

# Synthesis and Characterization of Fe (II), Co (II), Ni (II), Cu (II), Cd (II) Mono-Complexes with Mannich Base 4-hydroxy-3-(4'-morpholinyl methyl) quinoline

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## Abstract

Mannich bases have received great attention in recent years because of their proven antimalarial, antifungal and anticancer activities, coordination certain metal ions to them have interested use in synthesizing and studying structural aspects of metal complexes with oxygen and nitrogen donor ligands.

The chelate ligand 4-hydroxy-3-(4'-morpholinyl methyl) quinoline was prepared by condensation of the formaldehyde with morpholine and 4-hydroxyquinoline in ethanol.

A methanolic solution of the prepared ligand was reacted with a methanolic solution containing metal ions Fe (II), Co (II), Ni (II), Cu (II), Cd (II) to give metal complexes.

The reaction of Fe (II), Co (II), Ni (II), Cd (II) ions with chelate ligand gave the mono-nuclear tetrahedral complexes, and mono-nuclear square planar complex with Cu (II).

Their molecular structures were studied by FTIR, <sup>1</sup>H-NMR and UV-Visible spectra, atomic absorption technique, elemental analysis have been performed using CHN, magnetic susceptibility and conductivity measurements.

## Introduction

The aminoalkylation of aromatic substrates by the mannich reaction is of considerable importance for the synthesis and modification of biologically active compounds, the reaction pathways of the mannich reaction depend on the nucleophilicity of substrate and the pH of reaction medium (1-4). The reaction of these ligands with some transition metal ions gave a great importance in coordination chemistry, because of their application in pharmaceutical, industrial and analytical aspects (5-8). In recent years mannich bases have attracted considerable attention due to formation of stable chelates with

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transition metal presents in cell, they are exhibit a wide variety of pharmacological properties such as antifungal, antimicrobial and anticancer (9-12). This work aims at synthesizing and characterizing new transition metal complexes with the ligand, acts as a bidentate through oxygen of hydroxyl group (phenol) and nitrogen atom of the aminomethyl group, derived from 4-hydroxyquinoline and investigate the coordination behavior of mannich base ligand toward Fe (II), Co(II), Ni (II), Cu (II) and Cd (II) ions.

## Experimental

### Chemicals:

All chemicals used in this work were reagent grade (BDH/Aldrich). The purity of the mannich base ligand and its complexes were checked by thin layer chromatography (TLC) was carried out using ferric chloride pre-coated sheets type poly gram silica gel and the plates were developed with iodine vapour.

### Instrumentation:

Melting points were recorded by using Gallenkamp M.F.B. 600.01 of melting point apparatus. Infrared spectra were recorded as CsI discs using FTIR 8300 Shimadzu. UV-Visible spectra were measured using Shimadzu UV-Vis 160A Ultra-violet spectrophotometer in the range (200-1100nm).

The metal percent in the prepared complexes were determined using Shimadzu 680 cc-flame. Elemental analysis C.H.N were performed using Carlo Erba 1106 elemental analyzer. Proton nuclear magnetic resonance analyses spectra were recorded using a Varian Unity plus (300MHz spectrometer) using  $CDCl_3$  as a solvent and tetramethylsilane as internal reference. Magnetic susceptibility for the prepared complexes were obtained at room temperature using magnetic susceptibility balance Johnson Matthey catalytic system division and molar conductivity measurements were carried out at room temperature at the concentration  $10^{-3}M$  using corning conductivity meter 220.

