

### Quantitative Analysis of Nickel (II) in Micellar Media by Spectrophotometry

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#### Abstract

A spectrophotometry method for the determination of nickel (II) with nicotino hydroxamic acid (NHA) in the presence of Triton X-100 was studied at pH 9.0 using phosphate-borax buffer medium. Molar absorptivity of the sensitized complex was  $1.37 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$  at 530 nm and adhered to Beer's law in the concentration range 0.43-8.56  $\mu\text{g mL}^{-1}$  of nickel (II). The molar composition of the sensitized complex was 1:2 (nickel-NHA). The possible interfering effects of various ions were studied. The method was used for the determination of nickel in tap water and alloy samples.

**Keywords:** Nickel (II); Triton X-100; Nicotino hydroxamic acid; Alloy samples

#### Introduction

Nickel is biologically important, being an essential trace element in human diet. Since nickel is extensively used in the preparation of alloys and catalysts, its determination may be considered as interesting research activity. The determination of nickel in various samples in which it is found at low levels requires the use of sensitive and selective procedures. The obvious reasons for determining nickel by spectrophotometric method is due to its experimental simplicity, rapidity and the wide applicability of the procedure. Many different organic compounds have been used as spectrophotometric reagents for the determination of nickel (II) in aqueous solutions [1-5]. Addition of some surfactants and protective colloids to nickel (II) complexes have been used to enhance their spectral intensity [6,7]. The use of micellar systems for separation and pre-concentration has attracted considerable attention in the last few years, mainly because it is in agreement with the "green chemistry" principles. In continuation with our development of suitable methods for the determination of metal ions, especially in natural samples [8-15], this paper reports a simple, sensitive and highly selective spectrophotometry method for determination of nickel. The method is based on the complex formation of Ni (II) with new and novel chromogenic reagent nicotino hydroxamic acid (NHA) in Triton X-100 micellar media.

#### Experimental

##### Apparatus

All absorbance measurements were conducted on Sytonics UV-VIS spectrophotometer using 1 cm matched glass cells. A ELICO LI- 120 digital pH meter equipped with glass-calomel electrode was used for pH measurements and pH adjustment, respectively.

##### Reagents

All the reagents used were of analytical reagent grade unless otherwise stated and double distilled water was used throughout the

experiment. A 0.001M solution of the reagent nicotino hydroxamic acid (NHA) was prepared by the reported procedure [16]. Stock solution of Ni (II) ( $3.6 \times 10^{-3}\text{M}$ ) was prepared in bidistilled water and the metal solution was standardised by known methods [17] and further diluted as required. A buffer solution of pH 9.0 was prepared from borax and potassium hydrogen phosphate at appropriate concentration, 10% w/v sodium hydrogen carbonate and 1% Triton X-100 solution was prepared in distilled water. A 5% (w/v) solution of alkali metal salts and 0.2% (w/v) solution of different metal salts were used to study the interference of diverse ions.

##### General procedure

A 2.0 mL volume of buffer solution of pH 9.0, 2.0 mL of (0.001) NHA solution, 1.0 mL of 10% w/v sodium hydrogen carbonate solution, an appropriate volume of Ni (II) and 2.0 mL of Triton X-100 were added to a 25 mL standard flask. The solution of each flask was made to mark with distilled water and the absorbance of portion of each solution was measured at its respective absorption maximum, against a reagent blank prepared under the similar conditions.

#### Results and Discussion

##### Absorption spectra

The absorption spectra of grass green colored Ni (II)-NHA complex was recorded in the presence of surfactant, Triton X-100 against a reagent blank prepared under the similar conditions. The complex absorbs strongly at the absorption maxima (530 nm) in the presence of the surfactant (Figure 1). Therefore, 530 nm was chosen as the analytical wavelength to carry out the determination of the metal ion.

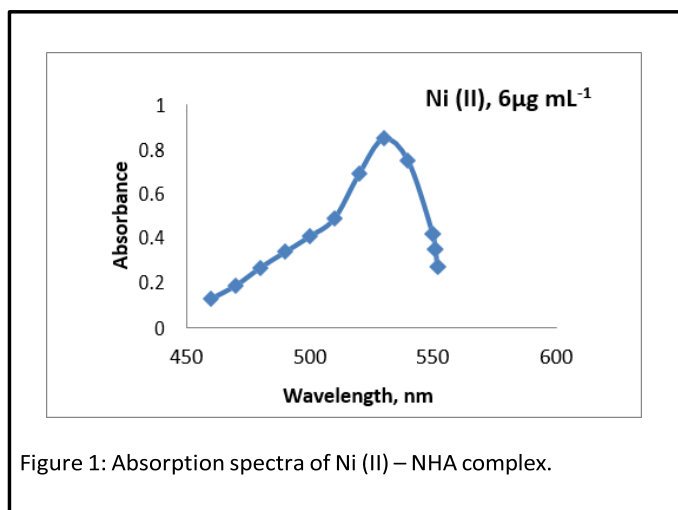


Figure 1: Absorption spectra of Ni (II) – NHA complex.

