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Study of Substituted 3,5-Diaryl Isoxazolines Complexes in 70% Dioxane Solvent Media pH-Metrically

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Abstract

Isoxazolines posses numerous medicinal activities. In present study complex formation between Pr(III)&Sm(III) metal ions and 3-(2-hydroxy-3-nitro-5-methylphenyl)-5-(2-phenylethenyl)isoxazoline[HNMP2EI]L_1,3-(2-hydroxy-3-nitro-5-methylphenyl)-5-(phenyl)isoxazoline [HNMP5PI]L_2,3-(2-hydroxy-3-bromo-4-nitro-5-methyl)-5-(3-nitrophenyl)isoxazoline[HBNM3NI]L_3have been studied at 0.1M Ionic Strength (26±0.1)^{o}C in 70% Dioxane water mixture by Bjerrum method as adopted by Calvin & Wilson .It is observed that Pr(III)&Sm(III) metal ions form 1:1 & 1:2 complexes with ligand

L_1, L_2&L_3.The data obtained were used to estimate & compare the values of proton ligand stability constant (p^K) & metal ligand stability constant (log K).From estimated data (p^K & log K),the effect of substituents were studied.

Keywords **:**Substituted 3,5-diarylisoxazoline,dioxane-water mixture,stability constant,complex formation,metal ions.

Introduction

The studies in metal ligand complexes in solution of a number of metal ion with carboxylic acids, oximes, phenol etc. would be interesting which throw light on the mode of storage and transport of metal ions in biological Kingdom.Metal with the view to understand the bioinorganic chemistry of metal ions,Agrawal et al [1] have studied thermodynamic & ionisation constant of 2,5-diaminopentanoic acid.Wadekar et al [2] have studied stability constant of substituted thiopyrimidine.Khambre et al [3] have studied proton and metal ligand stability constant of some substituted schiff's bases and dibromo chalcone.Meshram et al [4] have studied the formation constant of Co(II),Ni(II),Cr(III)&Fe(III)ion complexes with some heterocycles by pH metrically.Recently Sonkamble [5] have studied stability constant of some synthesized ligand at 0.1M ionic strength. Recently Tihile [6] studies on interaction between Cu (II), Cr(II), Nd(II) and Pr(II) metal ions and substituted hydroxyl chalcones at 0.1 M ionic strength pH metrically. Thakur et al [7,8] have studies the influence of dielectric constants of medium on the complex equilibrium of substituted hydroxyl-1,3- propandiones with Cr(II) metal ions and studies on interaction between Cu(II), Cr(II) and Ni(II) metal ions at 0.1M ionic strength pH metrically. Shamsher etal [9] have studied the determination of thermodynamic parameters from the dissolution of calcium hydroxide in mixed solvent systems by pH-metric method.Stability constants of transition metal complexes with piperacilin drug and alanine,glycine amino acids have been studied by Magare et al [10]. Parmar P.J. [11] have been studied the determination of proton- ligand stability constants of some substituted pyrazolines pH metrically. Parhate V.V.et al [12] have studied the effect of dielectric constants of dioxane-water mixtures on proton-ligand dissociation constants (pK) and formation constants of Cu (II) complexes with 1, 3-diphenyl thiazines pH-metrically at 0.1M ionic strength. Ante Mili~evi and Nenad Raos [13] have studied the estimation of stability constants of cadmium(II) bis-complexes with amino acids by model based on $3\chi\ v$

connectivity index. Tayade D.T.et al [14] have studied the pH metric studies of interaction of synthesized ligands 2-amino-4-hydroxy-6-methylpyrimidine and 1-(4-hydroxy-6-methylpyrimidino)-3-phenylthiocarbamide with Cu(II),Cd(II), Cr(II), cations At 0.1 M ionic strength. Patil M. P. [15] have studied metal-ligand stability constant of some substituted isoxazolines by pH-metric technique. Isoxazolines posses medicinal activities such as anti-inflammatory [16] , antibacterial, anticonvulsant [17],antibiotic [18], antituberculer [19] , antifungal [20] and anxiolytic activity [21]. The Isoxazole nucleus is a prominent structural mofit found in numerous natural products and synthetic compounds with vital medicinal value [22], also possess as anti-Influenza virus activity [23]. Isoxazoline derivatives controlled botrytis cinera on cucumbers [24] has been found to have antiviral properties against herpes type 2 virus [25]. Penicillin derivatives containing isoxazole ring are found to be antibacterial [26]. Isoxazole derivatives are used as corrosion inhibitors for fuels and lubricants [27]. Its derivatives also show a good potency in animal models of thrombosis [28].

