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Polarographic Studies on Interaction of 3-Hydroxy-3-Phenyl-1-p-Sulfonato (Sodium Salt) Phenyltriazene with Ni (II) in Aqueous Medium

Neelam Pareek, Pooja Joshi, Dipen Upadhyay, G.P.Singh, Amit Bhandari, Anita Mehta, R. S. Chauhan and A. K. Goswami*
Coordination Chemistry Lab, Department of Chemistry, M.L. Sukhadia University, Udaipur – 313001 (Raj.), India
*E-mail : akumargoswami@rediffmail.com

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ABSTRACT

Polarographic studies of Ni (II) - 3-hydroxy-3-phenyl-1-p-sulfonato (sodium salt) phenyltriazene (HPST) have been done in aqueous medium. Ni (II) forms 1:2 complex with HPST and the electrochemical reduction of the complex is diffusion controlled in nature between the pH 7.5 to 8.5. Well defined waves are obtained and the $E_{1/2}$ shifts to more negative side with the addition of HPST. The reduction mechanism indicates two electron reversible reduction process and the stability constant Log β value found is 10.88.

Keyword: 3-hydroxy-3-phenyl-1-p-sulfonato (sodium salt) phenyltriazene, Polarographic study.

INTRODUCTION

In the present work HPST – Ni (II) complexes have been examined polarographically and stability constants calculated are in very good agreement with results of spectrophotometric studies of this system. The structure of 3-hydroxy-3-phenyl-1-p-sulfonato (sodium salt) phenyltriazene (HPST) is given below.

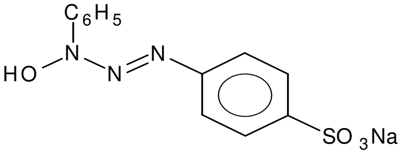

Fig.-1: HPST

MATERIALS AND METHOD

Apparatus and solutions

A Systronics polarograph 1632 was used for obtaining current- voltage curves. Metal solution (1mM) was prepared using nickel sulphate heptahydrate and ligand solution was prepared by dissolving HPST (.01M) in double distilled water. Citric acid and Na_2HPO_4 solutions were used as buffer to maintain pH of test solution. Ionic strength was kept constant by using KCl as supporting electrolyte. Gelatin (.002%) was used as maximum suppressor.
The D.M.E. had the following characteristics-
m = 1.35 mg/sec. t = 1 sec per drop.
The electrochemical behaviour of Ni (II) – HPST has been studied at d.m.e. in aqueous medium. Solution was deaerated by purging of oxygen free nitrogen through the polarographic cell. Temperature was maintained at 298 K.

Synthesis

3-hydroxy-3-phenyl-1-p-sulfonato (sodium salt) phenyltriazene has been synthesized as per reported method[3]. In this method nitrobenzene (12.3 ml) was reduced with Zn dust (20 g) in the presence of NH_4Cl (5.3 g) at 40-60°C to obtain phenyl hydroxylamine. The diazotized product was obtained by adding sodium nitrite (6.9 g) to sulphanilic acid (17.3 g) dissolved in 20 ml HCl and 100 ml water in small lots at 0-5°C under constant mechanical stirring. The diazonium compound was coupled with phenyl hydroxylamine at 0-5°C under mechanical stirring with occasional addition of sodium acetate solution for maintaining pH close to 5 during coupling process. After complete addition of diazonoum salt NaCl was added in sufficient quantity for salting out. The hydroxytriazene was obtained as yellowish brown micro crystals after crystallization from double distilled water. Its purity was checked by m.p. determination and CHN analysis. M.P. was found 157°C. The theoretical and experimental values of %C, %N and %H were obtained 43.2, 12.6, 3.6 and 42.4, 12.6, 3.6 respectively.

Further the compound was subjected to IR spectral analysis which yielded the characteristics bands reported for hydroxytriazenes[4] and their values for $\nu_{O\text{-}H}$, $\nu_{N\text{-}H}$, $\delta_{N\text{-}H}$ and $\delta_{N\text{-}OH}$ are 3450, 3190, 1590 and 940 respectively. The IR spectra confirmed their presence establishing purity of compound.

Determination of half wave potential of Ni (II) with supporting electrolyte 1×10^{-3} M Ni (II) solution in N/10 KCl has been used to obtain polarogram of Ni (II). This showed an $E_{½}$ at -1.100V vs. SCE.

Study of Ni (II) – HPST System

Solution of Ni (II) 1 mM at various concentration of HPST was prepared from the stock solution and polarographed. The shift of half-wave potentials towards a more negative value with increasing concentration of ligand indicated complex formation and the diffusion current was found to decrease regularly with increase of HPST concentration. To avoid any precipitation, the polarographic behaviour of Ni (II) – HPST system has been studied in a solution containing a ten fold excess of ligand.

RESULTS AND DISCUSSION

A single well defined wave was obtained for Ni (II) – HPST system between pH 7.5 to 8.5. Diffusion controlled nature of each wave was verified by id Vs C and id Vs $\sqrt{h}$ plots. The slope value of linear plots of $\log\left(\frac{i}{id - i}\right)$ Vs E_{de} was found to be in the range of 30-32 mV, thereby showing the reversible nature of reduction process involving two electrons.

Determination of coordination number

The plots of half wave potential ($E_{1/2}$) Vs log C_x (where C_x is equal to concentration of complex in mole/lit) have been found to be a straight line showing the formation of most stable complex. The value of j (coordination number) as determined by slope is 4. This shows that the complex composition is in 1:2 (M: L) ratio.

$$\frac{d\left(E_{\frac{1}{2}}\right)_c}{d \log C_x} = -J\frac{.0591}{n} \qquad (1)$$

Determination of stability constant

The stability constant log β of the Ni (II) – HPST complex has been determined by classical method of Lingane[2], as this method is applicable for maximum coordination number and for the stability constant of highest complex formed. The $\Delta E_{1/2}$ has a linear correlation with ligand concentrations; which shows that there is only one complex formed in the solution. The following equation has been used to calculate the stability constant of the complex studied.

$$\Delta E_{\frac{1}{2}} = \frac{.0591}{n}\log\beta + j\frac{.0591}{n}\log C_x \qquad (2)$$

Here,

$\Delta E_{\frac{1}{2}}$ = Difference of half wave potentials of simple metal ion and complexed ion.
n = Number of transferred electrons
Log β = Stability constant of complex formed.
j = Coordination number
C_x = Concentration of ligand

Thus, the value of log β has been found to be 10.88. The value of log β fall in good agreement with results of hydroxytriazene - Ni (II) complexes studied spectrophotometrically[1,5]. The data are presented in **Table I**.

CONCLUSION

The results of Polarographic study of Ni (II) – HPST system is similar to the results found in spectrophotometric study of the same system.

Table-1: Polarographic study of Ni (II)–HPST system in aqueous medium Ni (II):1 mM

S.No.	C_x (mol/lit)	log (C_x)	$E_{1/2}$ (-V vs SCE)	log β
1	00.00		1.150	
2	0.010	-2.0000	1.250	11.38
3	0.015	-1.8239	1.255	10.85
4	0.020	-1.6980	1.260	10.52
5	0.025	-1.6020	1.265	10.30
6	0.030	-1.5228	1.278	10.42
7	0.035	-1.4559	1.285	10.40
8	0.040	-1.3979	1.325	11.52
9	0.045	-1.3467	1.335	11.65

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