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Study of Interaction of Cadmium With Imine Derivative of 2-Amino-2 -Methyl -1-Propanol In DMF -Water System (40%V/V)

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Abstract: Cadmium (II), highly toxic metal ion that acts as a carcinogen in mammals, inhibits growth of plants and decreases uptake of water and minerals. Thus there is an emergency to develop promising reducing methods. In this present research deford- hume's formulation was applied to investigate the complexation of Cadmium with newly synthesized imine derivative 2-(3,4,5 tri methoxybenzylidene amino)-2-methyl propan-1-ol using an electrochemical technique polarography in di methyl formamide-water system. Imine derivative was synthesized by condensation of 3,4,5-trimethoxybenzaldehyde with 2-Amino-2-Methyl -1-Propanol.Characterised by electronic and IR spectroscopy. Solutions were taken in the H-type cell of Lingane and Laitimer for recording polarograms. Millicoulometric method was applied to know the number of electrons transferred. From these studies it was observed that Cd(II) complex showed irreversible two electron reduction at d.m.e. electrode and 1:4 metal to ligand ratio with consecutive stability constants as $\beta_1 = 0.85 \times 10^2$; $\beta_2 = 4.50 \times 10^3$; $\beta_3 = 1.50 \times 10^5$; $\beta_4 = 3.48 \times 10^{6-}$,This study confirmed that imine derivative shows a weak interaction with cadmium.

Keywords: Deford-Humes method, Cadmium, stability constants and Polarography

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1. INTRODUCTION

Cadmium (II) is generally known as a non-essential, highly toxic metal ion that acts as a carcinogen in mammals, inhibits growth of plants by interfering with photosynthesis and nitrogen metabolism, and decreased uptake of water and minerals[1]. A well-known example of cadmium poisoning is the *Itai-Itai*disease (*Itai* = pain in Japanese), which was caused by cadmium released from mining waste into the Jinzu river in Japan, contaminating large agricultural areas[2]. Literature revealed that not much work has been carried out in establishing the complexing ability of 2-Amino-2-Methyl-1-Propanol (AMP), towards metal ions. Even the available few references on metal complexes of AMP not fully established the coordinating ability of AMP towards metals, but only emphasized the applications of those complexes in biological fields. Metal complexes prepared by the reaction of tetra chloroaurate with AMP in aqueous media were recommended as antitumor agents[3]. Werner coordination complexes[4] prepared by reacting benzotriazole with a monoepoxide to form a mixture of 1 and 2-hydroxy alkyl benzotriazoles and esterifying mixture with an alkaline succinic anhydride to form a monoester and converting the monoester to a salt of metal selected from IB, IIB, IVB and VII groups of the periodic table and complexing the salt with alkanolamines including AMP form a chelate. The chelate thus obtained was employed as detergent and anti wear additive for lubricants references. Structural and bonding properties of Cu (II) complexes in presence of AMP employing X-ray crystallographic techniques have been reported. Muhonen et al. Studied the magnetic properties of (2-amino-2-methyl-1-propanolato) chloro copper (II) and bromo Copper (II) tetramers[5]. The studies on Ni (II) complexes with AMP and otheralkanolamines have been carried out by using potentiometric methods. pH- titration method is also employed in studying the Ni (II) (substituted alkanolamines) systems and relative stabilities reported in the literature[6]. The electrochemical behaviour of Copper (II) complexes of five aminoalcohols including 2-amino-2-methyl-1-propanol (AMP) were studied[7] in aqueous solutions by maintaining pH (10.5-13.0). Exchange interaction in a linear tri nuclear Copper (II) cluster complex with a single oxygen bridge between neighbouring Copper (II) ions using AMP as a ligand have been reported [8,9]. The stability constants and molar absorptivities of complexes of Copper (II) with AMP in aqueous medium were determined employing Spectrophotometric method[10]. Specification of Copper in presence of AMP as a complexing agent has been studied by DC Polarography and Differential Pulse Polarographic techniques[11]. Complex formation equilibria of heavy metals (Cd, Pb and Hg) with aminoalcoholic biological buffers have been discussed[12]. A Potentiometric study of zinc (II) complexes and spectrophotometric study of Copper (II) complexes with AMP in aqueous media has been reported [13,14]. Synthesis, Structural, Magnetic and Spectral properties of alkanolamine complexes of, Ni, Ag, Au andZn.have been reported[15]. A determination and correlation of stability constants of various Copper (II) complexes with ethanolamines, the isopropanolamines and 3-hydroxy substituted amines of AMP and ethanol propanolamines have been reported[16]. The synthesis, structural and catalytic ethylene oligomerizationbehaviour of Nickel complexes with substituted diphenylPhosphinomethyloxazoline has been studied by Braustein et.al[17]. A nucleating tetra-dentate Schiff base ligand, bis (O-Vanillin) benzidine (O-v₂bzHz), and its seven new binuclear complexes have been synthesized and characterized on the basis of elemental analysis, IR, NMR, Electronic, Magnetic, Thermal studies and conductance measurements. The 3D molecular modeling and analysis for bond lengths and bond angles have also been carried out by Mouryaet al[18]. Spectroscopic analysis on interaction of O-Vanillin-D-Phenylalanine, o-Vanillin-L-Tyrosine and O-Vanillin-L-Levodopa Schiff bases with bovine serum albumin (BSA) has been investigated by Gao J and Wang J and et.al [19],The crystal structures of the ligand (L2) and complexes [Ni(L2)2] and [Cu(L2)2] have also been determined by using X-ray crystallographic technique has been reported by CemalSenoland et al[20].Nagaraju Reddy G, Synthesised and characterized of mo (vi) and vo (iv) new schiff base Metal complexes [21]. Schiff base derived from 2,4,6-trimethyl-m-phenylenediamine with o-vanillin and its metal complexes were studied by Kassim Karimah[22].A new tetra-nuclear coordination complex $[Cu_4(HL)_4]$ containing Cu_4O_4 cubane core has been synthesized by using Schiff base ligand $[(OH)C_6H_4CHNC(CH_3)(CH_2OH)_2]$ (H_3L), obtained by the 1:1 condensation of 2-amino-2-methyl-1,3-propanediol with salicylaldehyde[23]. Sam Norrihan was synthesized, Organotin(IV) complexes with-vanillin-2-hydrazinopyridine (VHP) [24].

