\pi \rightarrow \pi^*$ transition. The results reveals (1:2) metal to ligand ratio. A chosen plots of were represented in (Figure-13) (Table- 7) and (Figure-14) (Table- 8) summarize the results obtained as a conditions for the preparation of the complexes. Pb(II) with Leucin and 8-ydroxyquinoline respectively. [26].

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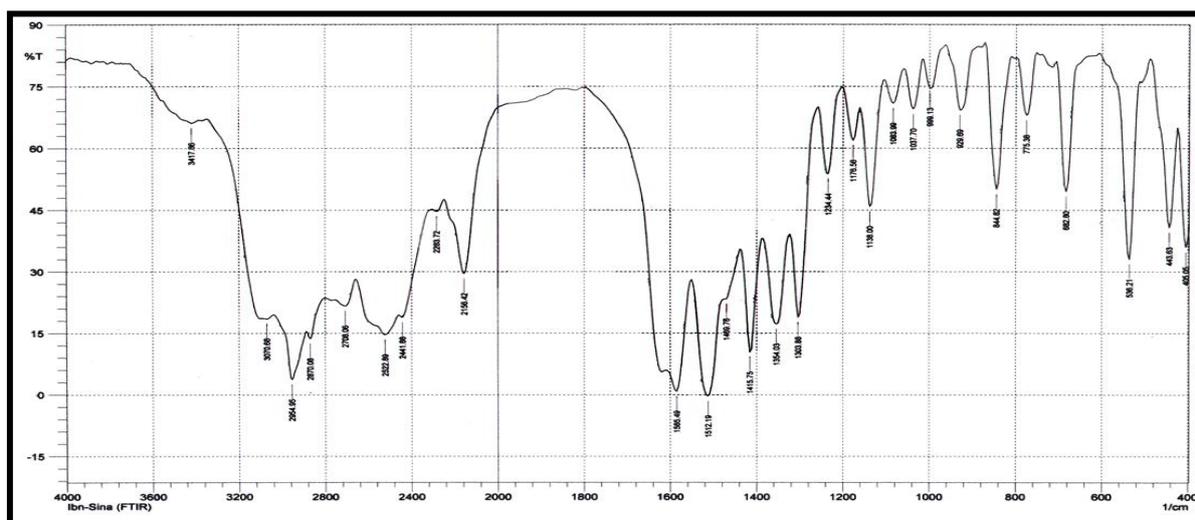


Figure (1) FT- IR Spectrum of L-leucin ($\text{C}_6\text{H}_{13}\text{NO}_2$)

