



DETERMINATION OF THERMODYNAMIC STABILITY CONSTANT OF BIVALENT TRANSITION METAL COMPLEXES WITH SCHIFFS BASE AS LIGANDS

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ABSTRACT

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 5-Methyl furan-2-carbaldehyde and 7- methoxy naphthalene- 2 – amine with bivalent transition metals i.e. Co(II), Ni(II), Cu(II) and Zn(II).

Schiff's base ligands were synthesized by the condensation reaction of 5-Methyl furan-2-carbaldehyde 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 reported methods. pH metric titrations were carried out in an inert atmosphere of nitrogen gas at constant ionic strength of 0.1M KNO₃ with the help of digital pH meter. Stability constant of complexes of these metals with the ligands synthesized were computed by Irving- Rossoti technique¹ modified by Calvin Bjerrum² 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.

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INTRODUCTION

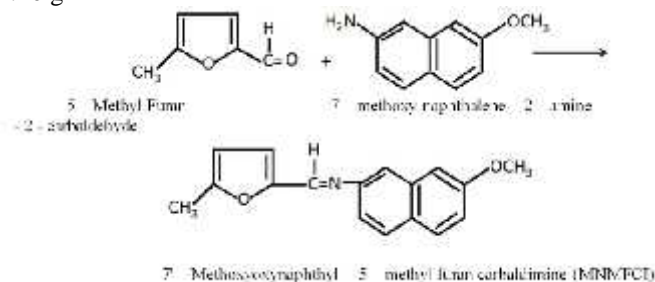
Schiff's base offers a versatile and flexible series of ligands capable to bind with variety of metal ions to give complexes with varying properties. These complexes are biologically active³, have wide potential applications in many fields such as catalysis⁴, electrochemistry⁵ and medicines like anti tumour, anti viral⁶, anti cancer⁷ and other many anti bacterial agents⁸. 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 chelation⁸. 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.

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 methods¹¹.

medium in the ratio 3:2 (v/v). Dioxane was purified by standard method.

Schiff's base ligands were synthesised by reported method¹² 1.5 g of 3-Methyl furan-2-carbaldehyde in solution were mixed with 3.5 g 7 – Hydroxy naphthalene – 2 – amine. The mixture was refluxed in the presence of glacial acetic acid for 3 hours. The solution was concentrated and cooled to 0^o C. The product obtained was filtered, washed several times and recrystallised from ethanol. The yield of product was nearly 2.40 g



Solution of metal salts, ligands, electrolytes etc were prepared in doubly distilled CO₂ free water. Strength of various solutions used were tabulated in Table - 1.

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pH metric titration of acid, acid + ligand and acid + ligand + metal ion solutions were done at constant ionic strength of 0.1 M KNO₃ at temperature 298 K 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.

A graph was plotted between pH meter reading [B] and volume of alkali added in each case. Graph no. 1. 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

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

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

$$\bar{n}_A = 1 + [(V_1 - V_2) / (V^0 + V_1)] (N^0 + E^0) / T_L^0$$

$$\bar{n} = [(V_3 - V_2) / (V^0 + V_1)] [(N^0 + E^0) / T_M^0] \times 1 /$$

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

The values of volumes V₁, V₂ & V₃ corresponding to the same pH values were read from acid, ligand and complex titration curves given in **graph – 1** at temperature 298 K.

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. 1**) at temperature 298 K.

The formation curves obtained from the plot of \bar{n}_A vs [B] (**graph NO. 2**) at temperature 298 K, show that values of \bar{n}_A lies between 0.42 and 0.878. This indicates that ligand is monoprotic.