2. MATERIALS AND METHODS

Standard cadmium solution was prepared using AR Cadmium acetate in a 250 ml volumetric flask and standardized gravimetrically. The $E_{1/2}$ of Cd (s) has been determined in similar conditions (for e.g. 40% DMF -water medium) employed for Cd (c) in order to avoid discrepancy due to liquid-junction potential. Potassium nitrate was used to kept ionic strength at 0.1M, 0.002% Triton-X-100 used as maximum suppressor. Double distilled mercury was employed for dropping mercury electrode. Purified mercury was employed for de oxygenation of all test solutions prior to recording. Polarography model CL-357 was used for recording the polarograms. Saturated calomel electrode (SCE) was used as reference electrode. An ELICO glass capillary having the following characteristics m = 1.7434 mg sec^{-1}, t = 4.00 sec at constant height of mercury head 67.0 cm (0.1M KNO$_3$ in open circuit) was used. The pH measurements and pH adjustments were made using 0.1 M HNO$_3$ and 0.1 M NaOH with ELICO digital pH meter model (LI 120). Solutions were taken in the H-type cell for recording polarograms. Thermostat type GL-15 was used to keep the temperature at 30±0.1. De oxygenation was employed for of all test solutions prior to recording. The electrochemical studies of imine in the presence of cadmium were carried out In BR buffer solutions of pH 2.1–10.1 Effect of Mercury column height was carried out at pH 4.10 Kinetic parameters of redox behavior were carried out at pH 2.10-10.10. The number of electrons involved in the redox behaviour of different functional groups was analyzed by Millicoulometric method. Effect of pH on current voltage curves was studied by taking 8.0 ml of the buffer solution of desired pH (1.1 – 10.1), 2 ml of the stock solution of the ligand (1.0 x 10^{-2} M) in dimethylformamide (DMF), 6 ml of dimethylformamide (DMF) and 4.0 ml of distilled water are mixed thoroughly in the polarographic cell and the polarograms are recorded after removing the dissolved oxygen by passing pure and dry nitrogen gas through the solution for about fifteen minutes. To study effect of ligand concentration 8.0 ml of required buffer solution and a known aliquot of the ligand solution (1.0 x 10^{-2} M) and the requisite volume of dimethyl formamide are mixed in the

polarographic cell to make the total volume 20 ml and the polarogram is recorded after deprecation. To effect of mercury column height 8.0 ml of the buffer solution (pH 4.1 and 8.1), 2.0 ml of the ligand (1.0×10^{-3} M) stock solution, 6.0 ml dimethylformamide (DMF) and 4.0 ml of distilled water are mixed in the polarographic cell and the polarogram is recorded at different heights (80, 70, 60, 50 and 40 cms) of mercury column[26].

