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Synthesis, Characterization and Analytical Study of Metal Complexes Derived from 1,2-Bis-(4′-Amino-2′,3′- Dimethyl-1′- Phenyl-Pyrazolinyl)-Diimino Ethane

J.M. Dhabab ,M. N. Al-Jboori ,H. H. AL-Bajalan

Department of Chemistry , College of Science. Al-Mustnsiriyah University

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Abstract

Six transition metal complexes of Cr (III), Mn(II), Co(II), Ni(II), Cu(II), and Zn(II) were prepared using 1,2-bis -(4-Amino-2,3-dimethyl-1- phenyl-pyrazolinyl)-diimino ethane(L) as ligand. These complexes were characterized by elemental analysis, magnetic susceptibility, UV/VIS and FT-IR spectroscopy. These data showed that the solid complexes of Mn(II), Co(II), Zn(II) were tetrahedral geometry, and Cr(III) was octahedral while the symmetry around Ni(II) and Cu(II) ions with the new ligand were square planar of the formula $[ML]Cl_2$, M=Ni(II) and Cu(II).

A simple method for the determination of trace amounts of copper as an example by spectrophotometer was described based on the formation of the Cu-L complex. The optimization were done on basis , reagent concentration , reaction time and pH medium for CuL- complex. The molar absorptivity of the complexes at pH 9.0 , molar ratio (1:1) (L:M) at 390 nm was $0.20x10^4$ L/mol.Cm, were determined after completion the stability of Cu(II) complex spectrophotometrically.

Key words : transition metal; complexes of transition metal; synthesized

of Schiff base ;4-amino antyprine; spectrophotometry

Introduction

Metal complexes of 4-amino antipyrine derivatives are extensively studied due to synthetic flexibility and sensitivity toward variety of metal ions [1]. They are found to be useful in catalyst, media as antibiotics and anti-inflammation agents[2-5], and as analytical reagent in determination of trace amounts of elements [6-7]. These derivatives, especially Schiff base having N_4 and N_2O_2 donor active sites These types of ligands and complexes were extensively studied because of their wide spectrum in applied analytical chemistry for determination of metal ions [8-15].

The present paper deals with the synthesis and characterization of new complexes of Cr (III), Mn(II) , Co(II), Ni(II), Cu(II), and Zn(II with 1,2-Bis-(4-Amino-2,3dimethyl-1-phenyl-pyrazolinyl)diimino ethane (L). Moreover, the spectroscopic analysis of Cu(II) ions were reported using the previous ligand as organic reagent after the optimization of the pH, λ_{max}, time and molar ratio (M:L), conditions.

Experimental

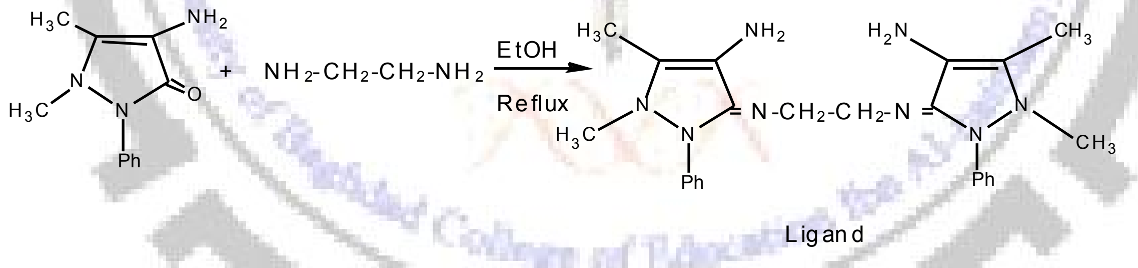
a - Materials and Methods

Reagent such as 4-amino antipyrine ,ethylenediamine ,metal chloride $CrCl_3.6H_2O$, $MnCl_2.4H_2O$, $CoCl_2.6H_2O$, $NiCl_2.6H_2O$, $CuCl_2.2H_2O$ and anhydrous $ZnCl_2$ were of commercial quality (Aldrich,Merck) and were used as received. Solvents were dried and purified using the standard technique. Micro analytical data (C.H.N.) were done using an elemental varia instrument(Carlo-Erba 1106 elemental analyzer in king university at Jordan). FT.IR spectra were recorded for solids in KBr and CsI media spectrophotometer. Electronic spectra of compounds solutions recorded in ethanol and DMF solvents on Varian UV-Visible spectrophotometer 100 conc type. Molar conductance of 10^{-3} solutions of the prepared complex in DMF were measured on Coring 220 conductivity meter. The percentages of metal in solid complexes were determined by Shimadzu A,A-670 spectrophotometer via F.A.A.S. Magnetic susceptibility measurements were obtained at 25 C° on the solid state applying Faradays method using Bruker BM6 instrument at al-Nahrain university .

b-Synthesis of Schiff base[1,2-Bis-(4-Amino-2,3-dimethyl-1-phenyl-pyrazolinyl)-diiminoethane]

The Schiff base (L) was synthesized by condensation of a inoantypyrine(4.066gm,20mM) in 80 ml of ethanol and (0.5gm,10mM) of ethylene diamine . The resulting reaction mixture was refluxed for 2 hours .The yellow solid precipitate of Schiff base was filtered ,washed with distilled water ,dried and recrystallized from ethanol ,and finally preserved in desiccator .yield: 75% (m.p. 195-197C°).