Dissociation of ligand may be given as,
 $LH \rightleftharpoons H^+ + L^-$

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

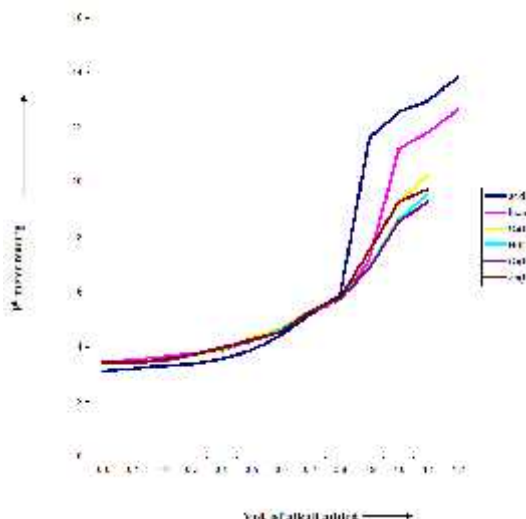
Metal ions	V ⁰ in mL	Y	N ⁰	E ⁰	T _L ⁰	T _M ⁰
Co (II)	100	1	1.0(M)	1.0 x 10 ⁻² (M)	2.5 x 10 ⁻³ (M)	5.0 x 10 ⁻⁴ (M)
Ni (II)	100	1	1.0(M)	1.0 x 10 ⁻² (M)	2.5 x 10 ⁻³ (M)	5.0 x 10 ⁻⁴ (M)
Cu (II)	100	1	1.0(M)	1.0 x 10 ⁻² (M)	2.5 x 10 ⁻³ (M)	5.0 x 10 ⁻⁴ (M)
Zn (II)	100	1	1.0(M)	1.0 x 10 ⁻² (M)	2.5 x 10 ⁻³ (M)	5.0 x 10 ⁻⁴ (M)

The value of proton ligand stability constant was calculated by half – integral method and it was further corroborated by linear plot method³⁻⁴ (log $\bar{n}_A / (1 - \bar{n}_A)$) vs [B] (**graph No.3**) at temperature 298 K .

Table - 2 Ligand - MNMFCI Temp. 298 ± 1 K
 $\sim^0 = 0.1(M)$ KNO₃, Water: Dioxane 3:2 (v/v)

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	3.12	3.42	3.42	3.42	3.42	3.43
0.1	3.22	3.52	3.42	3.42	3.42	3.4
0.2	3.32	3.64	3.54	3.46	3.46	3.54
0.3	3.36	3.74	3.72	3.72	3.7	3.7
0.4	3.56	3.86	3.86	3.94	3.98	3.92
0.5	3.88	4.24	4.34	4.22	4.22	4.26
0.6	4.42	4.66	4.68	4.66	4.56	4.56
0.7	5.17	5.27	5.29	5.28	5.28	5.3
0.8	5.88	5.72	5.74	5.76	5.76	5.78
0.9	11.6	7.16	7.47	6.88	6.86	7.53
1.0	12.57	11.22	9.27	8.68	8.58	9.28
1.1	12.96	11.82	10.3	9.56	9.3	9.76
1.2	13.86	12.66				

The values of volumes V₁, V₂ & V₃ corresponding to the same pH values were read from acid, ligand and complex titration curves given in graph – 6.16 page no. 249 at temperature 298 K.



Graph No. 1 Experimental Curve with ligand MNMFCI
 conc. 1.0 x 10⁻² (M) (1.0 x 10⁻²) KNO₃, Water-Dioxane medium (V:V) 3:2

Co(II) –MNMFCI System

Complex titration curves separated from ligand mixture curve at pH = 6.18 the curves diverge at pH above 8.68.

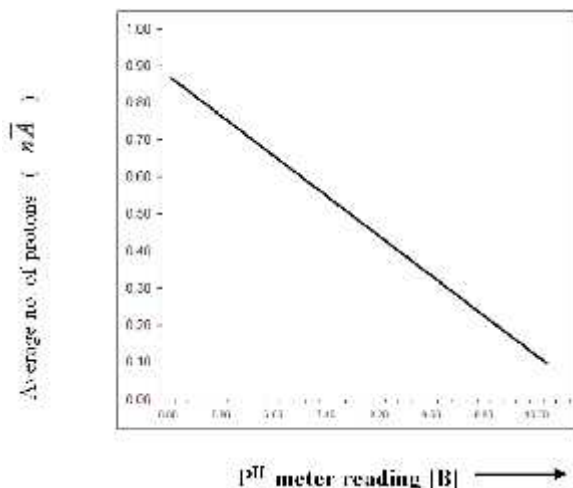
The value of \bar{n} lies between 0.09 and 1.84 (table no.4, graph no 4a at temperature 298 K. indicating the formation of ML and ML₂ types of complexes.

From the formation curves (graph no 4) the values of log K₁ and log K₂ were calculated by half – integral method at given temperature. The values were further corroborated by mid point slope method and linear plot of log $\bar{n} / (1 - \bar{n})$ vs P^L (Table no. 5 graph no. 5a) at temperature 298 K and plot of log (2 - $\bar{n}) / (\bar{n} - 1)$ vs P^L. (Table no. 5 graph 6a) at temperature 298 K.

