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Physico – Chemical Study of Transition Metal Complexes with Schiff's Base derived from Naphthaldehyde and Substituted Aromatic Amines

PREM MOHAN MISHRA[1] and VEENA MISHRA[2]

[1]Department of Chemistry, M.L.S.M. College, Darbhanga, India.
[2]L.N. Mithila University, Darbhanga, Bihar – 846004, India.
*Corresponding author E-mail: mishrapm6@gmail.com

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ABSTRACT

In continuation of the previous work here we are going to report stability constant values of transition metals like Cu(II) , Ni(II), Co(II) and Zn (II) with Schiff;s base ligand. Schiff's base ligands were synthesized by the condensation reaction of β- Naphthaldehyde with 7- Hydroxy naphthalene – 2 – amine .Nitrate salts of divalent cobalt, nickel, copper and zinc were estimated by usual methods. Ligand was analysed for elements by standard method. pH metric titrations were carried out with the help of digital pH meter and stability constant of complexes of these metals with the ligands synthesized were computed by Irving- Rossotti technique modified by Calvin-Bjerrum. The stability constant values of metals for the given ligand 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.

Keywords : Schiff's base, complex compound, Stability constant, Irving – Rossotti titration technique, Naphthaldehyde, Thermodynamic parameters.

INTRODUCTION

These days considerable attention is being paid to the chemistry of complex compounds of Schiff's base containing nitrogen and other donor atoms.[1-4] Schiff's base offer 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[5] and have wide potential applications in many fields such as catalysis[6], electrochemistry[7] and medicines Studies[8] have shown that metal complexes act as antitumour, antiviral, anti cancer[10] and other many anti bacterial agents.

A large number of polydentate Schiff's base compounds have been synthesized and their complexes have been structurally characterized and extensively investigated. But little is known for their stability in aqueous solution in which it is used. Hence, the title project have been under taken. Here in the stability constant of complexes of divalent

transition metals i.e. Co(II), Ni(II), Cu(II) and Zn(II) with Schiff's base ligands have been determined.

Experimental

Nitrate salts of divalent Co, Ni, Cu and Zn all were E. Merck. All other chemicals used were AnalR grade and used without further purification. Elemental analysis of metal salts were done by volumetric and gravimetric methods. 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 synthesized by the condensation of β–Naphthaldehyde with 7-Hydroxy naphthalene – 2 – amine. 3.5g of aldehyde in solution was mixed with nearly 3.0 g amine. The mixture was boiled under reflux in the presence of glacial acetic acid for about 2 hours. The solution was concentrated and cooled to 0^0C. The product obtained was filtered, washed several times and re-crystallized from ethanol. The yield of product was nearly 2.6 g.

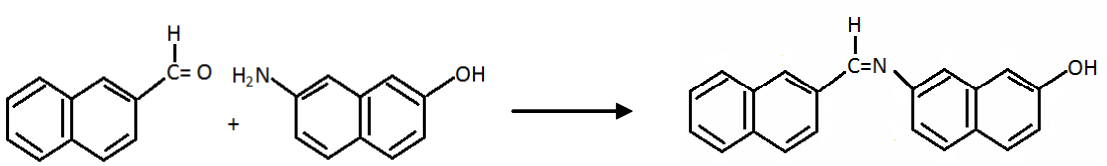


Table 1: Concentrations used in the experiment

Metal / Ions	V^o (mL)	Y	N^O	E^O	T^O_L	T^O_M
Co (II)	100	1	1.0 (M)	1.0×10^{-2} (M)	2.4×10^{-3} (M)	5.0×10^{-4} (M)
Ni(II)	100	1	1.0 (M)	1.0×10^{-2} (M)	2.4×10^{-3} (M)	5.0×10^{-4} (M)
Cu(II)	100	1	1.0 (M)	1.0×10^{-2} (M)	2.4×10^{-3} (M)	5.0×10^{-4} (M)
Zn(II)	100	1	1.0 (M)	1.0×10^{-2} (M)	2.4×10^{-3} (M)	5.0×10^{-4} (M)

Calvin – Bjerrum pH metric titration of acid, acid + ligand and acid + ligand + metal ions solutions were done at constant ionic strength of 0.1 M KNO_3 at 298 K temperature 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, (Figure - 1.) Three titration curves obtained for each metal ions are acid titration curve (a), ligand titration curve (b) and 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 (a), (b) and (c) respectively obtained from the experiment at temperature 298 K given in Figure **- 1.**