2.1   METHODS
All chemicals used were of Analar Grade.

2.2   Synthesis Of 2-(3,4,5 Tri Methoxybenzylidene Amino)-2-Methyl Propan-1-ol

Imine derivative of AMP was synthesized by adding 0.05moles 3,4,5-Trimethoxybenzaldehyde (merk) to a mixture of 50ml of methanol and 2-Amino-2-methyl-1-propanol 5ml; 0.05mole and 50ml of distilled water. The mixture of reactants was poured into a clean 250 ml RB flask and stirred well with magnetic stirrer. Then the reaction mixture was refluxed for 8 hours. A yellow colored product was formed. It was separated by filtration and washed several times with hot water and methanol and dried in vacuum. This product was recrystallized from methanol[25]. The percentage of yield was 70% and melting point of the compound was 82^0C. Schiff bases were used as complexing agents. As the ligands were not freely soluble in water, 40% V/V DMF -water medium was maintained uniformly throughout the experiment to establish the complexation towards Cadmium.

3.   STATISTICAL ANALYSIS

The data obtained was analyzed by using DeVries and Kroon[27] method is used to arrive at the number of electrons involved in reduction.The heterogeneous rate constant ($k_{f,h}$) calculated from the Meites–Israel equation[28], Graphs   were drawn by using MS excel. Functions of [x], F1 [x] and F2 [x], F3[x] and F4 [x] were computed by deford-hume formulation.

4.   RESULTS AND DISCUSSION

The IR spectra of the newly synthesized Schiff base was recorded in KBr Pellets in theIR range of 4000-400 cm^{-1} and IR data establishes the Molecular structure of.   3420cm^{-1} (intramolecular H- bonding), 3020 cm^{-1} (Ar-H, stretching), 2929cm^{-1} (Aliphatic C-H stretching), 1568cm^{-1} (>C=N stretching), 1343 cm^{-1} (C-O stretching vibrations of Alcohol / Phenol shown in the (Fig 1).

Fig 1. Infrared spectra of imine derivative of AMP
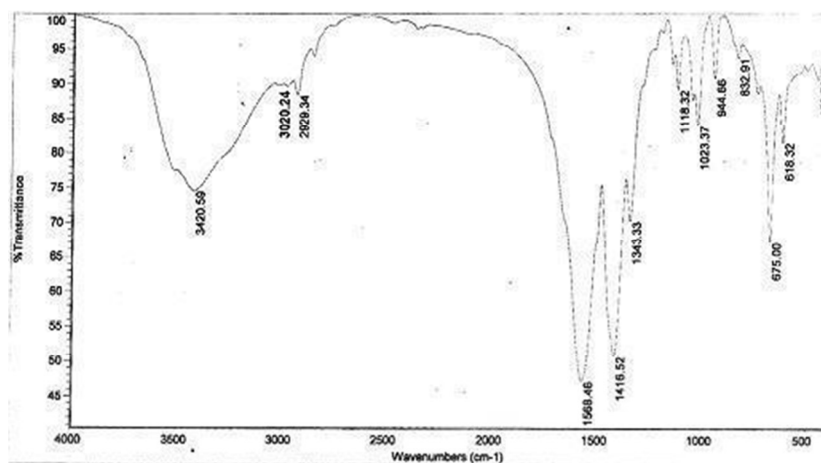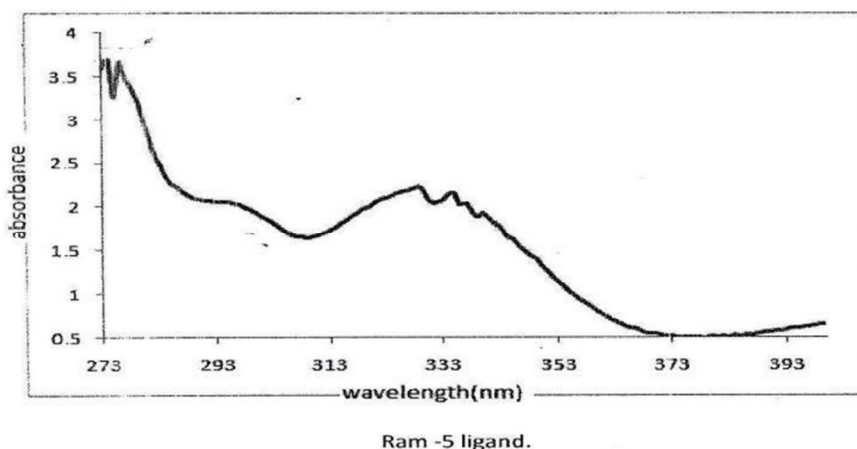

