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ORIGINAL ARTICLE

Physico – chemical study of Transition metal complexes with Schiff's base ligand (1-phenyl – 3 - (2' – mercaptophenyl) – N – (3" – hydroxy – 5" – ethyl – 1", 2", 4" – triazolyl) propene – 1 – imine (PMHETPI) at temperature 298 K.

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ABSTRACT

In the continuation of the previous work (P^H – metric investigation on the complexation behaviour of Schiff's based ligand) here we are going to report stability constant value of transition metal like Co(II), Ni(II), Cu(II), Zn(II) and Cd(II) with Schiff's base ligands were synthesized by the condensation reaction of Chalcone with 3 – hydroxy – 5 – ethyl- 4 – amino – 1, 2, 4 triazole. Nitrate salt of divalent cobalt, Nickel, Copper, Zinc and Cadmium were estimated by usual methods. Ligand was analysed for elements by standard method P^H – metric titration were carried out with the help of digital P^H- meter and stability constant of complexes of these metal with the ligands synthesized were computed by Irving – Rossotti technique modified by Calvin – Bjerrum. The stability constant values of metal for the given ligand were found to be in the order Cu(II) > Ni (II) > Co(II) > Zn (II) > Cd (II). This result is in agreement with the natural order proposed by Irving – William.

KEY-WORDS:- *Schiff's base, complex compound, chalcone, Irving – Rossotti, stability constant, Acetophenone, 2-mercapto Benzaldehyde.*

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INTRODUCTION

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 days considerable attention being paid to the chemistry of complex compounds of Schiff's base containing nitrogen and other donor atom. These complexes are biologically active and have wide potential application in many fields such as catalysis, Electrochemistry and medicine studies have shown the metal complexes act as antitumour, antiviral and other many antibacterial agents [4-7].

A large number of Schiff's base compounds have been synthesized and their complexes have been structurally characterized and extensively investigated. But little in known for their stability in aqueous solution. 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), Zn(II) and Cd (II) with 1 – phenyl – 3 – (2' – mercaptophenyl) – N – (3" – hydroxy – 5" - ethyl – 1", 2", 4" – triazolyl) propene – 1 – imine have been determined at 298 K.

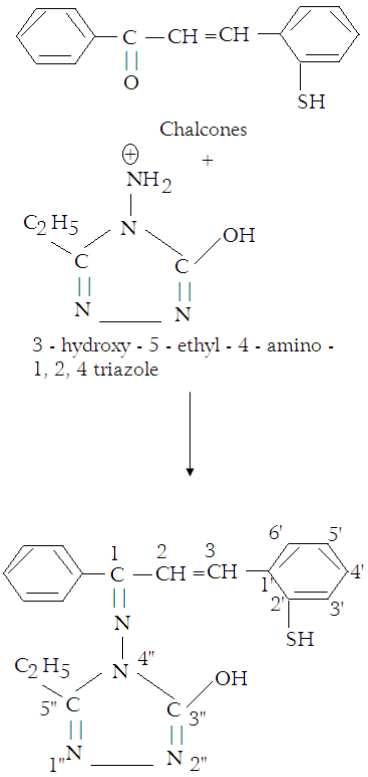
EXPERIMENTAL

All the chemicals used were AnalR grade and of highest purity available. Elemental analysis of metal slats were done by volumetric and gravimetric. Double distilled and deionised water was used throughout the experiment. All titrations were done in aqueous – dioxane medium in the ratio 50:50 (v/v).

Schiff's base ligands were synthesized [1-3] by the condensation of chaleone with 3-hydroxyl – 5 – ethyl – 4 – amino – 1, 2, 4 triazole and mole of chaleone in 30 ml of ethanol was taken in round bottom flask and 3 – hydroxyl – 5 – ethyl – 4 – amino – 1, 2, 4 triazole was added. The reaction mixture was refluxes for 6 – 7 hours. solvent was evaporated on water bath and solid separated was collected. The product was

washed with absolute alcohol and crystallized from rectified spirit to obtain yellow coloured crystals yield of the product was 1.80 g.

Reaction involved in the preparation of ligand (PMHETPI)



(PMHETPI)

Table No. – 1: Concentrations of metal ions, ligand, acid and salt.

Metal ions	V^0 in mL	Y	N^0	E^0	T^0_L	T^0_M
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)
Cd (II)	100	1	1.0(M)	1.0×10^{-2}(M)	2.5×10^{-3} (M)	5.0×10^{-4}(M)

Calvin – Bjurrum P^H metric titration of acid, acid + ligand and acid + ligand + metal ion solutions were done at constant ionic strength 0.10 KNO_3 at 298 K temperature. The same process of titration were repeated for all the five (Co, Ni, Cu, Zn and Cd) metal ions. The change in colour and appearance of turbidity at particular P^H value were recorded simultaneously.

The change in pH of the solutions[4-7]with each addition of alkali was recorded in Table No. – 2.

