Iraqi National Journal of Chemistry

Journal homepage:
http://iqnjc.com/Default.aspx

Preparation, Characterization and Antibacterial Activity of Schiff base Ligand and their Complexes

Batool qusai [1]; Mohammed Hamid Said [2,*]; Rasha alfaham [1]

[1]Department Of Chemistry , Faculty Of Education For Women , University Of Kufa.

[2]Department of pharmaceutical Chemistry, College of pharmacy University of Babylon

Email: mohammed2912197132@yahoo.com

Abstract:

A new Schiff base ligand has been synthesized from condensed 6.Amino Penicillinic acid with Benzyldehyde , then it mixed with number of the transition metal to form complexes. The newly prepared Schiff base ligand and metal complexes have been characterized by various techniques such as ^{1}H NMR, ^{13}C NMR , UV/ Visible, FT IR , Mass spectral ,Magnetic moment, molar conductance method , and it tested the effectiveness of inhibitory compounds prepared against four types of bacteria S.aureus , pseudomonas aeruginosa , Streptococcus Facials and Proteus Mirabils then it isolated from different classes of ulcerative infections.

Key Word: β-lactam , Schiff base , Antibacterial.

1-Introduction:

The first of the synthesis of compounds beta lactam compounds by the scientist Staudinger in 1907(1), beta lactam compounds consist of a four ring connected with six- membered of Dihydrothiazin ring called Cephalosporins but if it connected with five-membered Thiazolidine ring called penicillins(2), it

can be classified as beta lactam derivatives depending on the ring system : Penam ,Cephem , clavam , carbapenem and monolactam[3], β-lactam antibiotics working on the inhibition of the synthesis of the bacterial cell wall [4] , the compounds containing azomethene group known schiff base configured by the intensification of the first amino with carbonyl compounds[5] ,there are many important applications for schiff base ligands free and its complexes in medical and pharmaceutical fields in addition to biological activity against bacteria , fungi and tumors[6] .

2.Experimental:

2.1 Chemical:

All chemicals used were reagent grade (Sigma Aldrich , B.D.H , C.D.H. and S.L. company) including 6.Amino Penicillinic acid ,Benzaldehyde , $CuCl_2.2H_2O$, $CoCl_2.6H_2O$,$NiCl_2.6H_2O$ and $ZnCl_2$ absolute ethanol , Pyridine , DMSO.

2.2 Instrumentation :

UV-Visible spectra were recorded on Shimadzu 1800 series spectrometer for ligand and complexes in DMSO solvent.FT-IR spectra were recorded on 8400 FTIR Simadzu spectrometer, The Magnetic Moment Measurement was using Auto Magnetic Susceptibility Balance Sherwood scientific , The Molar Conductance Measurement in DMSO solvent using Digital conductivity meter alpha -800,addation to mass spectrum , The proton ^{1}H and carbon ^{13} C Nuclear Magnetic Resonance (NMR) spectra were recorded in DMSO were recorded on a Bruker Advance II 400 Spectrometer at room temperature the chemical shifts are reported in ppm relative to TMS (tri methyl silane).

2.3 Preparation of Schiff base ligand :

The Ligand were synthesized according to the general method by dissolving 6.Amino Penicillinic acid (2.16g,0.01mole) in absolute ethanol (medium basic) , then it added to (1.06g , 0.01mole) of benzaldehyde of climb thermally for 12hrs then it filtered out put formed , dried and recrystallization from absolute ethanol .

2.4 Preparation of complexes:

The complexes were prepared in a M:L ratio 1:2 by dissolving 0.608g , 0.001mole of Schiff base ligand in 15 ml ethanol absolute , then it added to (0.170g , 0.2379g ,0.2377g and 0.136g) 0.001 mole of $CuCl_2.2H_2O$, $CoCl_2.6H_2O$,$NiCl_2.6H_2O$ and $ZnCl_2$ respectively . The mixture was refluxed for 30min , and then filtered, dried and recrystallization from absolute ethanol .