Ram -5 ligand.

Fig 2. Electronic spectra of imine derivative of AMP

Electronic spectra(fig 2) of the TMB-AMP Schiff base in UV region show an intense band at 275 nm and weaker band at 323 nm hich are assigned to the   π → π* and n → π* Transitions respectively[15]. Current voltage curves of Schiff's base ligand andSchiff's base in presence of cadmium were recorded. The results in the current voltage curves show

that the half-wave potential increases with increase in pH in the acid range but it remains unaltered in the alkaline pH range (Table;1) The plot of the half-wave potential and the pH was a straight line up to pH 8.1 and the slope lies between 80-110 mV (fig 4) .The p values (Table-3) were low and non-integers. This confirms that the proton transfer in the reduction process was a heterogeneous process.
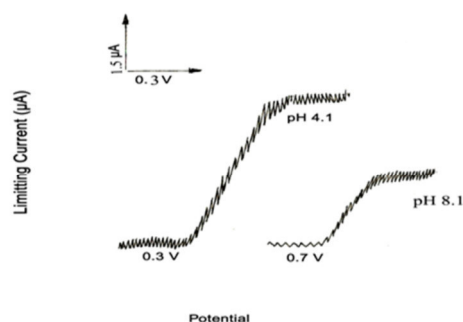
Fig 3.Polarograms of imine derivative of AMP at pH 4.1 and 8.1

The shift in the half-wave potentials with pH of the solution can therefore be ascribed due to the following reasons. Both protonated form and unprotonated form of the depolarizer were electro active. There exists equilibrium between protonated and Unprotonated forms.   The unprotonated form is reduced at more negative potentials than protonated form of the azomethine group[13].

Table:1 Polarographic results of TMB-AMP in Briton Robinson buffer solutions (1x10-3 mM) Medium: Aqueous DMF (40% v/v)		
pH	$E_{1/2}$ vs SCE	Limiting Current i_l(μA)
2.1	0.48	3.4
3.1	0.57	3.4
4.1	0.66	3.4
5.1	0.75	3.5
6.1	0.84	3.3
7.1	0.93	3.2
8.1	1.02	3.0
9.1	1.02	3.0
10.1	1.02	3.0

The pH dependence of the half-wave potential was caused not only by the antecedent chemical acid-base reaction but also by the consumption of protons in the reduction process[6]. In the presence of electric field the inequality was observed (pHs)<(pHo) as the equilibrium shifted towards the unprotonated form[25], the $E_{1/2}$ remains constant. It is seen that above pH 8.1, the shift in half-wave potential with pH was not so marked as in the acidic pH range. $E_{1/2}$ – pH of Polarographic wave was √ shaped in the pH range of study. The $E_{1/2}$ becomes practically constant in the alkaline pH range and this constancy in $E_{1/2}$ may be due to the fact that both acidic and basic forms of the depolariser were electroactive. But in the pH range where the protonation rate decreases, the half-wave potentials of both the protonated form (acidic) and the unprotonated form (basic) were so close to each other that the waves merged and a single wave was observed. $E_{1/2}$vs pH graph consists of two linear segments intercepting each other and the point of interception of the two linear plots was approximately equal to pK$_1$. The effect of mercury column height on the limiting current indicates the diffusion controlled nature of the polarographic wave[11]. The i_l versus h$^{1/2}$ plots[12] are linear and passing through the origin. The values of i_l/h$^{1/2}$ were constant as shown in the three Polarographic reduction waves that were found to be diffusion controlled waves. The i_l versus concentration graphs were linear and passing through the origin.   The constancy of i_l/C values serves not only as a test for the diffusion controlled nature of the wave but also indicates the applicability of the polarographic method for quantitative determination.

