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Research Article

# ANALYTICAL APPLICATION OF 3-HYDROXY-3-ISOPROPYL-1-(4-SULPHONAMIDOPHENYL) TRIAZENE IN THE SPECTROPHOTOMETRIC DETERMINATION OF NICKEL (II)

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**ABSTRACT**

3-Hydroxy-3-isopropyl-1-(4-sulphonamidophenyl)triazene has been used for spectrophotometric determination of Nickel(II) at 395 nm. The pH range was observed between 6.7 to 7.3. The Beer's law is obeyed in the range $1x10^{-5}$ to $6x\ 10^{-5}$ M. The molar absorptivity and Sandell's sensitivity values are 7076 $dm^3\ mol^{-1}\ cm^{-1}$ and 8.29 ng $cm^{-2}$, respectively. The Nickel (II) has been determined successfully even in presence of upto 100 ppm of various interfering cations and anions. The reagent forms complex with iron at ratio of 1:2. The composition of complex was determined by Job's method and Mole ratio method of Yoe and Jones. The value of log β found from two different methods were 9.46 and 9.43 respectively.

## 1. INTRODUCTION

A number of hydroxytriazenes[1-7] have been used as spectrophotometric as well as metalochromic indicators. We report here spectrophotmetric determination of Nickel (II) using one such hydroxytriazene, 3-hydroxy-3-isopropyl-1-(4-sulphonamidophenyl) triazene.

## 2. EXPERIMENTAL

3 - Hydroxy - 3 -isopropyl-1- (4 - sulphonamidophenyl) triazene was synthesized by the reported method[8].

### 2.1 Synthesis of 3-hydroxy-3-isopropyl-1-(4 - sulphonamidophenyl) triazene

***2.1.1 Synthesis of isopropylhydroxylamine*:** In a one litre beaker 19.00g (0.30 mol) of nitroisopropane, 30 g of $NH_4Cl$ and 300 ml of distilled water were mixed, stirred mechanically and cooled to 0°C by surrounding the beaker with ice salt mixture. To the reaction mixture 30 g Zn dust was added in small lots in few minutes interval taking about an hour for complete addition. During this period, the temperature was maintained between 0° to 15°C by adding ice to the reaction mass occasionally. The reaction mixture was stirred mechanically for another 40 min keeping temperature between 0 to 15°C. The solution was filtered under suction and washed with 100 ml ice cold water. The filtrate was taken in a beaker and kept in freezer and used as such for coupling with diazotized product.

***2.1.2 Diazotization of sulphanilamide*:** In a 500 ml beaker 0.2 mol ofl sulphanilamide was dissolved in warm mixture of 50 mL of concentrated HCl and 50 mL of water. After constant stirring the mixture was kept in a freezer to cool. In another beaker 13.9g of $NaNO_2$ was dissolved in 40 mL of distilled water and kept it in the freezer. The beaker which contained sulphanilamide solution was put in an ice bath to maintain temperature between 0 to 5ºC. To this The $NaNO_2$ solution was added drop by drop with continuous stirring. The diazotized product so obtained was directly used for coupling.

***2.1.3 Coupling*:** The isopropylhydroxylamine prepared in step (a) was coupled with the diazotized product of (b) step 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 3-hydroxy-3-isopropyl-1-(4-sulphonamidophenyl) triazene was obtained as yellowish green 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. The compound was subjected to IR spectral analysis and following bands were observed. IR (KBr) cm-1: 3290 (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. Physical and analytical data are given in Table 1 and the structure has been given in Figure 1.

**Table 1: Elemental analysis of 3 - hydroxy - 3 -isopropyl - 1 – (4 -sulphonamidophenyl) triazene (HISPT)**

| Molecular Formula of HISPT | Molecular Weight | M.P. °C | Elemental Analysis | | | |
|---|---|---|---|---|---|---|
| | | | | C% | N% | H% |
| $C_9H_{14}N_4O_3S$ | 258.30 | 193 | Th. | 41.85 | 21.69 | 5.46 |
| | | | Exp. | 41.00 | 21.20 | 5.03 |