### Preparation of the Mannich Base Ligand:

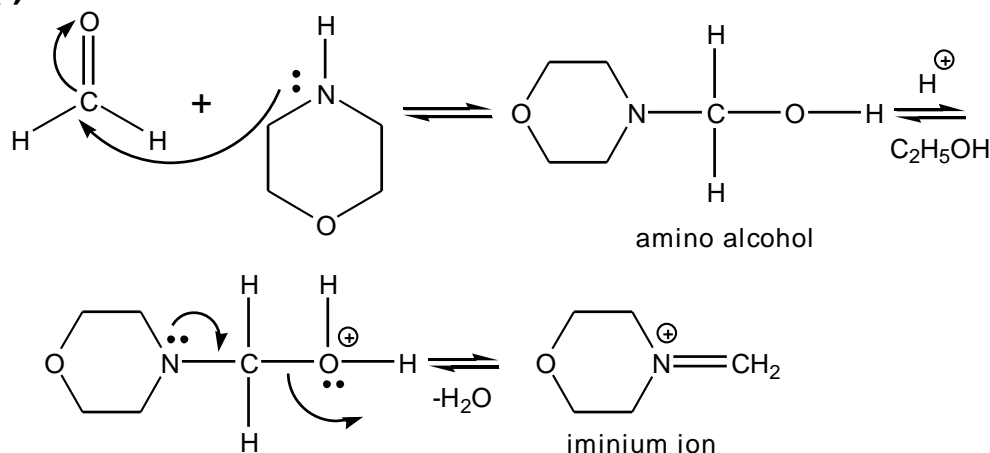
To a solution of 4-hydroxyquinoline (0.1 mole) and formaldehyde (0.1 mole) in an absolute ethanol (15 ml) was added the morpholine (0.1 mole) at room temperature.

Then, the resulting mixture was heated at reflux for 20 hours. After cooling, yellow precipitate was collected by filtration, washed several times with ethanol and dried under vacuum. The yellow fine powder product was identified by FTIR spectroscopy and TLC technique.

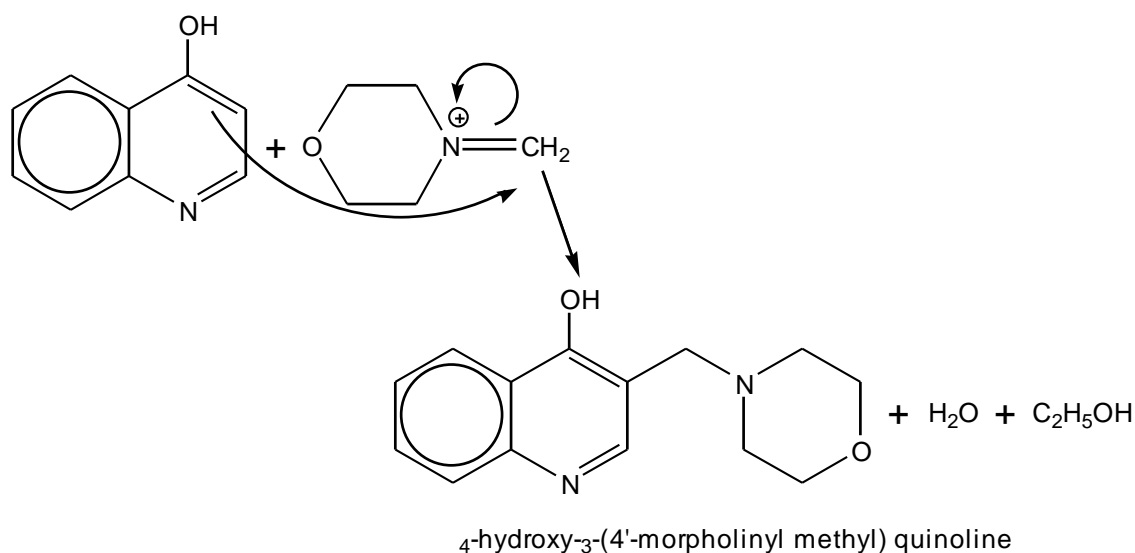
The suggested mechanism of mannich base ligand is as follows:-

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(I)



(II)



**Preparation of the Complexes:**

Methanolic solutions of metal salts ( $\text{FeCl}_2 \cdot 6\text{H}_2\text{O}$ ,  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ ,  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ ,  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ,  $\text{CdCl}_2 \cdot 2\text{H}_2\text{O}$ ) was added to a methanolic solution of the ligand 1:2 (metal:ligand) mole ratio and refluxed for 3 hours .After cooling the resulting precipitate was filtered , washed several times with methanol until the washing becomes colorless and dried at  $60^\circ\text{C}$ .