Table: 2 Effect of Mercury Column height (h) on the limiting current (i) of TMB-AMP(1x10-3 M) Medium: aqueous dimethyl formamide (40%v/v)				
Mercury column height (h)(cm)	pH = 4.1 First wave		pH = 8.1 First wave	
	i_l (μA)	$i_l/\sqrt{h}$	i_l (μA)	$i_l/\sqrt{h}$
80	3.8	0.420	3.4	0.38
70	3.5	0.42	3.1	0.38
60	3.0	0.40	2.80	0.36
50	2.8	0.40	2.50	0.36

The experimental results observed from the effect of concentrations and mercury column height (Table-2) on the limiting current confirmed the diffusion-controlled nature of the limiting current. The electrode process was found to be irreversible at low acidic pH values unlike the reversible electrode process observed for simple azo and azomethine compounds under comparable conditions. The irreversible nature of the process observed in the present investigations was also further confirmed by The slopes of (0.08-0.15) of the semi-logarithmic plots (Ede Vs log $\frac{i}{id-i}$) do not confirm the values expected for two or four electrons reversible reduction processes respectively. $E_{1/2}$ shifts towards more negative potentials with increase in the depolariser concentration. The heterogeneous rate constant ($k^0_{f,h}$) calculated from the Meites–Israel equation[28], the $K^0_{f,h}$ values

in the present investigations were less than 10^{-4}. (d) The G* values computed from the relation $k^0_{f,h} = KT/h ©_0 exp (\Delta G*/RT)$ ..(4) Increases in pH range 1.1 – 7.1 and remains unaltered in alkaline pH range 8.1 – 10.1. These observations suggest that the Polarographic reduction of azomethine (>C=N-) was found to be irreversible in nature[6]. This may be due to the presence of bulky groups at the end of the >C=N- groups. A mercury pool cathode was used for determining the number of electrons (n) in the millicoulometer described by DeVris and Kroon[27]. $F_o[x]$ (Fig 5), $F_1 [x]$ (Fig 6) $F_2 [x]$ (Fig 7) $F_3 [x]$ (Fig 8) and $F_4 [x]$(Fig 9) were computed[11] from the $E_{1/2}$ data of Cadmium at different concentrations of the ligand (Table-4), The obtained data confirmed the 1:4 metal to ligand ratio.

Table: 3 Polarographic characteristics and kinetic parameters of TMB-AMP (1x 10-3 mM) Medium: Aqueous dimethyl formamide (40% v/v)

pH	$-E_{1/2}/pH$ (Mv)	α_{na}	No. of protons p	$D \times 10^{-6}$ cmsec^{-1}	$I* \times 10^3$	K^0fh cm sec^{-1}	$\Delta G*$ k cal mole^{-1}
2.1	0.086	0.41	0.51	2.85	4.04	4.25x 10^{-4}	9.20
4.1	0.086	0.46	0.58	1.92	3.34	2.30x 10^{-4}	9.77
6.1	0.086	0.51	0.64	1.11	2.54	1.09x 10^{-4}	10.49
8.1	0.086	0.58	0.69	0.55	1.84	4.43x 10^{-5}	11.04
10.1	0.086	0.58	0.69	0.55	1.84	4.25x 10^{-5}	11.04

Table 4. Derived functions for Cadmium-ligand complex system = 1.0 mMpHβ_1 = 0.85 x 10^2; β_2 = 4.50 x 10^3; β_3 = 1.50 x 10^5; β_4 = 3.48 x 10^6