Table 3

Ligand: MNMFCI
 [L] = 0.1 M KNO₃ Temp: 298 ± 1 K
 Water: Dioxane 3:2 (v/v)

[B]	V ₂ - V ₁	\bar{n}_A	$\log \bar{n}_A / (1 - \bar{n}_A)$
5.0	0.030	0.878	0.8996
5.2	0.032	0.8794	0.8642
5.4	0.034	0.8716	0.8326
5.6	0.036	0.8639	0.8020
5.8	0.038	0.8560	0.7732
6.0	0.040	0.8528	0.7594
6.2	0.042	0.8436	0.7526
6.4	0.044	0.8328	0.6940
6.6	0.046	0.8189	0.6582
6.8	0.049	0.8076	0.6262
7.0	0.050	0.7928	0.5821
7.2	0.054	0.7788	0.5483
7.4	0.056	0.7682	0.5190
7.6	0.060	0.7528	0.4822
7.8	0.064	0.7366	0.4374
8.0	0.068	0.7242	0.4178
8.2	0.072	0.7156	0.4019
8.4	0.076	0.7037	0.3746
8.6	0.078	0.6928	0.3528
8.8	0.080	0.6756	0.3179
9.0	0.084	0.6592	0.2882
9.2	0.808	0.6394	0.2486
9.4	0.094	0.6192	0.2122
9.6	0.101	0.5993	0.1744
9.8	0.102	0.5884	0.1538
10.0	0.106	0.5682	0.1178
10.2	0.110	0.5526	0.0888
10.4	0.116	0.5266	0.0478
10.6	0.124	0.4984	0.0026
10.8	0.132	0.4675	-0.0558
11.0	0.144	0.4210	-0.1422



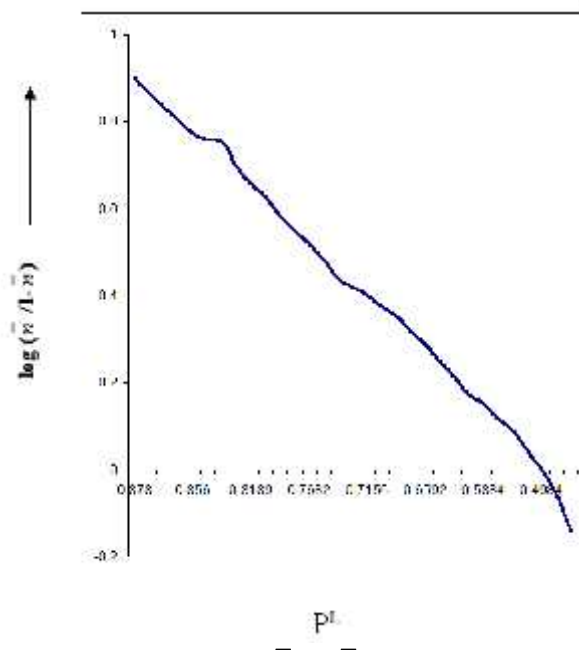
Graph No. 2 Formation Curve of ligand MNMFCI

Plot of \bar{n}_A Vs [B] ~ = 0.1 M KNO₃ Water: Dioxane 3:2(v/v)

Ni(II) – MNMFCI System

The complex titration curves crossed the ligand titration curve at pH = 5.16 indicating the start of complexation. The curve increases regularly upto pH = 7.78 indicating quick but incomplete dissociation of ligand.

No turbidity appears, hence hydrolysis does not take place values of \bar{n} falls between 0.45 and 1.972 (graph 4b, Table no. 6.) at temperature 298 K indicating the formation of ML and ML₂ type of complexes.



Graph No. 3 Linear Plot of $\log (\bar{n}_A / (1 - \bar{n}_A))$ Vs P^L Ligend: MNMFCI

Temp: 298 ± 1K ~ = 0.1 M KNO₃ Water: Dioxane 3:2(v/v)

From the formation curves, (graph 4b) the values of log K₁ and log K₂ were calculated by half integral method at given temperature and verified by mid – point slope method and linear plot of $\log (\bar{n} / (1 - \bar{n}))$ vs P^L (graph 5b, table no. 7 at temperature 298 K and liner plot of $\log (2 - \bar{n} / (\bar{n} - 1))$ vs P^L (graph no. 6b table no. 7) at temperature 298 K.