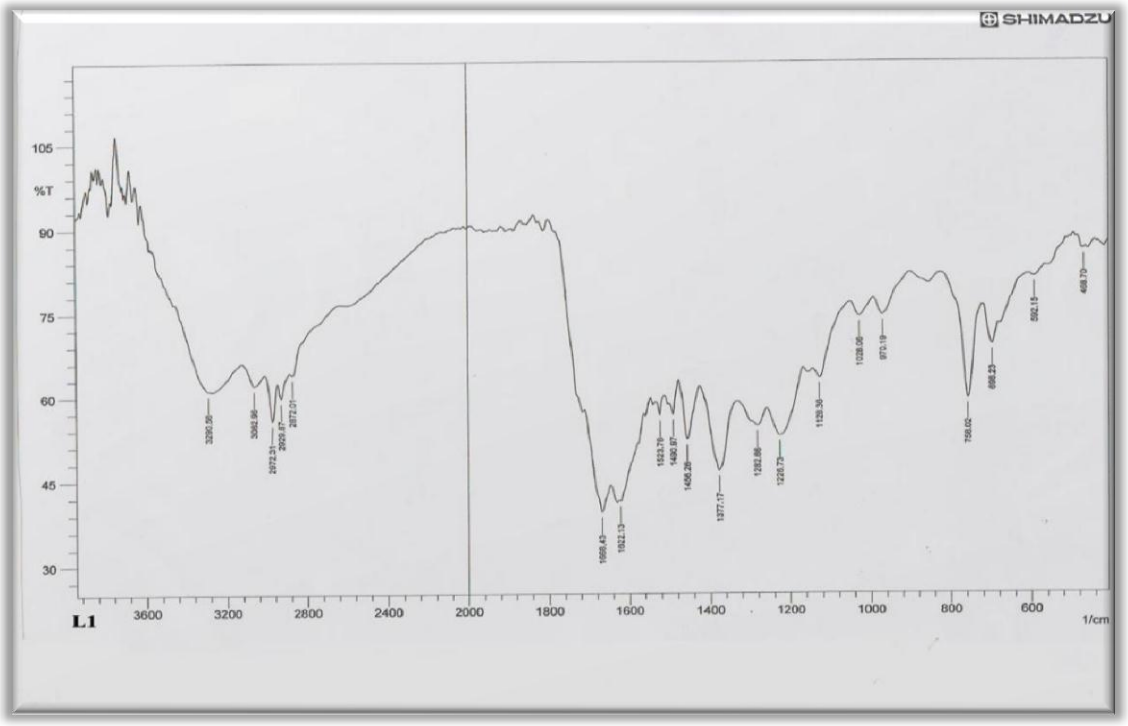
Tube(1):**Data ligand and complexes show chemical and physical characterizations**

Compound symbol	M.wt	M .p°C	Color
L_1	304	134-132	Orange
$Cu(L_1)_2$	671.54	122-120	Green Light
$Co\ (L_1)_2$	666.69	142-140	Green
$Ni\ (L_1)_2$	666.93	154-152	Brown
$Zn(L_1)_2$	673.39	157-155	Brown Light

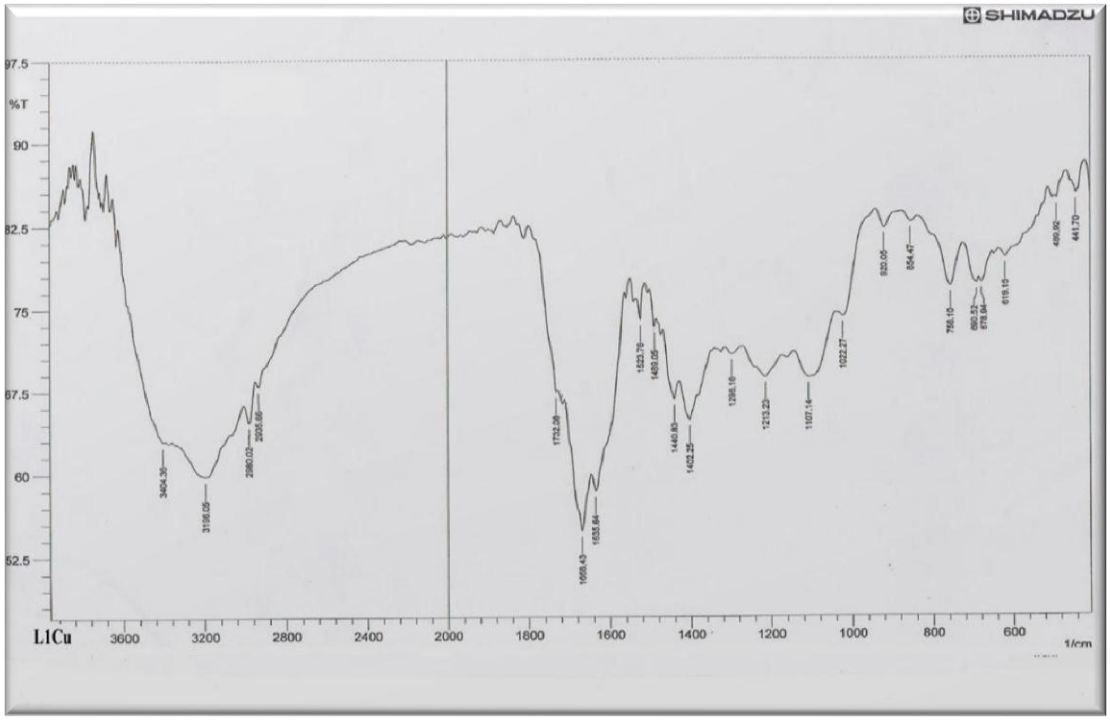
3. Results and Discussion:

3.1 Infrared spectra:

The IR spectra of the ligand show bands in the range 3290cm¯ - 1622cm¯corresponding to hydroxyl group and azomethene group of schiff base ligand, these packages shifted towards higher frequencies when you get consistency with some ions first transition chain elements in addition to the emergence of new packages in the spectrum of the complexes between the frequencies 590-497cm¯ and 460-437cm¯ attributed to v(M-O) and v(M-N) , the packages at the frequency 1720cm¯ belonging to the group of beta lactam of schiff base ligand [7,8].



