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Research Article

DETERMINATION OF STABILITY CONSTANT AND RELATED THERMODYNAMICS OF BIVALENT TRANSITION METAL COMPLEXES WITH DERIVATIVE OF SCHIFFS BASE AS LIGANDS

Prem Mohan Mishra[1]., Kapileshwar Mishra[2]., Sri Lalan Kumar Jha[3] and Sri Naresh Mohan Yadav[4]

[1]Department of Chemistry M L S M College, Darbhanga, Bihar-846004
[2]Department of Chemistry, M L S M Coleege, Darbhanga
[3,4]M L SM College, Darbhanga

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A B S T R A C T

Schiff's base ligands were synthesized by the condensation reaction of 5-Methoxy Furan with 7-Methoxy naphthalene-2-amine. Nitrate salts of divalent cobalt, nickel, copper and zinc were estimated by standard volumetric and gravimetric methods. Ligand was analysed for elements by literature methods. pH metric titrations were carried out in an inert atmosphere of nitrogen gas at constant ionic strength of 0.1M KNO_3 with the help of digital pH meter and stability constant of complexes of these metals with the ligands synthesized were computed by Irving- Rossoti technique[1] modified by Calvin Bjerrum[2] at temperatures 298K.

The stability constant values of metals for the given ligand at the given temperature were found to be in the order Cu(II) > Ni(II), Co(II) > Zn (II).

This result is in agreement with the natural order proposed by Irving-William.

INTRODUCTION

In continuation of our previous work here also, we have been interested in studying the stability of complexes of transition metals with biologically active ligands. Here we are presenting determination of stability constant of complex compounds using ligands synthesised from of 5-Methoxy Furan carbaldehyde with 7- Methoxy naphthalene-2- amine with bivalent transition metals i.e. Co(II), Ni(II), Cu(II) and Zn(II).

Schiff's base offers a versatile and flexible series of ligands capable to bind with variety of metal ions to give complexes with varying properties. These complexes are biologically active[3], have wide potential applications in many fields such as catalysis[4], electrochemistry[5] and medicines like anti tumour, anti viral[6], anti cancer[7] and other many anti bacterial agents[8].

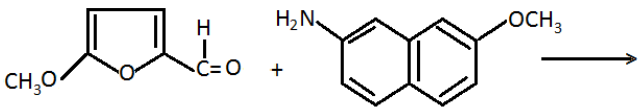
Metal ions play vital role in a vast number of widely different biological processes. The interaction of these ions with biologically active ligands is a subject of considerable interest. Some of the biologically active compounds act via Chelation[8]. A large number of such chelates have been synthesized, their structure and activity have been studied, but little is known about its stability in solution which will be its functioning condition.

Corresponding author:* **Prem Mohan Mishra
Department of Chemistry M L S M College, Darbhanga, Bihar-846004

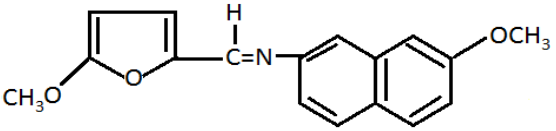
Experimental

Nitrate salts of divalent Co, Ni, Cu and Zn all were E. Merck. All other chemicals used were Anal R grade and used without further purification. Elemental analysis of metal salts were done by volumetric and gravimetric methods[11]. Double distilled and deionised water was used throughout the experiment. All titrations were done in aqueous-dioxane medium in the ratio 3:2 (v/v). Dioxane was purified by standard method.

Schiff's base ligands were synthesised by reported method[12] 2. g 2-mehoxy furan carbaldehyde in solution were mixed with 3.5 g 7 –methoxy naphthalene-2-amine. The mixture was refluxed in the presence of glacial acetic acid for 2-2.5 hours. The solution was concentrated and cooled to 0^0 C. The product obtained was filtered, washed several times and recrystallised from ethanol. The yield of product was nearly 2.40 g



5-Methoxy Furan - 2 - carbaldehyde 7-Methoxy naphthalene-2-amine



7' –Methoxynaphthyl-5-methoxy furan Carbaldimine (MNMFCI)

Solution of metal salts, ligands, electrolytes etc were prepared in doubly distilled CO_2 free water strength of various solutions used were tabulated in table-1.

pH metric titration of acid, acid + ligand and acid + ligand + metal ion solutions were done at constant ionic strength of 0.1 M KNO_3 at temperature 298k in an inert atmosphere of nitrogen.