The preparation of the ligand was shown in scheme (1), as below:



Scheme (1)

Bis,1,2-(4´-Amino-2´,3´dimethyl-1´-phenyl-pyrazolinyl)diimino ethane (L).

Synthesis of Complexes

A solution of metal chloride in (15ml) ethanol (0.01 mole) was refluxed with an ethanolic solution(20ml) of the ligand (L) (0.01mole) for 2 hours.

The solution was then reduced to half on water bath . the solid complex precipitated was filtered , washed thoroughly with ethanol and dried in vacuum.

Study of complexes formation in solution

Complexes of the Schiff base (L) with metal ions Cr(III), Mn(II), Co(II), Ni(II),Cu(II), and Zn(II) were studied in solution using ethanol as solvent, in order to determine [M:L] ratio in the complex following molar ratio[16]. A series of solutions were prepared having a constant concentration 10^{-3} of the metal ion and L. Absorbencies were measured against blanks prepared separately for each concentration of chelating agent at the formed complex. The results of complexes formation in ethanol solution were listed in table (4) .

C-General Procedure of determination of Cu metal by uv.vis. spectrophotometry.

$1x10^{-3}$M of copper chloride (5ml) were transferred into a 25 mL of calibrated flask and $1x10^{-3}$ M (5ml) of ligand solution was added. The pH of the solution was adjusted with solution acetate buffer to pH 9.0, then the whole solution was mixed and diluted with DMF to the mark. After 5 min, the absorbance of this solution was measured at 390 nm by spectrophotometer against a reagent blank.

Results and Discussion

The complexes were prepared by reaction of ligand with metal chloride of Cr(III), Mn(II), Co(II), Ni(II), Cu(II), and Zn(II) in ethanol under reflux. these complexes are stable in this solution .The analytical and physical data wers listed in table (1). The higher decomposition points of complexes in comparison with free ligand suggest the thermal stability of complexes which increases with the increase of electro negativity of metal in the period (Mn,Co,Ni,and Cu).

I.R spectrum

The IR spectrum of the Schiff base shows a medium band at 3250 cm^{-1} which assigned to -NH_2 vibration. The ligand has no absorption at 1700 cm-1 which confirms the formation of ligand upon condensation with ethylene diamine. The strong absorption at 1615 cm^{-1} region is assigned to C=N vibration[17], fig. (1).

In the spectra of shift base complexes, the $\nu(NH_2)$ and $\nu_{(C=N)}$ modes are found to shift to lower or higher wave numbers, which indicate the linkage of four nitrogen atoms of amino acid , isomethine groups to metal atoms, all the complexes exhibited bands around (430-530 cm^{-1})[18], which could be assigned to the vibrations of ν(M-N),figure (2).furthermore, the chromium(III) complexes exhibited weak absorption in region (290-350 cm^{-1}) which is assignable to ν(Cr-Cl) vibrations[19].

The UV.Visble spectra of complexes

The electronic spectrum of ligand (10^{-3} M) in ethanol and complexes (10^{-3} M) in DMF, fig.(3), shows distinct peaks at 40000,31250 and 27777 cm^{-1} petaining to $\pi\rightarrow\pi^*$,$n\rightarrow\pi^*$, and charge transfer transitions of ligand [20].

The Cr(III) complex solution in DMF exhibits two weak bands at 14305 and 16129 cm^{-1} which may be assigned to $A_{2}g^{4} \rightarrow T_{2}g^{4}$ ($10D_q$) and $A_{2}g^{4} \rightarrow T_{1}g^{4}(p)$ respectively . These bands confirm the octahedral environment around Cr(III) ion[21] .

The electronic spectra of Mn (II) complex showed two spin allowed transition at 35714 and 26881 cm^{-1}, these bands have been assigned to $\pi\rightarrow\pi^*$ and metal to ligand charge transfer (MLCT), suggesting tetrahedral geometry around Mn(II) ions[22].

As well as Co(II) complex in DMF showed absorption bands in the visible region 16949, 16181 and 14598 cm^{-1} attributed to $A_2^4 \rightarrow T_2^4$, $A_2^4 \rightarrow T_1^4$, $A_2^4 \rightarrow T_1^4(P)$ respectively[23], are in conformity with tetrahedral rearrangement for Co(II) ion.