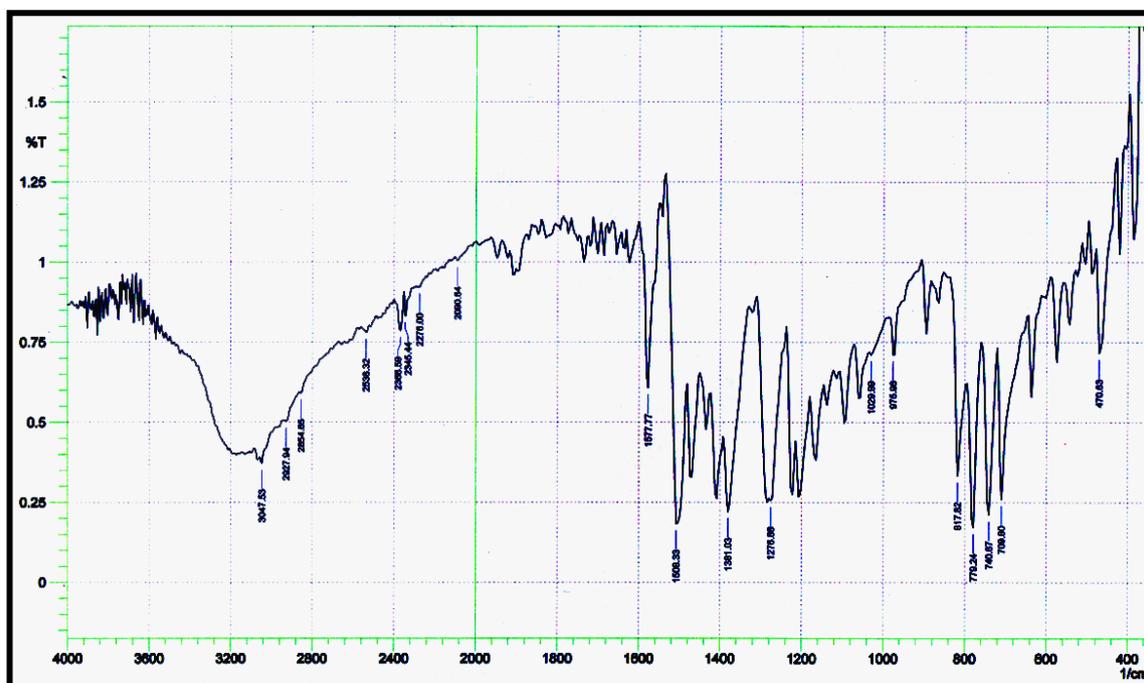


Figure (2) : FT-IR spectrum of 8-Hydroxyquinoline ($\text{C}_9\text{H}_6\text{NO}$)

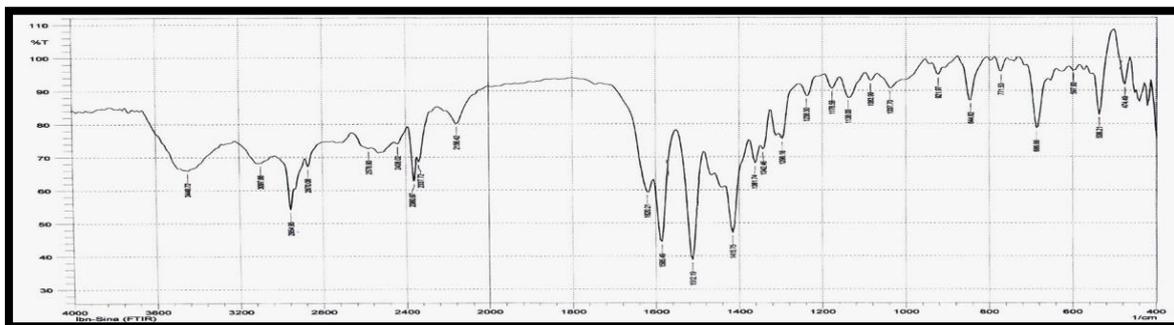


Figure (3) : FT IR Spectrum of $[\text{Sn}(\text{C}_6\text{H}_{12}\text{NO}_2)_2]$

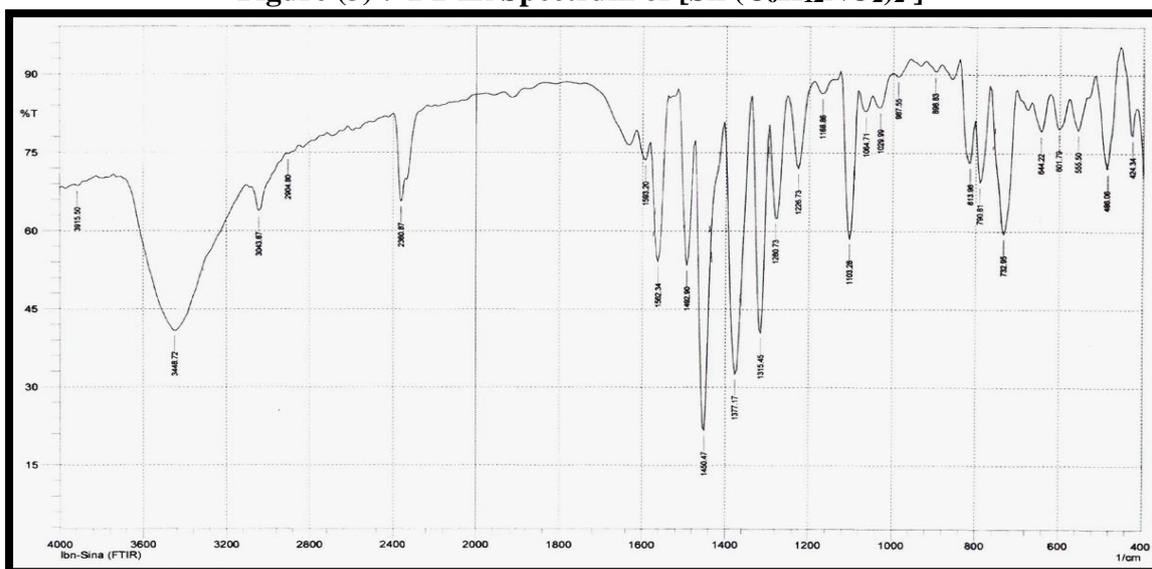


Figure (4) : FT IR Spectrum of $[\text{Pb}(\text{C}_6\text{H}_{12}\text{NO}_2)_2]$