The same process of titration were repeated for all the four Co, Ni, Cu and Zn metal ions. The change in colour and appearance of turbidity at particular pH value were recorded simultaneously. The change in pH of the solutions with each addition of alkali was recorded in Table no. 2.

RESULTS

A graph was plotted between pH meter reading [B] and volume of alkali added in each case, graph - 1. The three titration curves obtained for each metal ions are acid titration curve (a), ligand titration curve (b) and complex titration curve (c) respectively.

A graph was plotted between pH meter reading [B] and volume of alkali added in each case. The three titration curves so obtained for each metal ions are referred as:

1. Acid titration curve (a)
2. Ligand titration curve (b)
3. Complex titration curve (c) respectively

The values of volumes V_1, V_2 & V_3 corresponding to the same pH values were read from acid, ligand and complex titration curves given at temperature 298 K.

Calculation of $\bar{n}_A$, $\bar{n}$ AND P^L

The $\bar{n}_A$, $\bar{n}$ & P^L are calculated using standard expressions

$$\bar{n}_A = 1 + [(V_1-V_2) / (V^O + V_1)] (N^O + E^O) / T_L^O$$

$$\bar{n} = [(V_3-V_2) / (V^O + V_1)][(N^O + E^O) / T_M^O] \times 1/ \bar{n}_A$$

$$P^L = \log$$

$$\left[\sum_{j=0}^{j} \beta_j^0 H \ (1/\text{anti}\log B)(V^O + V_3)/(T_L^O - \bar{n} T_M^O)V^0\right]$$

Proton-Ligand Stability Constant

The ligand titration curve separates from acid titration curve at pH 5.16 at temperature 298 K. The ligand titration curves run parallel to the acid titration curves indicating the smooth dissociation of ligand.

The value of $\bar{n}_A$ at various pH reading [B] were calculated from the acid and ligand titration curves (table no.3,) at temperature 298 K.

The formation curves obtained from the plot of $\bar{n}_A$ vs [B] at temperature 298 K, show that values of $\bar{n}_A$ lies between 0.528 and 0.889. This indicates that ligand is monoprotic.

Dissociation of ligand may be given as,

$$LH \rightleftharpoons H^+ + L^-$$

The value of proton ligand stability constant was calculated by half-integral method and it was further corroborated by linear plot method[3-4] ($\log \bar{n}_A/(1-\bar{n}_A)$ vs [B] (graph 7.18,) at temperature 298 K.

Table 1 Concentrations of solutions of metal ions, ligand, acid and salt.

Metal ions	V^0 in mL	Y	N^0	E^0	T_L^0	T_M^0
Co (II)	100	1	1.0(M)	1.0×10^{-2}(M)	2.5×10^{-3} (M)	5.0×10^{-4}(M)
Ni (II)	100	1	1.0(M)	1.0×10^{-2}(M)	2.5×10^{-3} (M)	5.0×10^{-4}(M)
Cu (II)	100	1	1.0(M)	1.0×10^{-2}(M)	2.5×10^{-3} (M)	5.0×10^{-4}(M)
Zn (II)	100	1	1.0(M)	1.0×10^{-2}(M)	2.5×10^{-3} (M)	5.0×10^{-4}(M)

Table 2 Volume of alkali consumed in different titrations

Ligand MNMFCI Temperature: 298 ± 1K

μ^0 = 0.10 (M) KNO_3 Water: dioxane = 3:2 (v/v)

	pH-meter reading [B]					
Vol. of alkali added in mL	H^+	$H^+ + L$	$H^+ + L$ +Co(II)	$H^+ + L + Ni(II)$	$H^+ + L$ + Cu(II)	$H^+ + L$ +Zn(II)
0.0	4.04	4.38	4.36	4.38	4.38	4.40
0.1	4.18	4.52	4.50	4.48	4.46	4.50
0.2	4.36	4.60	4.60	4.62	4.60	4.64
0.3	4.46	4.80	4.82	4.80	4.78	4.82
0.4	4.66	5.02	4.98	4.98	5.02	5.00
0.5	5.02	5.34	5.29	5.32	5.30	5.34
0.6	5.40	5.68	5.68	5.66	5.68	5.70
0.7	6.10	6.30	6.28	6.32	6.31	6.28
0.8	6.96	6.98	7.02	7.00	6.98	7.00
0.9	9.56	9.68	8.20	8.28	8.84	8.86
1.0	10.46	10.62	9.52	9.46	9.18	9.30
1.1	10.92	11.04	9.66	9.84	9.86	9.92
1.2	12.14	12.71				