In present work an attempt has been made to study the interactions between Pr(III)&Sm(III) Cations At 0.1 M Ionic Strength with Ligand at 0.1 ionic strength,pH metrically in 70% Dioxane-water mixture.

Materials and Methods

The ligands L_1,L_2,&L_3 was synthesized in the laboratory by known literature method. The purity of these compounds exceeds 99.5% and structures were confirmed by NMR, IR and melting points. The stock solutions of the ligand was prepared by dissolving required amount of ligand in a minimum volume of dioxane subsequently diluted to final volume. Metal ion solution was prepared by dissolving metal nitrate (Sigma - Aldrich) and standardized by EDTA titration method as discussed in literature . Carbonate free sodium hydroxide solution was prepared by dissolving the Analar pellets in deionised water and solution was standardized 22. The stock solution of percholric acid was prepared and used after standardization 23.

Measurements

All measurements were carried out at (26±0.1) ^{0}C. Systronic microprocessor based pH meter with magnetic stirrer and combined glass and calomel electrode assembly used for pH measurements. The sensitivity of pH meter is 0.01 units. The instrument could read pH in the range 0.00 to 14.00 in the steps of 0.005. The pH meter was switched on half an hour before starting the titration for initial warm up of the instrument. It was calibrated before each titration with an aqueous standard buffer solution of pH 7.00 and 9.20 at (26±0.1) ^{0}C prepared from a 'Qualigens' buffer tablets. The hydrogen ion concentration was measured with combined glass electrode.

Procedure

The experimental procedure involved the titrations of

i. Free acid $HClO_4$ (0.01 mol.dm^{-3})

ii. Free acid $HClO_4$ (0.01 mol.dm^{-3}) and ligand (20 x 10^{-4} mol.dm^{-3})

iii. Free acid $HClO_4$ (0.01 mole dm^{-3}) and ligand (20 x 10^{-4} mol.dm^{-3}) and metal ion (4 x 10^{-4}mol.dm^{-3}) against standard carbonate free sodium hydroxide

(0.15 mol.dm$^{-3)}$ solution using Calvin-Bjerrum and Calvin-Wilson pH titration techniques. The ionic strength of all the solutions were maintained constant by adding appropriate amount of $NaClO_4$ solution. All titrations were carried out in 70 percentages of Dioxane-water mixtures and reading were recorded for each 0.1 ml addition. The curves of pH against volume of NaOH solution were plotted (fig 1-3). The Proton-Ligand constants were calculated from pH values obtained from the titration curves using the Irvin-Rossotti method and MATLAB computer program (Table 1) .

Results and Discussion

The extent of deviation may be the dissociation of -OH group. 3-(2-hydroxy-3-nitro-5-methylphenyl)-5-(2-phenylethenyl)isoxazoline[HNMP2EI]L_1,3-(2-hydroxy-3-nitro-5-methylphenyl)-5-(phenyl)isoxazoline [HNMP5PI]L_2 ,3-(2-hydroxy-3-bromo-4-nitro-5-methyl)-5-(3-nitrophenyl)isoxazoline[HBNM3NI]L_3 may be considered as a monobasic acid having one replaceable H^+ ion from phenolic -OH group and can be represented as

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

The titration data were used to construct the curves [acid curve (A), acid + ligand curve (A+L) and acid + ligand + metal ion curve (A+L+M)] between volume of NaOH against pH.The proton-ligand formation number n_A were calculated by Irving and Rossotti expression (Table1)

$$\bar{n}_A = \gamma - \frac{(V_2 - V_1) \quad (N + E^o)}{(V^o + V_1) \ (T_L{}^o)} \qquad ....01$$