[TMB-AMP] M	i_d μA	Slope mV	$E_{1/2}$ -V vs SCE	$\Delta E_{1/2}$-V vs SCE	Log I_M / I_C	F_o [TMB-AMP]	F_1 [TMB-AMP] X 10^2	F_2 [TMB-AMP] X 10^3	F_3 [TMB-AMP X 10^5	F_4 [TMB-AMP] X 10^6
0.000	5.88	29.41	0.549	-	-	-	-	-	-	-
0.005	5.52	31.82	0.552	0.003	0.0274	1.3445	0.6889	-	-	-
0.008	5.46	32.00	0.555	0.006	0.0322	1.7159	0.8949	0.5610	-	-
0.010	5.34	31.58	0.558	0.009	0.0418	2.2143	1.2143	3.6440	-	-
0.020	5.28	31.25	0.567	0.018	0.0467	4.5039	1.7519	4.5095	-	-
0.030	5.22	30.77	0.579	0.030	0.0517	11.5659	3.5220	8.9066	1.4688	-
0.040	5.10	30.00	0.588	0.039	0.0618	23.8081	5.7020	12.1301	1.9075	1.0187
0.050	4.98	30.77	0.597	0.048	0.0721	49.0307	9.6061	17.5122	2.6024	2.2048
0.060	4.92	30.43	0.606	0.057	0.0774	99.8187	16.4698	26.0330	3.5888	3.4813
0.080	4.86	29.63	0.618	0.069	0.0827	256.5098	31.9387	38.8608	4.2951	3.4939
0.100	4.56	28.57	0.627	0.078	0.1104	549.8548	54.8855	54.0855	4.9585	3.4585
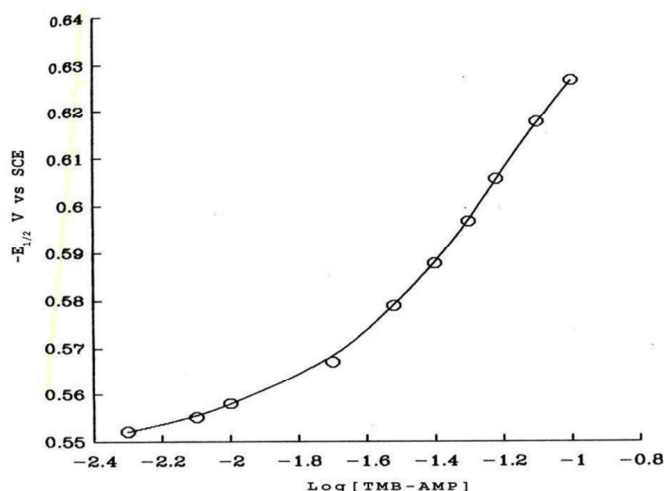
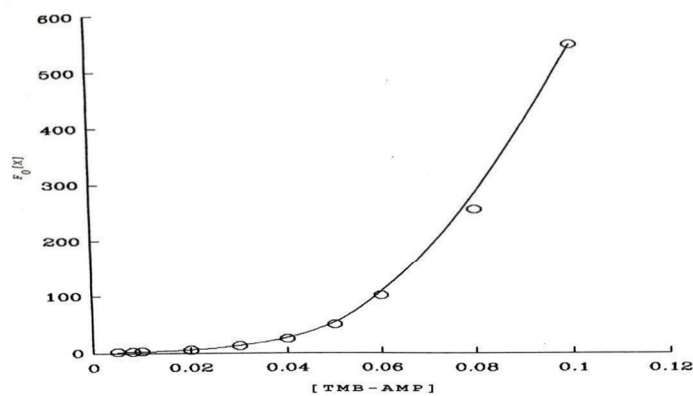**Fig 4. Plot of $E_{1/2}$ vs log [TMB-AMP] in 0.1 M KNO$_3$ at pH 8.0 for 1.0 mM Cadmium**

Fig 5. The function of F₀[X] plotted as function of ligand concentration for the Cadmium-TMB-AMP system
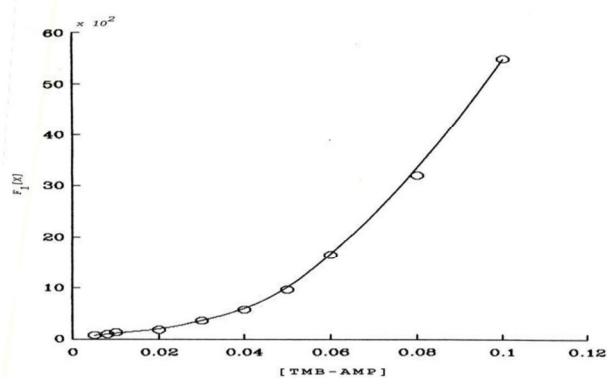

Fig 6. The function of F₁[X] plotted as function of ligand concentration for the Cadmium-TMB-AMP system