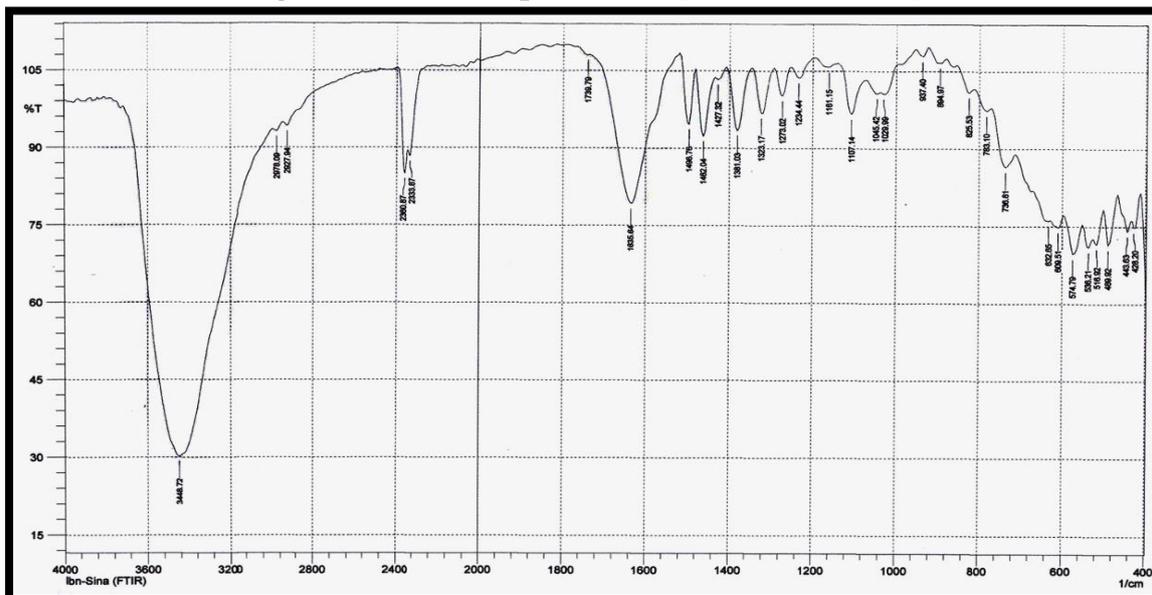


Figure (5) : FT IR Spectrum of $[\text{Pb}(\text{C}_9\text{H}_6\text{NO})_2]$

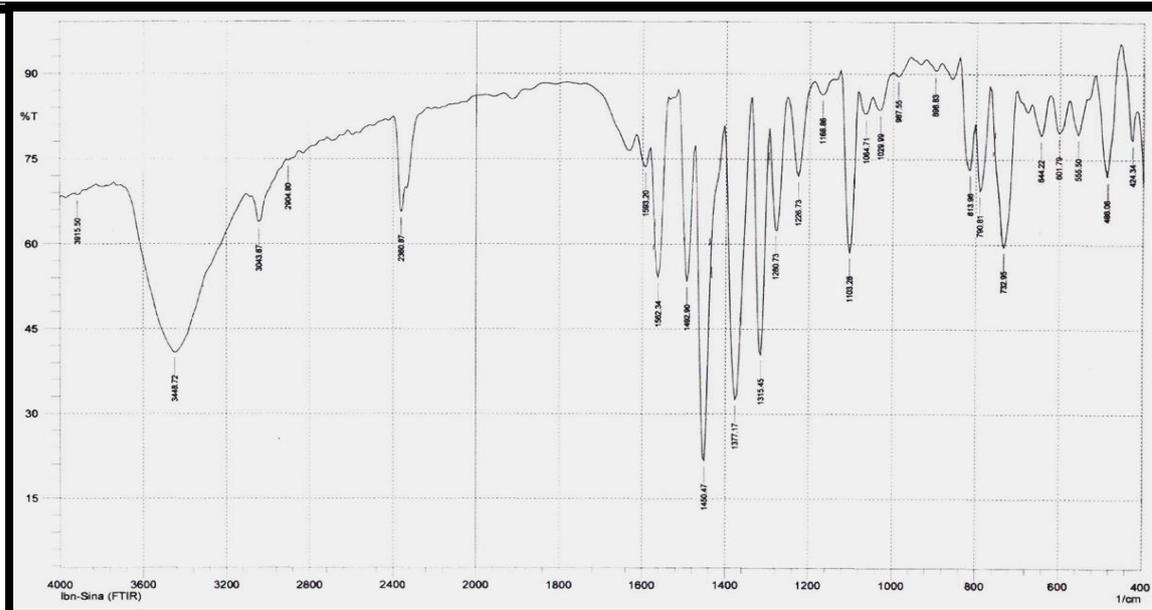


Figure (6) : FT IR Spectrum of $[Sn(C_9H_6NO)_2]$

