



## Synthesis and Characterization of Some New Mixed-Ligand Complexes Containing Schiff Base and 3-Picoline with Some Metal Salts

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### Abstract

Some metal ions ( $Mn^{+2}$ ,  $Co^{+2}$ ,  $Ni^{+2}$ ,  $Cu^{+2}$ ,  $Zn^{+2}$  and  $Cd^{+2}$ ) complexes of quodridentats Schiff base derived from (2-hydroxy benzaldehyde and 4,4'-methylenedianiline as primary ligand and 3-picoline (3-pic) secondary ligand have been synthesized and characterized on the basis of their  $^1H$ ,  $^{13}C$ -NMR, FT-IR, UV-Vis spectroscopy, conductivity measurements, elemental analysis, and magnetic moments, metal to ligands ratio in all complexes has been found to be (1:1:2) (M:Schiff base:3-pic), Schiff base behaves as neutral tetra dentate ligand with ( $N_2, O_2$ ) system from the results obtained, the following general formula has suggested for the prepared complexes  $[M^{+2}(2-mbd)(3-pic)_2]$  and octahedral stereochemistry, Where  $M^{+2} = (Mn, Co, Ni, Cu, Zn \text{ and } Cd)$ , 2-mbd = 2,2'-(1E,1'E)-(4,4'-methylene bis (4,1-phenylene) bis (azan-1-yl-1-ylidene) bis(methan-1-yl-1-ylidene)diphenol (2-mbd), 3-pic = 3-picoline.

**Key words:** Mixed ligand, Schiff bases, metal ions, 3-pic.

## Theoretical

Schiff bases represent one of the most widely used families of organic compound and their chemistry is essential material in many organic chemistry text books[1,2] and are well known to have antifungal antitumor and herbicidal activities[3,4] the complex behavior of Schiff base as ligands, with transition metal is very useful for bio-inorganic chemists, several complexes of palladium were synthesized and used as anti cancer and having biological activities[5,6] Schiff base containing nitrogen and oxygen donor atoms play an important role in biological system and represent interesting models for metalloenzymes as in the complex behavior of Schiff bases with various first series transition metal ions ( $\text{Cu}^{+2}$ ,  $\text{Co}^{+2}$ ) metal complexes were synthesized and characterized and these complexes have shown good anti bacterial and antifungal activities[7].

We have investigated in this paper, the preparation and properties of some metal ion complexes with Schiff base (2-mbd) and amine adduct (3-picoline).

## Experimental

All reagents used were analar or chemically pure grade by British drug house (BDH), Merck and Fluka. Metal salts ( $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$ ,  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ ,  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ ,  $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ ,  $\text{ZnCl}_2$  and  $\text{CdCl}_2 \cdot \text{H}_2\text{O}$ ), 3-Picoline, 2-Hydroxybenzaldehyde, 4,4'-Methylenedianiline, Glacial acetic acid, DMSO, Ethanol.

## Instruments

$^1\text{H}$  and  $^{13}\text{C}$ -NMR were recorded using ultra shield 300 MHz Switzerland at University of Al-Albyt, Jordan, conductivity measurements were carried out Philips PW digital meters conductivity in DMSO at  $10^{-3}$  M, FT-IR spectra were recorded as KBr discs in the range (4000-400)  $\text{cm}^{-1}$  using Shimadzu FT-IR, UV-Visible spectra were recorded by Shimadzu UV-8300 vis160A ultraviolet spectrophotometer the range of (200-1000) nm at  $10^{-3}$  M in DMSO. Metal contents of the complexes were determined by atomic absorption using (Shimadzu at 6806) atomic absorption spectrophotometer, magnetic susceptibility ( $\mu_{\text{eff}}$ , B.M) were recorded by faraday method using balance magnetic susceptibility model (MSB-MKT). Melting point was determined by using (Stuart-melting point apparatus).

## General method for the synthesis of the Schiff base (2-mbd)

to solution of 4,4'-methylenedianiline (3gm) (0.015mole) was added (3.67gm) (0.03mole) of 2-hydroxy benzaldehyde, few drops of glacial acetic acid have been added to the reaction mixture, the color of reaction mixture changed immediately to pale yellow. The final mixture reflux for 3hrs, then allowed to cool at room temperature, the precipitated pale yellow powder has been filtered off and recrystallization from ethanol, scheme[1], yield (91%), (M.P =163-165)  $^{\circ}\text{C}$ , %C found (79.005) while calculate (79.78), %H found (5.191) while calculate(5.46), %N found (7.063) while calculate(6.89).