RESULT AND DISCUSSION

A graph was plotted between pH meter reading [B] and volume of alkali added in each case (Graph – 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 volume (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 $\overline{n}_A$, $\overline{n}$ and P^L are calculate using standard expressions [8-9].

Table – 2: Volume of alkali consumed in different titrations of acid, acid + ligand and acid + ligand + metal ion solution with (PMHETPI)

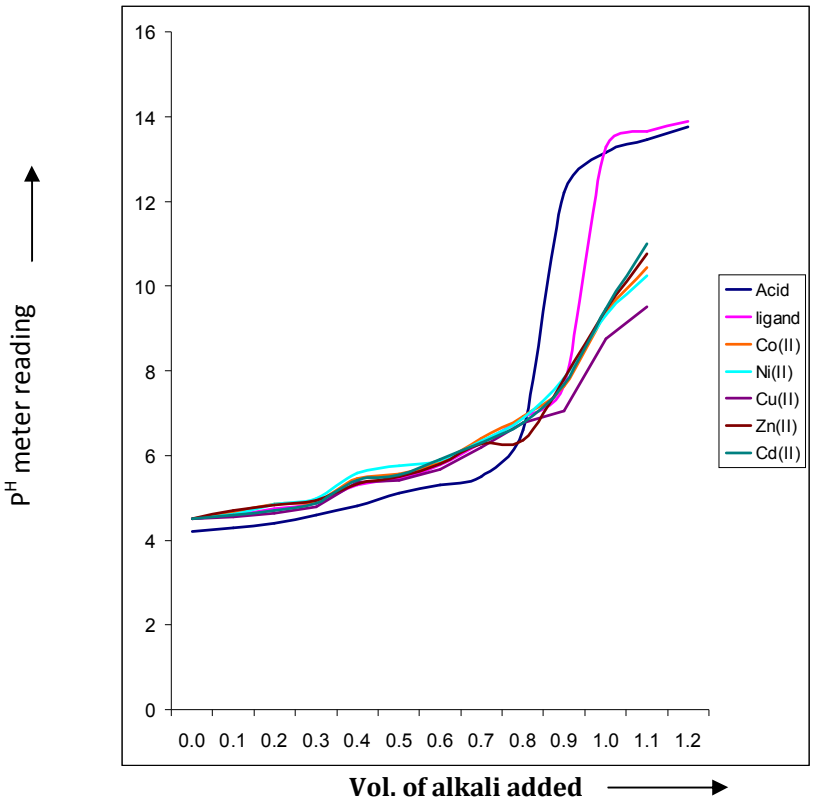
μ^0 =0.1(M) KNO_3, Water–Dioxane medium (V/V) = 50:50

Temperature 298 ± 1K

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)	$H^+ + L$ + Cd(II)
0.0	4.2	4.5	4.5	4.5	4.5	4.5	4.5
0.1	4.3	4.6	4.66	4.62	4.55	4.7	4.59
0.2	4.4	4.75	4.85	4.85	4.64	4.82	4.7
0.3	4.6	4.88	4.92	4.99	4.78	4.94	4.88
0.4	4.8	5.3	5.45	5.58	5.37	5.32	5.42
0.5	5.1	5.46	5.57	5.76	5.42	5.49	5.55
0.6	5.3	5.78	5.85	5.88	5.68	5.79	5.9
0.7	5.5	6.3	6.41	6.35	6.18	6.28	6.3
0.8	6.6	6.9	6.93	6.88	6.78	6.36	6.78
0.9	12.2	7.66	7.63	7.82	7.05	7.8	7.68
1.0	13.15	13.29	9.35	9.32	8.75	9.44	9.46
1.1	13.46	13.66	10.44	10.25	9.5	10.75	11
1.2	13.75	13.88					

Graph - 1 Experimental Curve with ligand PMHETPI

Temp: 298 ± 1K



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 = 5.08 at temperature 298 K. The ligand curves run parallel to the acid titration curve indicating the smooth dissociation of the ligand.

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

Table 3: The formation curve obtained from the plot of $\bar{n}_A$ vs [B] lies between 0 and 1.0 (Graph – 2) at 298 K.