Where γ denotes the number of dissociable protons, N is the concentration of sodium hydroxide(0.15 mol.dm-3), (V_2-V_1) is the measure of displacement of the ligand curve relative to acid curve, where V_2 and V_1 are the volume of alkali added to reach the same pH reading to get accurate values of (V_2-V_1): the titration curves were drawn on an enlarged scale: E^0 and $T_L{}^0$are the resultant concentration of perchloric acid and concentration of Ligand, respectively. V_0 is

the initial volume of reaction mixture (50 cm^3). Proton-Ligand stability constant p^k values of Ligand were calculated by algebraic method point wise calculation and also, estimated from formation curves n_A Vs p^H (Half integral method) by noting p^H at which $n_A = 0.5$[Bjerrum 1957] (Table 2).

Metal-Ligand stability constants (log k) were determined by the half integral method by plotting ṅ Vs pL. The experimental ṅ values determined using expression

$$\bar{n} = \frac{(V_3 - V_2) \ (N + E^o)}{(V^o + V_2) \ \bar{n}_A \, T_M^o} \qquad .....01$$

Where N, E^0, V_o and V_2have same significance as in equation (1), V_3 is the volume of NaOH added in the metal ion titration to attain the given p^H reading and $T_M{}^0$ (4×10^{-4} mol dm-3) is the concentration of metal ion in reaction mixture. The stability constants for various binary complexes have been calculated (Table 3).

Metal Ligand Stability Constant (Log K)

It is observed that (Table3 a-c) sufficiently large difference between log K_1& logK_2Values of Sm(III)for ligand L_1 & L_2&Pr(III) for ligand L_1&L_3 indicates the stepwise formation of complex between metal ion and ligand except Pr(III)for ligand –L_2 & Sm(III)for ligand L_3. It showed that less difference between log K_1 & log K_2 values indicates complexes are occurring simultaneously. The higher value of ratio(Log K1/ Log K2) forPr(III)- Ligand- L_1 &L_3 & Sm(III)-ligand-L_1&L_2 complex indicates the more stable stepwise complex formation as compare to Sm(III) –Ligand-L_3 &Pr(III)-Ligand L_2 complexes.

Proton-Ligand stability constant (pK)

It is observed from titration curve in (fig.1,2,3)shows that the ligand curves starts deviating from free acid (HClO4) curves at pH > 2.12,2.14,&2.0 respectively. The extent of deviation s may be the dissociation of –OH group completely.

Conclusion

From the titration curve, it is observed that the departure between (Acid + Ligand) curve & (Acid+Ligand +Metal) Curve for all system of L_1,L_2,&L_3 started from pH=2.12 to 3.38, this indicate the commencement of complex formation. Also change in color from yellow to brown in pH range from 3.35 to 10.07 during the titration showed the complex formation between Metal & Ligand.

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Table no.1 :Proton Ligand Formation number ($\dot{n}_A$) at $(26\pm0.1)^0$C and at ionic strength $\mu=0.1$ moldm^{-3} $NaClO_4$ in 70%Dioxane-Water mixture.

a)System : HNMP2EI(L_1)

P^H	V_1	V_2	$V_2 - V_1$	$\dot{n}_A$
4.42	3.2518	3.4117	0.1599	0.7597
4.70	3.2519	3.4501	0.1982	0.7023
5.07	3.2743	3.5030	0.2287	0.6566
5.14	3.2743	3.5039	0.2296	0.6552
5.21	3.2743	3.5060	0.2317	0.6520
5.42	3.3000	3.5327	0.2327	0.6507
5.63	3.3330	3.5659	0.2329	0.6504
6.00	3.3330	3.5660	0.2330	0.6503
6.14	3.3330	3.5660	0.2330	0.6503
6.21	3.3413	3.6083	0.2670	0.5996
6.28	3.3413	3.6293	0.2880	0.5681
6.35	3.3572	3.6568	0.2996	0.5506
6.37	3.3589	3.6589	0.3000	0.5502
6.42	3.3660	3.6807	0.3147	0.5282
6.49	3.3661	3.6889	0.3228	0.5608
6.70	3.3662	3.6977	0.3315	0.5030
6.84	3.4496	3.7824	0.3328	0.5018
7.00	3.4582	3.7912	0.3330	0.5016
7.35	3.4662	3.8159	0.3497	0.4767
7.42	3.4662	3.8159	0.3497	0.4767
7.56	3.4867	3.8464	0.3597	0.4619
7.70	3.4867	3.8509	0.3642	0.4552
8.00	3.5000	3.8670	0.3670	0.4512
8.35	3.5000	3.8670	0.3670	0.4512
8.42	3.5330	3.9112	0.3782	0.4349
8.56	3.5332	3.9119	0.3787	0.4342
8.70	3.5660	3.9502	0.3842	0.4260
9.00	3.6330	4.0330	0.4000	0.4037
9.35	3.6660	4.1227	0.4567	0.3192
9.70	3.7661	4.2487	0.4826	0.2805