Figure(1): FTIR spectra of Ligand L_1



Figure(2): FTIR spectra of Copper (II) complex

Table(2): FTIR data Of Schiff base ligand and metal complexes

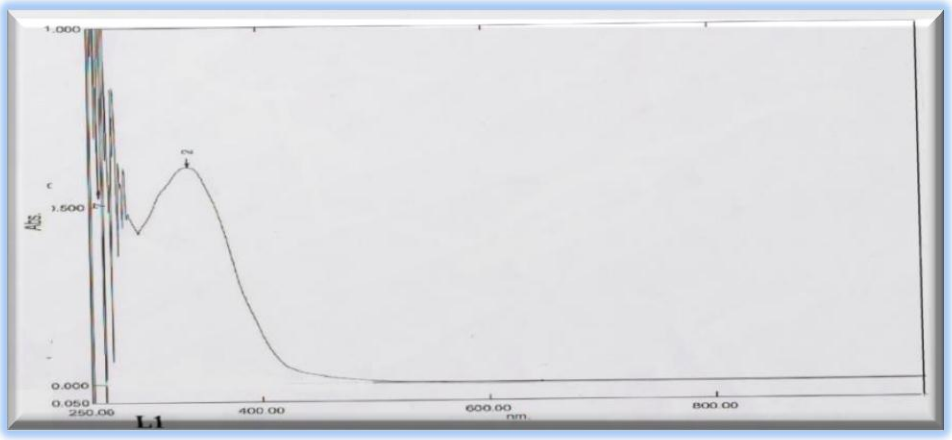
Compound	v(OH)	$v(C-H)_{alph}$	$v(C-H)_{arm.}$	v(C=O) Lactam	(C=N) Azomethene	M-N M-O
L_1	3290	2972	3062	1720	1622	-
$Cu(L_1)_2$	3404	2980	3060	1716	1635	499 440
$Co(L_1)_2$	3412	3064	2974	1716	1637	590 460
$Ni(L_1)_2$	3385	2974	3064	1716	1629	497 437
$Zn(L_1)_2$	3444	2972	3070	1716	1643	522 453

3.2 Electronic spectra :

Electronic spectra for ligand free includes the emergence of two wave lengths, first 262nm due to transmission electron to π-π^* (phenyl ring) and 325 nm due to transmission electron n-π^* (azomethene group) respectively[9,10] ,Uv-Vis data of the schiff base ligand **fig.3**.

Table(3): Electronic Spectra data Of Schiff base ligand and metal complexes

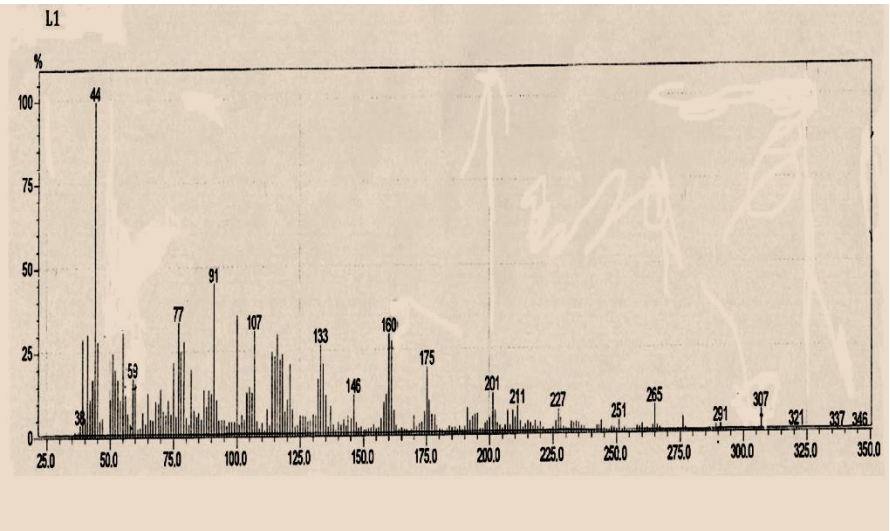
Complexes	Absorption Band nmλ_{max}	Assignment
L_1	325,262	n-π*, π-π*
$Cu(L_1)_2$	404,393	C.T
$Co(L_1)_2$	427,288	C.T
$Ni(L_1)_2$	497,311	C.T
$Zn(L_1)_2$	382,288	C.T