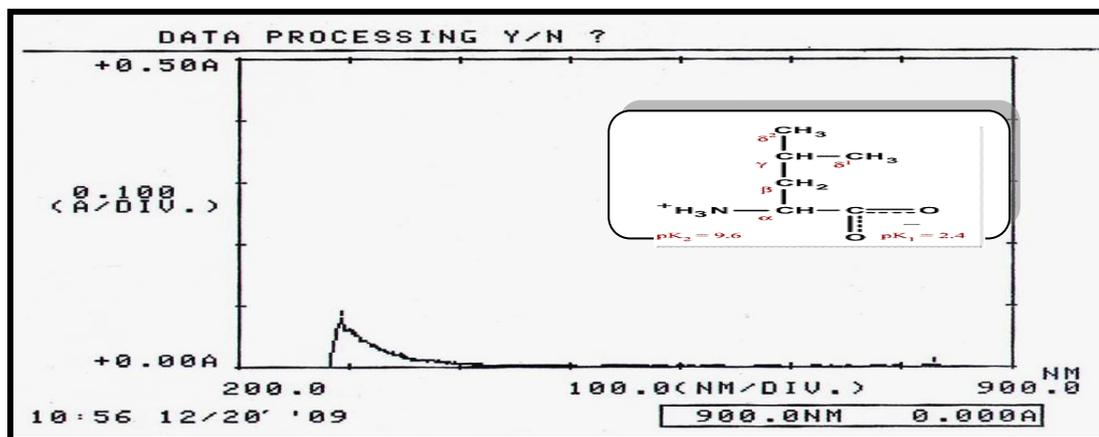


Figure (7) :The (UV-Vis) Spectrum of L-leucin

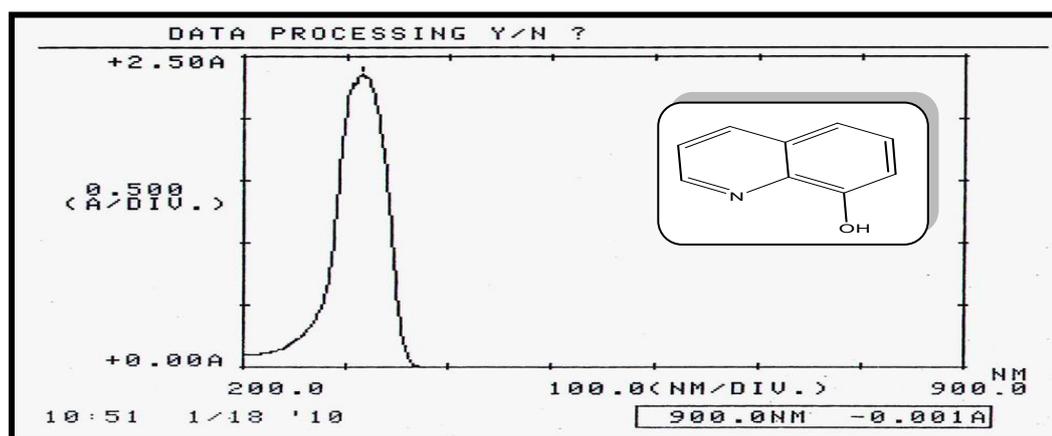


Figure (8) The (UV-Vis) Spectrum of (C_9H_6NO)