### Effect of pH

Absorbance for the complex was studied over a wide range of pH from 2.0-10.0. The studies showed that the absorbance was maximum in the pH range 8.5-9.5 (Figure 2). Hence, further studies were carried out at pH 9.0.

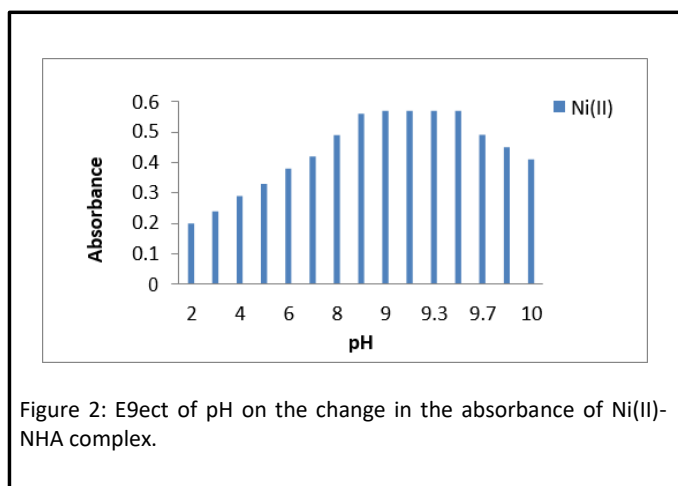


Figure 2: Effect of pH on the change in the absorbance of Ni(II)-NHA complex.

### Nature of surfactant

The effect of nature and concentration of different surfactants such as Sodium lauryl sulphate (SLS), Cetyltrimethylammonium bromide (CTAB), Cetylpyridinium bromide (CPB), Triton X-100, Tween-80, and Tween-20 on the absorbance of Ni(II)-NHA complex was studied and it was observed that complex formation is faster, stable and maximum in presence of Triton X-100, so it was selected as micellizing agent for further studies. The effect of concentration of Triton X-100 on sensitivity of the method was studied by varying its percent concentration from 0.5-3.0% (w/v) and its volume from 0.5-3.5 mL. The maximum absorbance was found with 1.5-2.0 mL of 1% Triton X-100. Hence 2.0 mL of 1% Triton X-100 was used for further studies.

### Statistical analysis of results

Under the optimum reaction conditions for the spectrophotometric determination of Ni(II) by chelation with NHA, obedience to Beer's

law was tested. On plotting the absorbance as a function of Ni(II) concentration, straight line was obtained from 1.71-8.56  $\mu\text{g mL}^{-1}$  using NHA in presence of borate buffer (Figure 3). Important analytical parameters of Ni(II) and NHA are incorporated in Table 1. The apparent molar absorptivity was  $1.371 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$  whereas the Sandell's sensitivity was  $0.538 \mu\text{g/mL/cm}^2$ . The high value of correlation coefficient and small value of standard deviation indicate the good linearity of calibration graph and the confirmatory of Beer's law to absorbance measurements.

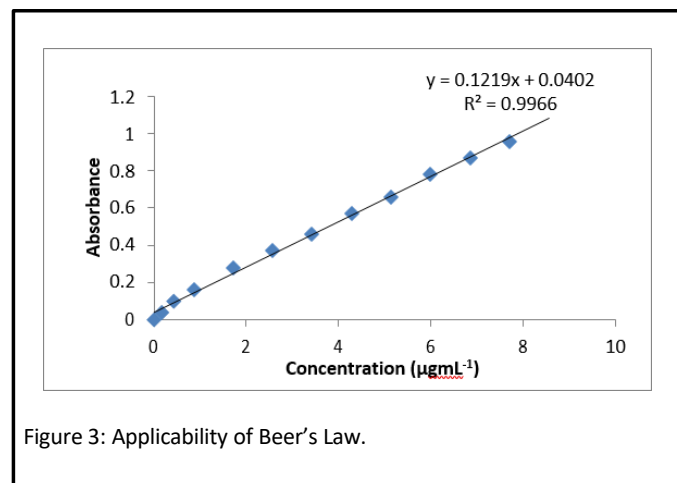


Figure 3: Applicability of Beer's Law.

