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Research Article**DETERMINATION OF STABILITY CONSTANT OF BIVALENT TRANSITION METAL COMPLEXES WITH SCHIFFS BASE LIGANDS DERIVED FROM METHYL FURAN WITH HYDROXY NAPHTHALENE AMINE****Prem Mohan Mishra., Lalan Kumar Jha and Naresh Mohan Yadav**

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DOI: <http://dx.doi.org/10.24327/ijrsr.2017.0810.0989>**ARTICLE INFO****Article History:**Received 20th July, 2017Received in revised form 29th

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Accepted 30th September, 2017Published online 28th October, 2017**Key Words:**

Substituted furan, Bivalent transition metals, Schiff's base ligand, coordination compound, stability constant, Irving-Rossoti technique modified by Calvin Bjerrum

ABSTRACT

In continuation of our previous work here also, we have been interested in studying the stability of complexes of bivalent transition metals with biologically active ligands.

Here we are presenting determination of stability constant of complex compounds using ligands synthesised from 2-Methyl furan with 7- Hydroxy naphthalene 2-amine with bivalent transition metals.

Schiff's base ligands were synthesized by the condensation reaction of 2-Methyl furan with 7-hydroxy 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 and stability constant of complexes of these metals with the ligands synthesized were computed at 298K. temperature by Irving- Rossoti technique¹ modified by Calvin Bjerrum²

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).

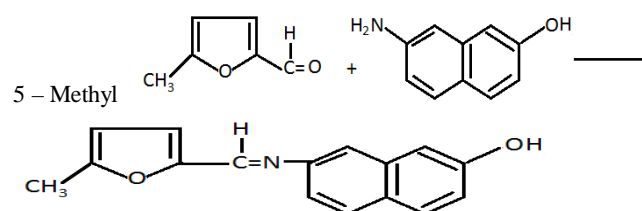
This result is in agreement with our previous studies and also the natural order proposed by Irving-William³**Copyright © Prem Mohan Mishra et al, 2017**, this is an open-access article distributed under the terms of the Creative Commons Attribution License, which permits unrestricted use, distribution and reproduction in any medium, provided the original work is properly cited.**INTRODUCTION**

Heterocyclic compounds display diverse pharmacological properties⁴. Metal ions play vital role in a vast number of widely different biological processes. Schiff's base offers a versatile and flexible series of ligands. These ligands bind with variety of metal ions to give complexes with varying properties. These complexes are biologically active⁵, have wide potential applications in many fields such as catalysis⁶, electrochemistry⁷ and medicines like anti tumour⁸ anti viral⁹, anti cancer¹⁰ and other many anti bacterial agent¹¹. The interaction of these ions with biologically active ligands is a subject of considerable interest. Some of the biologically active compounds act via Chelation¹². 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 methods¹³. 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 method¹³ 1.5 g 2-Methyl furan in solution were mixed with 3.0 g 7-Hydroxy naphthalene-2-amine. The mixture was refluxed in the presence of glacial acetic acid for 2 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 2.30 g. Reactions involved may be represented as



7-Hydroxy naphthyl-5-methyl furan 2-carbaldimine (HNMFCl)

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Solution of metal salts, ligands, electrolytes etc were prepared in doubly distilled CO₂ free water. Strength of various solutions used were tabulated in table no.-1.

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 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. (Graph no. 1). The three titration curves so obtained for each metal ions are referred as: i) Acid titration curve (a) ii) Ligand titration curve (b) and iii) 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

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

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

$P^L = \log$

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

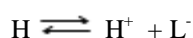
Proton-Ligand Stability Constant

The ligand titration curve separates from acid titration curve at pH 5.10 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] (Graph no. 2) at temperature 298 K, show that values of \bar{n}_A lies between 0.4576 and 0.8920 (Table no. 3) This indicates that ligand is monoprotic.