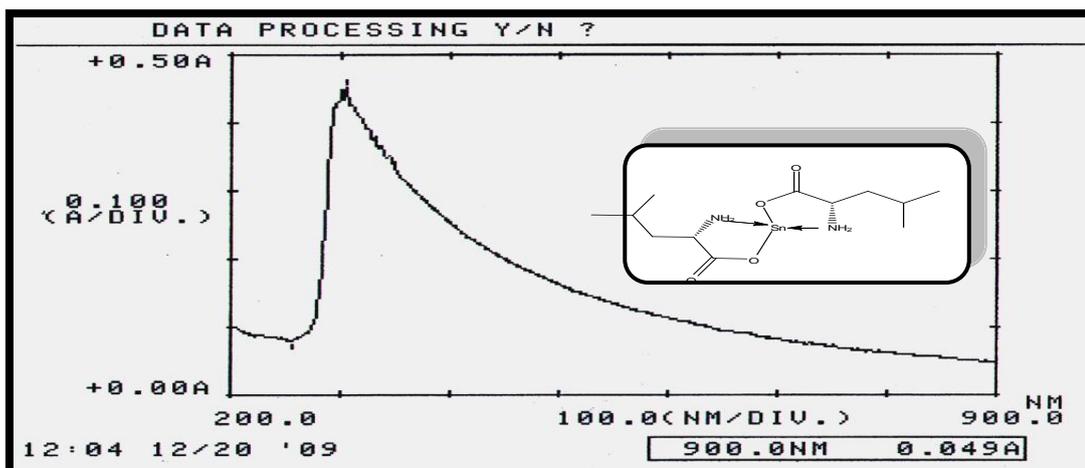


Figure (9) The (UV-Vis) Spectrum of $[Sn(C_6H_{12}NO)_2]$

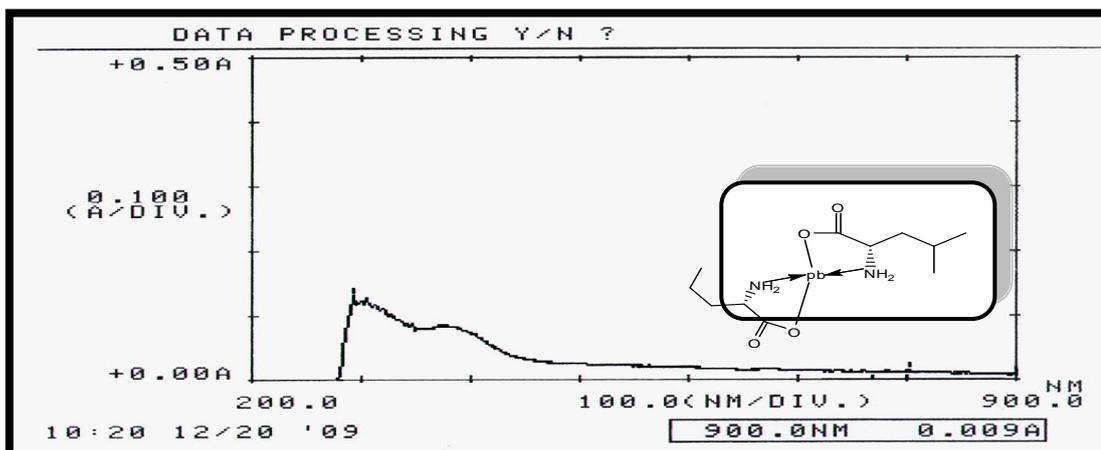


Figure (10) The (UV-Vis) Spectrum of $[Pb(C_6H_{12}NO)_2]$

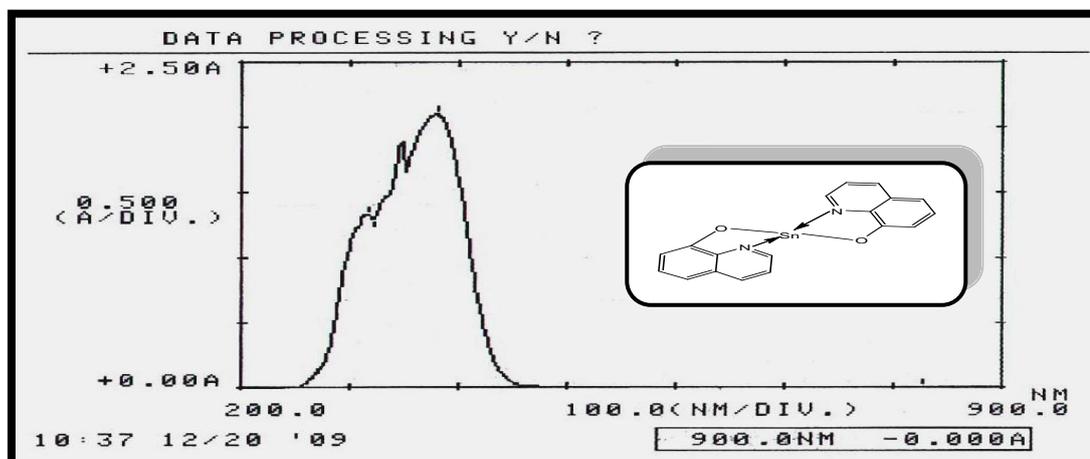