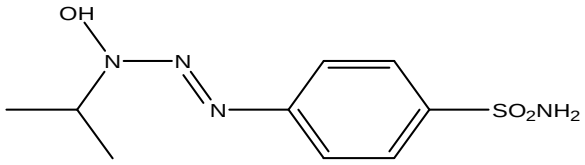



**Figure :1 Structure of 3 - hydroxy - 3 -isopropyl- 1 - (4 -sulphonamidophenyl) triazene**

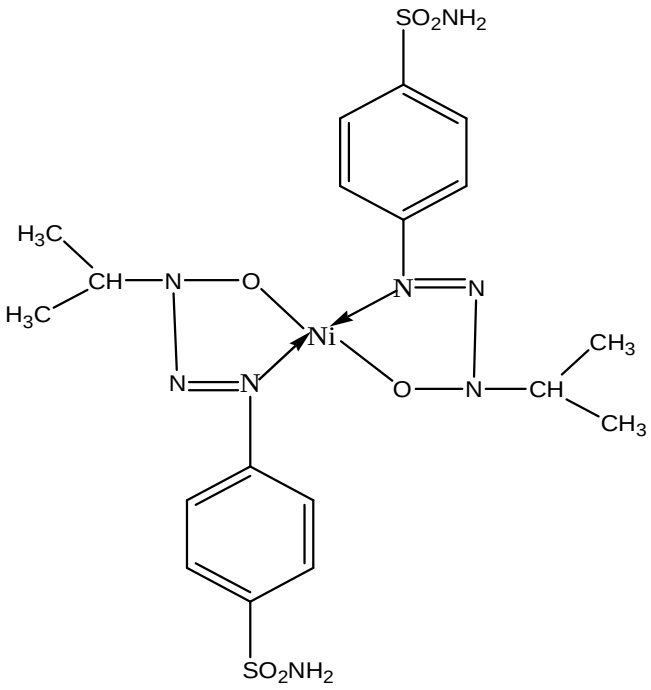
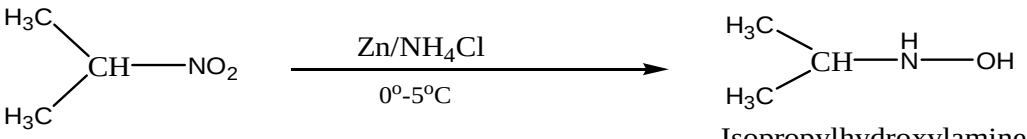



**Figure-2: The tentative structure of 1:2 complex of Ni (II) with 3-hydroxy-3-isopropyl-1-(4-sulphonamidophenyl)triazene**

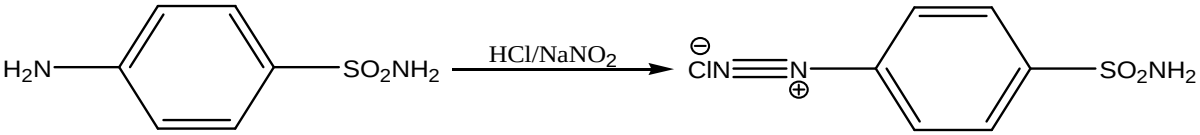
**Scheme:1**

**1.Synthesis of isopropylhydrolamine**




Nitroisopropane Isopropylhydroxylamine

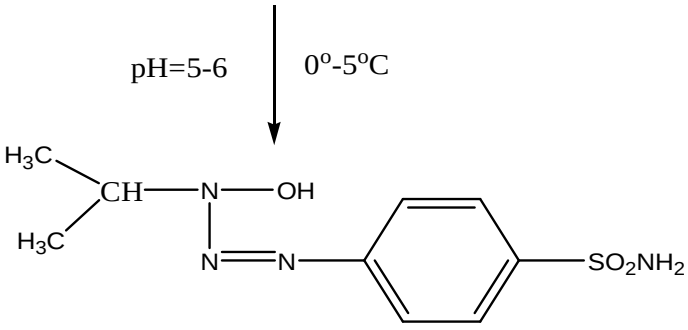
**2.Diazotization of sulphanilamide**




Sulphanilamide 4-Sulfamoyl-benzenediazonium; chloride

**3.Coupling**


Isopropylhydroxylamine 4-Sulfamoyl-benzenediazonium; chloride




3-Hydroxy-3-isopropyl-1-(4-sulphonamidophenyl)triazene

**2.2 SPECTROPHOTOMETRIC DETERMINATION OF NICKEL (II) COMPLEX WITH HISPT**

**2.2.1 Stock Solution**

A 1.0x $10^{-2}$ M stock solution of Nickel chloride hexa hydrate (BDH) was prepared in distilled water. Few drops of (1 M) concentrated hydrochloric acid were added to prevent hydrolysis. The solution was standardized with EDTA solution at pH 10-11 using murexide[9] as an indicator. A 1x$10^{-2}$ M solution of the reagent 3-hydroxy-3-isopropyl-1-(4-sulphonamidophenyl) triazene was prepared in alcohol. Tris-buffer (1%, w/v) was prepared. A UV/VIS. Systronic 106 Spectrophotometer and a pH Scan 2 tester were used.