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

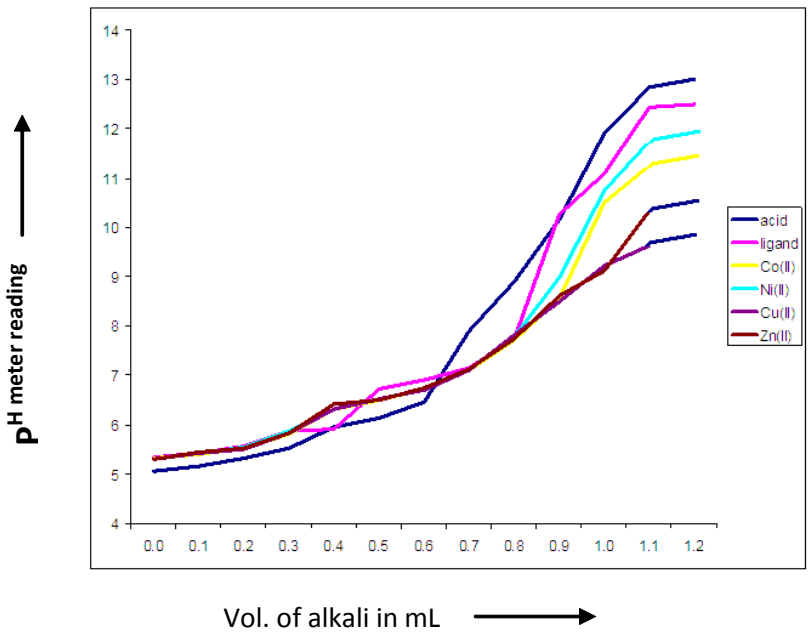
$$\bar{n}_A = 1 + [(V_1 - V_2) / (V^O + V_1)] (N^O + E^O) / T^O_L$$

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

Table 2: Volume of alkali consumed in different titrations

Ligand - HNNCI (L_1) Temp. 298 ±1K
μ° = 0.10 (M) KNO_3 Water : dioxane medium (v/v) = 3:2

Vol. of alkali added in mL	pH – meter reading (B)					
	H^+	H^++ L	H^+ +L + Co(II)	H^+ +L + Ni(II)	H^+ + L + Cu(II)	H^+ + L + Zn(II)
0.0	5.05	5.35	5.32	5.3	5.3	5.3
0.1	5.15	5.43	5.4	5.42	5.42	5.44
0.2	5.33	5.57	5.52	5.55	5.5	5.52
0.3	5.53	5.87	5.8	5.86	5.82	5.82
0.4	5.95	5.91	6.32	6.32	6.32	6.42
0.5	6.13	6.73	6.5	6.52	6.52	6.5
0.6	6.45	6.9	6.72	6.72	6.7	6.74
0.7	7.9	7.15	7.1	7.12	7.1	7.12
0.8	8.9	7.75	7.72	7.79	7.82	7.74
0.9	10.15	10.27	8.55	8.98	8.5	8.62
1.0	11.9	11.1	10.5	10.75	9.22	9.12
1.1	12.85	12.43	11.25	11.75	9.62	10.34
1.2	13.15	12.57	11.70	12.10	10.20	10.56



Temp. 298 ±1 K
μ°= 0.10(M) KNO_3 Water : dioxane = 3:2(v/v)

Fig. 1: Experimental curve with ligand HNNCI

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

Proton ligand stability constant

The ligand titration curve is above the acid titration curve showing the basic nature of ligand and it is well separated from the acid titration curve at pH=6.3 at temp 298 K. The ligand curves run parallel to the acid titration curve indicating the smooth dissociation of the ligand.

The values of $\bar{n}_A$ at various pH reading [B] was calculated from the acid and ligand titration curves and recorded in table 3.

The formation curve obtained from the plot of $\bar{n}_A$ vs [B] extends from 0.43 to 0.88 (Figure **2**) at temp 298 K.

The dissociation of ligand may be represented as

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

The value of proton ligand stability constant was calculated by half integral method and it was further corroborated by linear plot method. (log $\bar{n}_A$ / (1 - $\bar{n}_A$) vs [B] Figure. – 3.

Table 3 : The values of $\bar{n}_A$ at various pH reading [B]