Figure (11) The (UV-Vis) Spectrum of $[Sn(C_9H_6NO)_2]$

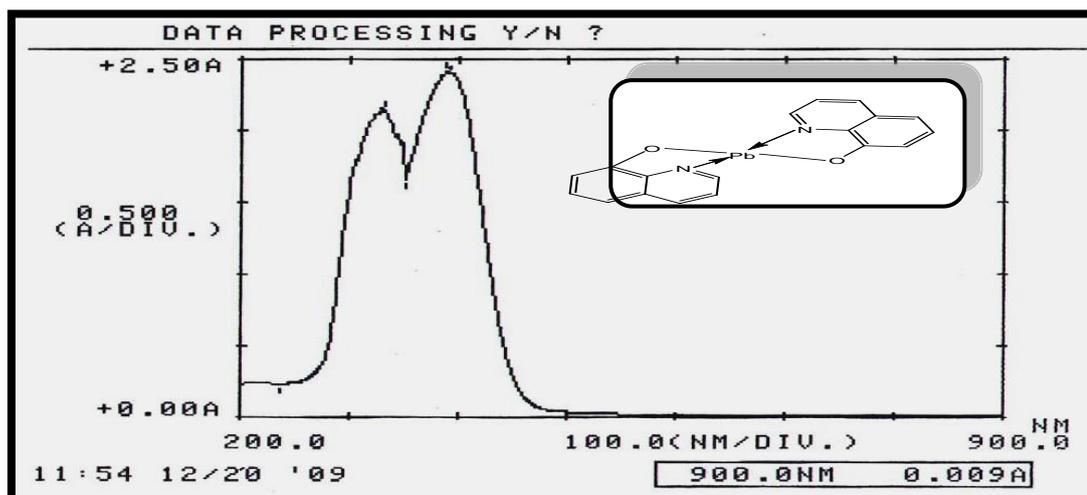
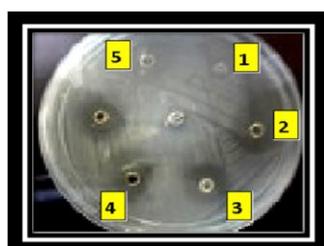
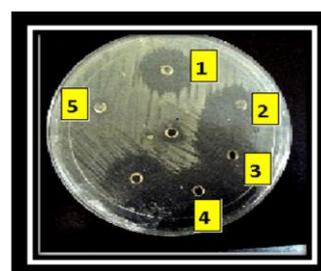


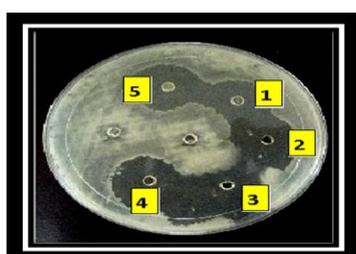
Figure (12)The (UV-Vis) Spectrum of $[Pb (C_9H_6NO)_2]$



