

**Polarographic Study of La(III)- 3-Hydroxy-3-p-Tolyl-1-p-Sulphonato(Sodium Salt) Phenyltriazene Complex**

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**ABSTRACT**

The electrochemical behaviour of complex of La (III) with 3- hydroxy-3-p-tolyl-1-p-sulphonato (sodium salt) phenyltriazene (HPST) was studied . It was observed that HPST forms 1:1 complex with La(III) in Citric acid and Na<sub>2</sub>HPO<sub>4</sub> buffer solution between pH

6.0 to 7.5. It was found that the reduction process of La (III) - HPST complex is two electron reversible reduction process. The stability constant of the hydroxy-3-p-tolyl-1-p-sulphonato(sodium salt) phenyltriazene complex was evaluated with the Lingane method at different ligand concentrations. The logarithm value of stability constant of 1:1 La(III)-3-hydroxy-3-p-tolyl-1-p-sulphonato(sodium salt) phenyltriazene complex is 10.05.

**Keywords :** Hydroxytriazene, Polarography, La (III)- HPST complex.

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**INTRODUCTION**

Hydroxytriazenes are well established chelating agents as revealed by reviews appearing on them during last few years[1,2,6,7]. These compounds have been used as spectrophotometric and complexometric reagents for determination of transition and non-transition elements[3,5,8]. In the present work complex formation of La (III) with HPST at D.M.E in aqueous and alcoholic medium has been studied polarographically. Overall stability constant of La(III)-HPST has been determined .

**MATERIALS AND METHODS****Synthesis of 3-hydroxy-3-p-tolyl-1-p-sulphonato (sodium salt) phenyltriazene (HPST)**

In a one litre beaker (0.1mol) of p-nitrotoluene, 5 gm of NH<sub>4</sub>Cl 50 ml water and 50 ml C<sub>2</sub>H<sub>5</sub>OH were mixed, stirred mechanically and cooled to 0° C. 20 gm Zn dust was added in small lots such that the temperature of reaction mixture remained between 50-60°C. The reaction mixture was stirred mechanically for another 15 min. The solution of p-tolylhydroxylamine was obtained after filtration. Thus, kept in freezer and used as such for coupling with diazotized product. In a 500 ml beaker (0.1 mol) of sulphanilic acid was dissolved in 20 ml Na<sub>2</sub>CO<sub>3</sub> solution and then NaNO<sub>2</sub> (6.9gm) was added to sulphanilic acid and dissolved this mixture in 20 ml HCl and 100 ml water in small lots at 0 to 5°C under constant mechanical stirring. The diazotized product so obtained was directly used for coupling. The p-tolylhydroxylamine was coupled with the diazotized product at 0 to 5°C under mechanical stirring with occasional addition of sodium acetate solution for maintaining the pH close to 5 during coupling process. Now sodium chloride (50 gm) was added to the reaction mixture. The compound of 3-hydroxy-3-p-tolyl-1-p-sulphonato(sodium salt) phenyltriazene was obtained as yellowish brown micro crystals after crystallization from double distilled water. C H N analysis corroborated the purity of compound. The compound was subjected to IR spectral analysis and following bands are given as: IR (KBr) cm<sup>-1</sup>: 3249 (O-H str.), 3078 (C-H str. Ar), 2981 (C-H str., CH<sub>3</sub>), 1632 (N=N str.), 1419 (N-N str.). The spectra showed the compound to be in pure state. IR spectra (KBr) were recorded on FT IR RX1 Perkin Elmer Spectrometer. A sytronics polarograph 1632 was used for obtaining C.V. curves. Physical and analytical data are given in Table-1.

**Polarographic study of La(III)-HPST complex**

Metal solution(1mM) was prepared using  $\text{La}(\text{NO}_3)_3$  and ligand solution was prepared by dissolving requisite quantity of HPST(.01 M) in double distilled water. Citric acid and  $\text{Na}_2\text{HPO}_4$  solution were used as buffer to maintain pH. Ionic strength was kept constant by using KCl as supporting electrolyte, gelatin (.002%) was used as maximum suppressor. The capillary had following characteristics  $t=1$  drop/sec .IR drop correction were applied.