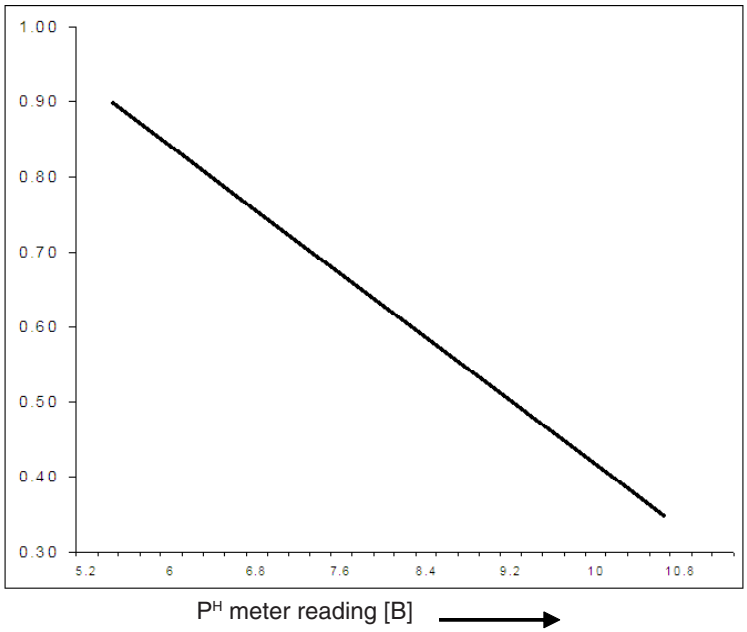
Ligand – HNNCl μ^o= 0.10(M) KNO_3		Temp.: 298 ± 1K Water : dioxane = 3:2(v/v)	
[B]	$V_2 - V_1$		log $\bar{n}_A$ / (1 - $\bar{n}_A$)
5.2	0.005	0.8878	
5.4	0.007	0.8840	
5.6	0.007	0.8832	
5.8	0.008	0.8812	
6.0	0.008	0.8814	
6.2	0.009	0.8742	
6.4	0.009	0.8724	
6.6	0.010	0.8682	
6.8	0.014	0.8610	
7.0	0.014	0.8442	
7.2	0.014	0.8362	
7.4	0.015	0.8360	1.2642
7.6	0.017	0.8282	1.2090
7.8	0.017	0.8196	1.0402
8.0	0.018	0.8121	1.0252
8.2	0.022	0.7986	0.8530
8.4	0.024	0.7964	0.7942
8.6	0.032	0.7882	0.7020
8.8	0.034	0.7834	0.6320
9.0	0.040	0.7602	0.4582
9.2	0.052	0.7120	0.3904
9.4	0.054	0.6794	0.3490
9.6	0.060	0.6555	0.2904
9.8	0.062	0.6274	0.2272
10.0	0.070	0.5996	0.4672
10.2	0.084	0.5674	0.4032
10.4	0.092	0.5322	0.3344
10.6	0.102	0.4883	0.2530
10.8	0.110	0.4782	0.0492
11.0	0.122	0.4672	0.0310
11.2	0.142	0.4394	-0.2050

Table 4: Values of $\bar{n}$ and P^L at various [B] values

Co (II) + HNNCl μ^o = 0.10 (M) KNO_3		Temp: 298 ± 1 K Water: Dioxane = 3:2(v/v)	
[B]	**$V_3 - V_2$**	$\bar{n}$	**P^L**
5.0	0.004	0.2222	8.1650
5.2	0.006	0.3244	7.9762
5.4	0.010	0.4265	7.7862
5.6	0.018	0.5914	7.6014
5.8	0.032	0.5764	7.4124
6.0	0.042	0.7870	7.2412
6.2	0.052	1.8662	7.0634
6.4	0.060	1.2842	6.8858
6.6	0.072	1.58404	6.7092
6.8	0.074	1.7964	6.5364
7.0	0.090	1.8682	6.3742

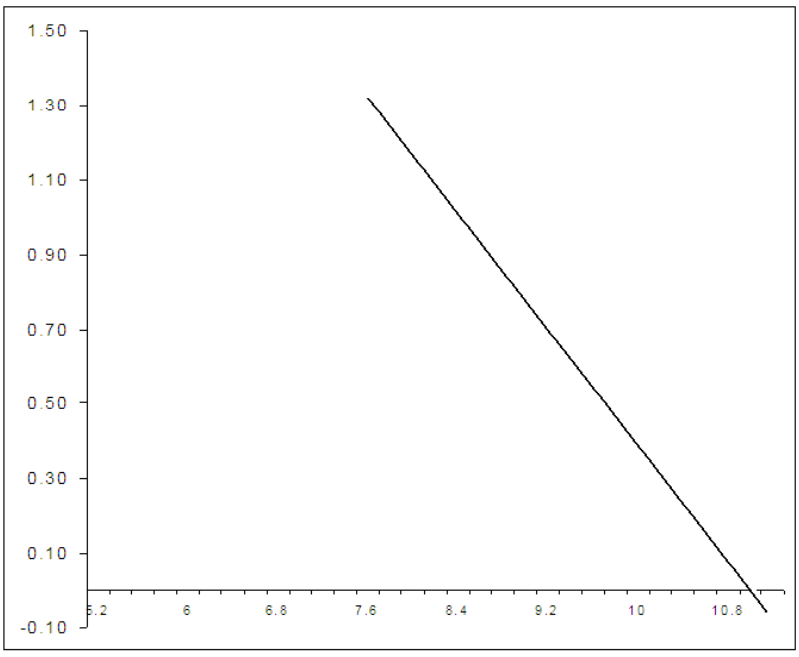
Table 5 : Values of P^L at various values of log $\bar{n}$/(1-$\bar{n}$) and log (2 - $\bar{n}$ /($\bar{n}$ -1)

