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Synthesis and Characterization of some New Complexes of Fe(II), Co(II) ,Ni(II) and Cu(II) Ions with Mixed Ligands Derived from Mannich Bases and Diamine Ligands

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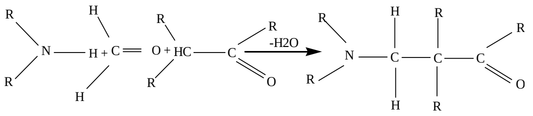
Abstract - **This paper describes the synthesis of a new series of complexes of Fe(II), Co(II), Ni(II) and Cu (II) ions with mixed Ligand involved Mannich Ligand (L) and diamine ligand (L'). L Ligands involved L_1,L_2,L_3 namely L_1=N-[1-Morpholino(3-methoxy,4-hydroxy) Benzyl] Acetamide (MBA), L_2=N-[1-Morpholino(3-methoxy, 4-hydroxy) Benzyl] benzamide (MBB), L_3= N-[1-Morpholino(3-methoxy,4-hydroxy) Benzyl] urea (MBU). L' ligand is 1,10-phenanthroline.These complexes were prepared from the direct reaction of the metal(II) chloride ,mannich bases (MBA, MBB, MBU) and diamine ligand namely with mole ratio of (1:1:2) of L:M:L' Categories of complexes were synthesis is mononuclear complexes of formula [M(L)(L′)] Cl_2 where: M= Fe(II), Co(II), Ni(II), Cu(II), L= mannich base MBA, MBB, MBU, L'=1-10 phenanthroline. These complexes were characterized on bases of their physical properties and spectroscopic data such as melting points, molar conductivity, elemental analysis (C.H.N) and atomic absorption techniques as well as spectral studies such as UV-Vis, IR, magnetic susceptibility also have been measured The completion of the reaction checked by using TLC. From all data all complexes exhibit octahedral geometry.**

Keywords- **Mannich base, transition metal (II) complexes, Chelates.**

I. INTRODUCTION

The coordination chemistry of amide group is one of the most active researchfields in organic chemistry due to its diverse coordinating behaviour and their role it plays in biological process [1]. Carl Mannich was the first to recognize the enormous significance of this reaction types and this reaction how arrives his name has developed into one of the most important C-C bond forming reactions in organic Chemistry [2]. Mannich reaction is an organic reaction and it is method for the preparation of β-amino ketones and aldehydes and is an example of nucleophilic addition of an amine to a carbonyl group followed by dehydration to the Schiff base [3]. The Schiff base is an electrophile which reacts in the second step with a compound containing an acidic proton as included in equation below. Mannich reaction is considered a condensation reaction [4] and is a threecompount condensation in which a compound containing an active H atom is allowed to react with aldehyde or ketone and a secondary amine [5]-[6]. The Mannich reaction is also used in the synthesis of medicinal compounds e.g (Mannich base of tetracycline, fluoxetine, tramadol and tolmetin) [7] .A number of studies have been done on the Mannich base complexes[8]-[20].There is no report on metal complexes with Mannich base L and L' were prepared in our knowledge. Hence, in this paper we describes the synthesis and characterization of transition metal (II) ions with Mannich base and 1,10 phenanthroline, which have improved by using physico chemical techniques. The ligand (Mannich base) coordinates with the metal ion in bidentate manner through the oxygen atom of carbonyl group and nitrogen atom of morpholine ring.



II. EXPEREMENTAL WORK

A- Materials and Methods

The transition metal ions were used as supplied by Fluka. All reagents used analytical grade. Molar conductance were carried out for 10^{-3}M solution in dimethyl sulfoxide (DMSO) using an electronic conductivity measuring device model LF_42 at 25C°. IR spectra were recorded by using KBr pellets. The electronic spectra of complex were recorded on Shimadzu uv.1800 spectrophotometer at 25 C° using 1 cm quartz cell. Magnetic susceptibility measurements were performed on MSB_MKI (magnetic susceptibility balance) Sherwood scientific using Gouy method Metal analysis was carried out by using an AA 240FSVarian instrument for atomic absorption Carbon, hydrogen and nitrogen contents were determined at the Ibn Haithem Centre for research, Baghdad, Iraq.