b)System : HNMP5PI(L_2)

P^H	V_1	V_2	$V_2 - V_1$	$\dot{n}_A$
3.00	3.1551	3.2131	0.0580	0.9126
3.28	3.1555	3.2217	0.0662	0.9003
3.35	3.2021	3.2719	0.0698	0.8950
3.37	3.2024	3.2878	0.0854	0.8716
3.56	3.2038	3.3368	0.1330	0.8000
3.70	3.2042	3.3527	0.1485	0.7767
4.00	3.2083	3.4083	0.2000	0.6992
4.35	3.2475	3.4642	0.2167	0.6745
4.37	3.2482	3.4649	0.21672	0.6745
4.42	3.2518	3.4818	0.2300	0.6545
4.49	3.2519	3.4849	0.2330	0.6499
5.07	3.2743	3.5570	0.2827	0.5754
5.14	3.2743	3.5570	0.2827	0.5754
5.21	3.2743	3.5570	0.2827	0.5754
5.42	3.3000	3.5848	0.2848	0.5725
5.63	3.3330	3.6247	0.2917	0.5622
6.00	3.3330	3.6247	0.2917	0.5622
6.14	3.3330	3.6247	0.2917	0.5622
6.21	3.3413	3.6272	0.2959	0.5562
6.28	3.3413	3.6372	0.2959	0.5562
6.35	3.3572	3.6572	0.3000	0.5501
6.37	3.3589	3.6589	0.3000	0.5502
6.42	3.3660	3.6779	0.3119	0.5323
6.49	3.3661	3.6780	0.3119	0.5324
6.63	3.3662	3.6918	0.3256	0.5119
6.84	3.4496	3.7938	0.3442	0.4839
7.00	3.4582	3.8026	0.3444	0.4832
7.35	3.4662	3.8108	0.3446	0.4829
7.42	3.4662	3.8108	0.3446	0.4829
7.56	3.4867	3.8354	0.3487	0.4784
7.70	3.4867	3.8437	0.3570	0.4660
8.00	3.5000	3.8582	0.3582	0.4643
8.35	3.5000	3.8617	0.3617	0.4591
8.42	3.5330	3.8972	0.3642	0.4559
8.70	3.5332	3.9024	0.3692	0.4485
9.00	3.6660	3.9408	0.3748	0.4413
9.35	3.6330	4.0660	0.4330	0.3545
9.70	3.7661	4.2016	0.4356	0.3506

c)System : HBNM3NI(L_3)