Graph No 1

Experimental curve with ligand MNMFCI

Temp. 298 ± 1 K

μ^0 = 0.10 (M) KNO_3 Water: dioxane= 3:2(v/v)

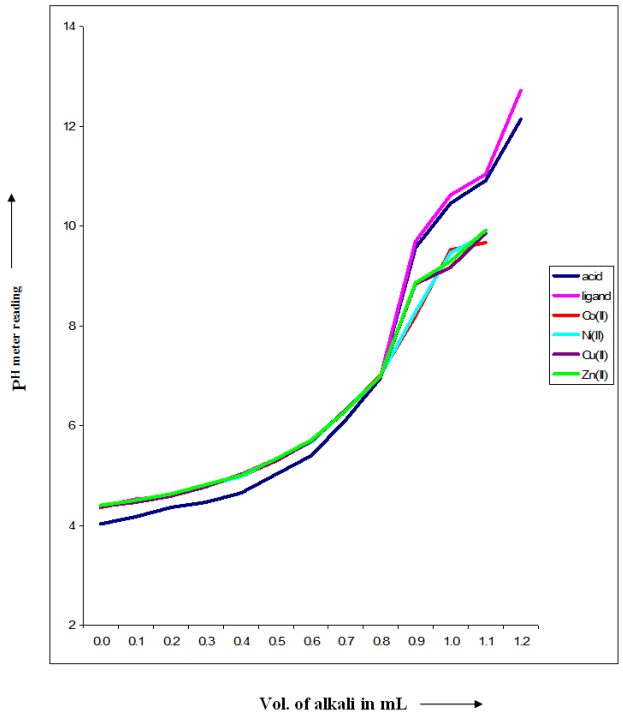


Table 3

Ligand: **MNMFCI** Temp: 298 ± 1K

μ^0 = 0.10 (M) KNO_3 Water: dioxane = 3:2(v/v)

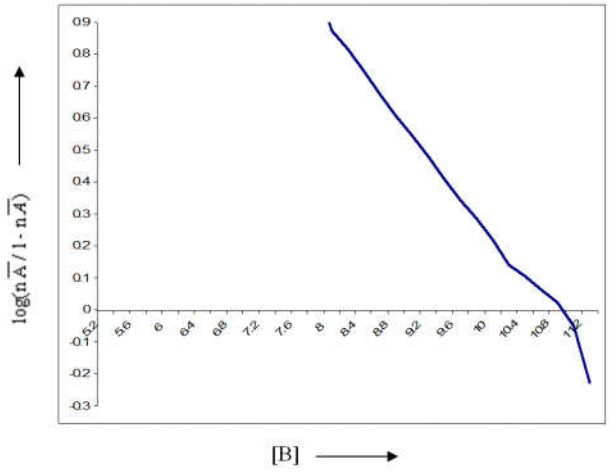
[B]	V_2-V_1	$\bar{n}_A$	log $\bar{n}_A$ / (1 - $\bar{n}_A$)
5.2	0.006	0.889	
5.4	0.006	0.885	
5.6	0.007	0.885	
5.8	0.007	0.882	
6.0	0.008	0.881	
6.2	0.008	0.877	
6.4	0.009	0.874	
6.6	0.009	0.869	
6.8	0.021	0.862	
7.0	0.013	0.853	
7.2	0.015	0.845	1.3262
7.4	0.015	0.842	1.2952
7.6	0.017	0.829	1.2094
7.8	0.021	0.816	1.0472
8.0	0.023	0.802	0.8732
8.2	0.027	0.793	0.8164
8.4	0.031	0.777	0.7488
8.6	0.033	0.756	0.6732
8.8	0.041	0.736	0.6068
9.0	0.043	0.716	0.5464
9.2	0.053	0.693	0.4804
9.4	0.057	0.665	0.4098
9.6	0.063	0.636	0.3444
9.8	0.071	0.608	0.2842
10.0	0.081	0.596	0.2186
10.2	0.097	0.564	0.1414
10.4	0.107	0.559	0.1054
10.6	0.111	0.549	0.0642
10.8	0.117	0.5389	0.0244
11.0	0.127	0.528	-0.05
11.2	0.141	0.528	-0.226

Graph No. 2 Formation curve of ligand MNMFCI

Plot of $\bar{n}A$ Vs [B]

Temp. 298 ± 1 K

μ^0 = 0.10 (M) KNO_3 Water:dioxane = 3:2(v/v)



Co(II)-MNMFCI System

The complex titration curve of the system crossed the ligand mixture curve at pH = 5.06 indicating the start of complex formation.