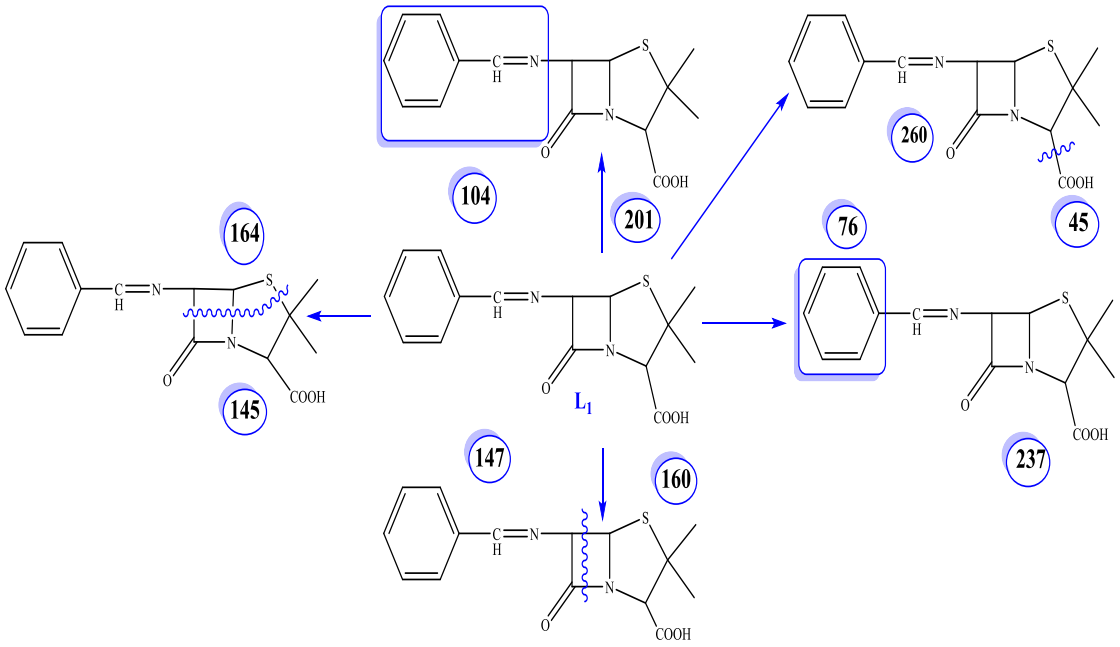
Figure(3):Electronic spectra of Schiff base ligand

3.3 Mass spectrum :

The mass spectrum includes of a base peak M^+ at 305, **fig.4** shows the proposed fragmentation of schiff base ligand free process [11,12].



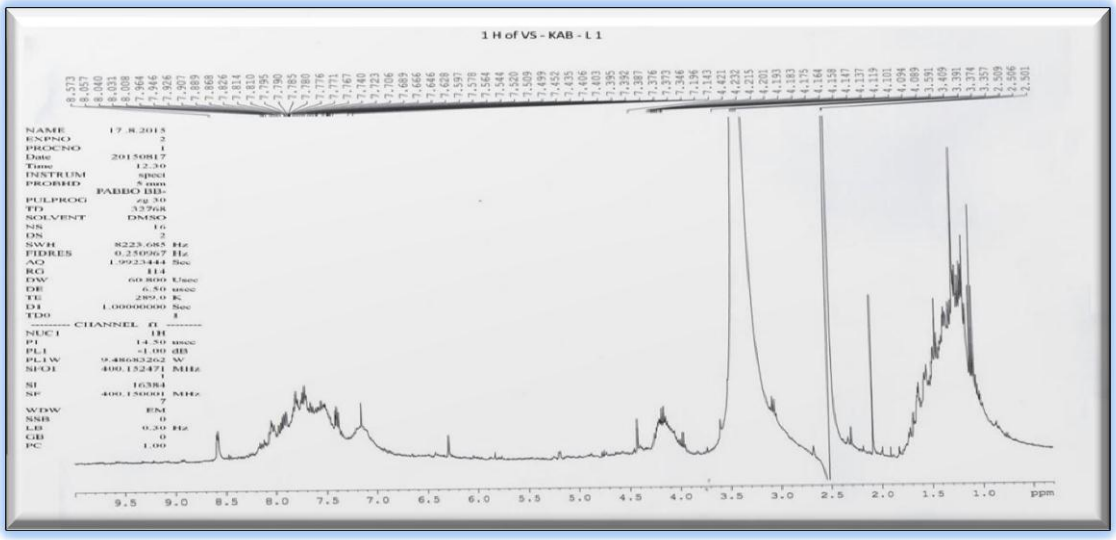
Figure(4): Mass spectrum of Ligand L_1



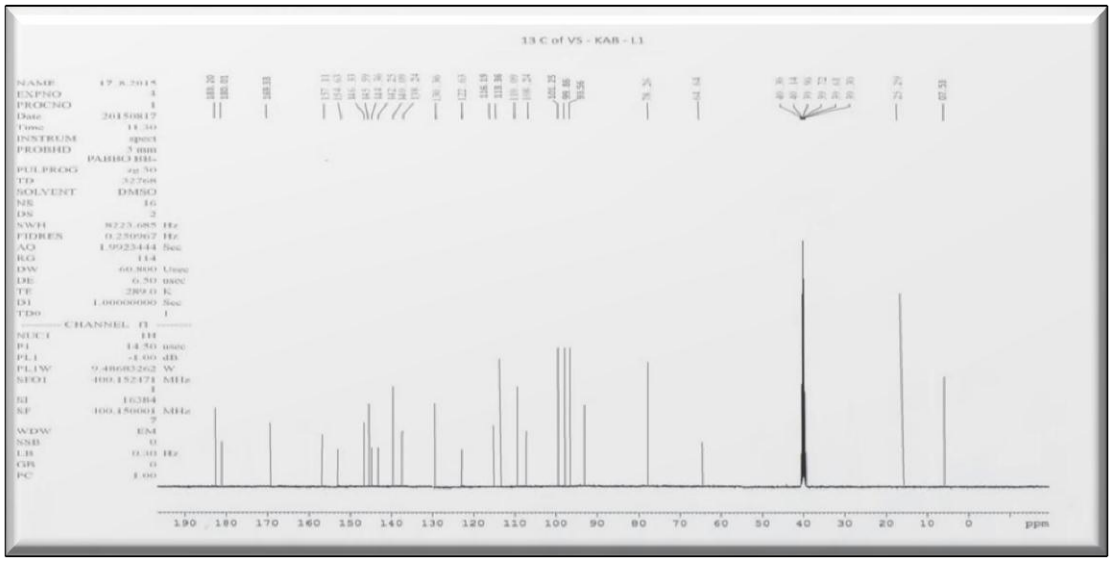
3.4 proton and carbon Nuclear magnetic resonance spectra of the schiff base ligand :