## Synthesis of metal complexes

(0.41gm) (1mmole) of Schiff base (2-mbd) was dissolved in 25ml of ethanol containing (0.12gm) (2mmole) of KOH, then the solution of following metal salts  $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$  (0.2gm, 1mmole),  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$  (0.24gm, 1mmole),  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$  (0.24gm, 1mmole),  $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$  (0.2 gm, 1mmole),  $\text{ZnCl}_2$  (0.14gm, 1mmole) and  $\text{CdCl}_2 \cdot \text{H}_2\text{O}$  (0.2gm, 1mmole) in ethanol were added dropwise to the solution of the Schiff base (2-mbd), the mixture was stirred for 2 hour at room temperature. Complexes were separated by adding an excess of (3-pic) (2mole, (1-2)

ml and treating the solution with diethyl ether until is completed precipitation, the precipitate was washed with distilled water and ethanol and dried under vacuum.

## Results and discussion

The isolated complexes were crystalline solids, soluble in some of the common solvents such as dimethyl formamide, dimethyl sulphoxide, they are relatively thermally stable, the conductivity measurements in DMSO indicated the non-electrolyte behavior, Table (1) in clouds the physical properties. The analytical data confirmed the (1:1:2) (metal:Schiff base: 3-pic) composition of the complexes, the magnetic measurements ( $\mu_{\text{eff}}$  B.M) for the complexes are also listed in Table (1).

## Spectral studies

### $^1\text{H}$ and $^{13}\text{C}$ -NMR spectra

1-  $^1\text{H}$ NMR spectrum for the Schiff bases (2-mbd) in ( $\text{CDCl}_3$ ) as solvent Fig. (1) showed the following signals: single peak at  $\delta(4.03-4.11)$  ppm for (2H,  $\text{CH}_2$ ), doublet peaks at  $\delta(6.95-7.05)$  ppm for (8H, aromatic protons), multiples peaks at  $\delta(7.24-7.41)$  ppm for (8H, aromatic protons), single peak at  $\delta(8.61-8.69)$  ppm for (1H,  $\text{HC}=\text{N}$ ), single peak at  $\delta(13.31)$  ppm for (1H, OH).

2- The carbon nuclear magnetic resonance spectrum for the Schiff bases was carried out using ( $\text{CDCl}_3$ ) as a solvent and the following peaks were detected, Fig. (2): signal at  $\delta(41.03)$  ppm for ( $\text{CH}_2$ ) signal at  $\delta(77.50)$  ppm for ( $\text{CDCl}_3$ ), signals at  $\delta(117.25-146.71)$  ppm for aromatic carbons, signal at  $\delta(161.13)$  ppm for ( $\text{CH}=\text{N}$ ), signal at  $\delta(162.19)$  ppm for (C-OH).

### Infrared spectra

FT-IR spectrum of the Schiff bases (2-mbd) Fig. (3), showed bands due to  $\nu(\text{OH})$ ,  $\nu(\text{C}=\text{N})$  and  $\nu(\text{C}-\text{O})$  respectively which absorbed at  $(3419)$   $\text{cm}^{-1}$ ,  $(1618)$   $\text{cm}^{-1}$  and  $(1270)$   $\text{cm}^{-1}$ [8-11]. The spectrum of free (3-picoline) showed band at  $(1613)$   $\text{cm}^{-1}$  was assigned to  $\nu(\text{C}=\text{N})$ [12]. The FT-IR spectra of complexes: a strong sharp absorption band around  $(1618)$   $\text{cm}^{-1}$  in spectrum of the Schiff base may be assigned to the  $\nu(\text{C}=\text{N})$  stretching, in spectra complexes, this band is shifted the range between  $(1600-1612)$   $\text{cm}^{-1}$ [10,11] upon complexation with metal, which may be attributed to the coordination of the imine nitrogen to the metal center[13]. The Schiff base shows band at  $(3419)$   $\text{cm}^{-1}$  due to  $\nu(\text{OH})$  stretching from phenolic group which disappears in the spectra complexes indicating the deprotonation of the Schiff base upon complexation[14]. The strong phenolic (C-O) absorption band at  $(1270)$   $\text{cm}^{-1}$  absorbed in the spectrum of Schiff bases shifts to higher frequency at range  $(1281-1369)$   $\text{cm}^{-1}$ , supporting the coordination of the deprotonated phenolic oxygen atoms to the metal centers in the complexes[14]. The stretching vibration band  $\nu(\text{C}=\text{N})$  appeared in the range  $(1504-1566)$   $\text{cm}^{-1}$  shifted to lower frequencies by  $(47-109)$   $\text{cm}^{-1}$  which means that the nitrogen atom of (3-pic) was involved in coordination[15] absorption bands in the  $(442-525)$   $\text{cm}^{-1}$  region are considered to be due to metal-nitrogen  $\nu(\text{M}-\text{N})$  vibrations whilst those accruing around  $(515-580)$   $\text{cm}^{-1}$  are through to a wise from metal-oxygen (M-O) vibration[16], Fig. (4) showed the FT-IR spectrum of  $[\text{Ni}(2\text{-mbd})(3\text{-pic})_2]$  and Table (2) describe the important bands and assignment for ligands and its complexes.