P^H	V_1	V_2	$V_2 - V_1$	$\dot{n}_A$
3.35	3.2021	3.2608	0.0587	0.9117
3.37	3.2024	3.2638	0.0614	0.9078
3.56	3.2038	3.2708	0.0670	0.8994
3.70	3.2042	3.3042	0.1000	0.8499
4.00	3.2083	3.3250	0.1167	0.8248
4.35	3.2475	3.3755	0.1280	0.8061
4.37	3.2482	3.3780	0.1298	0.8048
4.42	3.2518	3.3818	0.1300	0.8047
4.49	3.2519	3.3819	0.1300	0.8047
5.07	3.2743	3.4321	0.1578	0.7630
5.14	3.2743	3.4332	0.1589	0.7614
5.21	3.2743	3.4333	0.1590	0.7612
5.42	3.3000	3.4641	0.1641	0.7537
5.63	3.3330	3.4982	0.1652	0.7522
6.00	3.3330	3.4982	0.1652	0.7522
6.14	3.3330	3.4991	0.1661	0.7520
6.21	3.3413	3.5077	0.1664	0.7517
6.28	3.3413	3.5082	0.1669	0.7507
6.35	3.3572	3.5242	0.1670	0.7496
6.37	3.3589	3.5259	0.1670	0.7496
6.42	3.3660	3.5330	0.1670	0.7496
6.49	3.3661	3.5332	0.1671	0.7493
6.70	3.3662	3.5333	0.1671	0.7493
6.84	3.4496	3.6333	0.1837	0.7243
7.00	3.4582	3.6569	0.1987	0.7010
7.35	3.4662	3.6665	0.2003	0.7003
7.42	3.4662	3.6828	0.2166	0.6755
7.56	3.4867	3.7033	0.2166	0.6757
7.70	3.4867	3.7034	0.2167	0.6759
8.00	3.5000	3.7503	0.2503	0.6256
8.35	3.5000	3.7506	0.2506	0.6253
8.42	3.5330	3.7922	0.2592	0.6126
8.56	3.5332	3.7930	0.2598	0.6117
8.70	3.5660	3.8328	0.2668	0.6039
9.00	3.6330	3.8998	0.2668	0.6039
9.35	3.6660	3.9329	0.2669	0.6037
9.70	3.7661	4.0661	0.3000	0.5534
9.84	3.7907	4.1248	0.3340	0.5024
10.00	3.8000	4.1660	0.3660	0.4557
10.35	3.9000	4.2988	0.3988	0.4081
10.70	4.0330	4.5316	0.4986	0.2617

Table 2: Proton Ligand Stability Constant p^K

System	p^K	
	Half integral method	Pointwise calculation method
HNMP2EI (L_1)	7.0028	7.0014
HNMP5PI(L_2)	6.6507	6.5728
HBNM3NI(L_3)	9.8442	9.7267

Table 3: Metal Ligand Stability Constant(Log K)

a) HNMP2EI (L_1)

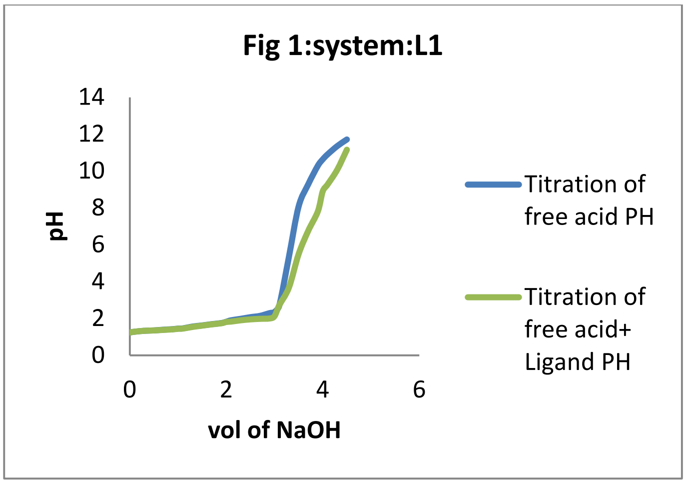
System	Log K_1	Log K_2	Log K1-LogK_2	LogK_1/LogK_2
Pr(III)	6.5807	3.8465	2.7342	1.7108
Sm(III)	6.7926	3.9788	2.8138	1.7071

b) HNMP5PI(L_2)

System	Log K_1	Log K_2	LogK1-LogK_2	LogK_1/LogK_2
Pr(III)	7.3972	5.7815	1.6157	1.2795
Sm(III)	7.7559	4.8924	2.8635	1.5853

c) HBNM3NI(L_3)

System	Log K_1	Log K_2	Log K1 - LogK_2	LogK_1/LogK_2
Pr(III)	9.9990	7.4911	2.5079	1.3347
Sm(III)	9.7658	7.4891	2.2767	1.3040