Cu(II) – MNMFCI System

The complex, titration curve separated from ligand mixture curve at pH 5.28 indicating the start of complex formation.

As the metal titration curves did not join up and run parallel to the ligand titration curves indicating liberation of extra proton due to hydrolysis. Precipitation was observed at pH = 8.36. Hence, in order to preclude error due to hydrolysis in the calculation of \bar{n} , only the lower pH region of titration curves were used.

The values of \bar{n} lies between 0.04 to 1.90 graph – 3 table no. 8 at temperature 298 K indicating the formation of ML and ML₂ type of complexes.

From the formation curves(graph – 4c and table no. 8) of \bar{n} vs P^L , the value of log K₁ and log K₂ at the given two temperatures were calculated by half – integral method. It was verified by the mid – point slope method and linear plot of $\log (\bar{n} / (1 - \bar{n}))$ vs P^L graph no. 5c p, table no. 9 and plot of $\log (2 - \bar{n}) / (\bar{n} - 1)$ vs P^L graph no.6c and table no. 9 at temperature 298 K.

Zn(II) – MNMFCI System

The complex curves separated from ligand titration curves at pH = 5.89 and diverges at pH above 8.65.

During the titration no turbidity appears, hence hydrolysis does not take place.

The values of \bar{n} lies between 0.04 to 1.81 (Table – 10, graph 4d at temperature 298 K indicating the formation of ML and ML₂ type of complexes.

From the formation curve, (graph – 4d) at 298 K values of log K₁ and log K₂ were calculated by half – integral method.

It was further verified by mid-point slope method and linear plot of $\log \bar{n}/(1-\bar{n})$ vs P^L graph no. 5d table no. 11 at temperature 298 K and plot of $\log (2-\bar{n})/(\bar{n}-1)$ vs P^L (graph no.– 6d , Table no.11) at temperature 298 K.

Table 4 Co (II) + MNMFCl Temp: 298 ± 1 K
 ~ = 0.1 M KNO₃ Water: Dioxane 3:2(v/v)

[B]	V ₃ – V ₂	\bar{n}	P ^L
6.0	0.006	0.0936	7.2140
6.2	0.008	0.1898	7.0226
6.4	0.010	0.2886	6.8318
6.6	0.015	0.4150	6.6396
6.8	0.020	0.5210	6.4540
7.0	0.024	0.6320	6.2646
7.2	0.032	0.7962	6.0820
7.4	0.034	0.9386	5.8964
7.6	0.042	1.1452	5.7192
7.8	0.046	1.3078	5.5374
8.0	0.054	1.5220	5.3636
8.2	0.065	1.8464	5.2080

Table - 5 Co(II) + MNMFCl Temp: 298 ± 1 K
 ~ = 0.1 M KNO₃ Water: Dioxane 3:2(v/v)

$\log \bar{n}/(1-\bar{n})$	P ^L	$\log (2-\bar{n})/(\bar{n}-1)$	P ^L
-0.8494	7.0226	0.7696	5.7192
-0.3910	6.8318	0.1978	5.5476
-0.1486	6.6396	-0.1846	5.3750
0.2350	6.2648	-0.7409	5.2078
0.5912	6.0814		

Table - 6 Ni (II) + MNMFCl Temp: 298 ± 1 K
 ~ = 0.1 M KNO₃ Water: Dioxane 3:2(v/v)

B	V ₃ – V ₂	\bar{n}	P ^L
5.2	0.003	0.0454	8.0096
5.4	0.004	0.1376	7.8178
5.6	0.008	0.2318	7.6266
5.8	0.012	0.3512	7.4374
6.0	0.020	0.4935	7.2476
6.2	0.024	0.5932	7.0618
6.4	0.028	0.6980	6.8716
6.6	0.036	0.8552	6.6868
6.8	0.040	1.0413	6.5070
7.0	0.048	1.2390	6.3732
7.2	0.056	1.4634	6.1568
7.4	0.064	1.6948	5.9864
7.6	0.070	1.9720	5.8244

Table – 7 Ni(II) + MNMFCl Temp: 298 ± 1 K
 ~ = 0.1 M KNO₃ Water: Dioxane 3:2(v/v)

$\log \bar{n}/(1-\bar{n})$	P ^L	$\log (2-\bar{n})/(\bar{n}-1)$	P ^L
-0.7956	7.8182	0.5026	6.3232
-0.5202	7.6264	0.0632	6.1518
-0.2668	7.4378	-0.3596	5.9860
0.1640	7.0668		
0.3642	6.8712		
0.7704	6.6816		