^{1}HNMR spectral data of the Schiff base was recorded in d^6-DMSO, (fig.6) shows different peaks at 8.5ppm and 1.5 ppm the site dates back to the first azomethene group proton and the second

goes back to the methyl group proton instance , the peaks between 7.5-7.7 ppm attributable to the Phenyl ring protons [13,14] , while the C^{13} NMR spectra includes the emergence of package at the site date back to 180ppm carbonyl group carbon back to beta lactam group either peaks between 122-140ppm and 157 ppm due to the first peaks phenyl group carbon and second attributable to azomethene group carbon respectively[15,16], forms 7 and 8 shows peak back to C^{13} NMR and ^{1}HNMR spectral was recorded in d^{6}-DMSO .



Figure(5): ^{1}H-NMR spectrum of Ligand L_1



Figure(6): C^{13}-NMR spectrum of Ligand L_1

Table(4): Magnetic moments and Molar Conductivity data Of Schiff base ligand and metal complexes

Complexes	Magnetic moment	Molar Conductivity	Geometry
$Cu(L_1)$	1.7	7.7	Octahedral
$Co(L_1)$	4.2	6.6	Octahedral
$Ni(L_1)$	3.2	8.1	Octahedral
$Zn(L_1)$	Diamagnetic	2.9	Octahedral

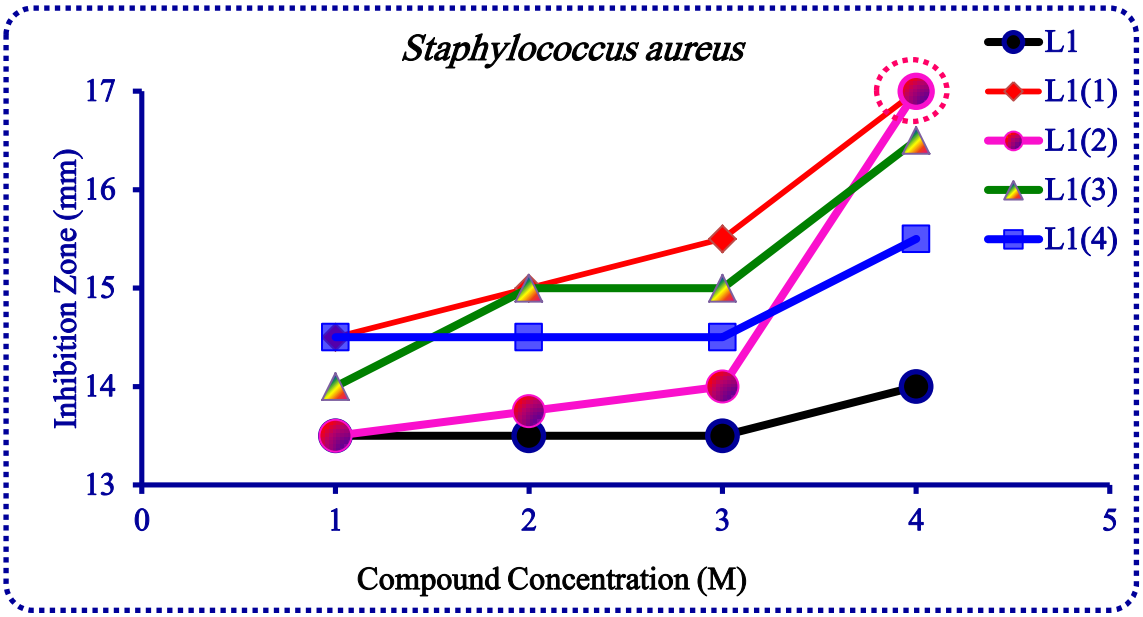
3.5 Anti-Bacterial Activity:

It was measured biological effectiveness free ligand against four types of bacteria ***S.aureus , Pseudomonas aeruginosa ,Streptococcus Facials*** **and** ***Proteus Mirabils*** will be illustrated as follows : Cu(II) Complex > Co(II) Complex >Schiff base ligand include biological effectiveness ligand free against bacteria of the type [*S.aureus*] of arrangement above it shows that copper and cobalt complexes higher efficiency of schiff base ligand free , either effective against bacteria of the type *Streptococcus Facials* showed a complex nickel effectiveness higher than the rest of the complexes in addition to ligand free , and for the type of bacteria *Pseudomonas aeruginosa* has to show that a complex of zinc higher effective either ligand free shall be the least effective Zn(II) Complex > Co(II) Complex >Cu(II) Complex> Ni(II) Complex >Schiff base ligand[17-20] , Cu(II) Complex >Ni(II) Complex >Co(II) Complex> Zn(II) Complex >Schiff base Ligand [Proteus Mirabils][21].