B- Preparation of N-(1-Morpholino (3-methoxy, 4-hydroxy) benzyl]acetamide (MBA)

For the preparation of MBA ligand employing the Mannich synthetic route [21,22]. Acetamide(0.1mol,2.9g) and vaniline (3-methoxy, 4-hydroxy benzaldehyde)(0.1mol,7.69g), morpholine (0.1mol,4.3 ml) were taken in (1:1:1) mole ratio ice cold condition Acetamide was dissolved in minimum quantity of ethanol and mixed with morpholine.and stirred in ice-bath at 5C° and stirred to get a clear solution. Vaniline was dissolved in ethanol and then added in dropwise with strring in ice-bath for an hour. The resulting reaction mixture was kept at room temperature for Ca.5d. The solid product form was filtered washed with water and ethanol removes the unreacted acetamide and morpholine. The product, MBA was then dried andrecrystallized in an ethanol purity of the compound was checked by TLC. The preparation of the ligand may be represented as fig.1.

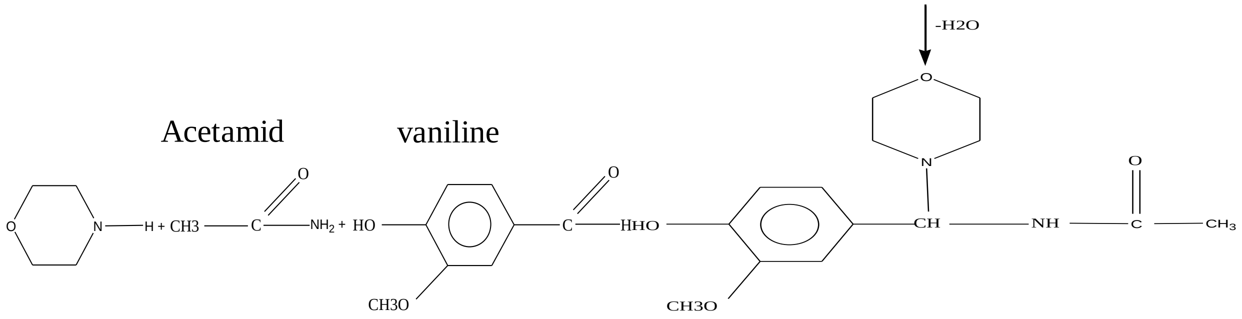


Fig.1: Preparation of ligand (MBA)

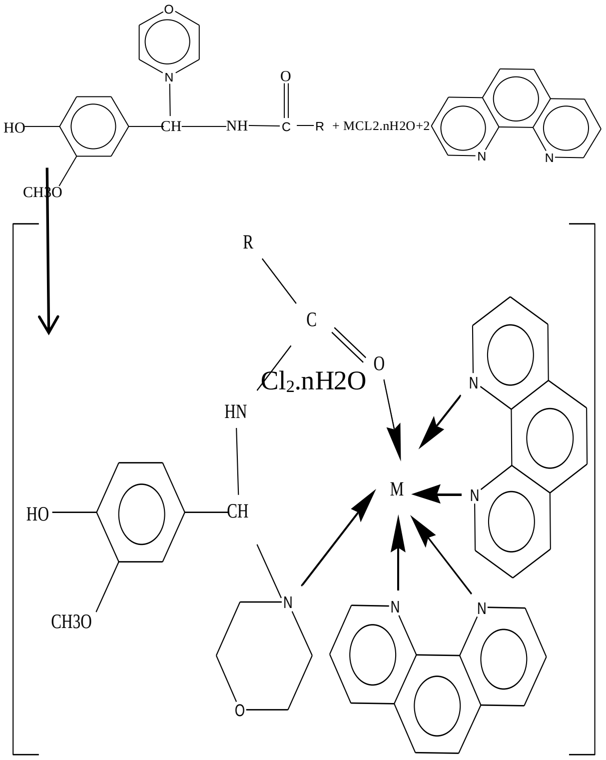
C-preparation of N- [1-Morpholino (3-methoxy,4-hydroxy) benzyl]benzamide (MBB)

This Ligand (MBB) was prepared by the same procedure in (B) by using (0.1mol, 6.1mol) benzamide to obtain orange crystals.