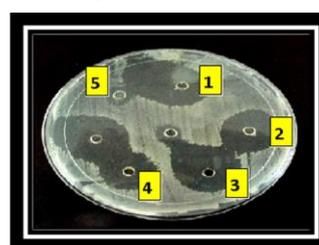
Escherichia coli



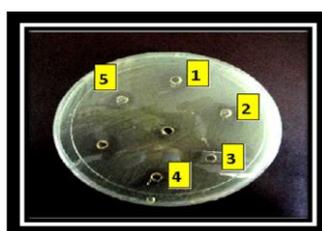
Staphylococcus aureus



Candida albicans



Bacillus cereus



Pseudomonas aeruginosa

Figure (13) shows the antimicrobial activity of chemical compounds (ligands, 3,4,) appear the inhibition zones against some pathogenic bacteria

Table 6: Solution preparation and absorbance measurements for L- leu -Pb (II) at 274nm.

Sample no.	V mL [leu] $1 \times 10^{-3} \text{M}$	V mL [pb ²⁺] $1 \times 10^{-3} \text{M}$	$\frac{V_m}{(V_m+V_L)}$	$\frac{V_L}{(V_m+V_L)}$	Absorbance at 274nm
					Pb (II)
0	0	10	1.0	0	0
1	1	9	0.9	0.1	0.125
2	2	8	0.8	0.2	0.283
3	3	7	0.7	0.3	0.391
4	4	6	0.6	0.4	0.44
5	5	5	0.5	0.5	0.563
6	6	4	0.4	0.6	0.715
7	7	3	0.3	0.7	0.594
8	8	2	0.2	0.8	0.398
9	9	1	0.1	0.9	0.201
10	10	0	0.0	1	0

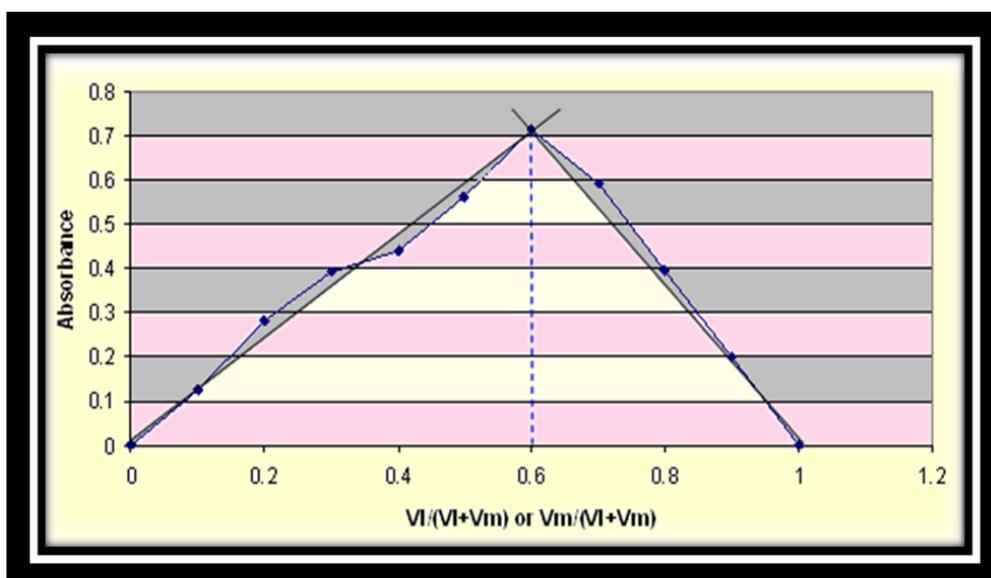


Figure 14: Complex formation curve for L- leu -Pb (II) at 274nm.

Table 7: Solution preparation and absorbance measurements 8-HQ -Pb (II) at 274nm.