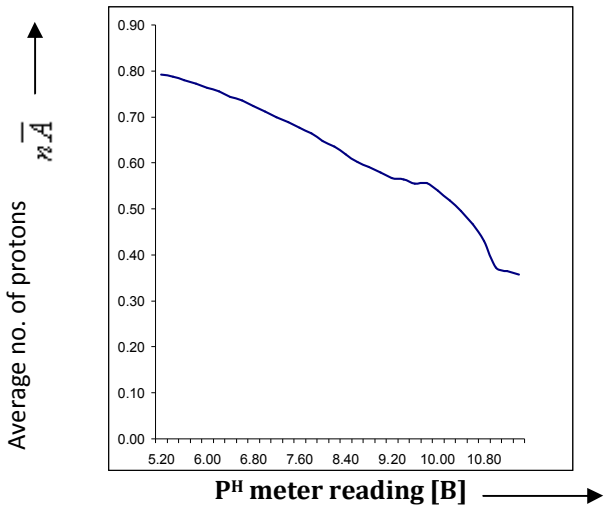
Ligand: PMHETPI Temperature : 298 ± 1 K

B	$V_2 - V_1$	$\bar{n}_A$	$\log \bar{n}_A / (1 - \bar{n}_A$
5.2	0.030	0.7922	0.8157
5.4	0.029	0.7872	0.7986
5.6	0.034	0.7794	0.7645
5.8	0.032	0.7722	0.7332
6.0	0.034	0.7634	0.7224
6.2	0.036	0.7562	0.6725
6.4	0.040	0.7436	0.6730
6.6	0.042	0.7354	0.6056
6.8	0.044	0.7242	0.5680
7.0	0.048	0.7122	0.5340
7.2	0.050	0.6994	0.5026
7.4	0.054	0.688	0.4690
7.6	0.056	0.6762	0.4342
7.8	0.058	0.664	0.4090
8.0	0.062	0.6476	0.3722
8.2	0.066	0.636	0.3442
8.4	0.700	0.6194	0.3092
8.6	0.078	0.6026	0.2736
8.8	0.080	0.5914	0.2506
9.0	0.082	0.579	0.2264
9.2	0.088	0.5674	0.2030
9.4	0.080	0.5642	0.1950
9.6	0.082	0.5554	0.1790
9.8	0.086	0.5562	0.1790
10.0	0.088	0.5394	0.1492
10.2	0.094	0.519	0.1130
10.4	0.014	0.4962	0.0682
10.6	0.108	0.467	0.0178
10.8	0.116	0.4298	0.0380
11.0	0.128	0.3728	-0.0540
11.2	0.130	0.3642	-0.0736
11.4	0.134	0.3574	-0.0838

Graph 2 : Formation Curve of ligand PMHETPI

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

Temp: 298 ± 1K



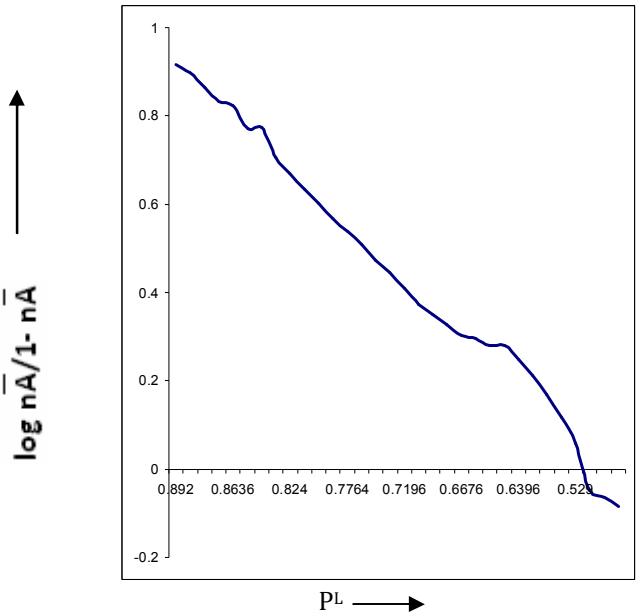
Dissociation of ligand may be given as

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

The value of proton ligand stability constant was calculated by half integral method and it was further corroborated by linear plot method [10-11](log $\bar{n}_A$ /1 - $\bar{n}_A$) vs [B], Graph – 3.

Graph – 3: Linear Plot of log($\bar{n}_A$/1 - $\bar{n}_A$) Vs P^L

Ligand : PMHETPI Temp: 298 ± 1K



The complex titration curve of the system separated from ligand mixture curve at pH 6.30 for Co(II), pH 5.20 for Ni(II), 6.30 for Cu(II), 5.86 for Zn(II) and 5.80 for Cd(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 ion.

In Co(II) system

The curve diverge at pH 9.70 hence the calculation of $\bar{n}$ only lower pH region of titration curve were used.

In Ni (II) system

The curve increased regularly up to pH 7.78 indicating quick but incomplete dissociation of ligand. No turbidity appears hence hydrolysis does not take place.

In Cu(II) system:

In Cu(II) system metal titration curve run parallel to the ligand titration curve indication liberate of extra proton due to hydrolysis.

In Zn(II) system

In case of Zn(II) the complex titration curve diverges ligand titration curve at pH = 7.86. No turbidity appears, hence hydrolysis does not take place.

In Cd(II) System:

In case of Cd(II) system complex titration curve run parallel to ligand mixture curve at pH = 8.68 and then diverged. No turbidity appeared shows that hydrolysis does not take place.