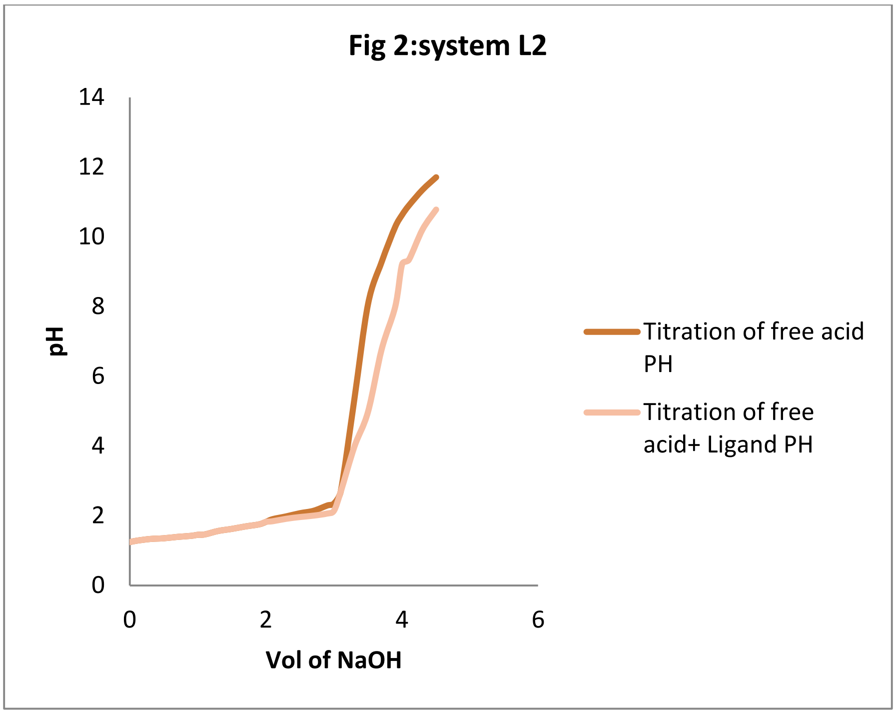


Fig 2:system L2

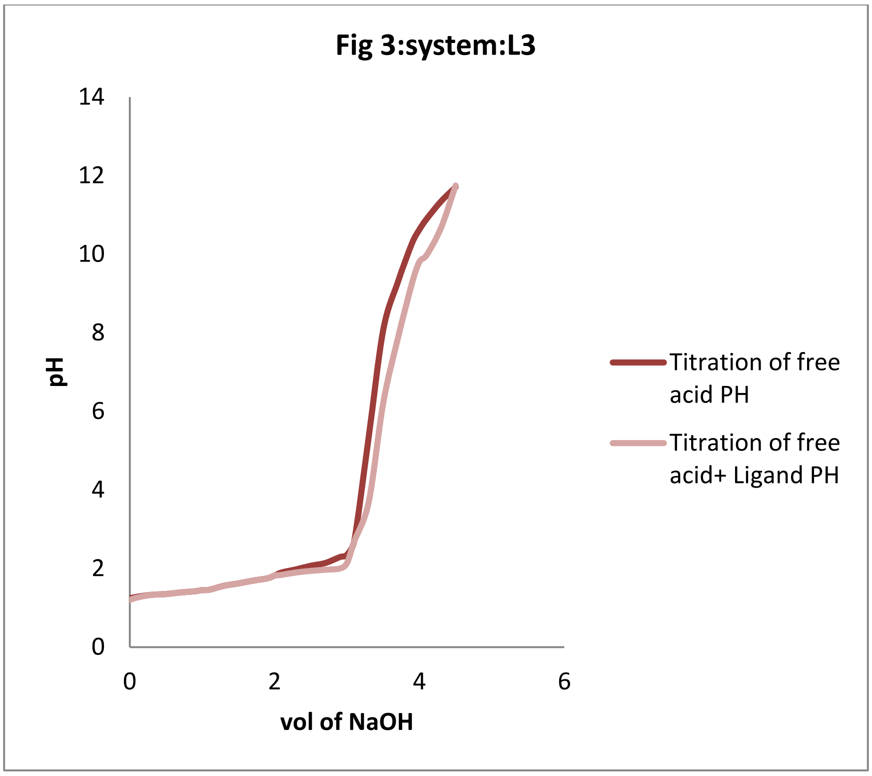


Fig 3:system:L3

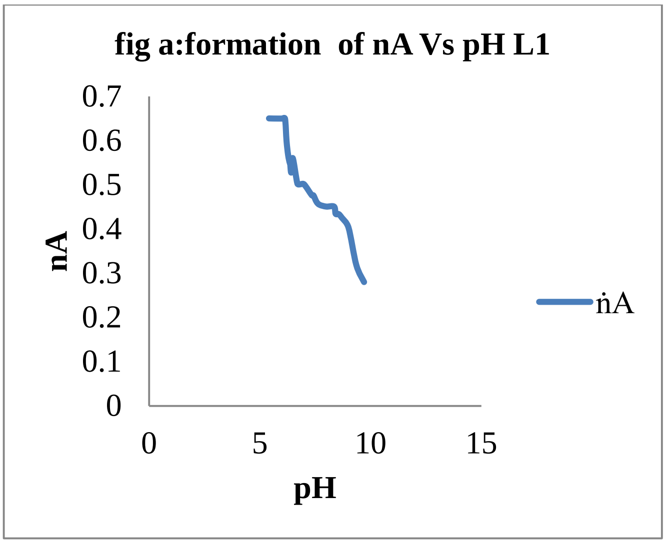


fig a:formation of nA Vs pH L1