Fig 7. The function of F₂[X] plotted as function of ligand concentration for the Cadmium-TMB-AMP system

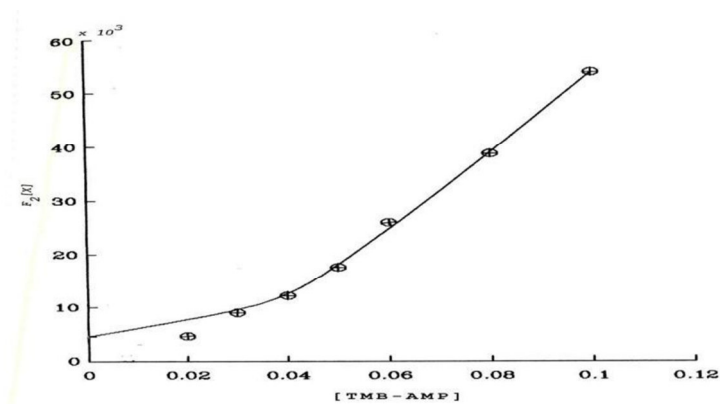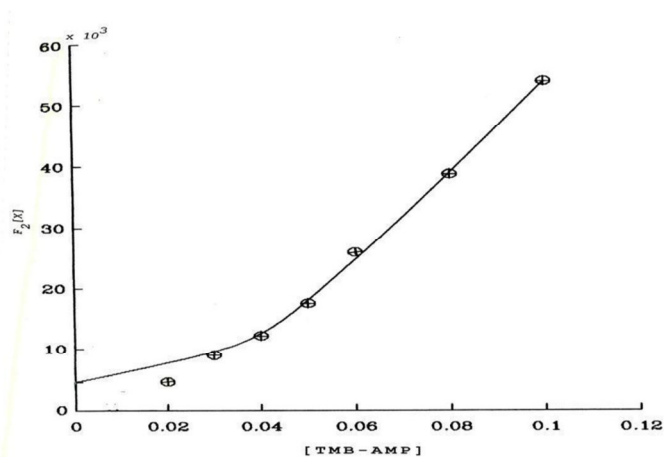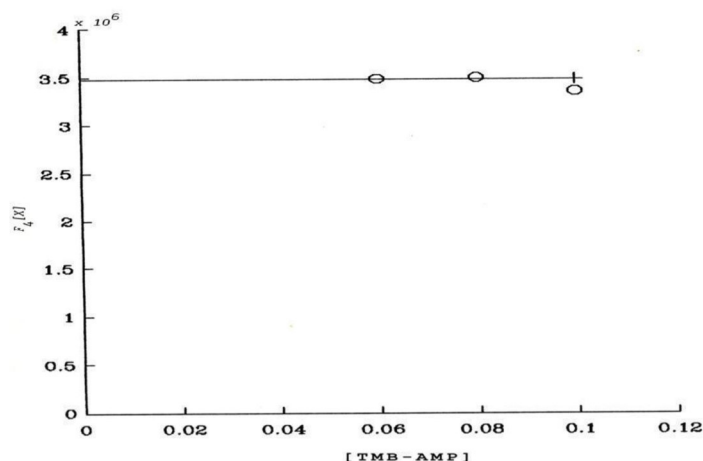**Fig 8. The function of F₃[X] plotted as function of ligand concentration for the Cadmium-TMB-AMP system**

Fig 9. The function of F$_4$[X] plotted as function of ligand concentration for the Cadmium-TMB-AMP system

5. CONCLUSION

Present investigation of interaction of cadmium with imine derivative of amp confirmed cadmium shows weak interaction with 2-(3,4,5 trimethoxybenzylidene amino)-2-methyl propan-1-ol. This study is further useful for pharmaceutical research as replacement of 2–amino- 2-methyl-1- propanol.

6. FUNDING ACKNOWLEDGEMENT

7. AUTHORS CONTRIBUTION STATEMENT

G.Ramamohan and P. Malleswarareddy conceptualized and gathered the data with regard to this work. Prof. J. Sreeramulu, D.Raju and N. Nagaraju analyzed these data and necessary inputs were given towards the designing of the manuscript. All authors discussed the methodology and results and contributed to the final manuscript.

8. CONFLICT OF INTEREST

Conflict of interest declared none.

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