Dissociation of ligand may be given as,



The value of proton ligand stability constant was calculated at temperature 298 K. by half-integral method and it was further

corroborated by linear plot method ($\log \bar{n}_A / (1 - \bar{n}_A)$) vs [B] (Graph no. 3)

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)

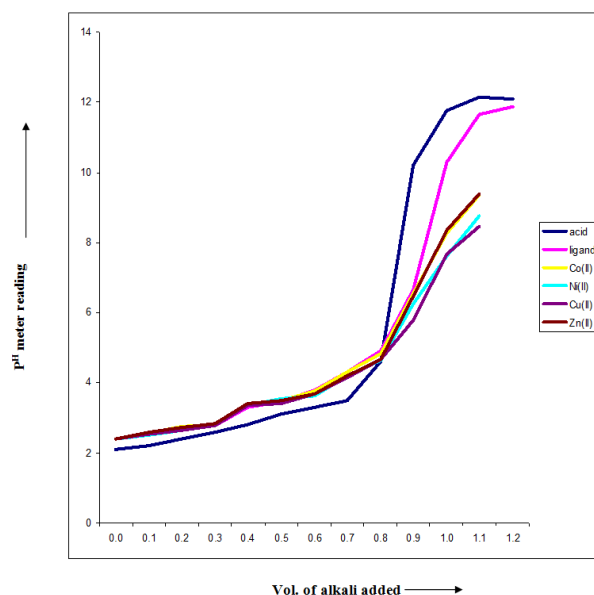
Table 2

Ligand: HNMFCI		Temperature 298 ± 1K				
$\mu^0 = 0.1(M) KNO_3$,		Water-Dioxane medium (V/V) = 3:2				
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	2.10	2.40	2.40	2.40	2.40	2.40
0.1	2.20	2.50	2.56	2.52	2.55	2.60
0.2	2.40	2.65	2.76	2.64	2.64	2.72
0.3	2.60	2.78	2.82	2.78	2.78	2.84
0.4	2.80	3.30	3.36	3.36	3.37	3.40
0.5	3.10	3.46	3.48	3.54	3.42	3.48
0.6	3.30	3.78	3.76	3.64	3.68	3.68
0.7	3.50	4.30	4.32	4.20	4.14	4.20
0.8	4.60	4.90	4.84	4.65	4.68	4.67
0.9	10.20	6.66	6.54	6.25	5.78	6.46
1.0	11.75	10.29	8.26	7.62	7.67	8.34
1.1	12.15	11.64	9.35	8.75	8.46	9.40
1.2	12.10	11.88				

$\mu = 0.1M KNO_3$

Temp: 298 ± 1K

Water: Dioxane 3:2 (v/v)



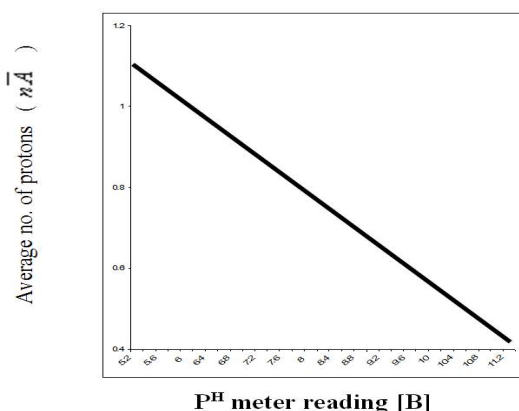
Graph No 1 Experimental Curve with ligand HNMFCI

Table No 3

Ligand-HNMFCI		Temperature 298 ± 1K	
$\mu^0 = 0.1(M) KNO_3$,		Water:Dioxane medium = 3:2 (v/v)	
B	V ₂ -V ₁	\bar{n}_A	$\log \bar{n}_A / (1 - \bar{n}_A)$
5.2	0.028	0.8920	0.9158