**Results and Discussion**

The physical properties and analytical data of the ligand Q with their metal complexes (M1-M5) are given in Table 1. The stoichiometry of the ligand and its complexes were confirmed by their C.H.N. analysis. The elemental analysis of the ligand and its complexes show good support with the proposed structures. The lower values of molar conductivity in DMF as a solvent at concentration  $10^{-3}$  M at room temperature  $25^\circ\text{C}$ , indicates the non-electrolyte behavior of these complexes, these values suggest that no anions present outside

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the coordination spheres. The molecular formula, symbols and name of complexes are given in Table 2.

**Table 1:** Physical characteristics and analytical data for ligand (Q) and its metal complexes.

symbol	Color	Melting Point, °C	Yield%	C.H.N Analysis, Found (Calc. %)				Molar Conductivity $\mu\text{s. cm}^{-1}$
				,M: Metal analysis				
				M	C	H	N	
Q	yellow	78-80	95	-	68.80 (68.85)	6.50 (6.55)	11.40 (11.47)	-
M1	Dark green	240-242	70	9.90 (9.97)	60.12 (60.02)	5.70 (5.71)	10.10 (10.00)	5.66
M2	green	260-262	80	10.40 (10.47)	59.60 (59.68)	5.60 (5.68)	9.89 (9.95)	5.00
M3	Yellowish-green	255-257	60	9.70 (9.71)	56.89 (56.18)	6.00 (6.02)	9.00 (9.36)	4.30
M4	green	225-227	90	10.36 (10.85)	56.98 (57.39)	5.90 (5.81)	9.54 (9.56)	6.00
M5	Light yellow	>300	60	17.00 (17.72)	51.99 (52.96)	5.03 (5.36)	8.63 (8.83)	4.10

**Table 2:** Symbols, molecular formula and names of the prepared ligand and complexes.

Symbol	Suggested Molecular formula	Name
Q	$\text{C}_{14}\text{H}_{16}\text{N}_2\text{O}_2$	4-hydroxy-3- (4'-morpholinyl methyl ) quinoline
M1	$[\text{Fe}(\text{Q})_2].\text{H}_2\text{O}$	Bis-(4-hydroxy-3-(4'-morpholinyl methyl) quinoline) iron (II) hydrate
M2	$[\text{Co}(\text{Q})_2].\text{H}_2\text{O}$	Bis-(4-hydroxy-3-(4'-morpholinyl methyl) quinoline) cobalt (II) hydrate
M3	$[\text{Ni}(\text{Q})_2].3\text{H}_2\text{O}$	Bis-(4-hydroxy-3-(4'-morpholinyl methyl) quinoline) nickel (II) trihydrate
M4	$[\text{Cu}(\text{Q})_2].2\text{H}_2\text{O}$	Bis-(4-hydroxy-3-(4'-morpholinyl methyl) quinoline) copper (II) dihydrate
M5	$[\text{Cd}(\text{Q})_2].2\text{H}_2\text{O}$	Bis-(4-hydroxy-3-(4'-morpholinyl methyl) quinoline) cadimium (II) dihydrate

## Infrared Spectra

### Infrared Spectra of Free Ligand(Q):

The characteristic vibrations and assignments of ligand (Q) and their complexes as (M1-M5) are described in Table 3, Ligand spectrum showed that some changes appeared on comparision with the initial materials spectra ((13a,b), 14, 8):-

1. Appearance of a band at 2922, 2854 and 1461, 713 $\text{cm}^{-1}$  due to  $\nu(\text{CH}_2\text{-N})$  and  $\delta(\text{CH}_2\text{-N})$  aliphatic and nonstrained cyclic hydrocarbons, respectively of aminomethyl moiety.