The red solution of Ni(II) complex exhibited absorption bands at 29411 and 18518 cm^{-1} which can be attributed to $A_1g^1 \rightarrow Eg^1$and $A_1g^1 \rightarrow B_2g^1$ transitions respectrivly,these bands confirm the square planner around Ni(II) ion[24].

Besides that the brownish solution of Cu(II) complex in DMF exhibited well–resolved bands at 16129 and 25316cm^{-1}, fig.(4), which are assigned to the transitions $B_1g^2 \rightarrow A_1g^2$ and $B_1g^2 \rightarrow B_2g^2$ respectively for a low spin square planner [24].

However , the Zn(II) complex exhibited only high intensity band at 27025cm-1 which is assigned as intra – ligand charge transfer (INCT) , this is in well agreement with tetrahedral symmetry of Zn(II) complex[25,26].

The molar conductance values of the soluble Mn(II) ,Co(II),Ni(II),Cu(II) and Zn(II) complexes in DMF solutions (10^{-3} M) were in the range (129-150)ohm^{-1}.cm^{2}.mol^{-1} indicated the electrolytic characters and confirm 1:2 ratio [27].

As well as the Cr(III) complex solution in DMF behaves as electrolyte in 1:1 ratio , having a molar conductance value of 95 ohm^{-1}.cm^{2}.mol^{-1} .

Magnetic Susceptibility Measurements

Magnetic moment for Cr(III),Mn (II)and Co(II) shows value of 3.30, 5.30 and 4.20

B.M, which agrees with tetrahedral symmetry [28] while Ni(II) and Zn(II) were diamagnetic and Cu(II) complex show decrease in magnetic property (1.05 BM) [29] due to the orbital contribution of square symmetry of Cu(II)ion. The magnetic mounts of the solid complexes at 300 K were consistent with the suggested geometries around the central metal ion.

The elemental analyses , molar conductance and the magnetic moment measurements agree with the proposed formula , and also confirmed the stoichiometries $[Cr^{III}LCl_2]Cl$, square planner of $[ML]Cl_2$ M=Ni^{II} and Cu^{II} and tetrahedral of $[ML]Cl_2$ M= Mn^{II} , Co^{II} and Zn^{II} fig.(8-10).

Determination of copper metal using the purposed method.

U.V-Vis spectrophotometry is an important instrumental method for the determination of metal ions in their inorganic or organic compounds[28] . The ligand reacts with several metal ions such as zinc, cobalt, copper, nickel, manganese and chromium to form metal complexes which were studies above . This chelating agent has been used to determine several metal ions by Uv-vis spectroscopy.

In order to high lights utility of the proposed method, it was used for the spectrophotometric determination of copper contents (as example)at optimal conditions fig.(5) , such as effect of pH and effect of time on the complex formation .

Effect of pH

The effect influences of pH of the solution on the formation of copper-L complex were investigated spectrophotometrically at 390 nm using various solutions of different pH values (2-9).

The results are given in Fig. 6. as complex begins to form at approximately pH 9.0, with maximum absorbance being reached at pH values between pH 2.0 to pH 9.0. In the light of these findings, complexes were found at pH 9.0 using acetate buffer solution.

Effect of time on the complex formation

The influence of time on the formation of the copper –L complex was also studied at the optimal working conditions.The development of the copper –L complex is completed in 5 minutes .The results are given in fig.(6) and the color is stable for at least 24 hour.

Calibration curve and detection limit

A calibration curve was constructed for copper in the range of $1x\ 10^{-1}$ to $1x\ 10^{-5}$M of copper at 305 nm according to the general procedure described above. The calibration curve can be represented by a linear regression equation: $y = 0.1047x + 0.0086$ ($r^2 = 0.999$) (y = absorbance, x = concentration).

The detection limits based on three times the standard deviations of the blank were $2x10^{-6}$ (N = 5). The relative standard deviations for the copper determinations were 2.3% (N = 5) at $1x10^{-4}$ Cu(II)

Suggested stereochemistry of somplexes

According to the results obtained from elemental analysis and spectral data, the structures of the above mentioned complexes can be illustrated as shown in fig.(8,9,10).

Stoichiometry of Cu- complex

Absorbencies were measured against blanks prepared separately for each concentration of chelating agent at the formed complex. The results of complexes formation in ethanol solution were listed in table (4). Fig.(5) showed the molar ratio of Cu:L was 1:1[30].

Conclusion

We have synthesized Cr(III), Mn(II), Co(II), Ni(II), Cu(II) and Zn(II) complexes of the monocyclic ligand formed by the condensation of 4-amino antipyrine and ethylene diamine.

The ligand behaves as tetradentate neutral via four nitrogen atoms of two amino groups of antipyrine ring and two nitrogen atoms of metal linkage to nitrogen donor give the complexes of thermal and kinetic stability.