## Electronic spectral

The absorptions and assignments related to the ligands and their complexes are listed in Table (3), the ligand Fig. (5) exhibited an absorption bands in (UV) region at wave number (37593)  $\text{cm}^{-1}$  which may be attributed to ( $\pi \rightarrow \pi^*$ ) transition, other band appeared at (29069)  $\text{cm}^{-1}$  was expressed at the ( $n \rightarrow \pi^*$ ) free (3-picoline) spectrum showed absorption band at (30769)  $\text{cm}^{-1}$  which was expressed as the ( $n \rightarrow \pi^*$ ) [17].

## Spectra of the complexes

[Mn(2-mbd)(3-pic)<sub>2</sub>] ( $d^5$ ): The spectrum of this complex showed four absorptions at (37593)  $\text{cm}^{-1}$ , (28985)  $\text{cm}^{-1}$ , (13513)  $\text{cm}^{-1}$  and (11111)  $\text{cm}^{-1}$  which may be attributed to (L.F), (C.T),  ${}^6A_{1g} \rightarrow {}^4T_{2g(G)}$  and  ${}^6A_{1g} \rightarrow {}^4T_{1g(G)}$  respectively [18,19].

[Co(2-mbd)(3-pic)<sub>2</sub>] ( $d^7$ ): The spectrum of the this complex, Fig. (6) exhibited bands at (37593)  $\text{cm}^{-1}$ , (28985)  $\text{cm}^{-1}$  (27855)  $\text{cm}^{-1}$ , (17857)  $\text{cm}^{-1}$  and (12345)  $\text{cm}^{-1}$  which have been assigned as (L.F), (C.T),  ${}^4T_{1g(F)} \rightarrow {}^4T_{1g(P)}$ ,  ${}^4T_{1g} \rightarrow {}^4A_{2g}$  and  ${}^4T_{1g(F)} \rightarrow {}^4T_{2g(F)}$  [20] respectively, and the  $B^-$  found to be at (578.46)  $\text{cm}^{-1}$  and  $\beta = B^- / B_0$  comes out to be (0.595).

[Ni(2mbd)(3-pic)<sub>2</sub>] ( $d^8$ ): The spectrum of this complex exhibited the following absorptions at (37593)  $\text{cm}^{-1}$ , (29068)  $\text{cm}^{-1}$ , (16260)  $\text{cm}^{-1}$  and (12437)  $\text{cm}^{-1}$  which have been assigned as (L.F),  ${}^3A_{2g} \rightarrow {}^3T_{1g(P)}$ ,  ${}^3A_{2g} \rightarrow T_{1g(F)}$  and  ${}^3A_{2g} \rightarrow {}^3T_{2g}$  respectively [21] and the  $B^-$  found to be at (534.5)  $\text{cm}^{-1}$  and  $\beta = B^- / B_0$  comes out be at (0.513).

[Cu(2-mbd)(3-pic)<sub>2</sub>] ( $d^9$ ): The spectrum of complex gave two bands at (37593)  $\text{cm}^{-1}$  and (28985)  $\text{cm}^{-1}$  which may be attributed to (L.F), (C.T) and another band shows at (12658)  $\text{cm}^{-1}$  which may be attributed to  ${}^2E_g \rightarrow {}^2T_{2g}$  transitions [22,23].

[Zn(2mbd)(3-pic)<sub>2</sub>] and [Cd(2-mbd)(3-pic)<sub>2</sub>] ( $d^{10}$ ): The complexes [Zn(2-mbd)(3-pic)<sub>2</sub>] and [Cd(2-mbd)(3-pic)<sub>2</sub>] confirmed the absence of any (d-d) transition [24].

According to spectral data as well as those obtained from elemental analysis, the chemical structure of the complexes may be suggested as oh octahedral Fig. (7) for  $[M^{+2}(2-mbd)(3-pic)_2]$  where  $M^{+2} = (\text{Mn}, \text{Co}, \text{Ni}, \text{Cu}, \text{Zn}$  and  $\text{Cd})$

2-mbd = 2,2'-(1E,1'E)-(4,4'-methylenebis(4,1-phenylene))bis(azan-1-yl-1-ylidene))bis(methan-1-yl-1-ylidene)diphenol

(3-pic) = 3-picoline

## Conclusions

Some metal ions ( $\text{Mn}^{+2}$ ,  $\text{Co}^{+2}$ ,  $\text{Ni}^{+2}$ ,  $\text{Cu}^{+2}$ ,  $\text{Zn}^{+2}$  and  $\text{Cd}^{+2}$ ) complexes of Schiff base (2-mbd) and 3-picoline have been synthesized and characterized on the basis of their ( ${}^1\text{H}$  and  ${}^{13}\text{C}$ -NMR, FT-IR, UV-Vis) spectroscopy. The complexes showed octahedral geometry around the metal.