Co (II) + HNNCl μ^o = 0.10 (M) KNO_3		Temp : 298 1 K Water: Dioxane = 3:2(v/v)	
log $\bar{n}$/(1- $\bar{n}$)	**P^L**	**log (2 - $\bar{n}$ /($\bar{n}$ -1)**	**P^L**
-0.9560	8.9754	0.0262	7.7092
-0.4134	8.7850	-0.3622	7.5364
0.4212	8.4132		
0.7904	8.2402		



Temp. 298 ±1 K
μ^{o} = 0.10 (M) KNO_3 Water: Dioxane = 3:2(v/v)

Fig. 2: Formation curve of ligand – HNNCl Plot of $\bar{n}_A$ Vs [B]



Ligand ;HNNCl Temp : 298 ± 1K
μ^{o}= 0.10 (M) KNO_3 Water: Dioxane = 3:2(v/v) [B]

Fig. 3: Linear plot of log(log $\bar{n}_A$ / (1 -) Vs [B]

The complex titration curve of the system crossed the ligand mixture curve at pH 5.35 for Co(II), pH 4.88 for Ni(II), at pH = 5.90 for Cu(II) and pH 5.68 for Zn(II) – ligand system indicating the start of complexation.

Metal titration curve run parallel to the ligand titration curve indicating the liberation of extra proton due to hydrolysis of metal ions.

Table 6: Values of $\bar{n}$ and P^L at various [B] values

Ni (II) + HNNCl μ° = 0.10 (M) KNO_3		Temp : 298± 1K Water: Dioxane = 3:2(v/v)	
[B]	**$V_3 - V_2$**	$\bar{n}$	**P^L**
5.0	0.004	0.0406	8.1590
5.2	0.006	0.1012	7.9644
5.4	0.008	0.2454	7.7764
5.6	0.014	0.4082	7.5930
5.8	0.022	0.6150	7.4124
6.0	0.034	0.8036	7.2322
6.2	0.042	0.9932	7.0520
6.4	0.052	1.2306	6.8786
6.6	0.062	1.4634	6.7068
6.8	0.086	1.6972	6.5368
7.0	0.094	1.9684	6.3734

Table 8 : Values of $\bar{n}$ and P^L at various [B] values

Cu (II) + HNNCl μ°= 0.10 (M) KNO_3		Temperature 298 ± K Water: Dioxane = 3:2(v/v)	
[B]	**$V_3 - V_2$**	$\bar{n}$	**P^L**
6.2	0.006	0.0814	8.5624
6.4	0.010	0.1640	8.3702
6.6	0.012	0.2672	8.1794
6.8	0.014	0.3722	7.9890
7.0	0.020	0.4796	7.7994
7.2	0.024	0.6150	7.6124
7.4	0.030	0.7632	7.4270
7.6	0.042	0.9412	7.2462
7.8	0.052	1.1434	7.0684
8.0	0.060	1.3270	6.8912
8.2	0.064	1.5364	6.7152
8.4	0.080	1.8240	6.5526

In, Co (II) System

Precipitation was observed at pH 8.9. Hence in the calculation of $\bar{n}$ only the lower pH region of titration curve were used.

In Ni(II) System

The curve increased regularly up to pH 7.56 indicating constant rate of release of proton

Table 7: Values of P^L at various values of log $\bar{n}$ /(1- $\bar{n}$) and log (2 - $\bar{n}$ /($\bar{n}$ -1)

Ni (II) + HNNCl μ° = 0.10 (M) KNO_3		Temp : 298 ± 1K Water: Dioxane = 3:2(v/v)	
log $\bar{n}$ /(1- $\bar{n}$)	**P_L**	**log (2- $\bar{n}$) / ($\bar{n}$ - 1)**	**P^L**
-0.9454	8.9640	0.6226	7.8782
-0.4882	8.7770	0.5626	7.7064
-0.1606	8.5924	0.4622	7.5362
0.2040	8.4124	0.3216	7.4832
0.6114	8.2322	0.2182	7.3218

.No turbidity appears, which indicates that hydrolysis does not take place.