The spectrophotometric analysis of copper ion using the ligand as reagent was studied at optimum conditions after determination the molar ratio which were pH = 9.0, λ_{max} = 390 nm and time of reaction was 10 minutes.

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Table(1): Physical properties, analytical data, magnetic moments and molar conductivities of the Prepared Compounds.

Λ* Ohm^{-1} cm^2 mol.$^{-1}$	μ eff. B.M	Yield %	Analysis Found(calc.%)				Compound
			N	H	C	M	
		70	26.04(25.99)	6.97(7.1)	66.97(66.99)		L
95	3.30	80	18.99(19.00)	5.08(4,91)	48.85(47.91)	8.99(8.71)	$[CrLCl_2]Cl$
146.5	5.3	80	20.14(21.01)	5.39(4.87)	51.79(50.79)	9.89(8.99)	$[MnL]Cl_2$
150.3	4.2	90	20.07(21.01)	6.01(5.91)	51.61(50.98)	10.12(9.81)	$[CoL]Cl_2$
135	0.0	85	20.81(21.91)	5,81(5.00)	50.91(49.92)	10.37(9.89)	$[NiL]Cl_2$
129	1.05	92	19.90(20.30)	4.92(3.81)	50.01(49.81)	11.27(10.95)	$[CuL]Cl_2$
141	0.0	73	20.31(20.61)	5.21(5.11)	51.00(50.81)	10.71(9.91)	$[ZnL]Cl_2$

*** the molar conductance were measured in 10^{-3} M solution (DMF).**

Table (2) : I.R spectral data of the ligand and its metals complexes (ν cm^{-1})

Compound	ν C=N	ν NH2	ν M-N	ν M-Cl	Other bands
L	1615(S)	3250(m)	---	---	1500-1600(C=C) 2970(C-H)
CrL	1590(s)	3180(br	500	290,350(w)	1400,1370.2970(C-H)
MnL	1690(s	3150(m)	460-490(m)	---	2980(C-H)
CoL	1575(s)	3200(br)	510-530(m)	---	2962(m)722(sCH2)
NiL	1575(s)	3250(br)	495(m)		
CuL	1580(m)	3185-3200(br)	490(m)	---	
ZnL	1630(s)	3200(w)	430 (m)	---	

s = strong, m = medium, br = broad and w = weak

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Table(3) :Electronic spectral data ν_{max} cm^{-1} of the prepared complexes

Compound	ν cm^{-1}	ε max. L.Mol^{-1}.cm^{-1}	Assignment	Proposed structure
L	27777 40,000	20500 12500	Charge transfer $\pi\rightarrow\pi^*$	---
CrL	28169 14305 16129	30500 230 80	Charge transfer $A_2g^4 \rightarrow T_2g^4$ $A_2g^4 \rightarrow T_1g^4$	Octahedral
CoL	37037 16949 16181 14598	13500 115 182 210	$\pi\rightarrow\pi^*$ $A^4_2\rightarrow T^4_{2\ (F)}$ $A^4_2\rightarrow T^4_{1\ (F)}$ $A^4_2(F)\rightarrow T^4_{1\ (P)}$	Tetrahedral
NiL	38461 29411 18518	10300 225 82	$\pi\rightarrow\pi^*$ $A_1g^1_{(D)}\rightarrow Eg^1_{(G)}$ $A_1g^1 \rightarrow B_1g^1_{(G)}$	Square planar
CuL	40816 25316 16129	3180 22500 118	$\pi\rightarrow\pi^*$ $B_1g^2\rightarrow A_1g^2$ $B_1g^2\rightarrow B_2g^2$	Square planar
MnL	35714 26881	1980 25000	$\pi\rightarrow\pi^*$ M→L. C.T	Tetrahedral
ZnL	34482 27025	3450 31200	$\pi\rightarrow\pi^*$ L→M C.T	Tetrahedral

Table (4) : Continuous variation data for Cu – L complex at λ_{max} (390 nm)

$V_L / V_{L+} V_M$	abs.
0.10	1.572
0.20	1.900
0.40	3.435
0.45	3.198
0.82	1.883
0.85	1.550
0.86	1.482