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**Table (1): Physical properties of ligand and its complexes**

Complexes	Color	M.P °C	M % Calculate (Found)	Molar conductivity ( $\text{Oh}^{-1}.\text{cm}^2 \text{mole}^{-1}$ ) in DMSO	$\mu_{\text{eff}}$ (B.M)
Schiff base (2-mbd)	Yellow	163-165 °C	-	2.8	-
[Mn(2-mbd)(3-pic) <sub>2</sub> ]	Brown	210-212 °C	8.50 (7.93)	5.20	5.72
[Co(2-mbd)(3-pic) <sub>2</sub> ]	Deep-violet	290-292 °C	9.07 (8.74)	5.78	4.35
[Ni(2-mbd)(3-pic) <sub>2</sub> ]	Deep-green	260-262 °C	9.03 (8.69)	7.50	3.26
[Cu(2-mbd)(3-pic) <sub>2</sub> ]	deep-brown	201-203 °C	9.71 (9.13)	4.45	1.72
[Zn(2-mbd)(3-pic) <sub>2</sub> ]	Yellow	312-314 °C	9.96 (8.95)	9.83	-
[Cd(2-mbd)(3-pic) <sub>2</sub> ]	Yellow	310-312 °C	15.98 (15.23)	7.18	-

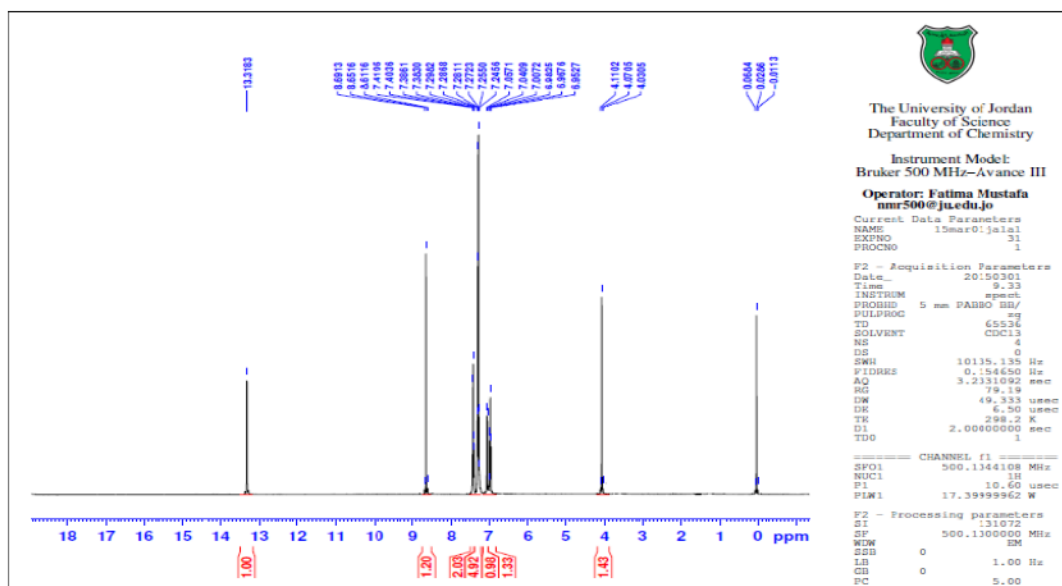
**Table (2): The characteristic infrared of the ligands (2-mbd), (3-pico) and their metal complexes**

Complexes	(C=N) (3-pico)	(C=N) Ligand	(OH)	(C-O) Phenolic	(M-N)	(M-O)
Schiff base (2-mbd)	-	1618 <sub>(s)</sub>	3419 <sub>(m)</sub>	1270 <sub>(s)</sub>	-	-
3-pico	1613 <sub>(s)</sub>	-	-	-	-	-
[Mn(2-mbd)(3-pic) <sub>2</sub> ]	1566 <sub>(m)</sub>	1600 <sub>(m)</sub>	-	1281 <sub>(s)</sub>	525 <sub>(w)</sub> 470 <sub>(w)</sub>	550 <sub>(w)</sub>
[Co(2-mbd)(3-pic) <sub>2</sub> ]	1523 <sub>(s)</sub>	1606 <sub>(s)</sub>	-	1311 <sub>(s)</sub>	457 <sub>(w)</sub> 432 <sub>(w)</sub>	519 <sub>(w)</sub>
[Ni(2-mbd)(3-pic) <sub>2</sub> ]	1504 <sub>(s)</sub>	1606 <sub>(s)</sub>	-	1311 <sub>(s)</sub>	515 <sub>(m)</sub> 462 <sub>(w)</sub>	580 <sub>(m)</sub>
[Cu(2-mbd)(3-pic) <sub>2</sub> ]	1504 <sub>(s)</sub>	1610 <sub>(s)</sub>	-	1369 <sub>(s)</sub>	447 <sub>(w)</sub> 465 <sub>(w)</sub>	519 <sub>(m)</sub>
[Zn(2-mbd)(3-pic) <sub>2</sub> ]	1529 <sub>(s)</sub>	1612 <sub>(s)</sub>	-	1321 <sub>(s)</sub>	451 <sub>(m)</sub> 463 <sub>(w)</sub>	519 <sub>(m)</sub>
[Cd(2-mbd)(3-pic) <sub>2</sub> ]	1504 <sub>(m)</sub>	1606 <sub>(s)</sub>	-	1317 <sub>(m)</sub>	448 <sub>(w)</sub> 442 <sub>(w)</sub>	519 <sub>(m)</sub>