In Cu(II) System

Similarly in Cu(II) system the curve increased regularly up to pH 9.356 indicating constant rate of release of proton .No turbidity appears, indicating that hydrolysis does not take place

Table 9 : Values of P^L at various values of log $\bar{n}$/(1- $\bar{n}$) and log (2 - $\bar{n}$ /($\bar{n}$ -1)

Cu (II) + HNNCl μ° = 0.10 (M) KNO_3		Temperature 298 ± K Water: Dioxane = 3:2(v/v)	
log $\bar{n}$ /(1- $\bar{n}$)	**P^L**	**log (2- $\bar{n}$) / ($\bar{n}$ - 1)**	**P^L**
-0.7056	8.3704	0.7752	7.0684
-0.4360	8.1796	0.3114	6.8902
-0.2262	7.9892	-0.0642	6.7152
-0.0353	7.7994	-0.6710	6.5364
0.2044	7.6130	-0.7128	6.3281
0.5090	7.4284	-0.8214	5.9216

In, Zn(II) system

In case of Zn(II) system complex titration curve diverges at higher pH which indicates incomplete dissociation of ligand. Therefore for the calculation of $\bar{n}$ only symmetrical region of the curve was used.

The value of $\bar{n}$ calculated for these metals are

Co(II)	-	0.22 to 1.86
Ni(II)	-	0.40 to 1.96
Cu(II)	-	0.081 to 1.82
Zn(II)	-	0.12 to 1.72

As $\bar{n}$ value did not go beyond 2 for any of the metal indicating the formation of ML and ML_2 type of complexes.

From the formation curve of $\bar{n}$ vs P^L (Figure no.- 4a,4b,4c and 4d) the values of $\log K_1$ and $\log K_2$ were calculated in each case. It was further corroborated

Table 10 : Values of $\bar{n}$ and P^L at various [B] values

Zn (II) + HNNCl Temp: 298 ± 1K
μ^o = 0.10 (M) KNO_3 Water: Dioxane = 3:2(v/v)

B	$V_3 - V_2$	$\bar{n}$	P^L
6.0	0.006	0.1226	7.3646
6.2	0.018	0.1852	7.1726
6.4	0.010	0.2914	6.9824
6.6	0.012	0.3750	6.7882
6.8	0.020	0.5092	6.6032
7.0	0.024	0.6366	6.4162
7.2	0.030	0.8344	6.2350
7.4	0.042	1.0352	6.0564
7.6	0.052	1.2612	5.8832
7.8	0.076	1.6668	5.7292
8.0	0.084	1.7215	5.5264

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 HNNCl

System Metal ions	Methods	Ligand HNNCl $\log K_1$	$\log K_2$
HNNCl(L_1)	A	10.96	
	b	-	
	c	10.96	
Co (II)	A	7.56	6.64
	b	7.58	6.66
	c	7.62	6.68
Ni (II)	A	7.52	6.66
	b	7.44	6.62
	c	7.46	6.68
Cu (II)	A	6.76	5.76
	b	6.64	5.04
	c	6.84	5.78
Zn (II)	A	6.62	5.76
	b	6.54	5.72
	c	6.68	5.86

Table 11: Values of P^L at various values of $\log \bar{n}/(1-\bar{n})$ and $\log (2-\bar{n})/(\bar{n}-1)$

Zn (II) + HNNCl Temp: 298 ±1K
μ^o = 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
-0.6424	8.1722	0.4526	6.8822
-0.3860	7.9816	-0.2626	6.7296
-0.2212	7.6896	-0.2012	6.5812
0.2430	7.3152	-0.1628	6.3158
0.7024	7.0125	-0.0831	6.2817

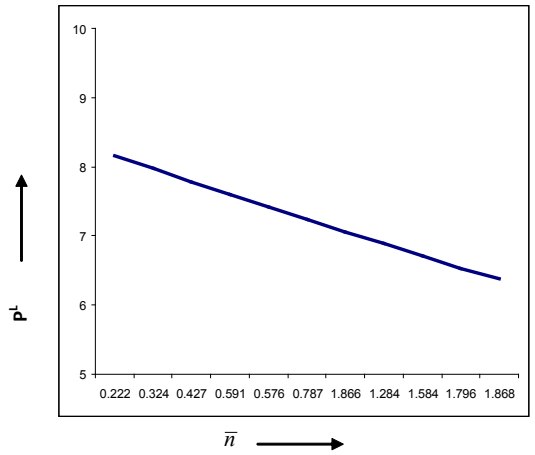
Table 13 : Stepwise and over all stability constant of complex compounds of various metals with ligand HNNCl at temperature 298K

Water – Dioxane medium (v/v) = 3:2
μ^o= 0.10(M) KNO_3

System	Ligand- MNNCl (L_1) $\log K_1$	$\log K_2$	log
HNNCl (L_1)	10.98	-	10.98
Co (II)	6.76	5.70	12.46
Ni (II)	7.48	6.68	14.10
Cu (II)	7.58	6.66	14.20
Zn (II)	6.60	5.78	12.34