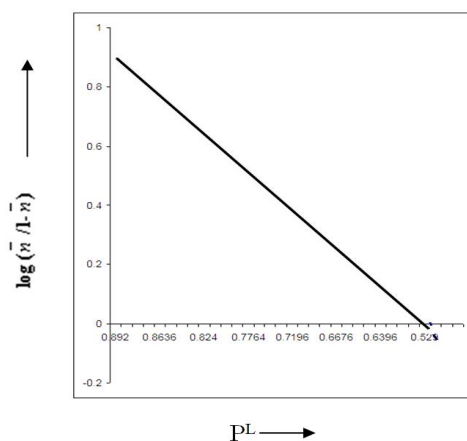
5.4	0.027	0.8870	0.8987
5.6	0.032	0.8796	0.8645
5.8	0.030	0.8720	0.8330
6.0	0.032	0.8636	0.8223
6.2	0.034	0.8560	0.7724
6.4	0.038	0.8438	0.7732
6.6	0.040	0.8356	0.7058
6.8	0.042	0.8240	0.6686
7.0	0.046	0.8120	0.6340
7.2	0.048	0.7996	0.6025
7.4	0.052	0.7882	0.5685
7.6	0.054	0.7764	0.5386
7.8	0.056	0.7640	0.5092
8.0	0.060	0.7478	0.4720
8.2	0.064	0.7362	0.4444
8.4	0.070	0.7196	0.4096
8.6	0.076	0.7028	0.3738
8.8	0.078	0.6916	0.3508
9.0	0.078	0.6795	0.3266
9.2	0.080	0.6676	0.3032
9.4	0.082	0.6640	0.2950
9.6	0.084	0.6556	0.2792
9.8	0.084	0.6560	0.2792
10.0	0.088	0.6396	0.2494
10.2	0.096	0.6192	0.2130
10.4	0.012	0.5960	0.1682
10.6	0.106	0.5672	0.1178
10.8	0.118	0.529	0.0574
11.0	0.130	0.4727	-0.0482
11.2	0.132	0.4640	-0.0642
11.4	0.136	0.4576	-0.0838

Ligand –HNMFCI
 $\mu^0 = 0.1(M) KNO_3$,
 Temperature $298 \pm 1K$
 Water:Dioxane medium = 3:2 (v/v)



Graph No 3 Formation Curve of ligand HNMFCI (Plot of \bar{n} Vs [B])

Ligand – HNMFCI
 $\mu^0 = 0.1(M) KNO_3$,
 Temperature $298 \pm 1K$
 Water:Dioxane medium = 3:2 (v/v)



Graph No 4 Linear Plot of $\log(\bar{n}_A/1 - \bar{n}_A)$ Vs P^L

Co(II) –HNMFCI System

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

The value of \bar{n} lies between 0.1174 and 1.78 (Table no. 4, Graph no. 4a) at temperature 298 K indicating the formation of ML and ML_2 types of complexes.

From the formation curves (Graph no.4a) the values of $\log K_1$ and $\log K_2$ 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) and plot of $\log(2 - \bar{n})/(\bar{n} - 1)$ vs P^L (Table no. 5, Graph 6a) at temperature 298 K.

Ni (II)-HNMFCI 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 0.06 and 1.89 (Graph no.4b, Table no. 6) at temperature 298 K indicating the formation of ML and ML_2 type of complexes. From the formation curves, (Graph no.4b) the values of $\log K_1$ and $\log K_2$ 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 no.5b, Table no.7,) at temperature 298 K and linear plot of $\log(2 - \bar{n})/(\bar{n} - 1)$ vs P^L (Graph no. 6b, Table no.7) at temperature 298 K.

Cu (II)-HNMFCI 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 1.9 (Graph no.–4c, Table no. 8) at temperature 298 K indicating the formation of ML and ML_2 type of complexes.