The values of $\bar{n}$ calculated for these metals falls between

Co(II)	-	0.21 to 1.68
Ni(II)	-	0.04 to 1.97
Cu(II)	-	0.04 to 1.90
Zn(II)	-	0.056 to 1.68
Cd(II)	-	0.08 to 1.68

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

From the formation curve of $\bar{n}$ vs P^{L12} (Graph 4a, 4b, 4c, 4d and 4e) the value of log K_1 and log K_2 were calculated in each case. It was further corroborated[13-14] by mid point calculation method and linear plot of log $\bar{n}$ / 1- $\bar{n}$ vs P^L (Graph – 5a, 5b, 5c, 5d and 5e) and also by plot of log 2- $\bar{n}$ / $\bar{n}$ -1 vs P^L Graph No. – 6a, 6b, 6c, 6d and 6e) at temperature 298 K.

Table 4: Values of $\bar{n}$ vs P^L at volume [B]

Co(II) PMHETPI Temp: 298 ± 1 K

B	$V_3 - V_2$	$\bar{n}$	P^L
6.0	0.004	0.2171	8.3162
6.2	0.006	0.2894	8.1224
6.4	0.012	0.3642	7.9262
6.6	0.015	0.4642	7.7992
6.8	0.016	0.5936	7.5514
7.0	0.022	0.6258	7.3642
7.2	0.026	0.6620	7.1776
7.4	0.032	0.8286	6.9952
7.6	0.038	1.2040	6.8143
7.8	0.046	1.2124	6.6385
8.0	0.052	1.4280	6.4644
8.2	0.064	1.6804	6.2732

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

Co (II) + PMHETPI Temp: 298 ± 1 K

$\log \bar{n}/(1-\bar{n})$	P^L	$\log (2 - \bar{n})/(\bar{n}-1)$	P^L
-0.9766	8.3162	0.8352	6.8142
-0.7302	8.1224	0.4432	6.6384
-0.5404	7.9292	-0.0586	6.4645
-0.3412	7.7386	-0.6504	6.2732
0.3232	7.3642		
0.5716	7.1766		

Table 6: Values of $\bar{n}$ vs P^L at volume [B]

Ni (II) + PMHETPI Temp : 298 ± 1 K

[B]	$V_3 - V_2$	$\bar{n}$	P^L
5.2	0.004	0.0776	9.1116
5.4	0.006	0.2362	8.9182
5.6	0.018	0.3394	8.7260
5.8	0.014	0.4242	8.5352
6.0	0.016	0.5454	8.3462
6.2	0.022	0.6692	8.1582
6.4	0.026	0.7961	7.9714
6.6	0.032	0.9506	7.7872
6.8	0.038	1.0214	7.6040
7.0	0.044	1.2014	7.4252
7.2	0.052	1.3974	7.2484
7.4	0.060	1.6256	7.0772
7.6	0.068	1.8922	6.9130

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

Ni(II) + PMHETPI Temp: 298 ± 1 K

$\log \bar{n}/(1-\bar{n})$	P^L	$\log (2 - \bar{n})/(\bar{n}-1)$	P^L
-0.7006	8.9176	0.5972	6.4254
-0.6256	8.7260	0.1798	6.2488
-0.4180	8.5352	-0.2236	6.0770
-0.0848	8.3464		
0.2216	8.1585		
0.4604	7.9712		
0.8552	7.7870		

Table 8: Values of $\bar{n}$ vs P^L at volume [B]

Cu(II) + **PMHETPI** system Temp: 298 ± 1 K

[B]	$V_3 - V_2$	$\bar{n}$	P^L
5.2	0.002	0.0452	9.3098
5.4	0.004	0.1130	9.1156
5.6	0.008	0.1822	8.9222
5.8	0.012	0.2750	8.7306
6.0	0.016	0.3942	8.5418
6.2	0.018	0.5164	8.3532
6.4	0.024	0.6646	8.1682
6.6	0.034	0.8162	7.9834
6.8	0.040	0.9964	7.8028
7.0	0.044	1.1836	7.6236
7.2	0.050	1.4020	7.2774
7.4	0.064	1.6262	7.2774
7.6	0.076	1.9090	7.1156

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

Cu(II) + **PMHETPI** Temp: 298 ± 1 K

$\log \bar{n}/(1-\bar{n})$	P^L	$\log (2 - \bar{n})/(\bar{n}-1)$	P^L
-0.9942	9.1154	0.7470	7.6232
-0.7524	8.9220	0.2724	7.4492
-0.2862	8.5412	-0.3244	7.2770
0.3968	8.1680		
0.5484	7.9832		

Table 10: Values of $\bar{n}$ vs P^L at volume [B]