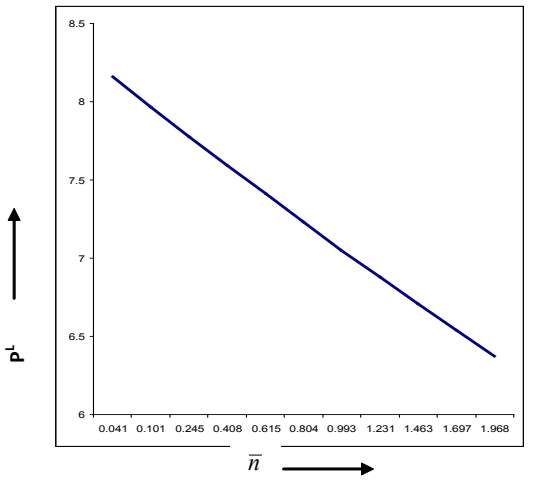
by mid point calculation method and linear plot of log $\frac{\bar{n}}{1-\bar{n}}$ vs P^L (Figure no.- 5a, 5b, 5c and 5d) and also by plot of log $\frac{2-\bar{n}}{\bar{n}-1}$ vs P^L (Graph no.-6a, 6b, 6c and 6d) at temperature 298 K.

The values of protonation constant and stepwise stability constant obtained by different



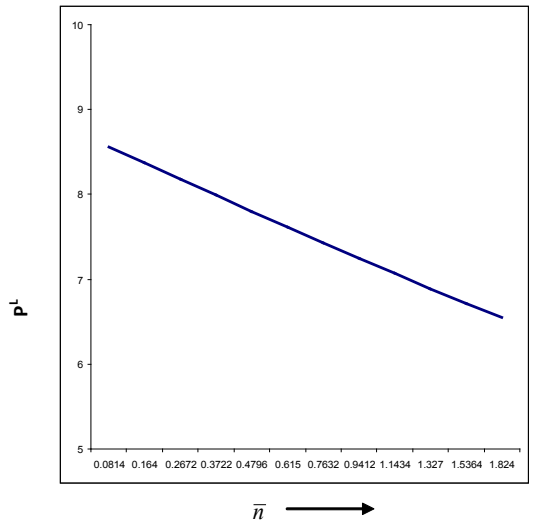
Ligand: HNNCl Temp. 298 ±1 K
μ° = 0.10 (M) KNO_3 Water: Dioxane = 3:2(v/v)

Fig. 4a: Formation curve of Co(II) Plot of $\bar{n}$ Vs P^L



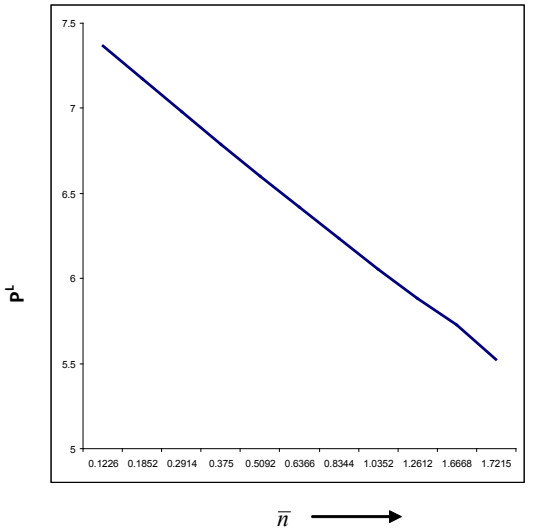
Ligand: HNNCl Temp. 298 ±1 K
μ° = 0.10 (M) KNO_3 Water: Dioxane = 3:2(v/v)

Fig.4b: Formation curve of Ni(II) Plot of $\bar{n}$ Vs P^L



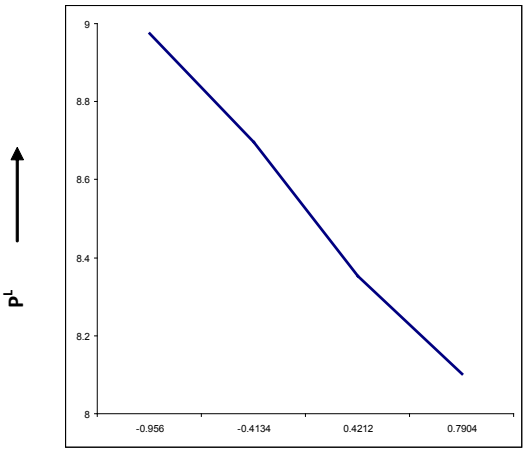
Ligand: HNNCl (L_1) Temp. 298 ±1 K
μ° = 0.10 (M) KNO_3 Water: Dioxane = 3:2(v/v)