Table - 8 Cu(II) + MNMFCl Temp: 298 ± 1 K
 ~ = 0.1 M KNO₃ Water: Dioxane 3:2(v/v)

B	V ₃ – V ₂	\bar{n}	P ^L
5.0	0.004	0.0451	8.2098
5.2	0.006	0.0912	8.0140
5.4	0.008	0.1606	7.8212
5.6	0.012	0.2316	7.5658
5.8	0.014	0.3742	7.3386
6.0	0.020	0.5174	7.2540
6.2	0.028	0.6410	7.0652
6.4	0.034	0.7946	6.8820
6.6	0.042	0.9772	6.7015
6.8	0.048	1.1410	6.5178
7.0	0.052	1.3662	6.3438
7.2	0.064	1.6178	6.1762
7.4	0.078	1.9040	6.1762

Table - 9 Cu(II) + MNMFCl Temp: 298 ± 1 K
 ~ = 0.1 M KNO₃ Water: Dioxane 3:2(v/v)

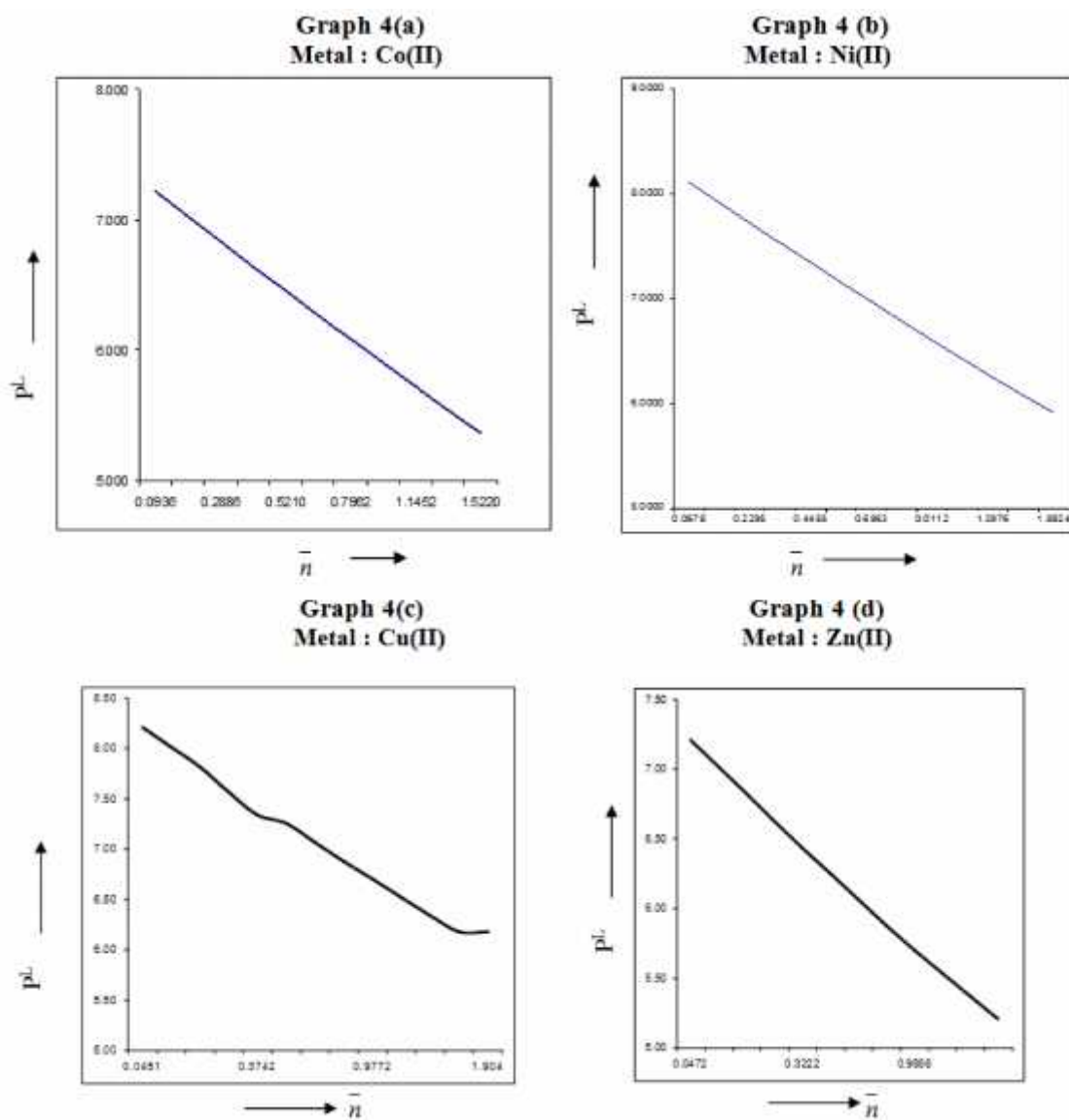
$\log \bar{n}/(1-\bar{n})$	P ^L	$\log (2-\bar{n})/(\bar{n}-1)$	P ^L
-0.7174	7.8202	0.7874	6.5188
-0.5202	7.6266	0.2396	6.3446
-0.2228	7.4396	-0.2082	6.1762
0.2512	7.0654		
0.5874	6.8816		