From the formation curve (Graph no.4c, and Table no.8) of \bar{n} vs P^L , the value of $\log K_1$ and $\log K_2$ 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, Table no. 9) and plot of $\log(2 - \bar{n})/(\bar{n} - 1)$ vs P^L (Graph no.5c, Table no. 9) at temperature 298 K

Zn (II)-HNMFCI System

The complex curves separated from ligand titration curves at pH = 5.66 and diverges at pH above 8.64. During the titration no turbidity appears, hence hydrolysis does not take place. The

values of \bar{n} lies between 0.46 to 1.78 (Table no.-10, Graph no. 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 vs P^L (Graph no.5d, Table no. 11) and plot of log (2 – \bar{n})/(\bar{n} – 1) vs P^L (Graph no.-6d,

Table no.-11

Table No 4

Ligand – HNMFCI, Metal Co(II), Temperature 298 ± 1K
 $\mu^0 = 0.1(M) KNO_3$, Water:Dioxane medium = 3:2 (v/v)

B	V ₃ – V ₂	\bar{n}	P ^L
6.0	0.006	0.1174	7.3164
6.2	0.008	0.1896	7.1226
6.4	0.010	0.2640	6.9264
6.6	0.014	0.3644	6.7990
6.8	0.018	0.4934	6.5515
7.0	0.020	0.6260	6.3644
7.2	0.028	0.7626	6.1778
7.4	0.034	0.9288	5.9954
7.6	0.040	1.1042	5.8144
7.8	0.048	1.3122	5.6384
8.0	0.054	1.5282	5.4646
8.2	0.066	1.7806	5.2730

Table No 5

Metal-Co(II)

Ligand – HNMFCI, Temperature 298 ± 1K
 $\mu^0 = 0.1(M) KNO_3$, Water: Dioxane medium = 3:2 (v/v)

log $\bar{n}/(1 - \bar{n})$	P ^L	log (2 - \bar{n})/(\bar{n} - 1)	P ^L
-0.8768	7.3160	0.9350	5.8144
-0.6300	7.1226	0.3430	5.6386
-0.4406	6.9294	-0.0488	5.4647
-0.2410	6.7388	-0.5506	5.2730
0.2234	6.3640		
0.4715	6.1768		

Table No 6

Metal : Ni(II)

Ligand – HNMFCI, Temperature 298 ± 1K
 $\mu^0 = 0.1(M) KNO_3$, Water:Dioxane medium = 3:2 (v/v)

[B]	V ₃ – V ₂	\bar{n}	P ^L
5.2	0.002	0.0678	8.1118
5.4	0.004	0.1364	7.9180
5.6	0.018	0.2295	7.7262
5.8	0.012	0.3244	7.5350
6.0	0.018	0.4455	7.3464
6.2	0.020	0.5694	7.1584
6.4	0.028	0.6963	6.9714
6.6	0.034	0.8508	6.7870
6.8	0.040	0.0112	6.6042
7.0	0.046	1.2016	6.4254
7.2	0.054	1.3976	6.2486
7.4	0.062	1.6258	6.0770
7.6	0.070	1.8924	5.9132

Table No-7

Metal: Ni(II)

Ligand – HNMFCI, Temperature 298 ± 1K
 $\mu^0 = 0.1(M) KNO_3$, Water: Dioxane medium = 3:2 (v/v)

Log $\bar{n}/(1 - \bar{n})$	P ^L	log (2 - \bar{n})/(\bar{n} - 1)	P ^L
-0.8006	7.9178	0.5972	6.4254
-0.5256	7.7262	0.1798	6.2488
-0.3180	7.5354	-0.2236	6.0770
-0.0948	7.3466		
0.1216	1585		
0.3604	6.9710		
0.7552	6.7872		

Table No 8

Metal Cu(II)

Ligand – HNMFCI, Temp: 298 ± 1 K
 $\mu = 0.1 M KNO_3$, Water: Dioxane 3:2 (v/v)

[B]	V ₃ – V ₂	\bar{n}	P ^L
5.2	0.004	0.0450	8.3098
5.4	0.006	0.1132	8.1156
5.6	0.010	0.1824	7.9222
5.8	0.014	0.2752	7.7306
6.0	0.018	0.3944	7.5418
6.2	0.020	0.5162	7.3532
6.4	0.026	0.6648	7.1682
6.6	0.036	0.8164	6.9834
6.8	0.042	0.9966	6.8028
7.0	0.046	1.1838	6.6236
7.2	0.052	1.4022	6.2774
7.4	0.066	1.6264	6.2774
7.6	0.078	1.9092	6.1156