As the metal titration curves did not join up and run parallel to the ligand titration curve indicating liberation of extra proton due to the hydrolysis of metal ions. Precipitation was observed at P^H = 9.0. Hence, in order to preclude error due to hydrolysis in the calculation of $\bar{n}$, only the lower P^H regions of titration curves were used.

The values of $\bar{n}$ extend between 0.20 to 1.81 (graph – 4a, & table no. – 4,) at temperature 298 K indicating the formation of), at temperature 298 K, the values of log K_1 and log K_2 at the given two temperatures were calculated by half integral method. It was further verified by the mid- point calculation method and the linear plot of log $\bar{n}$ / (1- $\bar{n}$) vs p^L (graph no. 5a, table no. 5) at 298 K and log (2- $\bar{n}$) / ($\bar{n}$ -1) vs p^L (graph 6a, Table no. -5,) at temperature 298 K.

Ni (II) – MNMFCI System

The complex titration curve on the system crossed the ligand mixture curve at pH = 4.94 indicating the start of complexation. The curve increased regularly upto pH = 7.48 indicating constant rate of release of proton and then complex titration curve diverges indicating quick but incomplete dissociation of ligand.

No turbidity appears, hence hydrolysis does not take place.

Values of $\bar{n}$ falls in the range of 0.10 to 1.96 at temperature 298 K (table no.– 6, graph – 4b,) indicating formation of ML and ML_2 type complexes.

Formation curve is almost symmetrical graph no. 4b.

The values of log K_1 and log K_2 were calculated by half integral method and verified by mid point slope method and linear plot of log $\bar{n}$ /(1- $\bar{n}$) vs P^L (graph no. 5b, Table No. 7,) at temperature 298 K log (2- $\bar{n}$)/($\bar{n}$ -1) vs P^L. (Table no.-7, graph 6b) at temperature 298 K.

Cu (II) – MNMFCI System

Complex titration curve crossed the acid titration curve and well separated from ligand titration curve at pH = 5.80. The curve increased regularly and run parallel to the ligand titration curve upto pH = 9.4.

During the titration equilibrium is attained very quickly, no turbidity appears, hence hydrolysis does not take place.

The values of $\bar{n}$ extended from 0.16 to 1.86 at temperature 298 K indicating the formation of ML and ML_2 type of complexes, at temperature (table no-8, ; graph 4c) at 298 K.

Formation curves (graph 4c,) is also very symmetrical, it gave the values of log K_1 and log K_2 by half integral method at given temperature. These values were further verified from mid-point slope method and the linear plot of log ($\bar{n}$ /1- $\bar{n}$) vs P^L (graph no.5c, ; table no. 9,) and plot of log (2 - $\bar{n}$)/($\bar{n}$ -1) vs P^L. (table – 9, ; graph – 6c ,) at temperature 298 K .

Zn (II) – MNMFCI System

Metal ligand titration curve is well separated from ligand titration curve at pH = 5.88 and complex titration curve diverges at higher pH, indicates the incomplete dissociation of ligand.

For the calculation of value of $\bar{n}$ only the symmetrical region of the curve were considered.

The values of $\bar{n}$ extended from 0.40 to 1.86 at temperature 298 K and, indicating the formation of ML and ML_2 type complexes only. (table no 10 . , ; graph 4d,) at temperature 298 K .

The value of log K_1 and log K_2 were calculated from the formation curves (graph-4d) at temperature 298 K by using half – integral method at given temperature. These values of log K_1 and log K_2 were further verified by the mid-point slope calculation method and straight – line plot of log $\bar{n}$ /(1- $\bar{n}$) vs P^L (Table no. 11, ; graph no. 5d, at temperature 298 K and plot of log (2- $\bar{n}$) / ($\bar{n}$ - 1). (table no. 11, ; Graph 6d,) at temperature 298 K .