Where: s = strong , m = medium , w = weak

Table (3): Electronic spectra data of ligands and its complexes

Compound	$\lambda_{\max}(\text{nm})$	Wav number $\text{cm}^{-1}$	$\epsilon_{\max}$ $\text{L.mol}^{-1}.\text{cm}^{-1}$	Assignment
Schiff base (2-mbd)	266	37593	921	$\pi \rightarrow \pi^*$
	344	29069	2254	$n \rightarrow \pi^*$
3-pic (ligand)	325	30769	600	$n \rightarrow \pi^*$
[Mn(2-mbd)(3-pic) <sub>2</sub> ]	266	37593	863	L.F
	345	28985	2108	C.T
	740	13513	16	${}^6A_{1g} \rightarrow {}^4T_{2g}(G)$
	900	11111	15	${}^6A_{1g} \rightarrow {}^4T_{1g}(G)$
[Co(2-mbd)(3-pic) <sub>2</sub> ]	266	37593	922	L.F
	345	28985	1754	C.T
	359	27855	198	${}^4T_{1g} \rightarrow {}^4T_{1g}(P)$
	560	17857	50	${}^4T_{1g} \rightarrow {}^4A_{2g}$
	810	12345	15	${}^4T_{1g} \rightarrow {}^4T_{2g}$
[Ni(2-mbd)(3-pic) <sub>2</sub> ]	266	37593	830	L.F
	344	29069	1994	${}^3A_{2g} \rightarrow {}^3T_{1g}(P)$
	615	16260	15	Mix (d with) C.T
	804	12437	10	${}^3A_{2g} \rightarrow {}^3T_{1g}(F)$ ${}^3A_{2g} \rightarrow {}^3T_{2g}(F)$
[Cu(2-mbd)(3-pic) <sub>2</sub> ]	266	37593	906	L.F
	345	28985	1754	C.T
	790	12658	60	${}^2E_g \rightarrow {}^2T_{2g}$
[Zn(2-mbd)(3-pic) <sub>2</sub> ]	266	37593	1034	L.F
	330	30303	1890	L.F
	407	24570	2124	C.T
[Cd(2-mbd)(3-pic) <sub>2</sub> ]	266	37593	723	L.F
	345	28985	1580	C.T

Figure (1): The <sup>1</sup>H-NMR spectrum of ligand Schiff base (2-mbd)



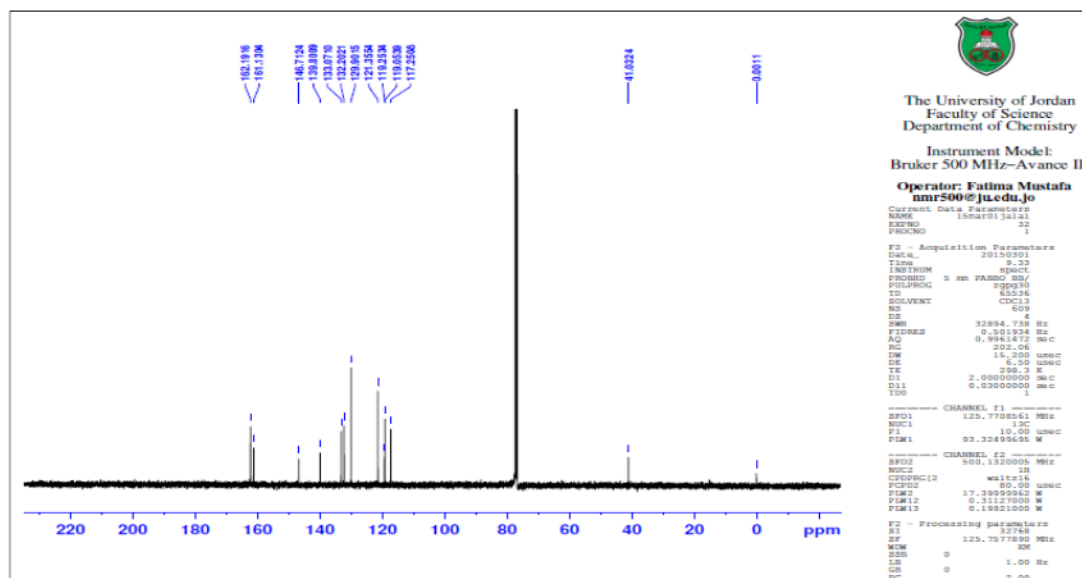


Figure (2): The  $^{13}\text{C}$ -NMR spectrum of ligand Schiff base (2-mbd)