Table 10 Zn(II) + MNMFCl Temp:298 ± 1 K
 ~ = 0.1 M KNO₃ Water: Dioxane 3:2(v/v)

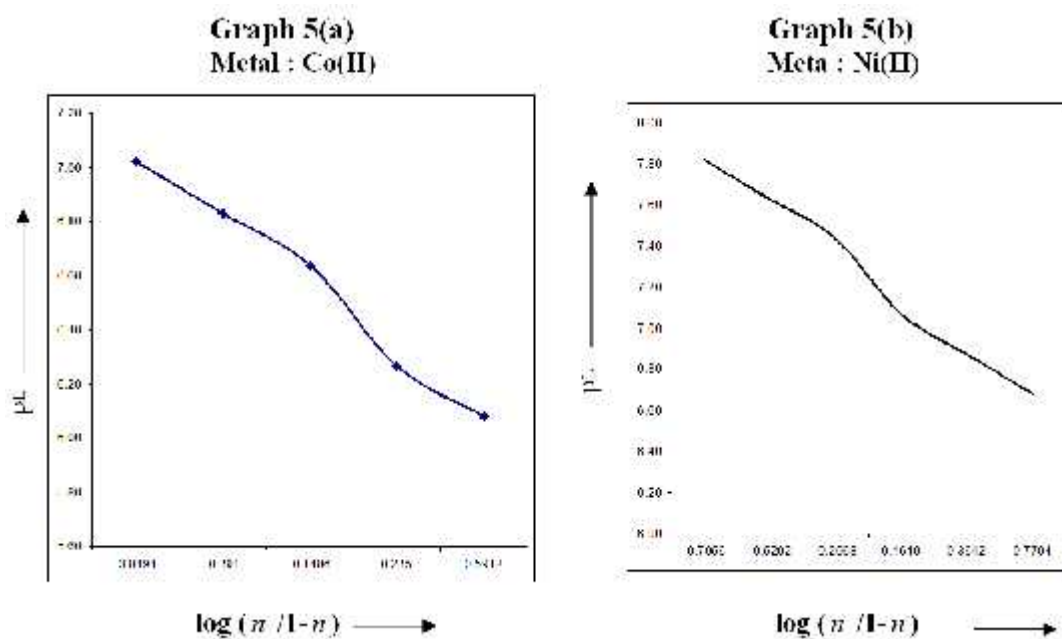
[B]	V ₃ – V ₂	\bar{n}	P ^L
6.0	0.004	0.0472	7.2102
6.2	0.006	0.0948	7.0166
6.4	0.008	0.1446	6.8188
6.6	0.010	0.2194	6.6254
6.8	0.012	0.3222	6.4352
7.0	0.016	0.4554	6.2478
7.2	0.022	0.5906	6.0604
7.4	0.028	0.7802	6.8798
7.6	0.036	0.9856	5.7014
7.8	0.044	1.2518	5.5313
8.0	0.052	1.5214	5.3645
8.2	0.066	1.8180	5.2036