Table 4

Co (II) + MNMFCI Temp. 298 ± 1 K

μ^0 = 0.10 (M) KNO_3 Water: dioxane = 3:2(v/v)

B	$V_3 - V_2$	$\bar{n}$	P^L
5.2	0.008	0.2019	9.0645
5.4	0.010	0.3037	8.8735
5.6	0.014	0.3275	8.6853
5.8	0.022	0.4015	8.5027
6.0	0.028	0.6057	8.3219
6.2	0.040	0.7449	8.1365
6.4	0.052	1.1035	7.9567
6.6	0.064	1.3409	7.7801
6.8	0.076	1.5727	7.6075
7.0	0.082	1.6759	7.4333
7.2	0.090	1.8171	7.2667

Table 5

Co (II) + MNMFCI Temp. 298 ± 1 K

μ^0 = 0.10 (M) KNO_3 Water: dioxane = 3:2(v/v)

log $\bar{n}$ /(1- $\bar{n}$)	P^L	log (2- $\bar{n}$) / ($\bar{n}$ - 1)	P^L
-0.9455	9.0645	0.5995	7.7799
-0.5923	8.8733	0.0579	7.6075
-0.3135	8.6849	-0.4191	7.4331
0.3793	8.3223		
0.7357	8.1361		

Table 6

Ni (II) + **MNMFCI** Temp: 298 ± 1K

$\mu = 0.1$ M KNO_3 Water: Dioxane 3 :2 (v/v)

B	$V_3 - V_2$	$\bar{n}$	P^L
5.0	0.007	0.1017	7.8647
5.2	0.013	0.2657	7.6795
5.4	0.021	0.4293	7.4945
5.6	0.033	0.6155	7.3131
5.8	0.037	0.8033	7.1321
6.0	0.045	0.9931	6.9521
6.2	0.053	1.2095	6.7767
6.4	0.065	1.4305	6.6025
6.6	0.081	1.6971	6.4363
6.8	0.089	1.9597	6.2723

Table 7

Ni (II) + **MNMFCI** Temp: 298 ± 1K

$\mu = 0.1$ M KNO_3 Water: Dioxane 3 :2 (v/v)

log $\bar{n}$ /(1- $\bar{n}$)	P^L	log (2- $\bar{n}$) / ($\bar{n}$ - 1)	P^L
-0.9457	8.8645	0.5757	7.7765
-0.4419	8.6797	0.1215	7.6024
-0.1237	8.4947	-0.3621	7.4364
0.2049	8.3133		
0.6117	8.1325		

Table 8

Cu(II) + MNMFCI Temp: 298 ± 1K

$\mu = 0.1$ M KNO_3 Water: Dioxane 3 :2 (v/v)

[B]	$V_3 - V_2$	$\bar{n}$	P^L
6.2	0.014	0.1642	7.2702
6.4	0.015	0.2470	7.0780
6.6	0.016	0.3720	6.8894
6.8	0.021	0.5004	6.7016
7.0	0.025	0.6315	6.5148
7.2	0.037	0.7847	6.3300
7.4	0.041	0.9374	6.1462
7.6	0.053	1.1222	5.9664
7.8	0.062	1.3118	5.7882
8.0	0.073	1.5723	5.6204
8.2	0.081	1.8630	5.4585

Table 9

Cu(II) + MNMFCI Temp : 298 ± 1K

$\mu = 0.1$ M KNO_3 Water :Dioxane 3 :2 (v/v)

log $\bar{n}$ /(1- $\bar{n}$)	P^L	log (2- $\bar{n}$) / ($\bar{n}$ -1)	P^L
-0.7070	7.5308	0.8564	6.9668
-0.3494	7.5308	0.3446	6.7886
-0.2272	7.5308	-0.1276	6.6204
-0.2330	7.5308	-0.7998	6.4592
0.5628	7.5308		

Table 10

Zn (II) + **MNMFCI** Temp: 298 ± 1K

$\mu = 0.1$ M KNO_3 Water: Dioxane 3: 2 (v/v)