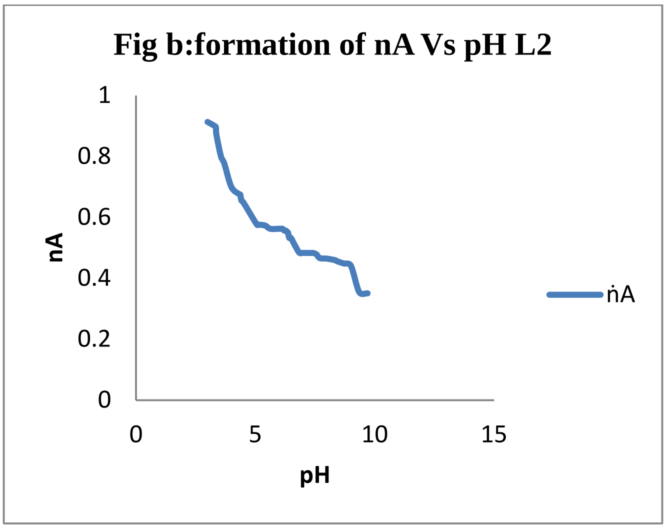


Fig b:formation of nA Vs pH L2

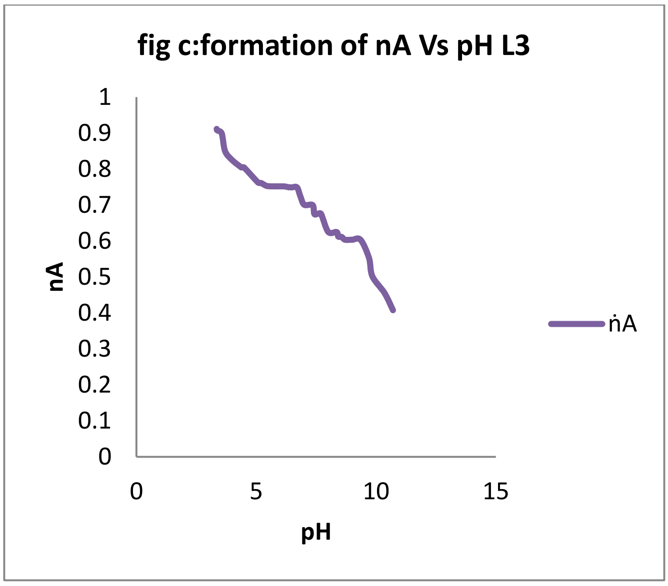


fig c:formation of nA Vs pH L3

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