Table(5): Vital effect of Ligand L_1 and Complexes in Vital Work of bacterial

Staphylococcus aureus

Concentration (M)	Compound Symbol				
	L_1	$L_1(1)$	$L_1(2)$	$L_1(3)$	$L_1(4)$
1×10^{-6}	+	+	+	+	++
1×10^{-5}	+	-	-	++	-
1×10^{-4}	+	+++	+	++	++
1×10^{-3}	+	+++	+++	+++	+++



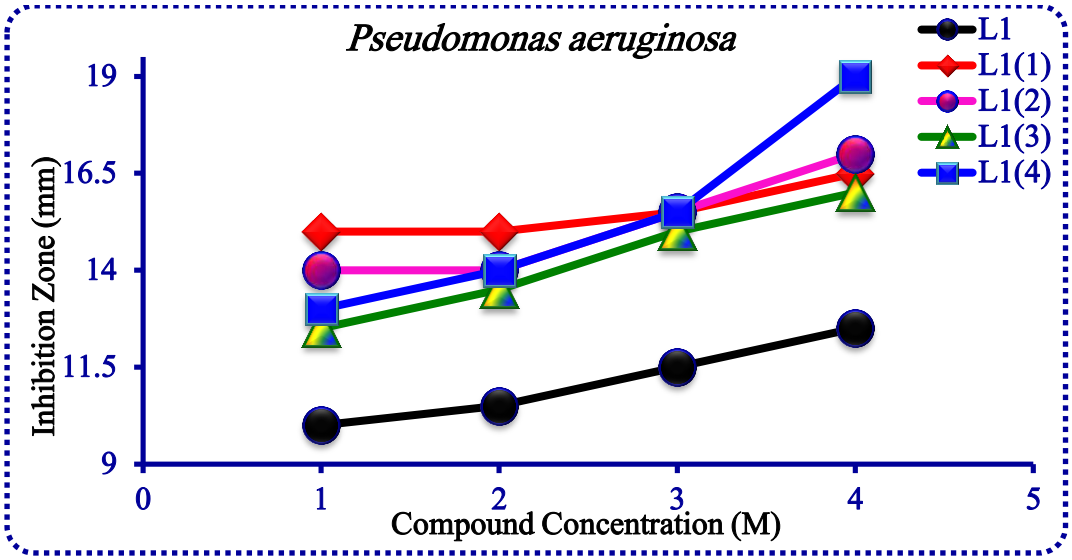
Figure(7): Vital effect of Ligand L_1 and Complexes in Vital Work of bacterial

Staphylococcus aureus

Table(6): Vital effect of Ligand L_1 and Complexes in Vital Work of bacterial

Pseudomonas aeruginosa

Concentration (M)	Compound Symbol				
	L_1	$L_1(1)$	$L_1(2)$	$L_1(3)$	$L_1(4)$
1×10^{-6}	-	++	+	-	-
1×10^{-5}	-	++	+	+	+
1×10^{-4}	-	+++	+++	++	+++
1×10^{-3}	-	+++	+++	+++	+++

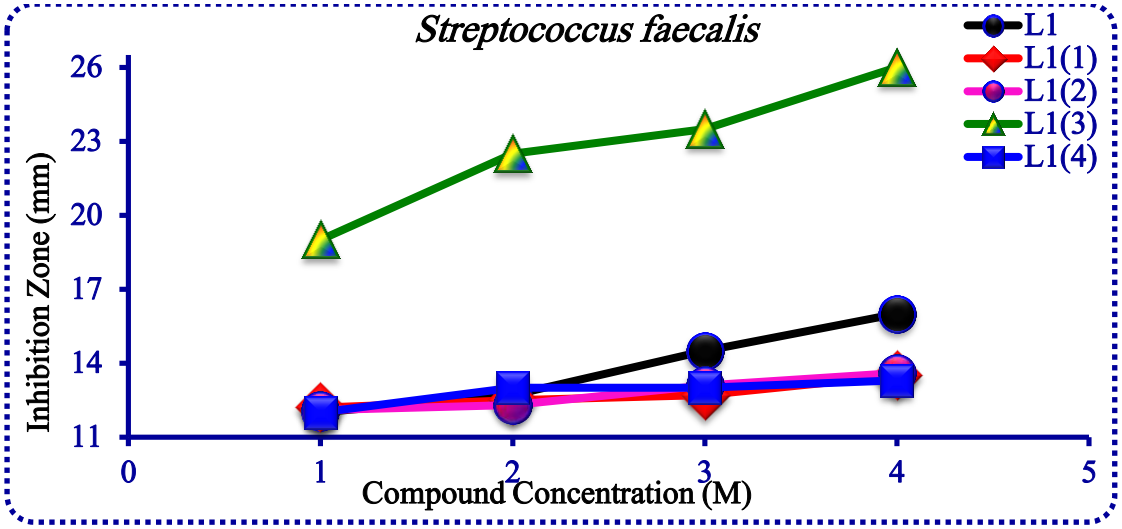


Figure(8): Vital effect of Ligand L_1 and Complexes in Vital Work of bacterial Pseudomonas aeruginosa