Zn(II) + **PMHETPI** Temp : 298 ± 1 K

[B]	$V_3 - V_2$	$\bar{n}$	P^L
6.0	0.002	0.0566	8.3100
6.2	0.004	0.0850	8.1142
6.4	0.006	0.2680	7.9206
6.6	0.010	0.3426	7.7276
6.8	0.012	0.4206	7.5344
7.0	0.016	0.3258	7.3448
7.2	0.024	0.5096	7.1626
7.4	0.030	0.8994	6.9820
7.6	0.038	1.0350	6.8056
7.8	0.042	1.3514	6.6290
8.0	0.054	1.5732	6.4584
8.2	0.066	1.6804	6.2982

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

Zn(II) + **PMHETPI** Temperature 298 ± 1 K

Log $\bar{n}/(1-\bar{n})$	P^L	$\log (2 - \bar{n})/(\bar{n}-1)$	P^L
-0.7945	7.9204	0.6212	6.6290
-0.5934	7.7274	0.0354	6.4584
-0.4260	7.5344	-0.4496	6.2980
-0.2302	7.3444		
0.2940	7.1626		
0.5012	6.9824		

Table 12 : Values of $\bar{n}$ vs P^L at volume [B]

Cd(II) + **PMHETPI** Temp: 298 ± 1 K

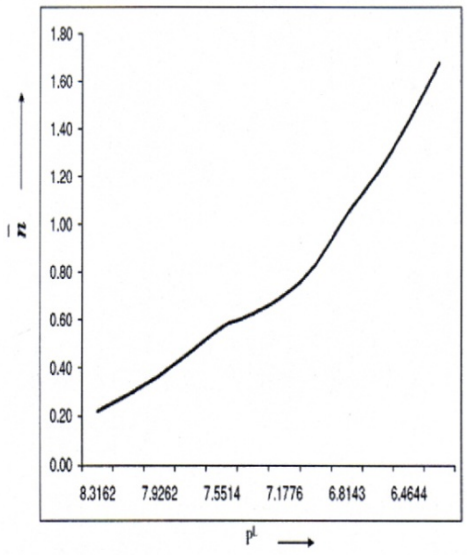
B	$V_3 - V_2$	$\bar{n}$	P^L
6.2	0.006	0.0813	8.1122
6.4	0.009	0.2444	7.9184
6.6	0.010	0.3424	7.7276
6.8	0.014	0.4456	7.5372
7.0	0.018	0.4505	7.3470
7.2	0.022	0.5847	7.1602
7.4	0.026	0.6488	6.9764
7.6	0.032	0.8438	6.7970
7.8	0.044	1.3054	6.6262
8.0	0.050	1.5695	6.4526
8.2	0.064	1.6804	6.2974

Table 13 : Values of P^L at various value of log $\bar{n}$ /1- $\bar{n}$ and log (2- $\bar{n}$ / $\bar{n}$ -1)

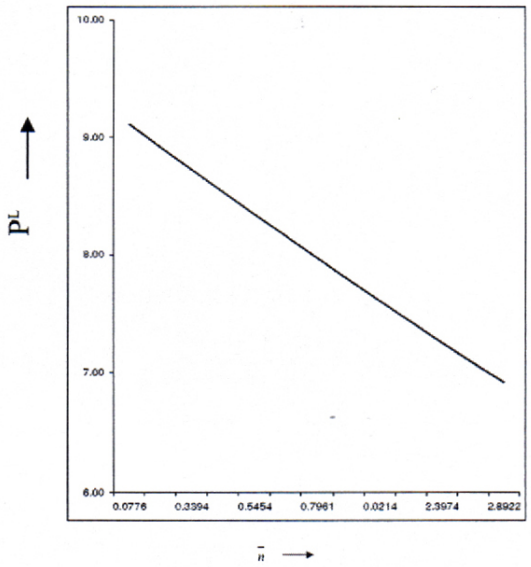
Cd(II) + **PMHETPI** Temp: 298 ± 1 K

$\log \bar{n}/(1-\bar{n})$	P^L	$\log (2 - \bar{n})/(\bar{n}-1)$	P^L
-0.7734	6.9186	0.5882	6.6260
-0.4932	6.7276	0.0524	6.4572
-0.2778	6.5372	-0.556	6.2974
-0.0866	6.3470		
0.1482	6.1602		
0.4736	5.9764		

Graph - . 4a
Formation Curve of Co(II)
Plot of $\bar{n}$ Vs [P^L]
Ligand : PMHETPI Temp: 298 ± 1K



Graph 4b
Formation curve of Ni(II)
Plot of $\bar{n}$ Vs [P^L]
Ligand : PMHETPI Temp: 298 ± 1K