Characteristics	Results
Colour	Grass green
$\lambda_{\text{max}}$ (nm)	530
pH range (optimum)	8.5-9.5
Mole of reagent required per mole of metal ion for full colour development	10 - folds
Molar absorptivity ( $\text{L mol}^{-1} \text{ cm}^{-1}$ )	$1.371 \times 10^4$
Sandell's sensitivity ( $\mu\text{g/mL/cm}^2$ )	0.538
Beer's law validity range ( $\mu\text{g/mL}$ )	2.57-6.85
Optimum concentration range ( $\mu\text{g/mL}$ )	1.74 - 8.56
Composition of complex (M:L) obtained in Job's and mole ratio method	1:2
correlation coefficient ( $\gamma$ )	0.996
Standard deviation (%)	0.11

Table 1: Analytical Parameters of [Ni(II)-NHA] complex.

### Job's method of continuous variation

A Series of solutions of varying mole fractions of Ni(II) and NHA were prepared keeping the total volume constant. The test tubes were shaken well and the absorbance values in each case were measured at 530 nm. A graph was plotted between absorption and mole fraction of the ligand and from the graph it is observed that two moles of nickel chelates with 4 moles of NHA indicating that the compositions of the complex as 1:2 in Ni-NHA complex.

## Interference analysis

A systematic study of the foreign ions in the determination of Ni (II) has been made under the experimental conditions. An ion was considered to interfere when its presence produced a variation in the absorbance of the sample greater than 5%. The data reveals that all the anions except EDTA did not interfere in the determination of Ni (II) even in 200 fold excess. Most of the cations do not interfere in the determination even in 100 fold excess. Fe(III) and Cu(II), Co(II) were tolerated in 40 fold and 20 fold excess in presence of phosphate, thiourea and thioyanate respectively.

## Applications

In order to test the reliability of the proposed method it was applied to the determination of Ni(II) in tap water and alloy samples.

### Preparation and analysis of water samples

Two tap water samples, municipal water and ground water were collected inside the College campus. The water samples were analyzed after filtering through Whatman filter paper. The samples were spiked with Ni(II) in order to validate the procedure. The results are presented in Table 2. It can be seen that the recovery of spiked samples is good. The results indicate that the proposed method is applicable for the determination of Ni(II) in real samples.

### Preparation and analysis of alloy samples

A 0.1 g of the samples are dissolved in a mixture of 2 mL HCl and 10 mL HNO<sub>3</sub>. The resulting solution is evaporated to a small volume. To this 5 mL of 1:1 H<sub>2</sub>O:H<sub>2</sub>SO<sub>4</sub>, mixture is added and evaporated to dryness. The residue is dissolved in 15 mL of distilled water and filtered through Whatman filter paper No.41, the filtrate is collected in a 100 mL volumetric flask and made up to the mark with distilled water. The solution is further diluted as required. Then a known aliquot of the sample was analysed by the general procedure. The results are presented in Table 3. It can be seen that the recovery of the alloy samples is good. The results indicate that the proposed method is applicable for the determination of Ni(II) in alloy samples.

Sample	Nickel (II) Concentration, $\mu\text{g mL}^{-1}$		Error(%)
	Spiked	Found	
Tap Water 1	2.57	2.60	101.2
Tap Water 2	1.71	1.70	99.4

Table 2: Determination of nickel in tap water samples.

Sample	Certified composition (%)	Amount of nickel (%)		Relative Error (%)
		Present	Found*	
Copper-nickel alloy	Cu 67; Fe 0.83; Mn 0.08; Si 0.29; Ni 31.2	31.20	31.32	+0.38
NTPC Ball bearing	Fe 65; Cr 15; Cu 4.5; Mn 2; Ni 10.00	10.00	10.04	+0.46

Table 3: Determination of nickel in alloy samples.

## Conclusion

The proposed method offers significant advantages over conventional methods because of its speed and ease of operation. The proposed method is simple, rapid, safe, easy to use and inexpensive. In comparison with solvent extraction methods, this procedure employs only a small amount of surfactant which from the perspective of green chemistry is environment friendly. The method gives a very low limit of detection and therefore can be applied to the determination of Ni(II) in various water and alloy samples.

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