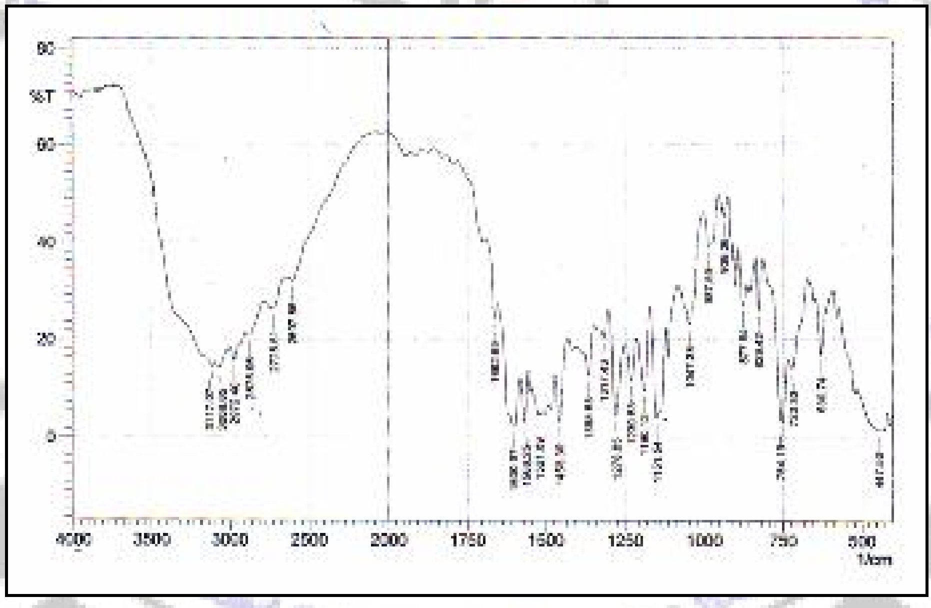


Fig. (1) : the FT-IR of ligand in KBr-disk

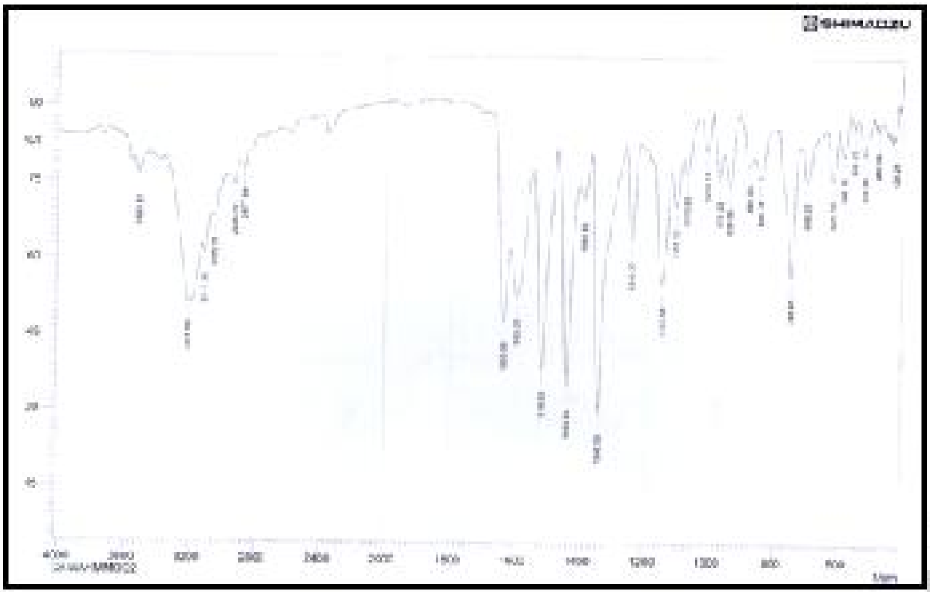


Fig. (2) : The FT-IR of Co (II) complex in CsI-disk

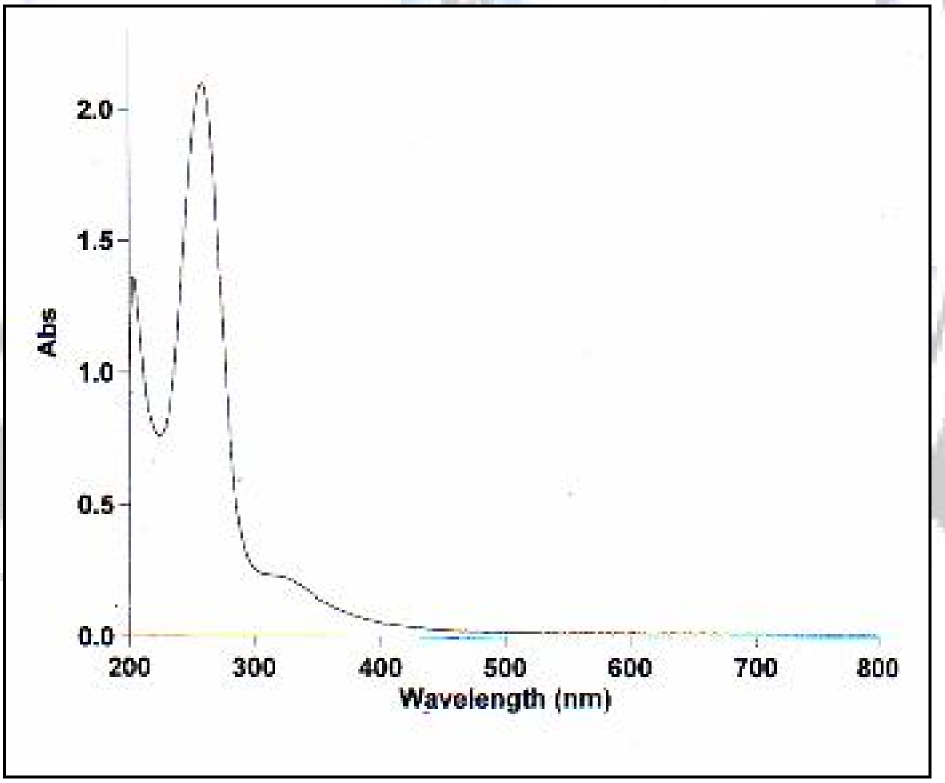