Fig. 4c: Formation curve of Cu(II) Plot of $\bar{n}$ Vs P^L



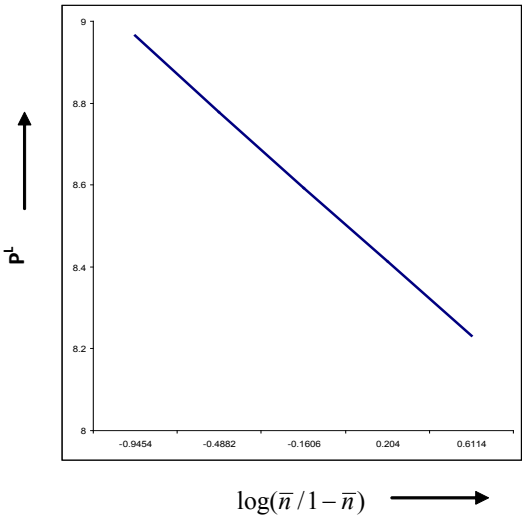
Ligand: HNNCl (L_1) Temp. 298 ±1 K
μ° = 0.10 (M) KNO_3 Water: Dioxane = 3:2(v/v)

Fig.4d: Formation curve of Zn(II) Plot of $\bar{n}$ Vs P^L



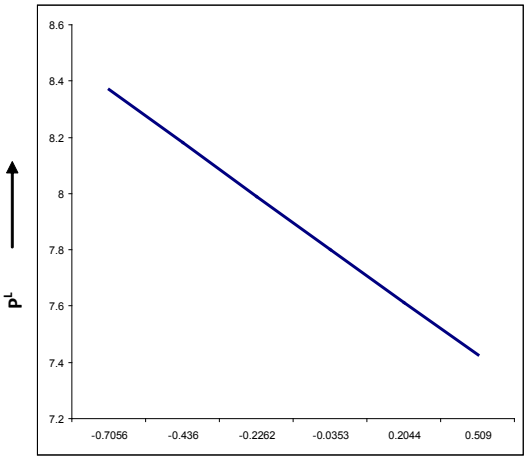
Ligand: HNNCI Temp. 298 ±1 K
μ° = 0.10 (M) KNO_3 Water: Dioxane = 3:2(v/v)

Fig.5a: Formation curve of Co(II) Plot of log ($\bar{n}$/1- $\bar{n}$ Vs P^L



Ligand: HNNCI Temp. 298 ±1 K
μ° = 0.10 (M) KNO_3 Water: Dioxane = 3:2(v/v)

Fig. 5b: Formation curve of Ni(II) Plot of log ($\bar{n}$/1-$\bar{n}$) Vs P^L



Ligand: HNNCI (L_1) Temp. 298 ±1 K
μ° = 0.10 (M) KNO_3 Water: Dioxane = 3:2(v/v)

Fig.5c: Formation curve of Cu(II)Plot of log ($\bar{n}$/1- $\bar{n}$) Vs P^L

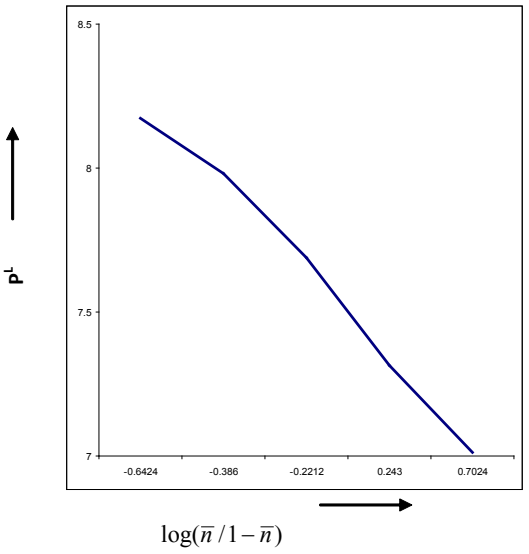
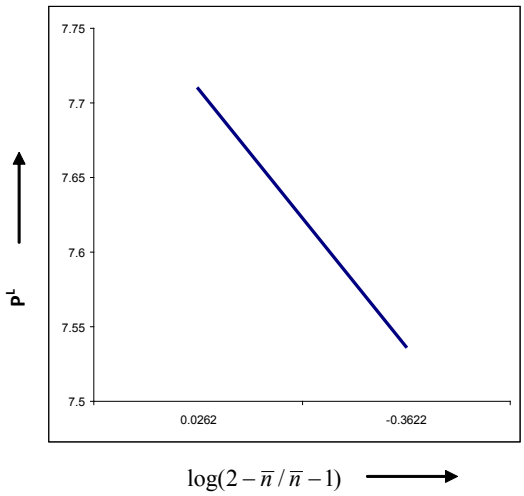