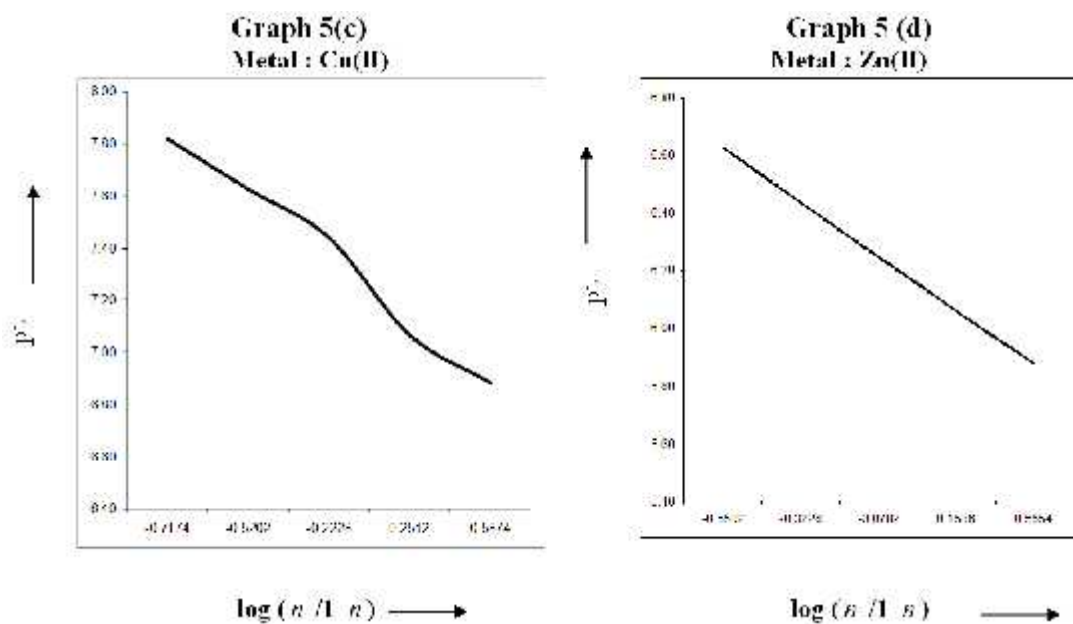
Table - 11 Zn(II) + MNMFCl Temp: 298 ± 1 K
 ~ = 0.1 M KNO₃ Water: Dioxane 3:2(v/v)

$\log \bar{n}/(1-\bar{n})$	P ^L	$\log (2-\bar{n})/(\bar{n}-1)$	P ^L
-0.5502	6.6254	0.4728	5.5314
-0.3228	6.4352	0.0376	5.3642
-0.0782	6.2474	-0.6530	5.2031
0.1586	6.0606	-0.7228	5.0321
0.5554	5.8798	-0.8315	4.9832
		-0.9232	4.8215