Fig. (3) : The UV. –Vis. of ligand in ethanol

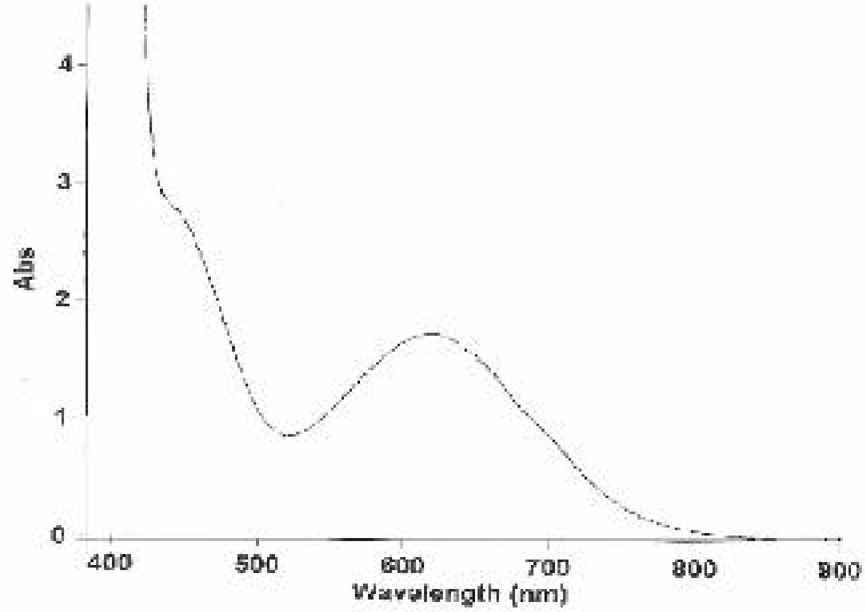


Fig. (4) : The UV. –Vis. of Cu-L complex in DMF

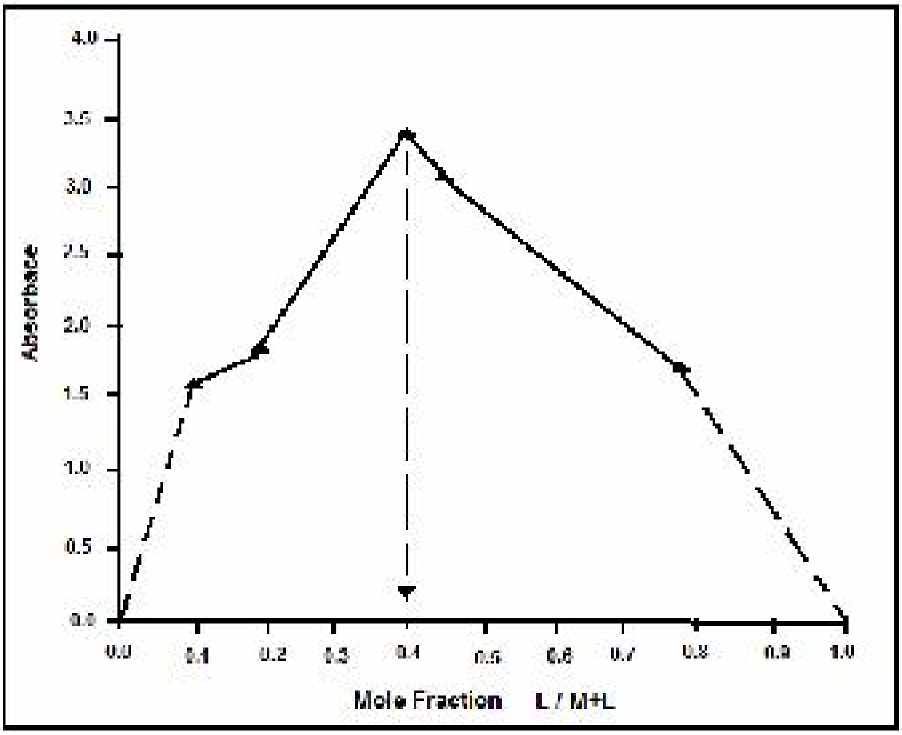


Fig.(5) :continuous variation plot for the Cu(II) complex at λ_{max} =390 nm .