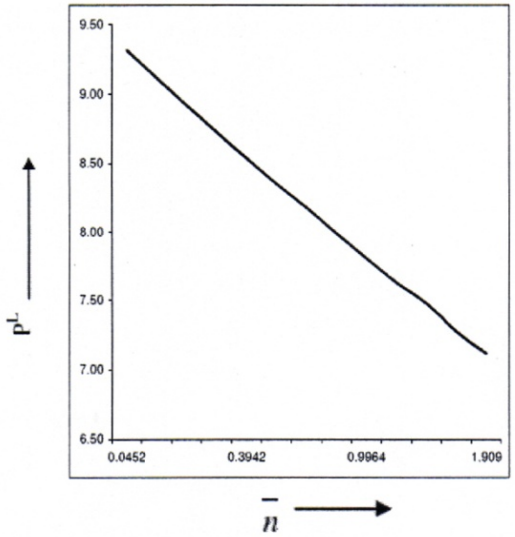


Graph - . 4c

Formation Curve of Cu(II)

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

Ligand : PMHETPI Temp: 298 ± 1K

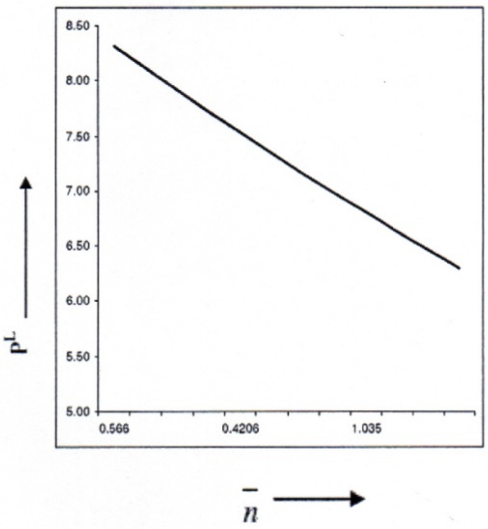


Graph 4d

Formation curve of Zn(II)

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

Ligand : PMHETPI Temp: 298 ± 1K

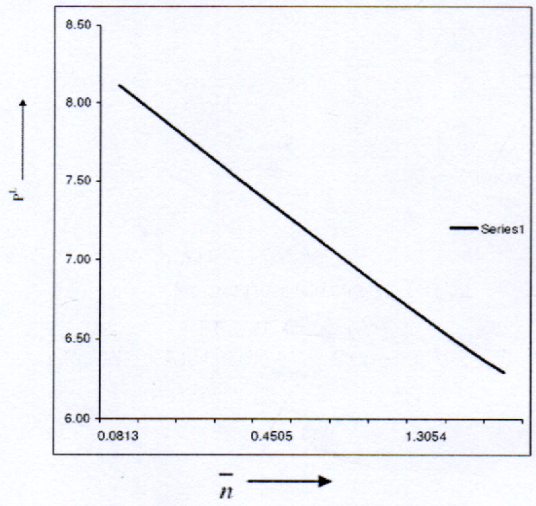


Graph - . 4e

Formation Curve of Cd(II)

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

Ligand : PMHETPI Temp: 298 ± 1K

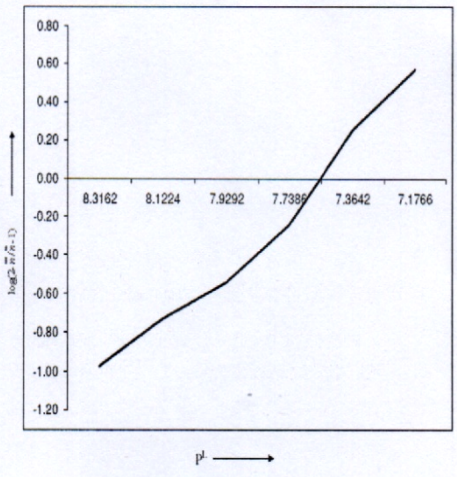


Graph 5a

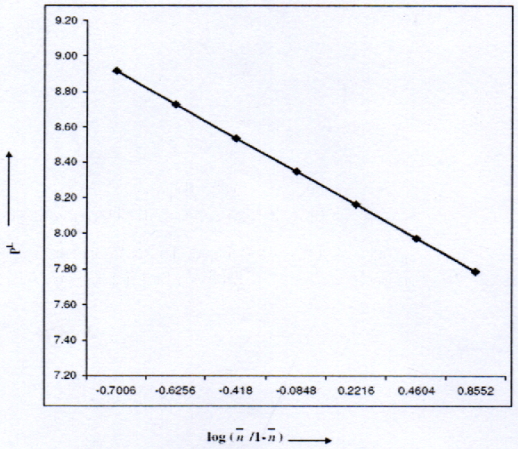
Formation curve of Co (II)