**2.2.2 Method**

Spectrum of 3-hydroxy-3-isopropyl-1-(4-sulphonamidophenyl) triazene was measured in the

wavelength region 360 – 460nm against solvent blank. Nickel and 3-hydroxy-3-isopropyl-1-(4-sulphonamidophenyl) triazene solutions were taken in 1:5 ratio and the spectrum of Nickel complex was recorded against reagent blank in the range 360-460 nm. The working wavelength was found to be 375 nm. A set of solutions containing Ni(II) and 3-hydroxy-3-isopropyl-1-(4-sulphonamidophenyl) triazene reagent in ratio 1:5 was prepared and ph was varied between 5 to 8. The pH range of constant maximum absorbance was found to be between 6.1 to 7.3. Composition of the complex was determined by Job's method and moles ratio method of Yoe and Jones. The study revealed that composition of Nickel (II) complex is 1:2 (M:R). Absorbance of set of six solutions containing Ni(II) to 3-hydroxy-3-isopropyl-1-(4-sulphonamidophenyl) triazene in ratio 1:5 was measured at corresponding working wavelength against reagent blank. Beer's law was obeyed in concentration range $1x10^{-5}$ to $6x10^{-5}$ M. Interference of 23 cations and anions in the determination of nickel was studied. To the set of solutions containing nickel to reagent 1:5 ratio, 10 ppm of different foreign ions were added at optimum conditions. Absorbance was measured against reagent blank. Those ions, which did not interfere at 10 ppm level their interference was again studied at 50 ppm level. In case no or little change in absorbance was seen as compared to the absorbance without any foreign ion, then for those ions interference was studied again at 100 ppm level. However tolerance of still higher concentration was not studied.

## 3. RESULTS AND DISCUSSIONS

Nickel(II) was found to form 1:2 complex with 3-hydroxy-3-isopropyl-1-(4-sulphonamidophenyl) triazene.

### Stability constants

Harvey and Manning's method[10] and Purohit's method[11] have been used to determine the stability constants. Validity of the methods can be confirmed from the value of log β obtained from both the methods. Value of log β obtained by Harvey and Manning method and Purohit's method were 9.33 and 9.55 respectively. The log β values agree quite well. Further the precision studies were carried out by measuring the absorbance of 10 sets of solution containing 5.86 ppm of Nickel (II), and title reagent in 1:5 ratio, under optimum conditions. The absorbance was measured against reagent blank at working wavelength (375 nm). Nickel was successfully determined at 5.86 ppm level with good precision. The value of ΔG obtained for the Harvey and Manning method and Purohit's method were –12.99 and –12.86(kcal/mole) at 27°C respectively. Interference of several cations and anions in the determination of nickel was studied at 10, 50 and 100 ppm level. Interference was studied using following 23 cations and anions viz. $Na^+$, $K^+$, $NH_4^+$,$Ba^{2+}$, $Mn^{2+}$ $Co^{2+}$, $Pb^{2+}$, $Cu^{2+}$, $Zn^{2+}$, $Cd^{2+}$, $Mg^{2+}$, $F^-$,$Cl^-$, $Br^-$, $I^-$, $NO_2^-$, $NO_3^-$, $SO_4^{-2}$, $WO_4^{-2}$, $CO_3^{2-}$, $S_2O_3^{2-}$ ,$C_2O_4^{2-}$ ,$CH_3COO^-$. It was seen that at 100 ppm level the ions which still did not interfere are, $Na^+$, $K^+$,$NH_4^+$, $Ba^{2+}$, $Mg^{2+}$, $Cl^-$, $Br^-$, $I^-$. $F^-$, $SO_4^{-2}$, $WO_4^{-2}$, $NO_2^-$ , $NO_3^-$, $CO_3^{2-}$, $CH_3COO^-$. However tolerance of higher concentration was not studied. Thus it can be seen that Nickel (II) can be determined even in presence of number of interfering species present at 100 ppm level. Thus from the above studies it can be concluded that 3-hydroxy-3-isopropyl-1-(4-sulphonamidophenyl) triazene can be used successfully for spectrophotometric determination of Nickel(II). The tentative structure of 1:2 complex of Ni (II) with HISPT has been given in Figure 2.

## 4. REFERENCES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