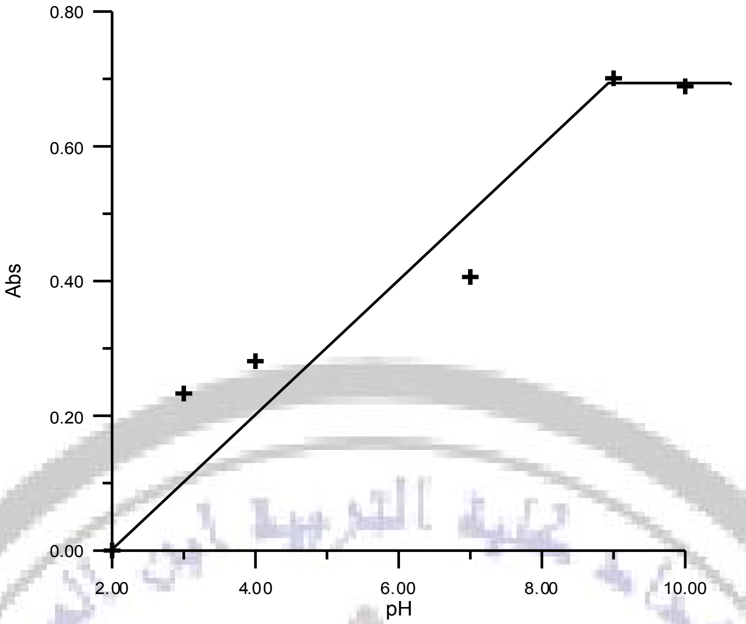


Fig. (6) : Effect of pH on formation of Cu : L complex

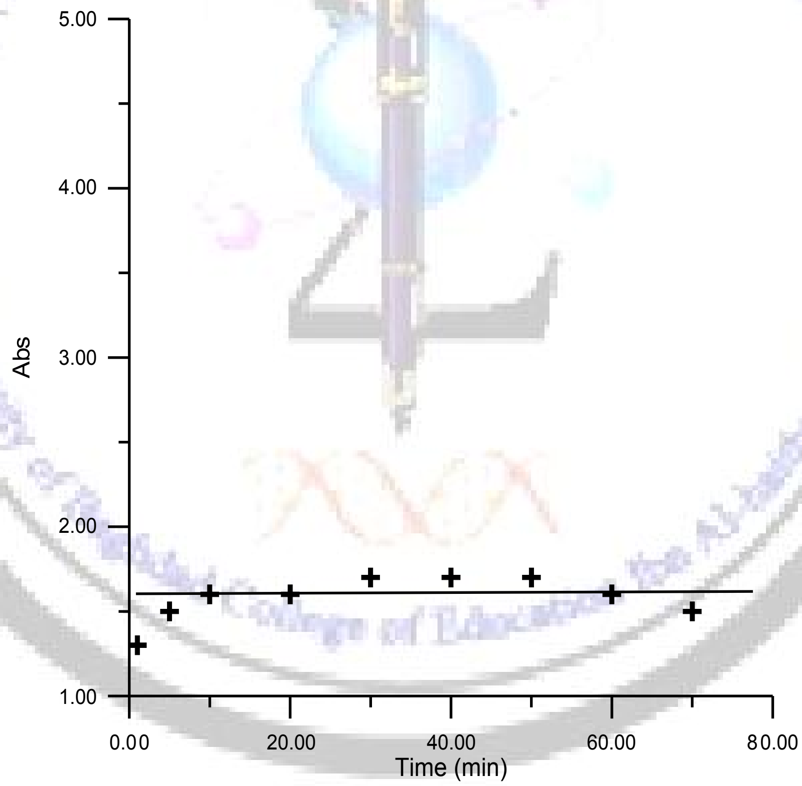


Fig. (7) : Effect of time on complex formation CuL

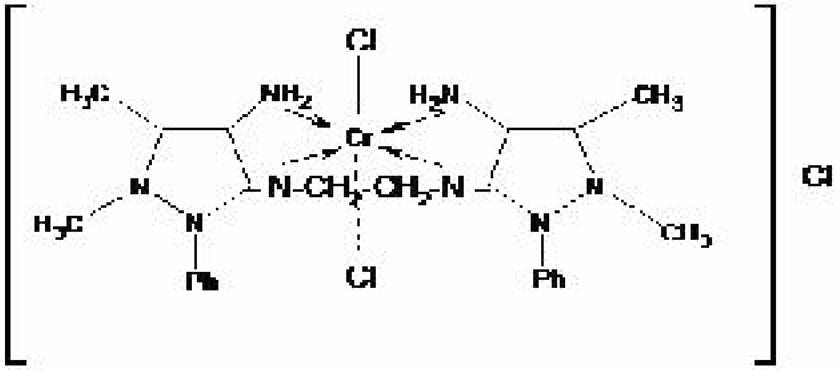
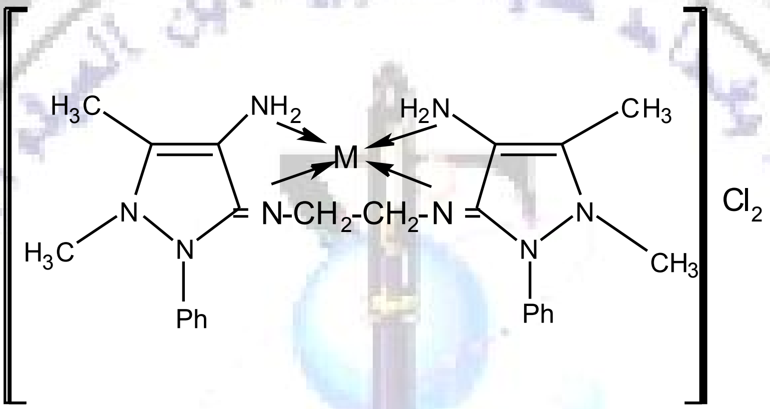
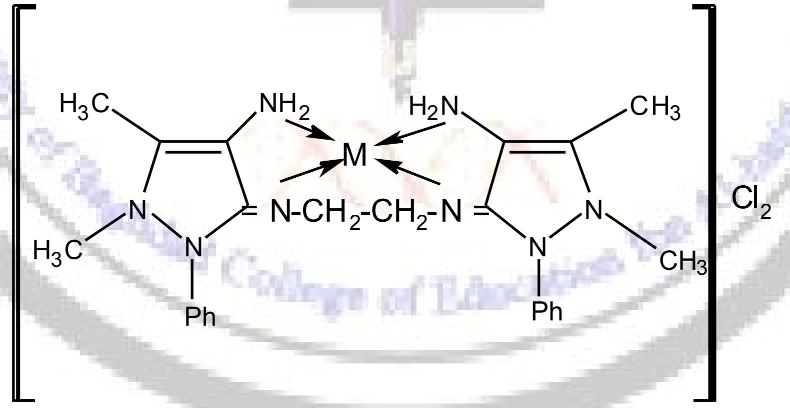


Fig. (8) : Proposed structure of Cr(III) complex



M : Mn^{+2} , Co^{+2} and Zn^{+2}

Fig. (9) : Proposed structure of Mn(II), Co(II) AND Zn(II) complexes



M : Ni^{+2} and Cu^{+2}

Fig. (10) : Proposed structure of Ni(II) and Cu(II) complexes

تشخيص ودراسة تحليلية لمعقدات بعض الفلزات المشتقات من 1-2ثنائي (4´-امينو-´2-´3 ثنائي مثيل - ´1-فنيل بايروزولين-ايل)-دايمينو ايثان

جميل موسى ضباب، محمود نجم الجبوري ، هناء حسن الباجلان

قسم الكيمياء ، كلية العلوم ،الجامعة المستنصرية

استلم البحث في : 7 شباط 2011 قبل البحث في : 18 تشرين الأول 2011

الخلاصة

حضرت معقدات جديدة لبعض ايونات عناصر السلسلة الانتقالية الاولى الكروم الثلاثي و المنغنيز و الكوبلت والنيكل والنحاس و الزنك الثنائية مع قاعدة شيف مشتقات من 4- امينو –انتي بايرين و اثلين ثنائي الامين.