D-preparation of N-[1-Morpholine- (3-methoxy , 4-hydroxy) benzyl] urea (MBU)

This ligand was prepared by the same procedure in (B) by using (0.1mol,3g) of urea. E- Preparation of Metal complexes. All the complexes were prepared in non aques medium. The Ligands (MBA, MBB,MBU) beinginsoluble in water. In typical procedure the ligand was dissolved in chloroform and

1,10 phenanthroline in ethanol are mixed with an ethanolic solution of the metal salt.MCl_2nH_2O[where:M=Fe(II),Co(II), Ni(II), Cu(II) in 1:1:2 mole ratio. The reaction mixture was gently warmed on water bath foran hour, in the resulting solid complex formed was filtered washed with distilled water and dried. The preparation of the metal complexes may be represented as fig.2.



M= Fe(II),Co(II),Ni(II),Cu(II).
R=CH_3,C_6H_5,NH_2

Fig.2: Preparation of complexes

III. RESULTS AND DISCUSSION

The physical properties, elemental analysis and molár conductivity of ligands and their complexes are given in table I. The complexes were air stable at room temperature and coloured solid .The physical properties, conductivity of complexes are listed in table(I). The molar conductivity was measured using DMSO at 10^{-3}M .The results obtained, (table I) indicates complexes as1:2electrolyte in nature [22,23, 24]. The ligands and their complexes were analysed for carbon, hydrogen and nitrogen. The percentage of the metal ions and results of the C.H.N were in a good agreement with the calculated values and with the proposed formula (Table I).

A-Infrared Spectral Studies

In order to study the binding made of the mannich base to the metal for in the complexes, the IR spectrum of the free ligand was compared with the spectra of the complexes by using KBr disc and listed in table (II). The ligand MBA, MBB, MBU shows bands between(3330-3348)cm^{-1}, (1637-1658)cm and (1118-1120) cm^{-1} which have been assigned to *v* (NH) amide *v*(C=0) and *v*(C-N-C) of morpholine group [25]. In the infrared spectra of all complexes, the *v*(NH) band remained at the same position as in the free Ligand indicating that the secondary nitrogen is not involved in the coordination to the metal ion [21] . The reduction in the *v*C=O stretching of the ligand MBA from 1640cm^{-1} to 1622-1629 cm^{-1} in the complexes and in Ligand MBB from 1637 cm^{-1} to 1622-1631 cm^{-1}in the complexes and in ligand (MBU) from 1658 cm^{-1}to 1629-1636 cm^{-1}in the complexes denotes the oxygen coordination of the ligand[13]. The *v*(C-N-C) of morpholine bands displayed substantial negative shifts with fairly low intensity indicating coordination through nitrogen of morpholine present in ligand [26]. The metal complexes showed band between 1035-1043 cm^{-1}shows that the bands are shifted to lower frequency characteristics for C-N-C of morpholine completed with metal salt. The presence of phenolic OH group in all complexes in between 3400-3461cm^{-1}indicates that which was not involved in coordination [27]. The rocking vibration of water is not observed in the IR spectra of complexes. This is confirmed that the water molecule is not coordinated [28]. New bands appeared for the prepared complexes in the region (422-598) and (414-462) cm^{-1}and were assigned to *v*M-O and *v*M-N streching vibration [28]-[29]-[30]. The band in the region (1290-1293) cm^{-1} are observed in the IR spectra of Ligands and complexes belonged to the methoxy group [31]. IR data concludes that Mannich base ligand (L_1,L_2,L_3) (Table II) in all complexes studied behaves as a chelating bidentate one and the binding sites are one carbonyl oxygen atom and the morpholine ring nitrogen atom [13]-[26]. The streching vibration of C=N in diamine Ligand (phen) observed in the region 1557 cm^{-1}but it shifted to low frequency (77–151) cm^{-1}in all complexes indicate the coordination to metal io n[32,33].