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

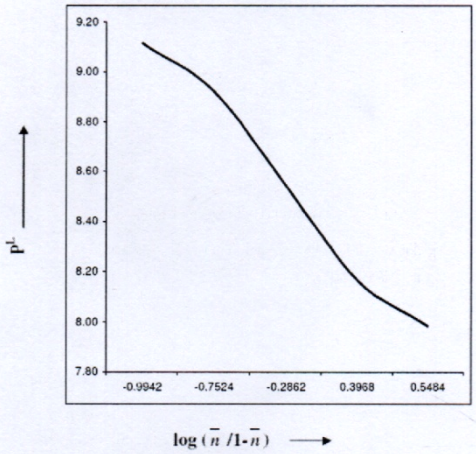
Ligand : PMHETPI Temp: 298 ± 1K



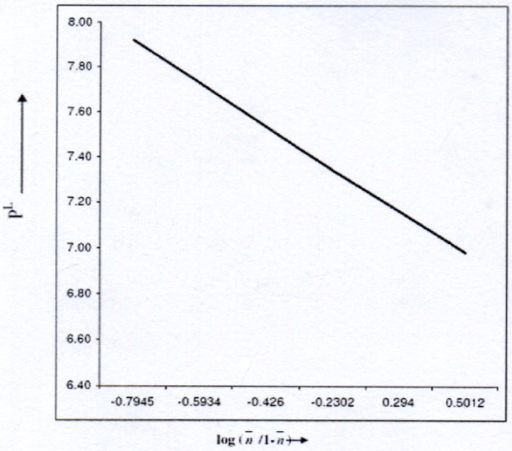
Graph 5b
Formation curve of Ni (II)
Linear Plot of log ($\bar{n}$ /1 - $\bar{n}$) Vs [P^L]
Ligand : PMHETPI Temp: 298 ± 1K



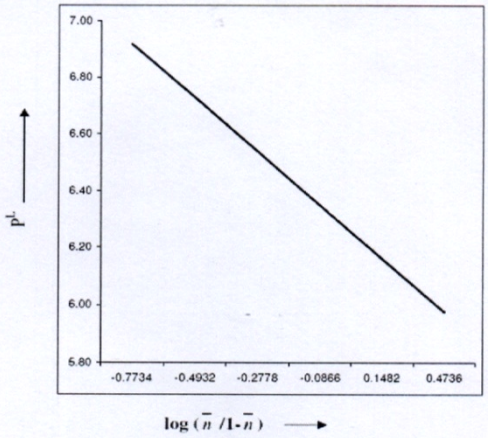
Graph 5c
Formation curve of Cu (II)
Linear Plot of log ($\bar{n}$ /1 - $\bar{n}$) Vs [P^L]
Ligand : PMHETPI Temp: 298 ± 1K



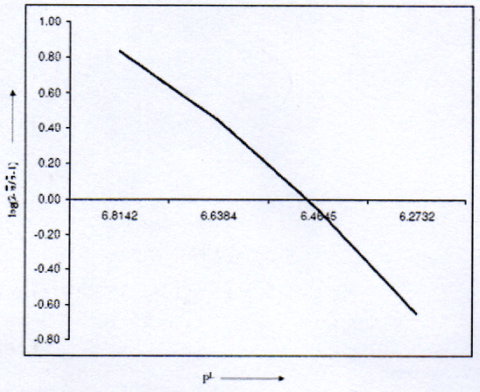
Graph 5d
Formation curve of Zn (II)
Linear Plot of log ($\bar{n}$ /1 - $\bar{n}$) Vs [P^L]
Ligand : PMHETPI Temp: 298 ± 1K



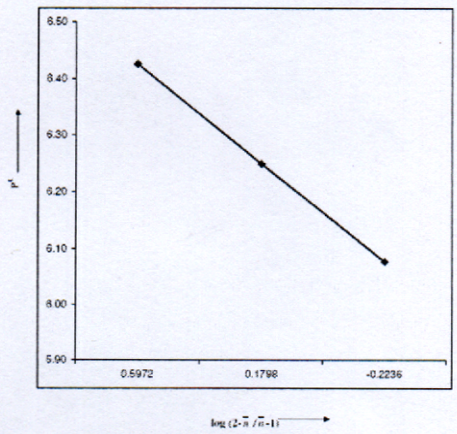
Graph 5e
Formation curve of Cd (II)
Linear Plot of log ($\bar{n}$ /1 - $\bar{n}$) Vs [P^L]
Ligand : PMHETPI Temp: 298 ± 1K

