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Development of Method for Extractive Spectrophotometric Determination of Ni (II) of 2-{4-(1H Benzimidazole-2-Yl) Phenyl Imino}-2-Hydroxy-4-Methoxy Benzaldehyde as an Analytical Reagent

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Abstract: A spectrophotometric method has been developed for the determination of Ni (II) using 2-{4-(1H benzoimidazole-2-yl) phenyl imino}-2-hydroxy-4-methoxy benzaldehyde (BPIHMB) as an extractive reagent. The reagent forms a coloured complex, which has been quantitatively extracted into *n*-butanol at pH-8.5. The method obeys Beer's law over a range from 1 to 10 ppm. The maximum wavelength for the reagent is 369nm. The Molar absorptivity and Sandell's sensitivity calculated were $0.1186 \times 10^4 \text{ LMol}^{-1}\text{cm}^{-1}$ and $0.13619 \mu\text{gcm}^{-2}$ respectively. The proposed method is very sensitive and selective.

Keywords: 2-{4-(1H benzoimidazole-2-yl) phenyl imino}-2-hydroxy-4-methoxy Benzaldehyde, Nickel, Spectrophotometric determination, *n*-butanol.

I. INTRODUCTION

Nickel is obtained from ores like pentlandite, garnierite and limonite. It is also found in meteorite. Pure nickel is very rarely found on earth. It plays a vital role in many fields such as industry, laboratory, medicine, food, surgical, computer component, dental prostheses and for constituent of alloy like stainless steel. It is also used for making turbine blades and nickel ferrites are used for making transformer. More than 65% of nickel produce is used for making stainless steel. Nickel has five stable isotopes and in which most common isotopes is nickel-58 and nickel-48 shows magic no of both protons 20 and neutron 28 in their nuclei which give enhanced stability.

Nickel works as a metal as resistant to moisture and air. It is not considered as a metal of biological importance until 1975, Zerner discovered the urease was a nickel enzyme [1]. Nickel plays a very important role in the biological. It is reportedly required for growth of bacterium (*Alcaligenes*), (*hydrogenomonas*) *eutrophus*. (Ross M. Welch). Nickel is very important for plant life. Some plants and microorganism required amount of nickel as a nutrient [2]. Nickel cannot avoid completely it is essential for our diet [3]. Some time it can cause skin disorder known as nickel-eczema [4]. Therefore, it is important to develop simple, sensitive methods for development of Ni (II) at trace levels.

Various reagents [5] such as hydrazane [6], imine [7], dimethyl glyoxime, dithizone and sodium-diethyldithiocarbamate etc have been used for the determination of nickel [8,9]. But these methods undergo from limitations such as requirement of masking agent [10], interference of ion, equilibrium for some ions, equilibrium for superior in sensitivity and selectivity to those reported in the literature is developed for extractive spectrophotometric determination of nickel with BPIHMB. The proposed method is free from limitation.

In these present investigations a novel BPIHMB is Schiff base derived from 2-hydroxy-4-methoxy benzaldehyde. Schiff bases are known to form stable complex with metal ion [11,12]. However, the use of Schiff base as extractant in liquid extraction is rare [13,14]. Solvent extraction is one of the most versatile methods of separation.

Solvent extraction preferred over other separation methods due to its simplicity, selectivity, and rapidity, low cost and moderate requirement. It will be applied for the determination of Ni (II) at trace level in synthesis mixture and alloys. We describe the spectrophotometric determination of Ni (II) using 2-{4-(1H benzoimidazole-2-yl) phenyl imino}-2-hydroxy-4-methoxy benzaldehyde as an extractive reagent.

II. EXPERIMENTAL

The reagent 2-{4-(1H benzoimidazole-2-yl) phenyl imino}-2-hydroxy-4-methoxy benzaldehyde(BPIHMB) was prepared by the given procedure. The stock solution of Nickel (II) was prepared by dissolving a weight amount of its nitrate in double distilled water containing dilute sulphuric acid, which was diluted to the desired volume with double distilled water and standardized by diethyldithiocarbamate method. Absorbance and pH measurement were carried out on a Shimadzu UV- Visible 2100 spectrophotometer with 1cm quartz cells and digital pH meter with combined glass electrode respectively.