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2. Absence of the single absorption band  $\nu(\text{N-H})$  of the secondary amine (morpholine) at  $3320\text{cm}^{-1}$ .
3. Stretching vibration of hydroxyl group (phenolic) appeared at  $3458\text{cm}^{-1}$  in the spectrum of 4-hydroxyquinoline, was shifted to lower frequency by  $4\text{cm}^{-1}$  in the spectrum of ligand due to the resonance with aromatic systems and strong band was found at  $1252\text{cm}^{-1}$  belong to the  $\nu(\text{C-O})$  (phenolic).
4. The FTIR spectrum of formaldehyde, showed absorption bands at  $1720$  and  $2830\text{-}2695\text{ cm}^{-1}$  region assigned to  $\nu(\text{C=O})$  and  $(\nu(\text{C-H}))$  two moderately intense bands), respectively disappeared in the spectrum of ligand.
5. The carbon-carbon stretching vibration within the ring absorbed at  $1581$  and  $1518\text{cm}^{-1}$  regions, imine group  $\text{C=N}$  stretch showed at  $1623\text{ cm}^{-1}$ , weak band of aromatic  $\text{C-H}$  stretching vibration at  $3063\text{cm}^{-1}$  was observed.
6. Two bands appeared at  $1157$  and  $1116\text{cm}^{-1}$  which could be due to ring  $\text{C-H}$  in-plane bending and the band  $788\text{cm}^{-1}$  which could be due to ring  $\text{C-H}$  out-plane bending frequency, the position of these three bands was confirmed by comparing the spectra of formaldehyde and 4-hydroxyquinoline (ortho substitution of quinoline ring). \* $\nu$ :stretching vibrations, $\delta$ :bending vibrations.

**Infrared Spectra of Complexes (M1-M5):**

The disappearing of band for  $\nu(\text{OH})$  vibration in the spectra of complexes is indicating the coordination of phenolic oxygen with metal ions, the spectra bands of complexes at  $1623\text{cm}^{-1}$  were characterized for the imine group which didn't suffer a shift. Thus, it is suggested that the  $\text{C=N}$  group is not coordinated to the metal ion. The bands due to  $\nu(\text{C-O})$  phenolic group and  $\nu(\text{CH}_2\text{-N})$  aminomethyl group were shifted to a lower frequencies by  $(26\text{-}134)\text{cm}^{-1}$  and  $(4\text{-}54)\text{cm}^{-1}$ , respectively, therefore the ligand (Q) behave as a bidentate coordinating with metal ion through oxygen of hydroxyl group and aminomethyl group. The appearance of new weak bands in the range of  $(410\text{-}464)\text{cm}^{-1}$  and  $(455\text{-}550)$  which was attributed to the  $\nu(\text{M-O})$  and  $\nu(\text{M-N})$ , respectively (15,16).

**Table 3:** The characteristic bands of infrared spectra of (Q)and (M1-M5)complexes.

Compound	$\nu(\text{O-H})$ phenol	$\nu(\text{C-H})$ aromatic	$\nu(\text{C=C})$	$\nu(\text{C=N})$ imine	$\nu(\text{CH}_2\text{-N})$ aminomethyl	$\nu(\text{C-O})$ phenol	$\nu(\text{M-O})$	$\nu(\text{M-N})$
Q	3454	3063	1581, 1518	1623	2854	1252	-	-
M1	-	3063	1581,1502	1623	2825	1126	418	455
M2	-	3063	1533,1517	1620	2850	1226	462	525
M3	-	3063	1573,1502	1623	2840	1118	435	500
M4	-	3063	1550,1500	1623	2800	1120	464	550
M5	-	3063	1571,1502	1623	2840	1126	410	500

## **Electronic Spectra**

### **Electronic Spectra of Free Ligand (Q):**

The electronic spectra of the prepared ligand (Q) in DMF solvent and their complexes were recodes in Table 4, the spectrum of (Q) showed a strong band at (330nm) may be attributed to  $\pi \rightarrow \pi^*$  electronic transition and the other band appearing at (385 nm) could be assigned to  $n \rightarrow \pi^*$  electronic transition (17, 18).

### **Electronic Spectra of Complexes (M1-M5):**

The electronic spectra of the prepared complexes showed different absorption from that of the free ligand, some of these bands was shifted to higher wavelength than the corresponding bands in the ligand.

#### **7. M1-complex**

The dark green complex of Fe (II) showed band at (335nm) which belong to  $\pi \rightarrow \pi^*$  and another band at (455nm) which caused by the electronic transition  ${}^5E_g \rightarrow {}^5T_{2g}$ .