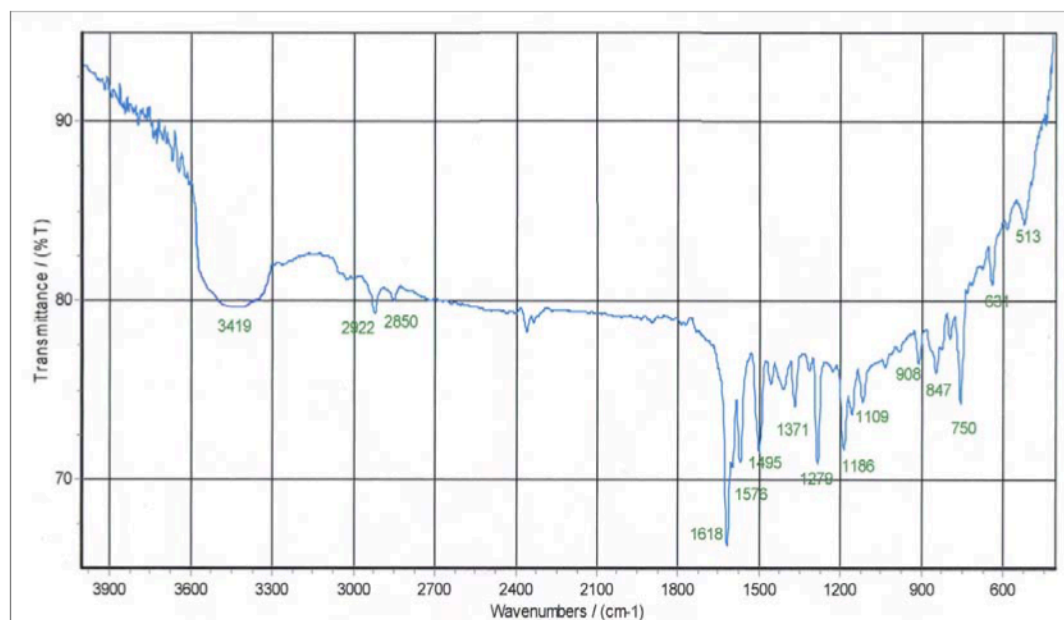


Figure (3): FT-IR spectrum of ligand Schiff base (2-mbd)



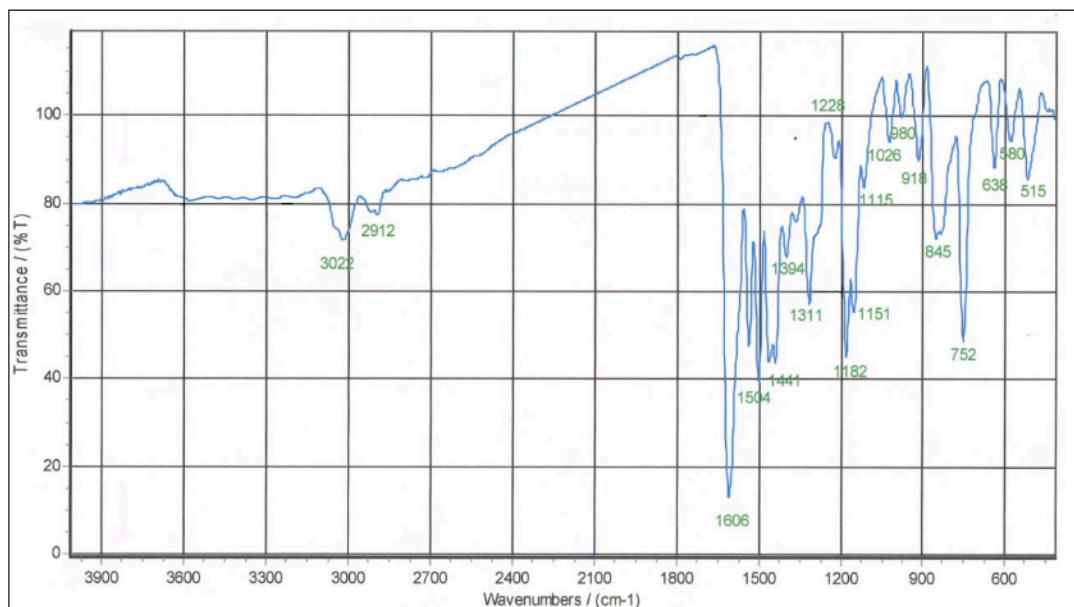


Figure (4): FT-IR spectrum of [Ni(2-mbd)(3-pic)<sub>2</sub>]