Table(7): Vital effect of Ligand L_1 and Complexes in Vital Work of bacterial

Pseudomonas aeruginosa

Concentration (M)	Compound Symbol				
	L_1	**$L_1(1)$**	**$L_1(2)$**	**$L_1(3)$**	**$L_1(4)$**
1×10^{-6}	-	-	-	+++	-
1×10^{-5}	-	-	-	+++	-
1×10^{-4}	++	-	+	+++	-
1×10^{-3}	+++	+	+	+++	+



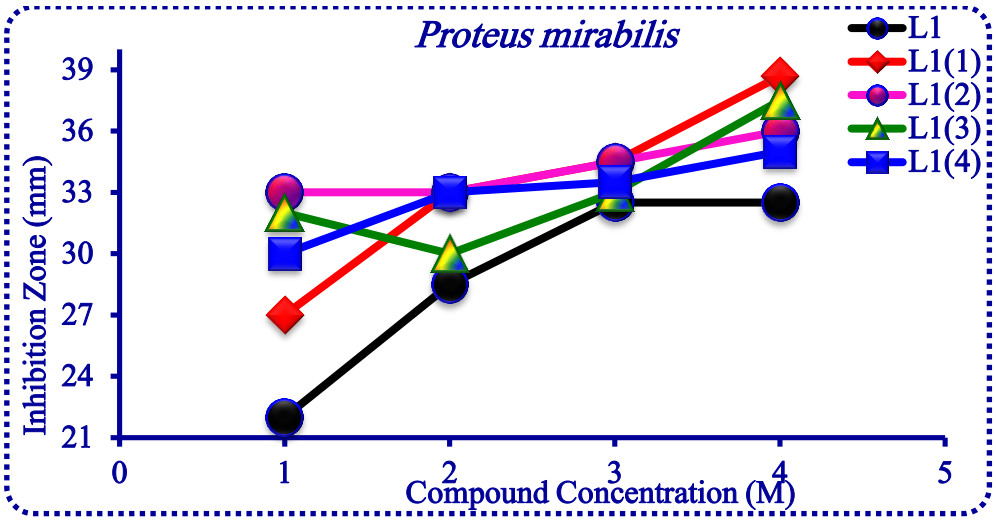
Figure(9): Vital effect of Ligand L_1 and Complexes in Vital Work of bacterial

Streptococcus Facials

Table(8): Vital effect of Ligand L_1 and Complexes in Vital Work of bacterial

Proteus Mirabils

Concentration (M)	Compound Symbol				
	L_1	$L_1(1)$	$L_1(2)$	$L_1(3)$	$L_1(4)$
1×10^{-6}	-	+	++	++	+
1×10^{-5}	+	++	++	+	++
1×10^{-4}	++	++	++	++	++
1×10^{-3}	++	+++	+++	+++	++



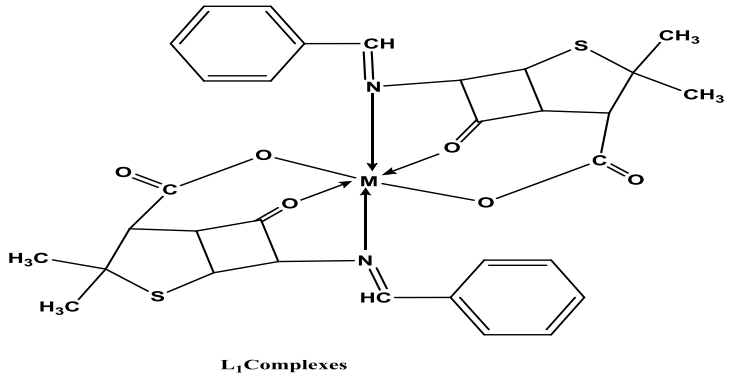
Figure(10): Vital effect of Ligand L_1 and Complexes in Vital Work of bacterial

Proteus Mirabils

Conclusion :

Novel Schiff base ligand have been preparation by the condensation of ^.Amino Penicillinic acid with benzaldehyde Identification by: FT-IR , Uv-Vis , mass spectrum , (C,H) NMR spectra , Molar Conductivity , Magnetic moment .

For sure, formation of ligand and mixed was metal elements for synthesis complexes showed the nitrogen and oxygen atoms coordination involving azomethene, Hydrogen group and β-Lactam respectively.



L_1Complexes

تحضير وتشخيص و فعالية مقاومة البكتيريا لليكاندات قواعد شيف و معقداتها

الخلاصة :-

تم تحضير ليكاند جديد من نوع قواعد شف من تكثيف (6.Amino Penicillinic acid) مع (Benzyldehyde) ثم تم مفاعلتها مع مجموعة من عناصر السلسلة الانتقالية الاولى لتحضير معقداتها ، شخص الليكاند والمعقدات بواسطة مجموعة من التقنيات الطيفية مثل ($^1H,C^{13}$) FT-IR , Uv-Vis , NMRوطيف الكتلة كما تم التوصيلية المولارية والخواص المغناطيسية للمعقدات المحضرة . تم قياس الفعالية البايلوجية لليكاند والمعقدات ضد انواع من البكتريا مثل (*S.aureus* , *pseudomonas aeruginosa* , *Streptococcus Facials* and *Proteus Mirabils*) والتي تم عزلها من التهابات مختلفة من قرح الفراش .