#### **M2-complex**

The electronic spectrum of Co(II) showed band at (351nm) which belong to  $\pi \rightarrow \pi^*$  transition and two bands at (630and 715 nm) which assigned to  ${}^4A_2(F) \rightarrow {}^4T_1(F)$  and  ${}^4A_2(F) \rightarrow {}^4T_1(p)$  transitions, respectively in tetrahedral geometry.

#### **M3-complex**

Ni (II) complex assigned to the transition  ${}^3T_1(F) \rightarrow {}^3T_1(p)$  at (428nm) and another band at (337nm) may be due to  $\pi \rightarrow \pi^*$ transition.

#### **M4-complex**

Spectrum of green Cu (II) complex showed abroad band at (620 nm) which may be assigned to  ${}^2E_g \rightarrow {}^2T_{2g}$  transition. The band at (390nm) may due to  $n \rightarrow \pi^*$  transition, Cu (II) ion in the present complex showed square planar geometry.

#### **M5-complex**

Cd (II) complex was diamagnetic as expected for  $d^{10}$  ions, so that no (d-d) transition can be expected in the visible region.

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**Table 4:** U. V-Visible spectra of (Q) and (M1-M5)complexes.

Compound	Band, nm	Transition	Suggested Structure
Q	330 385	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$	-
M1	335 455	$\pi \rightarrow \pi^*$ ${}^5E_g \rightarrow {}^5T_{2g}$	Tetrahedral
M2	351 630 715	$\pi \rightarrow \pi^*$ ${}^4A_2(F) \rightarrow {}^4T_1(F)$ ${}^4A_2(F) \rightarrow {}^4T_1(p)$	Tetrahedral
M3	337 428	$\pi \rightarrow \pi^*$ ${}^3T_1(F) \rightarrow {}^3T_1(p)$	Tetrahedral
M4	390 620	$n \rightarrow \pi^*$ ${}^2E_g \rightarrow {}^2T_{2g}$	Square planar
M5	300	$\pi \rightarrow \pi^*$	Tetrahedral

### Magnetic Properties

The experimental magnetic moment for each metal complexes is listed in Table5. The magnetic properties are due to the presence of unpaired electrons in the partially filled d-orbital in the outer shell of transition metals. These magnetic properties give an idea about the electronic state of the metal ion in the complex. The magnetic moment of  $Fe^{+2}$  and  $Co^{+2}$  were found to be 5.3B.M. and 4.5B.M. respectively, which gave a good agreement for tetrahedral structures (19, 20). The  $\mu_{eff}$  value for  $Ni^{+2}$  complex 2.5 B.M., this value refers to regular tetrahedral structure. The  $\mu_{eff}$  value of  $Cu^{+2}$  complex was found to be 1.9B.M. lead to square planar structure. Finally,  $Cd^{+2}$  complex was diamagnetic as expected for  $d^{10}$  ions (17).


**Table 5:** The magnetic properties of (M1-M5) complexes at 25°C.

Complexes	No. of electrons	No. of unpaired electrons	Term Symbol	Ground state term symbol	$\mu_{eff}$ , B.M.
M1	$d^6$	4	${}^5D$	${}^5E_g$	5.3
M2	$d^7$	3	${}^4F$	${}^4A_2$	4.5
M3	$d^8$	2	${}^3F$	${}^3T_1$	2.5
M4	$d^9$	1	${}^2D$	${}^2E_g$	1.9
M5	$d^{10}$	-	${}^1S$	-	0.0

### Proton Nuclear Magnetic Resonance:

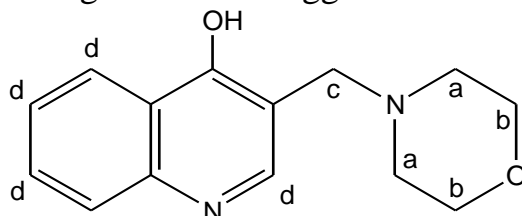
The  ${}^1H$ -NMR data of the compounds are presented in Table 6.  ${}^1H$ -NMR spectrum for free ligand (Q) it can be conclude that the aminomethyl group is