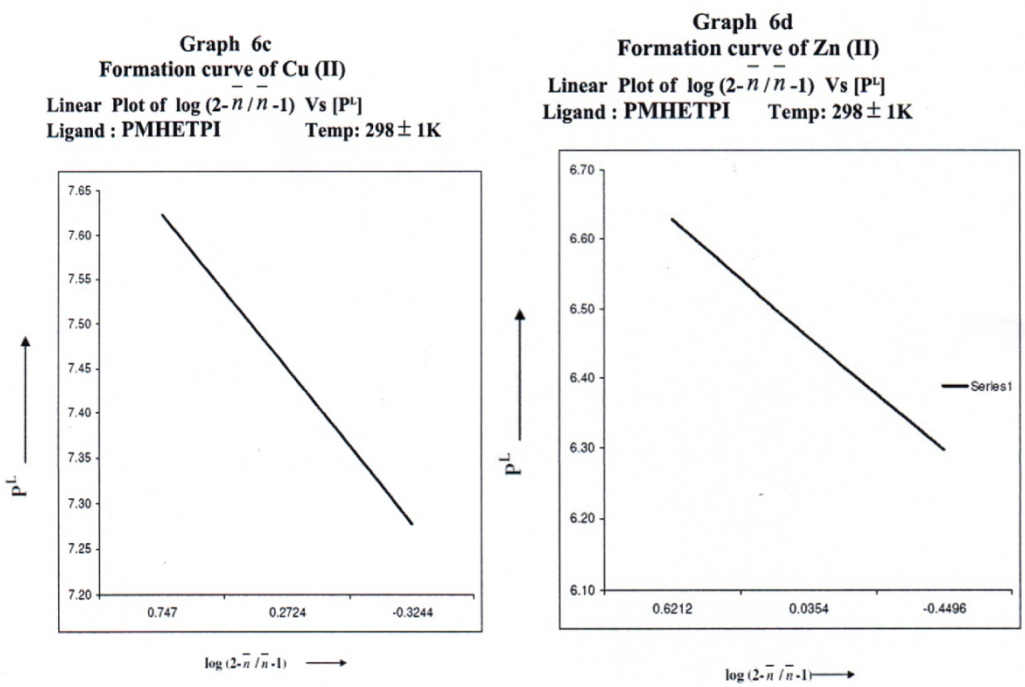


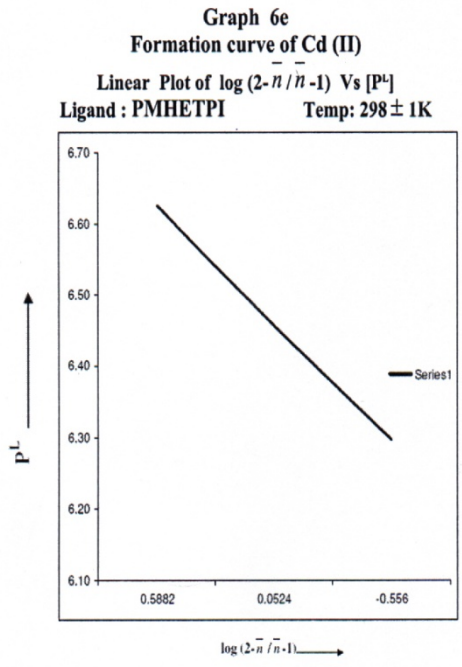
Graph 6a
Formation curve of Co (II)
Linear Plot of log (2-$\bar{n}$ / $\bar{n}$ -1) Vs [P^L]
Ligand : PMHETPI Temp: 298 ± 1K



Graph 6b
Formation curve of Ni (II)
Linear Plot of log (2-$\bar{n}$ / $\bar{n}$ -1) Vs [P^L]
Ligand : PMHETPI Temp: 298 ± 1K







The values of protonation constant and stepwise stability constant obtained by different computational method at temperature 298 K summarized in table no. – 14.

Table – 14: The values of protonation constant and stepwise stability constant

		Temperature – 298 K	
		log K_1	log K_2
H – **PMHETPI**	A	11.16	-
	b.	-	-
	c.	11.08	-
Co(II)	A	7.20	6.25
	b.	7.38	6.38
	c.	7.30	6.20
Ni(II)	A	7.24	6.06
	b.	7.26	6.23

	c.	6.38	5.44
Cu(II)	A	6.24	5.32
	b.	6.34	5.44
	c.	6.38	5.40
Zn(II)	A	6.26	5.38
	b.	6.40	5.48
	c.	6.38	5.40
Cd(II)	b.	6.20	5.32
	c.	6.35	5.44

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

The values of stepwise stability constant [15] and over all stability constant are give in table no. – 15 .

Table – 15: The values of stepwise stability constant and over all stability constant

Ligand –**PMHETPI**

μ^0 = 0.10 (M) KNO_3 Water – dioxane medium (V/V) = 50:50

System	Temp 298 K		
	log K_1	log K_2	log β
H – PMHETPI	11.10	-	11.10
Cu (II) – PMHETPI	7.31	6.31	13.62
Ni (II) – PMHETPI	7.22	6.14	13.36
Co (II) – PMHETPI	6.49	5.42	11.91
Zn(II) – PMHETPI	6.24	5.42	11.66
Cd (II) – PMHETPI	6.21	5.39	11.60

For the given ligand the stability constants of metals show the sequence Cu(II) > Ni(II) > Co(II) > Zn(II) > Cd (II)

This is natural order given by Irving – William. This method is valid for both aqueous and non-aqueous medium.

The stability of Chelates is greatly affected by the electron density around the imino constant nitrogen (-C = N -). Higher the electron density around 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 is conformity of our previous studies.

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