الكلمات المفتاحية :- بيتا- لاكتام ، قواعد شف ، مضادات البكتريا

Reference:

1. **Kaura ,Lalit Sharma and V. J. Dhār** , Int. J. Sci. 9(4) ,ISSN 0972-768x , 2011.
2. **S. Papanov M. Georgieva , D. Obreshkova and P. Atanasov** , Pharacia ,Vol. 61 , No. 2/2014 .
3. **Mahmdouh S. Masoudi , Alaa E. Ali and Nessma M. Nasr** ,Journal Of Chemical and Pharmaceutical Research , ISSN 0975-7384 ,Vol.6(11) , Pg.(28-58) , 2014
4. **Kevin Lorcheim** , Applied Biosafety Vol.16 , No. 1 , 2011.
5. **Abdulbaset A. Elgellal** , Journal of Medical and Bioengineering Vol. 3 , No.3 , 2014.
6. **Usharani M. , Akila E. and Rajavel** , International Journal Recent scientific Research, Vol.4 , Issue 9, pg.(1385-1390) , 2013.
7. **Aurora Reiss , Ariana Samide , Georgeta Ciobanu and Irina Dabuleanu** ,J. Chil. Chem. Soc , Vol.60 , Pg.(3074-3079), 2015.
8. **Mohammad S. Iqbal , Iftikhar H. Bukhari and Muhammad Arif** , Appl. Organometal , Chem. Vol.19 , Pages (864-869) , 2005.
9. **Noureddine Charef , Fouzia Sebti, Lekhmici Arrar , Meriem Djarmouni , Naouel Boussoualim , Abderrahmane Baghiani , seddik Khennouf , Ali Ourari , Murad A. Aldamen Mohammad S. Mubarak and Dennis G. Peters** , Journal Homepage :www. Elsevier Com /Locate /Poly , Vol. 85 , Pg.(450-456) , 2015.
10. **E. Akila , M. Usharani , R. Ashokan and R. Rajavel** , International Journal Of Metallurgical and Materials , ISSN 2278-2516 , Issue 4 , Vol. 3, Pg.(27-38) , 2013.
11. **Raj Kaushal , Sheetal , Rajeev Kaushal and Kiran Nehra** , International Journal Of Pharmacy and Pharmaceutical Sciences , ISSN 0975 -1491 ,Issue 2, Vol. 6, 2014.
12. **C. H. Latha Saranya , J. C. Thejaswini , B. M. Gurupadayya and B. Y. K. Sruthi** , IOSR Journal Of Pharmacy , ISSN 2319-4219 ,Issue 2 , Vol.4 , Pg.(12-18) ,2014.
13. **Taghreed H. Al-Noor , Manhel Reemon Aziz and Ahmed T. Al-Jeboori ,**Journal Of Chemical and Pharmaceutical Research , ISSN 0975-7384, Vol.6(4) ,Pg.(1225-1231), 2014.
14. **Muhammad Aslam , Itrat Anis , Nighat Afza , Ajaz Hussain ,Muhammad Safder, Shazia Yasmeen , Asif Hanif Chaudhry, Mehroze Ahmed Khan and Muhammad Niaz**, International Journal Of Current pharmaceutical Research , ISSN 0975-7066 , Issue 4 , Vol.4, Pg.(51-53) , 2012.
15. **Jabbar S. Hadi , Abdulelah A. Almyah and Ali G. Swadi** , Research Journal Of Pharmaceutical Biological and Chemical Sciences , ISSN 0975-8585 , Vol.5(4) , Pg.(233-246) , 2014.
16. **Daniel L. M. De Aguiar , Rosane A.S. San Gil ,Leandro B. Borre , Monica R.C. Marques and Andre L. Gemal** , International Journal Of

Pharmacy and Pharmaceutical Sciences , Issue 3 , Vol. 3 , Pg.(293-298) ,2011.

17. **Sengul Alpay-Karaoglu 2 and Neslihan Demirbas 1 Arch**., Pharm. Chem. Life Sci. 2013 , Vol.346 , Pg.(1-21).
18. **Susanne Sutterlin Evatano , Agneta Bergsten , Anna Brita Tallberg and Asa Melhus** , Acta Der Venereol , Vol. 92 , Pg.(34-39) , 2012.
19. **Suzanne J. Templer , Do. Maxino O**. **Brito** , MD Bacterial SSTIs , Vol.26 ,Pg.(9-16) , 2009.
20. **Yuji Morita** , **Junko Tomidandy and Oshiaki Kawamura**, Department Of Microbiology , School Of Pharmay , Aichi Gakuin University , Nagoya , Japan Microbiology , 2014, Vol.4 , Pp.(30-37).
21. **Ajay Kumar 1 and C.S. Vinod Kumar 2** , Int. J. Curr. Microbial. App. Sci. , 2016, Vol.5(1) , Pg.(200-208).