B	$V_3 - V_2$	$\bar{n}$	P^L
6.0	0.008	0.405	7.4592
6.2	0.010	0.1019	7.2696
6.4	0.012	0.1853	7.0780
6.6	0.016	0.3717	6.8894
6.8	0.022	0.5005	6.7012
7.0	0.028	0.6312	6.5148
7.2	0.036	0.7845	6.3300
7.4	0.042	0.9374	6.1462
7.6	0.054	1.1222	5.9664
7.8	0.066	1.3114	5.7882
8.0	0.078	1.5726	5.6204
8.2	0.086	1.8638	5.4586

Table 11

Zn (II) + **MNMFCI** Temp: 298 ± 1K

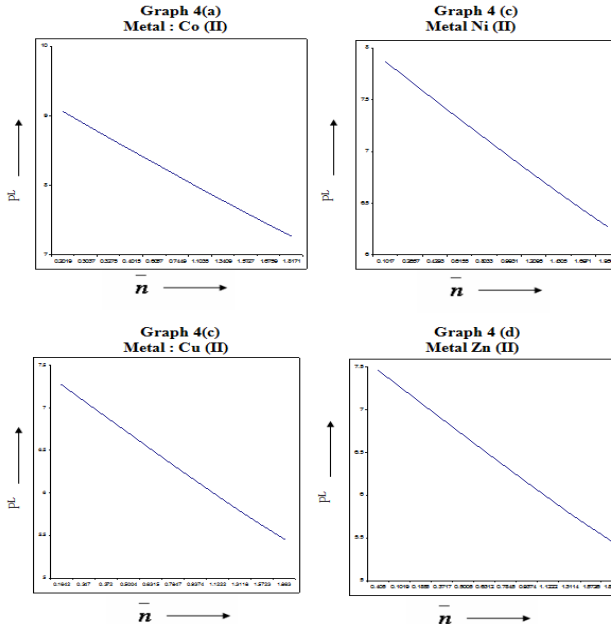
$\mu = 0.1$M KNO_3 Water :Dioxane 3 :2 (v/v)

$\log \bar{n}/(1-\bar{n})$	P^L	$\log (2-\bar{n})/(\bar{n}-1)$	P^L
-0.7072	8.2702	0.8568	6.9668
-0.3496	8.0784	0.3442	6.7886
-0.2267	7.8894	-0.1272	6.6204
-0.2332	7.5148	-0.7994	6.4582
0.5626	7.3304		

Graph No 4

Formation curve Plot of $\bar{n}$ Vs P^L

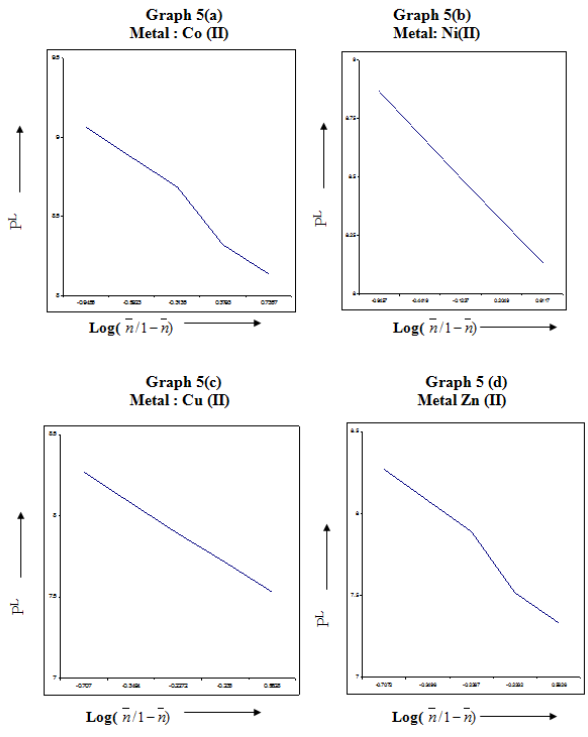
Ligand : MNMFCI $\mu^0 = 0.10$ (M) KNO_3 Temp. 298 ± 1 K Water : dioxane = 3:2(v/v)



Graph No. 5

Linear plot of log $(\bar{n}/1-\bar{n})$ Vs P^L

Ligand : MNMFCI $\mu^0 = 0.10$ (M) KNO_3 Temp. 298 ± 1 K Water : dioxane = 3:2(v/v)



Graph No. 6

Linear plot of log $(2-\bar{n}/\bar{n}-1)$ Vs P^L

Ligand : MNMFCI $\mu^0 = 0.10$ (M) KNO_3 Temp. 298 ± 1 K Water : dioxane = 3:2(v/v)