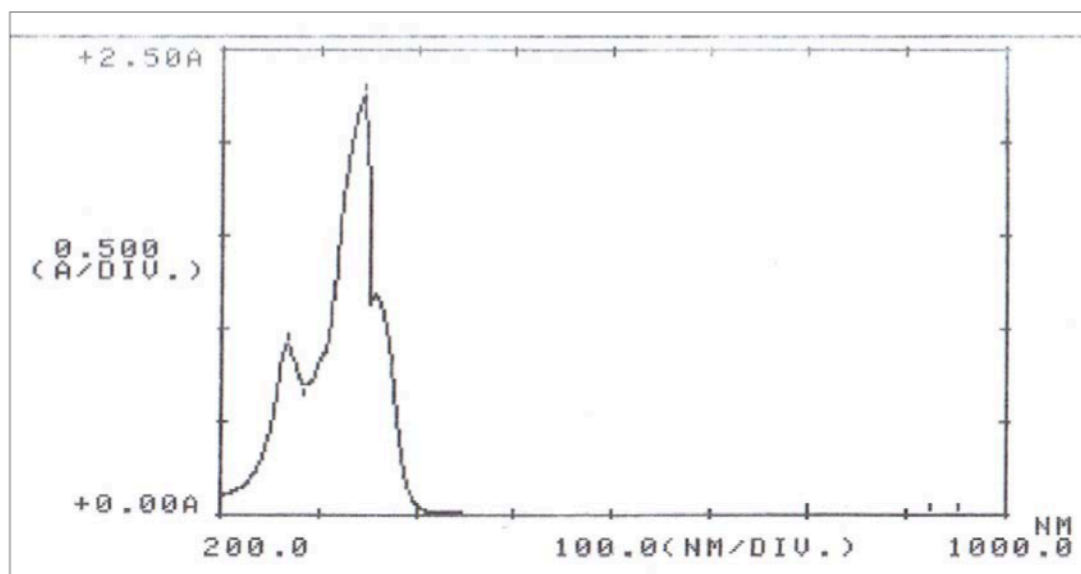


Figure (5): UV-vis spectrum of ligand (Schiff base 2-mbd)

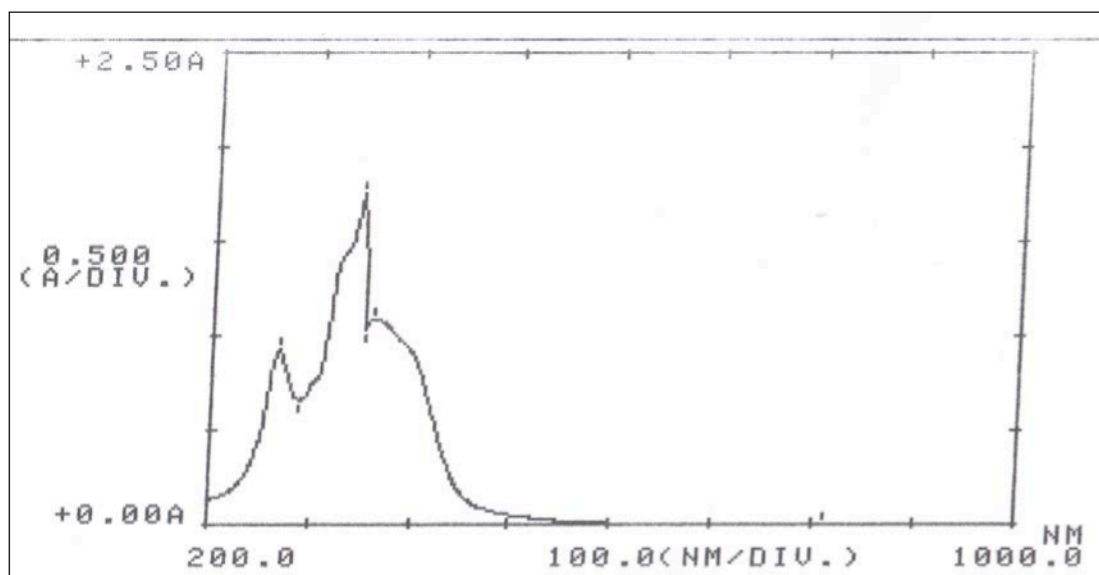


Figure (6): UV-vis spectrum of complex [Co((2-mbd)(3-pic)<sub>2</sub>)]