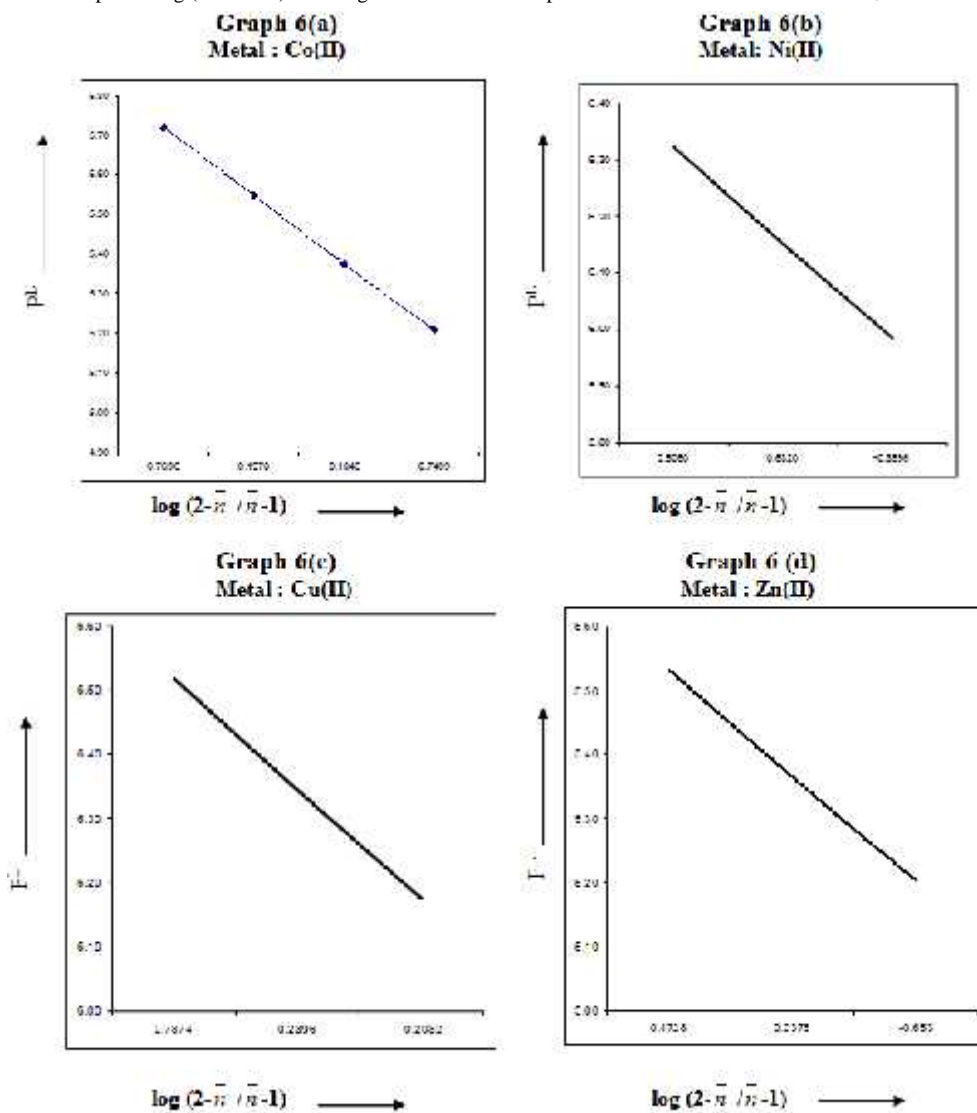


Graph No. 4 Formation curve Plot of \bar{n} Vs pL Ligand: MNMFCI Temp: 298 ± 1 K $\sim = 0.1$ M KNO_3 Water: Dioxane 3:2(v/v)





Graph No. 5 Linear plot of $\log(n-1/n)$ Vs P^H Ligand : MNMFCI Temp: 298 ± 1 K ~ 0.1 M KNO_3 Water: Dioxane 3:2(v/v)



Graph No. 6 Linear plot of $\log(2-n/n-1)$ Vs P^H Ligand MNMFCI Temp: 298 ± 1 K ~ 0.1 M KNO_3 Water : Dioxane 3:2(v/v)

The value of protonation constant and stepwise stability constant obtained from different computational methods at temperatures 298 K are summarized in table no. 12. The different computational methods are :-

1. Half - integral method
2. Mid - point slope calculation method and
3. Straight - line plot method.

Table no 12 Values of protonation constant of ligand and stepwise stability constant of complexes of Co(II), Ni(II), Cu(II) & Zn(II) with ligand (MNMFCI) at temperatures 298 K.

Metal ions	Ligand MNMFCI	
	log K ₁	log K ₂
	11.20	-
MNMFCI	11.22	-
Co(II)	7.18	6.18
	7.22	6.20
Ni(II)	7.18	6.10
	7.06	5.95
	7.18	6.24
Cu(II)	6.44	5.36
	6.30	5.36
	6.46	5.46
Zn(II)	6.16	5.42
	6.04	5.38
	6.12	5.36
	6.14	5.32

The most representative values of log K₁ and log K₂ are given in table no.13.

Table - 13 log of stepwise and overall stability constants of complex compounds of various metals with ligand MNMFCI at 298K. $\sim^0 = 0.10$ (M) KNO₃ Water - dioxane medium (V/V) = 3:2

System	Ligand (MNMFCI)		
	log K ₁	log K ₂	log S
HNMFCI			
MNMFCI	11.88		11.88
Cu (II)	7.46	7.42	14.88
Ni (II)	7.34	7.26	14.60
Co (II)	6.75	6.68	13.43
Zn(II)	6.58	6.42	13.0

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. 13. 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 - William¹⁵. 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 KNO₃ solution. The advantage of Irving and Rossotti method is 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 stoichiometric concentration of neutral salts added to maintain ionic strength. This method is valid for both aqueous and non-aqueous medium.

The nitrate (NO₃⁻) ion has very slight complexing tendency. Therefore, competition between nitrate ion and the ligand under study is of no importance¹⁶.

The stability of the chelates is greatly affected by the electron density around the imino nitrogen (- C = N -)¹⁷. 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 ML₂ chelates take place. The results obtained are in conformity of our previous studies and the study of other workers¹⁷⁻²⁴

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