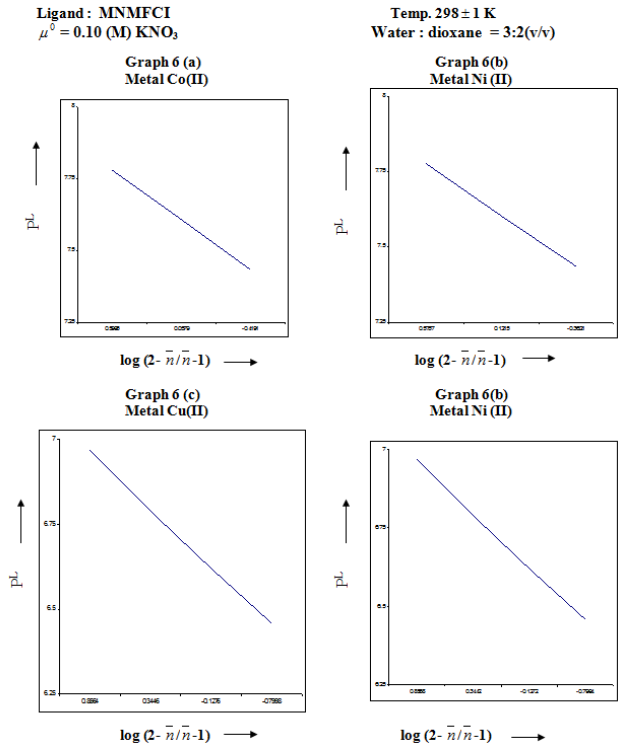


Table 12 Values of protonation constant of ligand and stepwise stability constant of complexes of Co(II), Ni(II), Cu(II) and Zn(II) with ligand at temperature 298.

System		Ligand MNMFCI	
		$\log K_1$	$\log K_2$
	A		10.84
	b		-
	c		10.84
	A	7.46	6.58
Co (II)	b	7.44	6.52
	C	7.47	6.57
	A	7.42	6.52
Ni (II)	b	7.34	6.54
	c	7.36	6.55
	A	6.77	5.63
Cu (II)	b	6.63	5.61
	C	6.74	5.42
	A	6.52	5.63
Zn (II)	b	6.52	5.73
	C	6.42	5.72

Table 13 Stepwise and over all stability constant at temperature 298 of complex compounds of various metals Co(II), Ni(II), Cu(II) & Zn(II) with ligand MNMFCI

System	Ligand MNMFCI		
	$\log K_1$	$\log K_2$	$\log \beta$
MNMFCI	10.86	-	10.84
Cu (II)	7.44	6.56	14.10
Ni (II)	7.38	6.56	13.90
Co (II)	6.68	5.68	12.36
Zn (II)	6.50	5.70	12.24

MNMFCI -Cu(II) > Ni (II) > Co (II) > Zn (II)

DISCUSSION

The values of stepwise stability constants and over all stability constants are given in table no. 12. For the given ligand the

stability constants of complexes for different metals show the sequence

Cu(II) > Ni(II) > Co(II) > Zn(II)

This is natural order given by Irving – William[12]. A theoretical justification of the order of stability constants follows from the consideration of the reciprocal of the ionic radii and 2nd ionization enthalpy of metal. Calvin – Bjerrum titration technique modified by Irving and Rossotti was used to determine the practical proton ligand and metal ligand stability constants at constant ionic strength maintained by using dilute KNO_3 solution. Irving and Rossotti pointed out that the formation constant of metal chelates can be obtained without converting the pH – meter reading [B] to stoichiometric hydrogen ion concentration and without knowing the stiochiometric concentration of neutral salts added to maintain ionic strength. This method is valid for both aqueous and non-aqueous medium.

The nitrate (NO_3^-) ion has very slight complexing tendency. Therefore, competition between nitrate ion and the ligand under study is of no importance[13].

The stability of the chelates is greatly affected by the electron density around the imino nitrogen (- C = N -)[14,15]. Higher the electron density around the nitrogen atom, stronger is the metal ligand bond.

The difference between the successive stepwise stability constant is large, which suggest that the formation of ML and ML_2 chelates take place. The results obtained are in conformity of our previous studies[16,17] and other workers[18,19]

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