B- Electronic Spectra and Magnetic Moment

The electronic spectra and magnetic moment of metal complexes are listed in Table(III). The electronic absorption spectra of the Ligands and its iron(II) , cobalt (II), nickel (II) and copper (II) were recorded at room tempture using 10^{-3}M solution of the complex prepared using DMSO as solvent. The electronic spectra of Fe(II) complex Table (III) show bands at 22123 cm^{-1} due to $^{5}T_{2}g$ ^{5}Eg and at 2890l cm^{-1} due to the charge transfer [34] that indicates as reported for octahedral [35]-[37]. The magnetic moment of Fe(II) complex (1) (4.52 B.M) is in agreement with hexa coordinated iron (II) complexes with octahedral geometry high spin [33]. The complex (5) show bands at 10161 cm^{-1} due to$^{5}T_{2}g$ ^{5}Eg and at 27322,35714 cm^{-1} due to charge transfer. The position of these bands is in agreement with hexa coordinated Iron (II) complexes with octahedral geometry [38]-[39]-[40] The complex (9) show bands at 16666cm^{-1} and (26455,30864) cm^{-1} due to $^{5}T_{2}g$$^{5}Eg$ and charge transfer [41, 42,38]. The magnetic moment for Fe(II) complexes (5,9) Table (III) indicates as reported for octahedral high spin[44]-[45]. In the present work, the electronic spectra of cobalt (II) complexes (2,6,10) show absorption bands at the range (10000-10111) cm^{-1} ,(14720-15230) cm^{-1} ,(20161-2083)3 cm^{-1} and (24449-32894) cm^{-1} which were assigned to $^{4}T_{1}g$ (F) $^{4}T_{2}g$ (F) (v_1), $^{4}T_{1}g$ (F) $^{4}A_{2}g$ (F) (v_2), $^{4}T_{1}g$ (F) $^{4}T_{1}g$ (P) (v_3) transitions and the fourth band is due to charge transfer transition Table(III). The position of these bands is in agreement with the described values for cobalt (II) high spin octahedral complexes[39]-[45]-[46]. The magnetic moments values for Co(II) complexes (2,6,10) Show μeff value of (4.67-4.81)B.M which indicates the presence of three unpaired electrons supporting octahedral structure [22]. The electronic spectrum of Ni (II) complexes (3,7,11) (Table (III) gave three spin-allowed transitions:

$^{3}A_{2}g$ (F) $\longrightarrow$ $^{3}T_{2}g$ (F) (V_1),

$^{3}A_{2}g$ (F) $\longrightarrow$ $^{3}T_{1}g$ (F) (V_2),

$^{3}A_{2}g$ (F) $\longrightarrow$ $^{3}T_{1}g$ (P) (V_3)

These transitions were observed in the region (10111-11248)cm^{-1}, (12195-23041) cm^{-1} , (23896-24449) cm^{-1} respectively. The four band may be due to charge transfer (Table III). The position of these bands suggests octahedral environment around nickel (II) ion [46]-[48]-[49]. The magnetic moment values for Ni(II) complexes (3,7,11) show magnetic effective value of (2.81-3.0) B.M. which indicates the presence of two unpaired electron supporting octahedral structure. The Low µeff values of will Ni(II) complexes may be due to super exchange anti ferromagnetic interactions [50]-[51]. The Cu(II) complexes (4,8,12) exhibit electronic absorption bands at (9090-9900 cm^{-1})υ_1,(11013-11627)υ_2,(13120-13880 cm^{-1})υ_3. The value of effective magnetic moments of the Cu (II) complexes (4,8,12) (Table III) is (1.14, 1.06, 1.15) B.M. representing an octahedral geometry of the ligand around the central metal ion which much less than the spin only value indicating the interaction between unpaired spins in the solid state[46]-[52].