شخص الليكاند (L) ومعقداته بواسطة التحليل الدقيق للعناصر (C.H.N) ، طيف الامتصاص الذري اللهبي فضلاً عن الطرائق الطيفية المعروفة FT-IR , UV-Vis ، قياس الحساسية المغناطيسية للمعقدات الصلبة ، واظهرت الدراسات الطيفية ان الليكاند يسلك رباعي الارتباط من خلال ذرات نتروجين مجاميع الايزوميثاين و الامين كما استنتج الشكل ثماني السطوح مثل معقد الكروم الثلاثي ،و الشكل المربع المستوي للنيكل والنحاس الثنائية فيما اظهرت معقدات المنغنيز والكوبلت والزنك تماثلا رباعي السطوح .

اجريت دراسة تحليلية طيفية لتعيين عنصر النحاس بأستعمال الليكاند المحضر كاشفا عضويا بعد تثبيت الظروف المثلى من زمن التفاعل ، الدالة الحامضية pH و دراسة النسبة المولية (L : M) ووجد ان الكاشف (L) يعطي انتقائية جيدة للنحاس الثنائي عند $pH = 9$ وزمن 10 دقيقة وبنسبة مولية(1:1) (L : M) وعند امتصاصية مولاريه $0.20x10^4$ L/mol.cm.

الكلمات المفتاحية: معقدات العناصر الانتقالية، قواعد شيف ، 4 -امينوانتي بايرين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