Sample no.	V mL [8-HQ] $1 \times 10^{-3} \text{M}$	V mL [pb ²⁺] $1 \times 10^{-3} \text{M}$	$\frac{V_m}{(V_m+V_L)}$	$\frac{V_L}{(V_m+V_L)}$	Absorbance at 274nm
					Pb (II)
0	0	10	1.0	0	0
1	1	9	0.9	0.1	0.128
2	2	8	0.8	0.2	0.299
3	3	7	0.7	0.3	0.398
4	4	6	0.6	0.4	0.467
5	5	5	0.5	0.5	0.578
6	6	4	0.4	0.6	0.754
7	7	3	0.3	0.7	0.566
8	8	2	0.2	0.8	0.378
9	9	1	0.1	0.9	0.245
10	10	0	0.0	1	0

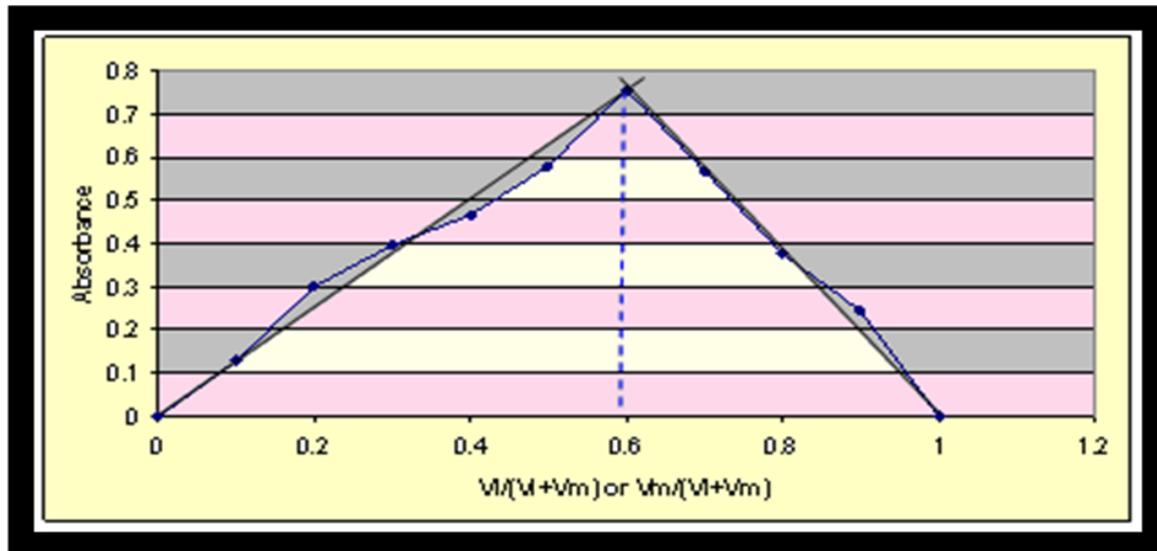


Figure 15: Complex formation curve for 8-HQ -Pb (II) at 274nm.

تحضير، تشخيص ودراسة الخواص الضديدة للبكتريا لمعدنات
الرصاص (II) والقصدير (II) مع (الليوسين و8-هيدروكسي كوينولين)

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الخلاصة:

يتضمن البحث تحضير وتشخيص نوعين من المعدنات بالصيغ العامة $[M(Q)_2]$ و $[M(Leu)_2]$. إذ ان الليوسين $(C_6H_{13}NO_2)$ بالرمز (LeuH) و8-هيدروكسي كوينولين (C_9H_7NO) بالرمز (8-HQ) و M(II) الايونين Sn(II) و (II) Pb وذلك بمفاعله كلا الليكاندين مع كلوريد الرصاص (II) و كلوريد القصدير (II) باستعمال الايثانول مذيباً وفي درجة حرارة المختبر وبنسب مولية 1:2 (ليكاند: فلز).

المعدنات المحضرة بلورات صلبة درست من النواحي الآتية: الاستقرار الحرارية، التوصيلية الكهربائية المولارية، الذوبانية، تقدير النسبة المئوية للأيون الفلزي في المعدنات بواسطة مطيافية الامتصاص الذري، الدراسات الطيفية: وتضمنت أطياف الأشعة تحت الحمراء، الأشعة فوق البنفسجية- المرئية، الخواص المغناطيسية ومحتوى الكلور) مع استعمال البرنامج (Chem. Office- Cs. chem- 3D pro 2006) في رسم اشكال المعدنات.