Table No 9

Metal Cu(II)

Ligand – HNMFCI, Temp: 298 ± 1 K
 $\mu = 0.1 M KNO_3$, Water: Dioxane 3:2 (v/v)

log $\bar{n}/(1 - \bar{n})$	P ^L	log (2 - \bar{n})/(\bar{n} - 1)	P ^L
-0.8942	8.1156	0.6472	6.6234
-0.6524	7.9222	0.1726	6.4490
-0.1862	7.5414	-0.2246	6.2774
0.2968	7.1682		
0.6484	6.9834		

Table No 10

Metal Zn(II)

Ligand – HNMFCI, Temp: 298 ± 1 K
 $\mu = 0.1 M KNO_3$, Water: Dioxane 3:2 (v/v)

[B]	V ₃ – V ₂	\bar{n}	P ^L
6.0	0.004	0.468	7.3102
6.2	0.006	0.0952	7.1144
6.4	0.008	0.1682	6.9208
6.6	0.012	0.2428	6.7278
6.8	0.014	0.3208	6.5346
7.0	0.018	0.4259	6.3447
7.2	0.026	0.6098	6.1628
7.4	0.032	0.7996	5.9822
7.6	0.040	1.0252	5.8058
7.8	0.044	1.2516	5.6292
8.0	0.056	1.4734	5.4586
8.2	0.068	1.7802	5.2984

Table No 11
Metal Zn(II)

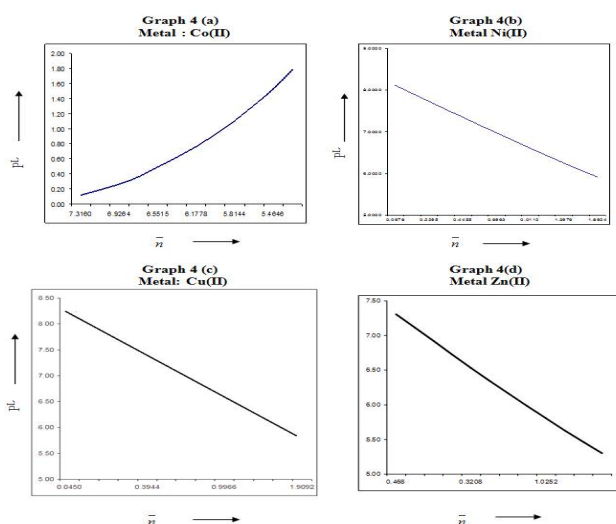
Ligand – HNMFCI
 $\mu^0 = 0.1 \text{ M KNO}_3$
Temp: $298 \pm 1 \text{ K}$
Water: Dioxane 3:2 (v/v)

$\frac{\log \bar{n}}{\bar{n}/(1-\bar{n})}$	P^L	$\frac{\log(2-\bar{n})}{\bar{n}/(\bar{n}-1)}$	P^L
-0.6946	6.9206	0.5214	5.6292
-0.4935	6.7278	0.0456	5.4586
-0.3262	6.5346	-0.5498	5.2984
-0.1304	6.3446	-0.6238	5.0834
0.1942	6.1628	-0.7386	4.9268
0.6016	5.9822	-0.8621	4.7325

Graph No 4

Formation Curve Plot of \bar{n} Vs $[P^L]$

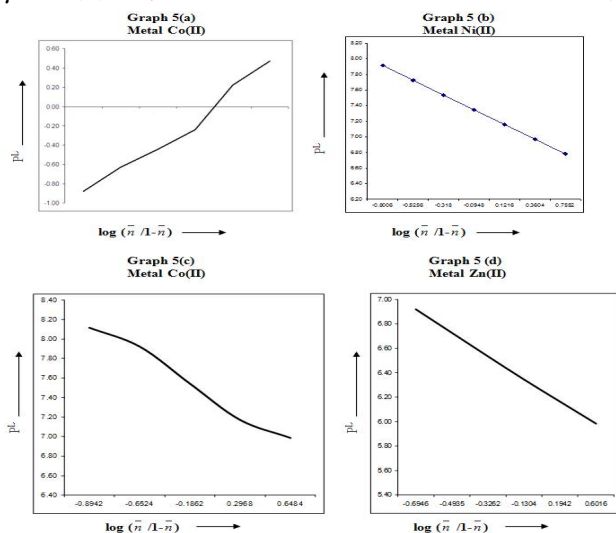
Ligand – HNMFCI
 $\mu^0 = 0.1 \text{ (M) KNO}_3$
Temperature $298 \pm 1 \text{ K}$
Water: Dioxane medium = 3:2 (v/v)



