

## 2-Hydroxy-5-Ethylacetophenone Oxime (HEAO) As An Analytical Reagent: Studies on Ni (II) Chelate

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**Abstract:** 2-Hydroxy-5-Ethylacetophenone Oxime (HEAO) has been used as a reagent for the study of Ni (II) chelate. The reagent gives green precipitate at pH 7.0 to 8.0. Ni (II) is quantitatively precipitated by HEAO between pH 5.5 to 10.0 and it was determined gravimetrically at pH 7.0 to 8.0. After drying the precipitate at 110<sup>o</sup> C its composition corresponds to the formula Ni(C<sub>10</sub>H<sub>12</sub>O<sub>2</sub> N)<sub>2</sub>. Structure of green coloured complex has been assigned on the basis of Job's method, mole- ratio method and IR spectra

**Key Words:** 2-Hydroxy-5-Ethylacetophenone Oxime (HEAO), Ni (II) chelate, Job's method, mole- ratio method and IR spectra.

### Introduction:

Many organic reagents like oximes(1-5),nioxime(6) ,dimethyl glyoxime(7)etc. are found to interact with metal ions giving precipitation due to the complex formation. The purpose of the present work is establishment of new ligand for nickel complex formation.

### Experimental:

The 0.05 M stock solution of Ni(II) has been prepared by dissolution of nickel chloride (AR) in distilled water and little acid. The amount of Ni (II) in this solution was determined volumetrically by EDTA(8).

- Ligand (HEAO) was prepared earlier for the study of Cu(II) chelate.(9)
- **Preparation of Ni(II)Heao Complex:**

A series of buffer solutions with pH values ranging from 4.0 to 10.0 were prepared by using acetic acid + sodium acetate, acetic acid + ammonia and ammonia + ammonium chloride. Then an ethanolic solution of HEAO ligand was mixed with an aqueous solution of nickel chloride in molar ratio 1:2. The pH of the

reaction mixture was adjusted by buffer solution. The green coloured complex was filtered, washed and dried at 110°C.

Spectrophotometric measurements were made on Bausch and Lomb spectrophotometer and Shimadzu UV spectronic. All the pH measurements were made with an Elico pH meter.

### Spectrophotometric Procedure:

The precipitates of Ni (II)-HEAO complex was insoluble in absolute ethanol or methanol. However, it was soluble in nonpolar solvents such as benzene, carbon tetrachloride, chloroform etc. The complex was therefore extracted in chloroform layer. The absorbance of the organic layer was recorded against the reagent blank prepared under similar conditions.

### Results and Discussion:

Spectrophotometric determination of Ni (II) optimum pH and selection of wavelength:

The pH of solution has a pronounced effect on the reaction between Ni (II) and HEAO and the stability of complex. On the other hand, the absorbance is dependent upon the wavelength used. Both the parameters were, therefore, controlled to get best results.

The absorbance measurements of the Ni (II)-HEAO complex show, that the absorbance of the coloured solution of complex increases continuously towards the shorter wavelength. The absorbance shows a shoulder at 385 nm. 385 nm wavelength is selected for present work.

For the selection of pH, absorbance of the organic layer containing Ni(II)-HEAO complex extracted from aqueous solution at different pH values were measured. From the results given in following table it is evident that the maximum absorbance occurs in the pH range 7-8. A pH 7-8 was selected for present work.

**Table no.-1**

pH	Buffer	Absorbance
5.5	CH <sub>3</sub> COOH+CH <sub>3</sub> COONa	0.232
6.0	CH <sub>3</sub> COOH+CH <sub>3</sub> COONa	0.468
7.0	CH <sub>3</sub> COOH+NH <sub>3</sub>	0.824
8.0	NH <sub>3</sub> +NH <sub>4</sub> Cl	0.824
9.0	NH <sub>3</sub> +NH <sub>4</sub> Cl	0.610
10.0	NH <sub>3</sub> +NH <sub>4</sub> Cl	0.383

### Validity of Beer's Law:

This Ni(II)-HEAO complex in CHCl<sub>3</sub> obeys Beer's law up to 23.094 ppm of nickel. Beyond it absorbance plot dose not exhibit linearity. The average molar absorptivity of the complex obtained from the absorbance data is found to be  $7.9 \times 10^2 \text{ lit. mol}^{-1} \cdot \text{cm}^{-1}$  at 385 nm. Sandell's sensitivity is 0.0782  $\mu\text{g}/\text{cm}^2$ .

### Stoichiometry and Stability Constant of the Complex:

The stoichiometry of the Ni(II)-HEAO complex was studied by following two methods.

- 1) Job's method of continuous variation (10)
- 2) Yoe and Jone's mole ratio method(11)

All the two methods gave metal: ligand ratio. It was found to be 1:2.

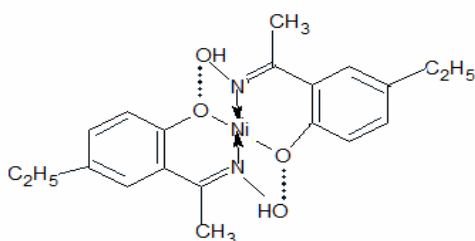
Gravimetric analysis also confirmed this ratio.

Table is given below.

**Table no.-2**

Ni(II) taken in g	Ni(II) complex in g	Ni(II) found in g	Error in g
0.00585	0.0411	0.00581	$-4 \times 10^{-5}$
0.01169	0.0825	0.01166	$-3 \times 10^{-5}$
0.01462	0.1029	0.01454	$-8 \times 10^{-5}$
0.02046	0.1449	0.02047	$+1 \times 10^{-5}$
0.02923	0.2062	0.02914	$-9 \times 10^{-5}$
0.03508	0.2475	0.03497	$-11 \times 10^{-5}$

Based on gravimetric analysis data, elemental analysis and IR spectrum results nickel complex with HEAO can be assigned the structure.



The stability constants for the complex for the different values on “n” were calculated. The stability constant is  $7.2 \times 10^8$ , the value of  $\Delta G$  is  $-12.076$  kcal/mole.

### Effect of Diverse Ion:

For the study of effect of foreign ions on the gravimetric determination of Ni(II), certain amount of various cations were added to a known amount of Ni(II) complex solution at pH 8.0. It was observed that Ca(II), Sr(II), Mg(II) do not interfere at this pH, but Cu(II), Mn(II) and Co(II) interfered seriously.

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