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# POLAROGRAPHIC DETERMINATION OF Co (II) COMPLEX WITH 3-HYDROXY-3-m-TOLYL-1-p-SULPHONATO(SODIUM SALT) PHENYLTRIAZENE

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**ABSTRACT**

The electrochemical behavior of complex of Co (II) with 3- hydroxy-3-m-tolyl-1-p-sulphonato (sodium salt) phenyltriazene (HTST) was studied . It was observed that HTST forms 1:3 complex with Co(II) in between pH 7.5-9.5 . It was found that the reduction process of Co(II)- HTST complex is two electron reversible reduction process. The logarithm value of stability constant of 1:3 Co(II)-3-hydroxy-3-m-tolyl-1-p-sulphonato(sodium salt) phenyltriazene complex is 11.66.

**Keywords:** Hyroxytriazene, Polarography, HTST-Co (II) complex, Stability constant.



## INTRODUCTION

Hydroxytriazenes are well established chelating agents as revealed by reviews appearing on them during last few years[1-4].These compounds have been used as spectrophotometric and complexometric reagents for determination of transition and non-transition elements[5-7]. In the present work complex formation of Co (II) with HTST at D.M.E in aqueous and alcoholic medium has been studied polarographically. Overall stability constant of Co (II)-HTST has been determined.

## EXPERIMENTAL

### Synthesis of hydroxytriazene 3-hydroxy-3-m-tolyl-1-p-sulphonato (sodium salt) phenyltriazene (HTST)

Synthesis of m-tolyl-hydroxylamine: In a one litre beaker (0.1mol) of m- nitrotoluene, 5 gm of $NH_4Cl$ 50 ml water and 50 ml $C_2H_5OH$ were mixed, stirred mechanically and cooled to 0° C by surrounding the beaker with ice salt mixture, 20 gm Zn dust was added in small lots such that the temperature of reaction mixture maintained between 50 to 60° C. The reaction mixture was stirred mechanically for another 15 min. The solution was filtered and filtrate was taken in a beaker and kept in freezer and used as such for coupling with diazotized product.

Diazonium salt of sulphanilic acid: In a 500 ml beaker 0.1 mol of sulphanilic acid was dissolved in warm mixture of 25 mL of concentrated HCl and 25 mL of water and in another beaker 6.9g of $NaNO_2$ was dissolved in 20 mL of distilled water. The beaker which contained sulphanilic acid solution was put in an ice bath to maintain temperature between 0 to 5º C. To this The $NaNO_2$ solution was added drop by drop with continuous stirring. The diazotized product so obtained was directly used for coupling.

Coupling: The m-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. The compound was obtained as yellowish fluffy powder after crystallization from ethanol.

Melting points of all synthesized compounds were taken in open capillaries and are uncorrected. C H N analysis corroborated the purity of compound. Further the compound was subjected to IR spectral analysis and following bands were observed :

IR (KBr) $cm^{-1}$: 3249 (O-H str.), 3078 (C-H str. Ar), 2981 (C-H str., $CH_3$), 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 Systronics Polarograph 1632 was used for obtaining current voltage curves. Physical and analytical data are given in Table-1.

**Polarographic study of Co(II)-HTST complex**

A systronics polarograph 1632 was used for obtaining current voltage curves. Metal solution (1mM) was prepared using $Co(NO_3)_2.6H_2O$ and ligand solution was prepared by dissolving requisite quantity of HTST(.01 M) in double distilled water. Citric acid and $Na_2HPO_4$ solution were used as buffer to maintain pH.Ionic strength was kept constant by using KCl as supporting electrolyte, gelatin (.002%) was use as maximum suppressor. The capillary had following characteristics t=1 drop/sec .IR drop correction were applied.

The polarographic study of Co(II)-HTST has been done at D.M.E in aqueous medium. Solution was deareated by purging of oxygen free nitrogen through the polarographic cell.

**Determination of half wave potential of Cu(II) with HTST**

A $1\times10^{-3}$ M Cu(II) solution in N/10 KCl has been used to obtain polarograms of Co(II).This showed an $E_{1/2}$ at 0.25 vs SCE. polarographic study was done on Co(II) with various concentration of HTST. 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 HTST concentration.

## RESULTS AND DISCUSSION

A single well defined wave was obtained for Co(II)-HTST system between pH 7.5-9.5. Diffusion controlled nature of each wave was verified from id vs C and id vs √h plots where id =diffusion current in μA; C=conc. In m mole $lit^{1-}$, h=height of mercury column.

Slope of the linear plots of log (i/id-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 Cx (where Cx = concentration of complex in m mole $lit^{1-}$ ) 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:

$d(E_{1/2})/d \log Cx = -j.0591/n$

where n = no. of electrons involved (here n =2).The value of j was found to be 6. This shows that composition of the complex is 1:3 (metal: ligand).

**Determination of stability constant Co(II)-HTST complex:**

The stability constant of the Co(II)-HTST complex has been determined by classical method of Lingane[8], 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.

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

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

j = Coordination number, Cx = concentration of ligand.

Thus the value of log β has been found to be 11.66. Polarographic data of Co (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 Co(II)-HTST complexes by D.C polarographic method. Stability constant ( log β) 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-m-tolyl-1-p-sulphonato (sodium salt) phenyltriazene

| Molecular formula | Melting point | | %C | %N | %H |
|---|---|---|---|---|---|
| $(C_{12}H_{10}N_3O_4.S.Na)\ H_2O$ | 180° C (d) | Th. | 43.2 | 12.6 | 3.6 |
| | | Exp. | 42.4 | 12.4 | 3.6 |

Table-2: Polarographic characteristics of Co(II)-3-hydroxy-3-m-tolyl-1-p-sulphonato (sodium salt) phenyltriazene .

| S.No | Cx | Log Cx | $E_{1/2}$ | Log β |
|---|---|---|---|---|
| 1 | 0.00 | 0.00 | 1.200 | - |
| 2 | 0.01 | -2 | 1.220 | 12.67 |
| 3 | 0.015 | -1.8239 | 1.240 | 12.16 |
| 4 | 0.020 | -1.6987 | 1.250 | 11.85 |
| 5 | 0.025 | -1.6020 | 1.260 | 11.65 |
| 6 | 0.030 | -1.5228 | 1.275 | 11.55 |
| 7 | 0.035 | -1.4559 | 1.280 | 11.12 |
| 8 | 0.04 | -1.3979 | 1.285 | 11.23 |
| 9 | 0.045 | -1.3467 | 1.290 | 11.10 |

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