Graph No 5

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

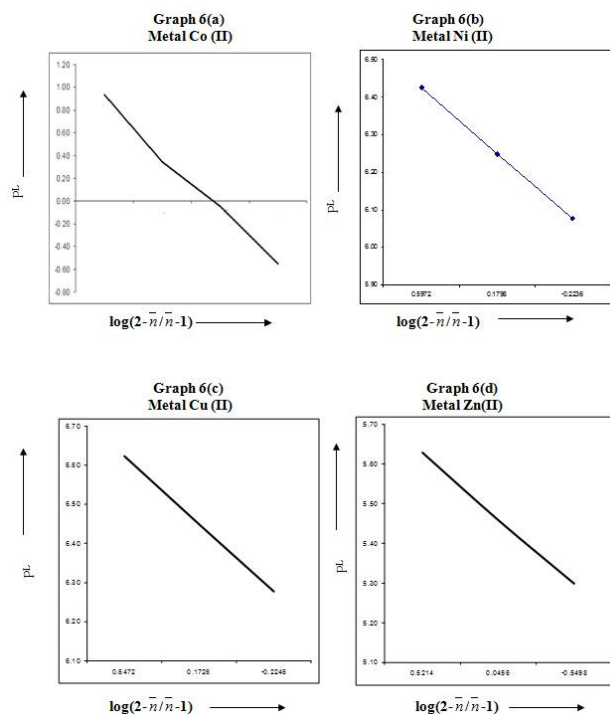
Ligand – HNMFCI
 $\mu^0 = 0.1 \text{ (M) KNO}_3$
Temperature $298 \pm 1 \text{ K}$
Water: Dioxane medium = 3:2 (v/v)



Graph No 6

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

Ligand – HNMFCI
 $\mu^0 = 0.1 \text{ (M) KNO}_3$
Temperature $298 \pm 1 \text{ K}$
Water: Dioxane medium = 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:-

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 HNMFCI at temperatures 298 K.

Metal ions	Ligand HNMFCI	
	$\log K_1$	$\log K_2$
HNMFCI	a	11.16
	b.	-
	c.	11.08
Co(II)	a	7.20
	b.	7.38
	c.	7.30
Ni(II)	a	7.24
	b.	7.26
	c.	6.38
Cu(II)	a	6.24
	b.	6.34
	c.	6.38
Zn(II)	a	6.26
	b.	6.40
	c.	6.38

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

$\text{Cu(II)} > \text{Ni(II)} > \text{Co(II)} > \text{Zn(II)}$

This is natural order given by Irving-William.

Table No 13 Log of stepwise and overall stability constants of complex compounds of various metals with ligand HNMFCI at 298K.

$\mu^0 = 0.10$ (M) KNO_3 Water – dioxane medium (V/V) = 3:2

System	Ligand (HNMFCI)		
	log K1	log K2	log β
HNMFCI	11.10	-	11.10
Cu (II)	7.31	6.43	13.83
Ni (II)	7.14	6.14	13.28
Co (II)	6.49	5.46	11.95
Zn(II)	6.24	5.58	11.82

Observed order of stability constant of complexes with ligand HNMFCI are-Cu(II) > Ni (II) > Co (II) > Zn (II)

DISCUSSION

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 stoichiometric 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 importance¹⁴.

The stability of the chelates is greatly affected by the electron density around the imino nitrogen ($-\text{C}=\text{N}-$)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 with our previous studies and also of other workers¹⁶⁻²⁴

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