IV CONCLUSION

From the above discussion, the following conclusion can be drawn: The Ligand Mannich base and the ligaud L' 1,10phenanthroline acts as bi dentate Ligands forming hexa -coordinate complexes, with the most probable octahedral Structure.

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Table I

Physical characterization analytical, molar conductance data of Ligands and their complexes

Compound No.	Compound	color	Yield (%)	Molting point (C$^{\circ}$)	Molar conductivity Ohm^{-1}.mol^{-1}.cm^{2}	%C.H.N.(found)calc			
						%C	%H	% N	%M
L_1MBA	$C_{14}H_{20}N_2O_4$	orange	75	198	----	59.984 (60.217)	7.191 (6.792)	9.993 (8.792)	---
L_2 MBB	$C_{19}H_{22}N_2O_4$	orange	64	192	---	66.650 (66.062)	6.476 (6.134)	8.181 (8493)	---
L_3 MBU	$C_{13}H_{19}N_2O_4$	orange	69	167	---	54.363 (53.275)	6.807 (5.964)	14.937 (14.043)	---
1	$FeC_{38}H_{36}N_6O_4]Cl_2.4$ $[H_2O$	Brownish Red	54	210*	68	54.363 (53.275)	4.321 (4.011)	10.010 (9.221)	6.651 (7.632)
2	$[CoC_{38}H_{36}N_6O_4]Cl_2.H_2O$	orange	85	228	63	57.877 (58.872)	4.761 (4.872)	11.875 (9.235)	7.473 (8.264)
3	$[NiC_{38}H_{36}N_6O_4]Cl_2.4H_2O$	orange	66	212 *	67	54.192 (55.032)	4.308 (5.562)	9.978 (8.958)	6.968 (7.247)
4	$[CuC_{38}H_{36}N_6O_4]Cl_2.4H_2O$	Pale brown	85	228	65	53.825 (52.229)	5.234 (4.647)	9.919 (8.881)	7.500 (7.806)
5	$[FeC_{43}H_{38}N_6O_4]Cl_2.2H_2O$	Dark red	74	232	67	59.666 (58.762)	4.424 (4.012)	9.709 (8.924)	6.451 (6.172)
6	$[CoC_{43}H_{38}N_6O_4]Cl_2.$ H_2O	Pale orange	83	220	65	60.713 (60.724)	4.739 (3.930)	9.879 (9.171)	6.928 (6.029)
7	$NiC_{43}H_{38}N_6O_4]Cl_2.8$ $[H_2O$	orange	90	226*	66	52.887 (50.329)	5.573 (4.647)	8.6o7 (8.905)	6.060 (6.987)
8	$CuC_{43}H_{38}N_6O_4]Cl_2.$ $[2H_2O$	Pale brown	78	228	65	60.385 (59.563)	4.478 (4.021)	9.826 (8.592)	7.429 (7.986)
9	$FeC_{38}H_{36}N_6O_4]Cl_2.$ $[4H_2O$	Brownish dark red	75	212	69	52.870 (53.108)	4.196 (3.891)	11.664 (12.781)	6.644 (7.569)
10	$[CoC_{38}H_{36}N_6O_4]Cl_2.$ H_2O	orange	60	208	68	53.827 (52.781)	4.761 (5.201)	11.875 (10.862)	7.464 (7.972)
11	$NiC_{38}H_{36}N_6O_4]Cl_2.3$ $[H_2O$	orange	68	216	70	53.788 (54.683)	5.000 (5.812)	11.879 (11.072)	7.111 (7.624)
12	$[CuC_{38}H_{36}N_6O_4]Cl_2.4H_2O$	Green	71	245*	69	52.391 (52.470)	4.158 (3.797)	11.558 (9.844	7.491 (8.575)