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present as  and absence of N-H hydrogen resonance of the secondary amine (morpholine) at  $\delta$ 1.9 ppm .  $\delta$ 2.51 (t, 4H) for H-a,  $\delta$ 3.50 (S, 2H) for H-c (aminomethyl) protons,  $\delta$ 3.64 (t, 4H) for H-b,  $\delta$ 7.34 (m, 1H) , $\delta$ 7.61 (m, 2H) and  $\delta$ 8.38 (m, 1H) for H-d and  $\delta$ 8.03 (S, 1H) which assigned to OH group.(13 a,b)

The chemical shift observed for the OH proton in the Q ( $\delta$ 8.03 ppm) was not observed in any of the (M1-M5) complexes.This confirms the bonding of oxygen to the metal ions. The multiplets of aomatic quinoline ring appeared within the range  $\delta$  (7.34-8.38 ppm) and they were not affected by chelation, the protons of the -CH<sub>2</sub> (aminomethyl) group shifted to higher field (21), the <sup>1</sup>H-NMR spectral data gave additional support for the composition of the complexes.

According to the results obtained from the chemical shifts spectrum, the molecular structure of the ligand can be suggested.



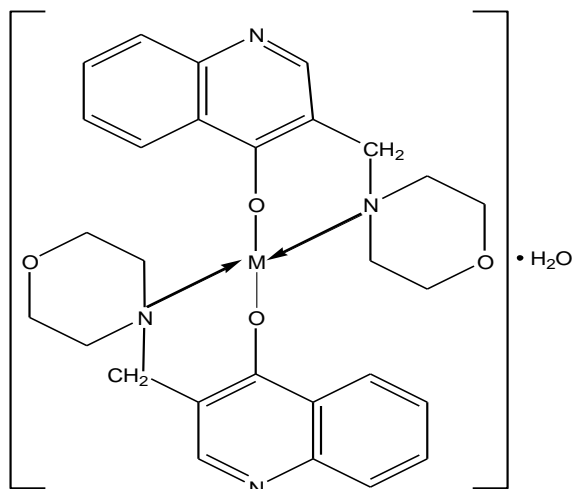
**Table 6:** <sup>1</sup>H-NMR spectral data ( $\delta$ , ppm) of the (Q)and (M1-M5) complexes.

Compound	OH	CH <sub>2</sub> Aminomethyl	CH <sub>2</sub> Morpholine	Aromatic
Q	8.03	3.50	2.51, 3.64	7.34- 8.38
M1	-	3.42	2.51, 3.66	7.34-8.38
M2	-	3.44	2.50, 3.64	7.34-8.36
M3	-	3.30	2.51, 3.65	7.30-8.35
M4	-	3.32	2.52, 3.64	7.31-8.35
M5	-	3.31	2.51, 3.66	7.30-8.35

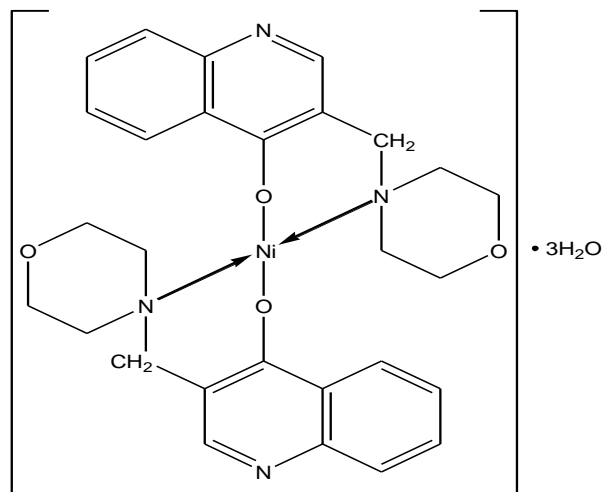
According to the results obtained from vibrational and electronic spectra, elemental analysis, proton-nuclear magnetic resonance analysis, magnetic moment and conductivity measurements the stereo chemistry around the metal ion has been suggested.