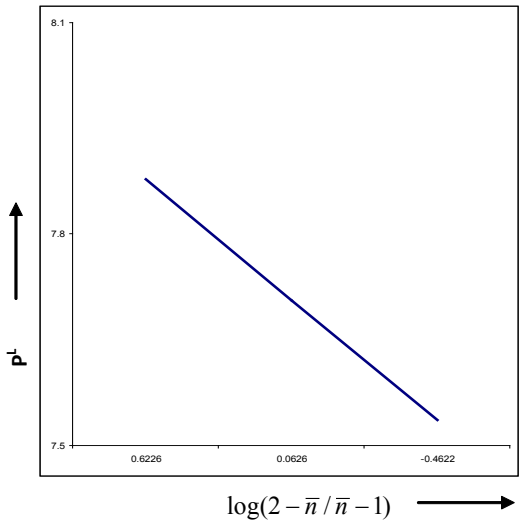
Fig.5d: Formation curve of Zn(II) Plot of log ($\bar{n}$/1-$\bar{n}$) Vs P^L

Ligand: HNNCI (L_1) Temp. 298 ±1 K
μ° = 0.10 (M) KNO_3 Water: Dioxane = 3:2(v/v)



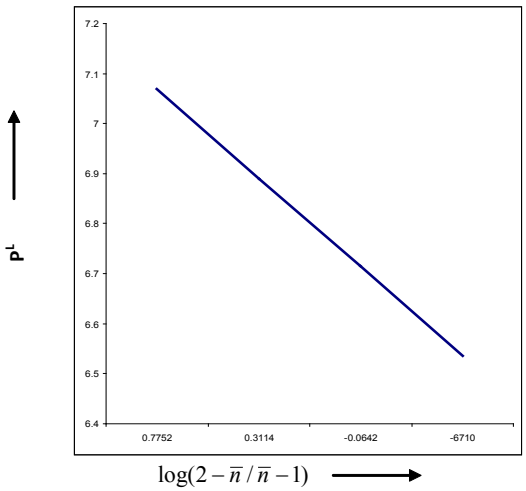
Ligand: HNNCI Temp. 298 ±1 K
μ° = 0.10 (M) KNO_3 Water: Dioxane = 3:2(v/v)

Fig. 6a: Formation curve of Co(II) Plot of log (2-$\bar{n}/\bar{n}-1$) Vs P^L



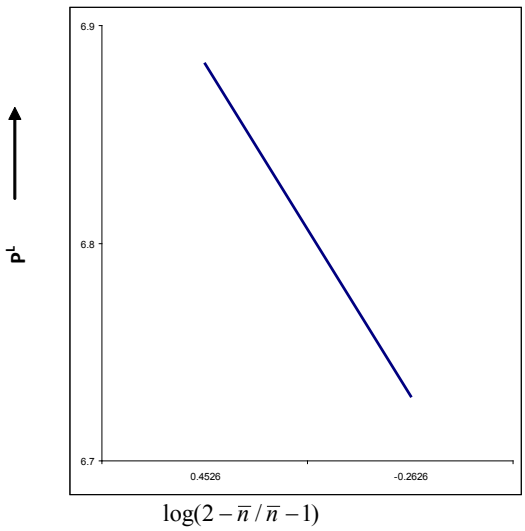
Ligand: HNNCI Temp. 298 ±1 K
μ° = 0.10 (M) KNO_3 Water: Dioxane = 3:2(v/v)

Fig.6b: Formation curve of Ni(II) Plot of log (2-$\bar{n}/\bar{n}-1$) Vs P^L



Ligand: HNNCI Temp. 298 ±1 K
μ° = 0.10 (M) KNO_3 Water: Dioxane = 3:2(v/v)

Fig.6c: Formation curve of Cu(II)Plot of log (2-$\bar{n}/\bar{n}-1$) Vs P^L



Ligand: HNNCI Temp. 298 ±1 K
μ° = 0.10 (M) KNO_3 Water: Dioxane = 3:2(v/v)

Fig.6d: Formation curve of Zn(II)Plot of log (2-$\bar{n}/\bar{n}-1$) Vs P^L

computational methods at temperatures 298 K are summarized in table no. 12

The different methods used are :-

a) Half – integral method
b) Mid – point calculation method
c) Straight line plot method.

The order of stability constant of various metals for the given ligand HNNCI are - Cu(II) > Ni(II) > Co (II) > Zn(II)

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 metals show the sequence

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

This is natural order given by Irving – William. 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.

The stability of the chelates is greatly affected by the electron density around the imino nitrogen (- C = N -). 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[12-15] and other workers[16-17].

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