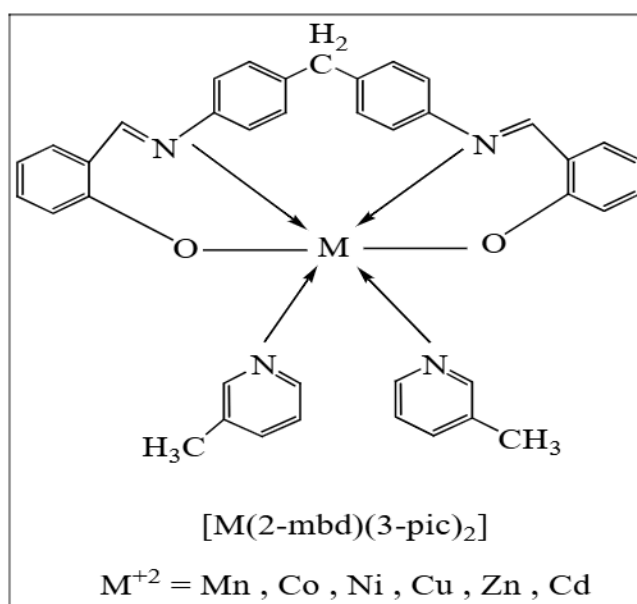
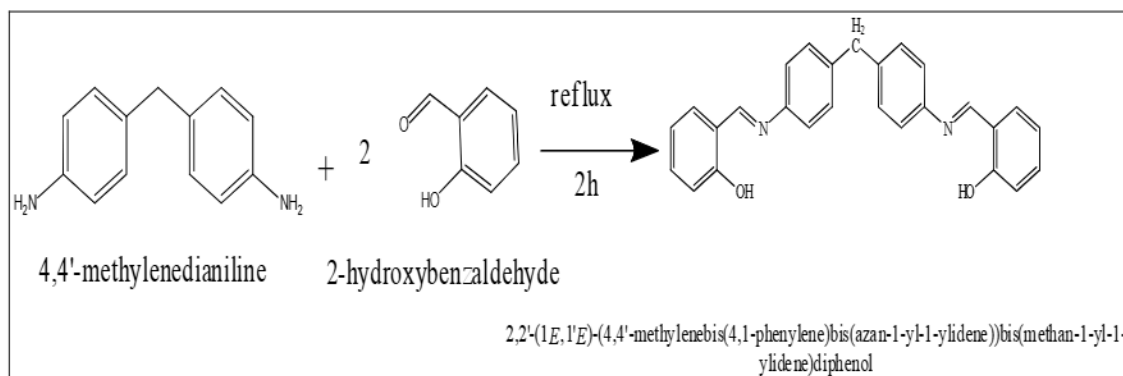


Figure (7): The proposed structural formula of the complexes



Scheme (1): Preparation of [Schiff base (2-mbd)]

## تحضير وتشخيص بعض المعقدات الفلزية مع ليكاندات مختلطة من قاعدة شف و

### (3- بيكولين)

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قسم الكيمياء / كلية التربية للعلوم الصرفة (ابن الهيثم) / جامعة بغداد

مظهر يونس محمد

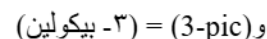
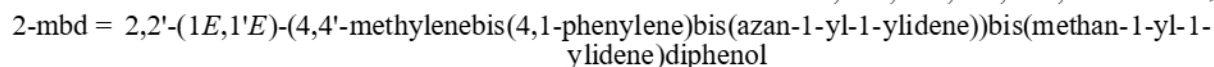
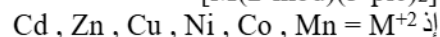
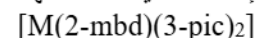
ثائر محمد سلمان

قسم الكيمياء / كلية التربية للبنات / جامعة تكريت

استلم في: 26/أيار/2015، قبل في: 30/حزيران/2015

### الخلاصة

يتضمن البحث تحضير وتشخيص بعض المعقدات الفلزية التي تحتوي على ليكاندات مختلطة حاوية على قاعدة شف المشتقة من (2- هايدروكسي سالسالديهايد و 4،4'- مثيلين ثنائي انيلين كليكاند اولي و 3- بيكولين كليكاند ثانوي وقد شخّصت ودرست بالطرائق الطيفية وهي طيف الرنين النووي المغناطيسي ( $^1\text{H}$  and  $^{13}\text{C}$ -NMR) والاشعة فوق البنفسجية-المرئية والاشعة تحت الحمراء، وقياس نسبة الفلز بواسطة طيف الامتصاص الذري فضلا عن قياس التوصيلية المولارية والحساسية المغناطيسية ومن نتائج هذه الدراسات التشخيصية اقترح الشكل الثماني السطوح لهذه المعقدات مع إعطاء الصيغة العامة لهذه المعقدات وكما يأتي:



إذ اثبتت النتائج ان قاعدة شف تسلك كليكاند رباعي السن اذ يرتبط عن طريق ذرتي الاوكسجين ضمن مجموعة الفينول وذرتي النتروجين ضمن الحلقة بينما يسلك 3- بيكولين كليكاند احادي السن اذ يرتبط عن طريق ذرة النتروجين في حلقة البيريدين.

الكلمات المفتاحية: ليكاندات مختلطة ، قاعدة شف ، 3- بيكولين ، المعقدات الفلزية .