Table II

Infrared dates (cm^{-1}) of the ligands and their complexes

No.	Compound	υ_{N-H}	υ_{O-H}	$\upsilon_{C=O}$	υ_{C-N-C}	υ_{O-CH3}	υ_{C-N}	υ_{M-O}	υ_{M-N}
	L_1(MBA)	3348	3461	1637	1120	1292	-------	------	------
1	$[Fe(MBA)(phen)_2]Cl_2.4H_2O$	3348	3461	1622	1043	1292	1433	538	456
2	$[Co(MBA)(phen)_2]Cl_2. H_2O$	3348	3461	1629	1041	1292	1423	536	457
3	$[Ni(MBA)(phen)_2]Cl_2.4H_2O$	3348	3461	1631	1039	1292	1425	486	447
4	$[Cu(MBA)(phen)_2]Cl_2.4H_2O$	3348	3461	1629	1041	1292	1425	486	426
	L_2 (MBB)	3348	3400	1640	1118	1290	------	------	------
5	$[Fe(MBB)(phen)_2]Cl_2.2 H_2O$	3348	3400	1629	1039	1290	1434	476	414
6	$[Co(MBB)(phen)_2]Cl_2. H_2O$	3348	3400	1627	1041	1290	1425	513	449
7	$[Ni(MBB)(phen)_2]Cl_2. 8H_2O$	3348	3400	1622	1041	1290	1425	476	414
8	$[Cu(MBB)(phen)_2]Cl_2. 2H_2O$	3348	3400	1627	1039	1290	1427	480	428
	L_3(MBU)	3330	3461	1658	1120	1292	------	-------	------
9	$[Fe (MBU)(phen)_2]Cl_2. 4H_2O$	3330	3461	1636	1035	1292	1425	492	422
10	$[Co (MBU)(phen)_2]Cl_2. H_2O$	3330	3461	1629	1041	1292	1436	499	462
11	$[Ni (MBU)(phen)_2]Cl_2. 3H_2O$	3330	3461	1629	1041	1292	1436	480	408
12	$[Cu (MBU)(phen)_2]Cl_2. 4H_2O$	3330	3461	1627	1043	1292	1423	507	426

Table III

Electronic spectra and magnetic moment

No.	Compound	$_{eff}$(B.M μ)	C.T. cm^{-1}	d→d transition cm^{-1}
1	[Fe (MBA)(phen)$_2$]Cl$_2$.4H$_2$O	4.52	28901	22123
2	[Co (MBA)(phen)$_2$]Cl$_2$. H$_2$O	4.67	24271,32894	10101,14720 , 20833
3	[Ni (MBA)(phen)$_2$]Cl$_2$.4H$_2$O	2.81	32467	10111-11248,12195 -23041, 23896 -24449
4	[Cu (MBA)(phen)$_2$]Cl$_2$.4H$_2$O	1.14	24038	9090,11627,24449
5	[Fe (MBB)(phen)$_2$]Cl$_2$.2H$_2$O	3.85	35714 ,27322	10161
6	[Co (MBB)(phen)$_2$]Cl$_2$.H$_2$O	4.77	24449	10111,14980,20449
7	[Ni (MBB)(phen)$_2$]Cl$_2$.8H$_2$O	2.88	27322 ,35714	10373-11261, 24096-20513
8	[Cu (MBB)(phen)$_2$]Cl$_2$.2H$_2$O	1.06	30450	9100,11013,13880
9	[Fe (MBU)(phen)$_2$]Cl$_2$.4H$_2$O	3.90	26455, 30864	16666
10	[Co (MBU)(phen)$_2$]Cl$_2$. H$_2$O	4.81	24630	10000,15230,20161
11	[Ni (MBU)(phen)$_2$]Cl$_2$.3H$_2$O	2.82	26455	10544,13185,24154
12	[Cu (MBU)(phen)$_2$]Cl$_2$.4H$_2$O	1.15	22573	9900,11240,13550