The polarographic study of La(III)-HPST has been done at D.M.E in aqueous medium. Solution was deareated by purging of oxygen free nitrogen through the polarographic cell. A  $1 \times 10^{-3}$  M Cu(II) solution in N/10 KCl has been used to obtain polarograms of La(III). This showed an  $E_{1/2}$  at 1.9 Vs SCE. Polarographic study was done on La(III) with various concentration of HPST. The polarogram showed the half wave potentials shifted towards more negative value with increasing concentration of ligand indicating complex formation and the diffusion current was found to decrease regularly with increase of HPST concentration.

## RESULTS AND DISCUSSION

A single well defined wave was obtained for La(III)- HPST system between pH 6.0-7.5. Diffusion controlled nature of each wave was verified from  $i_d$  Vs C and  $i_d$  Vs  $\sqrt{h}$  plots where  $i_d$  = diffusion current in  $\mu\text{A}$ ; C=conc. In m mole  $\text{lit}^{-1}$ , h=height of mercury column.

Slope of the linear plots of  $\log (i/i_d - i)$  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. The plot of half wave potential  $E_{1/2}$  Vs  $\log C_x$  (where  $C_x$  = concentration of complex in m mole  $\text{lit}^{-1}$ ) have been found to be a straight line showing the formation of most stable complex. The coordination no. (j) of the metal complex is obtained from the slope of this plot, as may be expressed by:  $d(E_{1/2})/d \log C_x = -j/0.0591/n$

where n = no. of electrons involved (here n =3).The value of j was found to be 2.This shows that composition of the complex is 1:1 (metal: ligand).

### Determination of stability constant

The stability constant of the La(III)--HPST complex has been determined by classical method of Lingane[4], as the method is applicable for maximum coordination number and for the stability constant of highest complex formed. The  $E_{1/2}$  has a linear correlation with ligand concentration; which shows that there is only one complex formed. The following equation has been used to calculate the stability constant of the complex studied.

$$\Delta (E_{1/2}) = 0.0591/n \log \beta + j 0.0591/n \log C_x$$

Here,  $\Delta (E_{1/2})$  =Difference of half wave potentials of simple metal ion and complexed ion, n =number of transferred electron,  $\log \beta$  = Stability constant of complex formed, j = Coordination number,  $C_x$  = concentration of ligand.

Thus the value of  $\log \beta$  has been found to be 10.05. Polarographic data of Cu (II)- 3-hydroxy-3-m-tolyl-1-p-sulphonato (sodium salt) phenyltriazene are given in Table-2.

## CONCLUSION

The present work has opened up possibility of studying La(III)--HPST complexes by D.C polarographic method. Stability constant (  $\log \beta$  ) was obtained with polarography .This proves the validity of polarographic techniques for studies of hydroxytriazenes metal complexes.

Table-1:Elemental analysis of 3-hydroxy-3-p-tolyl-1-p-sulphonato (sodium salt) phenyltriazene

Molecular formula	Melting point		%C	%N	%H
(C <sub>12</sub> H <sub>10</sub> N <sub>3</sub> O <sub>4</sub> .S.Na) H <sub>2</sub> O	180° C (d)	Th.	43.2	12.6	3.6
		Exp.	42.4	12.4	3.6

Table-2: Polarographic characteristics of La (III)- 3-hydroxy-3-p-tolyl-1-p-sulphonato (sodium salt) phenyltriazene .

S.No	Cx	Log Cx	E <sub>1/2</sub>	Log β
1	0.00	0.00	1.900	-
2	0.01	-2	1.950	12.16
3	0.015	-1.8239	1.970	11.15
4	0.020	-1.6987	1.985	10.47
5	0.025	-1.6020	2.005	10.32
6	0.030	-1.5228	2.020	9.54
7	0.035	-1.4559	2.040	9.20
8	0.04	-1.3979	2.055	8.91
9	0.045	-1.3467	2.070	8.65

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