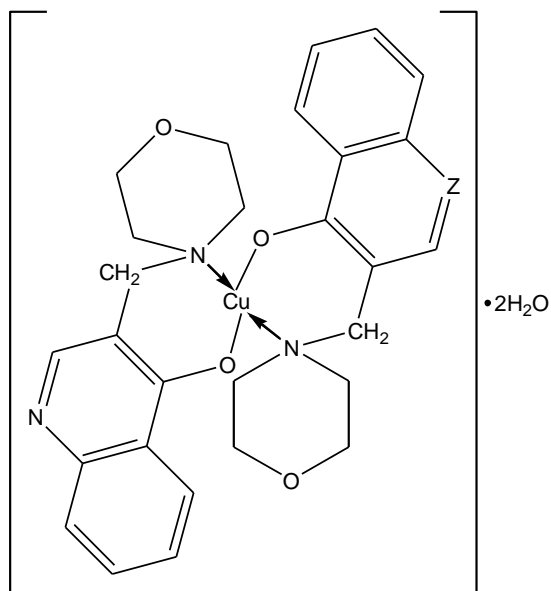
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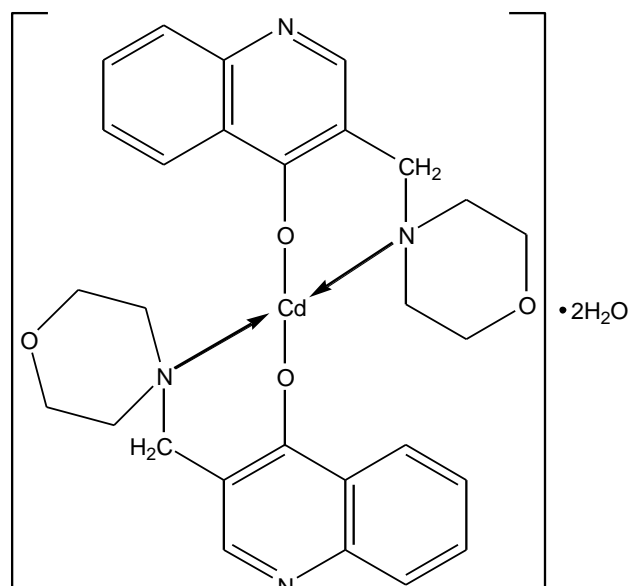
M1 and M2-complexes  
where, M = Fe<sup>2+</sup>, Co<sup>2+</sup>



M3-complex



M4-complex



M5-complex

**Conclusion:**

In this work bidentate mannich base ligand with new transition metal complexes were synthesized and characterized. The molar conductivity of the prepared complexes in DMF solution were non-electrolyte and the configurations were performed to coordinate the mannich base ligand through nitrogen and oxygen atoms. Therefore, from the presented results the mono-nuclear complexes have tetrahedral and square planar configuration.

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تحضير وتشخيص معقدات

Fe (II), Co (II), Ni (II), Cu (II), Cd (II)

أحادية النواة مع قاعدة مانخ

4-hydroxy-3-(4'-morpholinyl methyl) quinoline

فرح مؤيد إبراهيم

قسم الكيمياء، كلية العلوم، جامعة النهرين

### الخلاصة

إن لدراسة قواعد مانخ ومعقداتها أهمية جلبت انتباه الباحثين في الآونة الأخيرة بسبب امتلاكها فعالية حياتية واسعة النطاق ضد الملاريا والفطريات والخلايا السرطانية. جرى تحضير قاعدة مانخ ثنائية السن من تكاثف الفور مالديهايد والمورفولين مع 4-هيدروكسي كوينولين في الإيثانول. واستعمل الليكاند الكلابي في تحضير عدد من المعقدات الجديدة مع بعض العناصر الانتقالية في وسط كحولي. أعطى تفاعل الليكاند الكلابي مع أيونات Fe (II), Co (II), Ni (II), Cd (II) معقدات ذات تركيب رباعي السطوح (أحادي النواة) ومع أيون Cu (II) أعطى معقد ذو تركيب المربع المستوي (أحادي النواة) تم دراسة تراكيب جميع المركبات المحضرة باستخدام أطياف الأشعة تحت الحمراء والمرئية - فوق البنفسجية وطيف الرنين النووي المغناطيسي وتقنية الامتصاص الذري لتقدير النسبة المئوية للفلز وتحليل العناصر CHN والقياسات المغناطيسية بالإضافة إلى قياسات التوصيلية الكهربائية.