A. Procedure for the Extraction

1.ml of aqueous solution containing 0.1 mg of nickel metal and 1 ml of reagent were mixed in 50 ml beaker. The pH of the solution adjusted to 8.5 keeping the volume 10 ml. the solution was transferred to 100 ml separately funnel. The beaker was washed twice with n-butanol and transferred to the same funnel. The two phases were shaken for two minutes and allowed to separate. The organic phase was collected in 10 ml measuring flask and made up to the mark with organic solvent if required. The amount of nickel present in the organic phase determine quantitatively by spectrophotometric method by taking absorbance at 369 nm and in aqueous phase was determined by dimethylglyoxime.

III.RESULT AND DISCUSSION

The reagent BPIHMB forms reddish yellow coloured complex with Ni (II), which was extracted into organic phase. The extraction of Ni (II) forms aqueous phase by BPIHMB in n-butanol is studied over a wide range experimental condition. The results of various studies are discussed below.

A. Extraction as a Function of pH

The extraction of Nickel with 2- {4-(1H benzoimidazole-2-yl) phenyl imino}-2-hydroxy-4-methoxy benzaldehyde has been studied over the pH range 1- 10 and was observed that percentage extraction of Ni (II) is maximum at pH 8.5.

B. Absorption Spectrum

The absorption spectrum of Ni (II): 2- {4-(1H benzoimidazole-2-yl) phenyl imino}-2-hydroxy-4-methoxy benzaldehyde in n-butanol shows the maximum absorption at 369 nm. The absorption due to reagent at this wavelength is nearly negligible. Hence the absorption measurements were carried out at 369 nm.

C. Influence of Diluents

The suitability of diluents was investigated using organic solvents such as chloroform, ethyl acetate, pentane, toluene, n-butanol, xylene, and hexane. The extraction of Ni (II) was quantitative with BPIHMB in n- butanol. Hence, n-butanol was used for further extraction studies as it gave better and quicker phase separation.

D. Effect of Salting out Agent

The presence of 0.1M salts of various alkali and alkaline metals does not show any effect over the absorbance value of Ni (II): 2-{4-(1H benzoimidazole-2-yl) phenyl imino}-2-hydroxy-4-methoxy benzaldehyde complex extract. Therefore, no salting out agent was required during the extraction.

E. Effect of Reagent Concentration

Various volumes of 0.1% reagent solution were added to the sample solution containing 100 μ g of nickel at respective pH values. The absorbance remained nearly constant when the volume of the reagent solution used was more than 1 ml. Therefore, 1 mL of 0.1 % reagent was chosen for the quantitative determination of the metal.

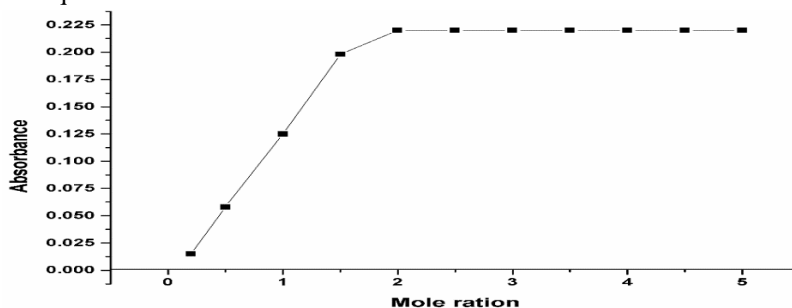


Fig. 1 Reagent concentration

F. Effect of Equilibrium time and Stability of the complex

The study of change in absorbance with variation in equilibrium time extraction of the complex into organic solvent shows that equilibrium time of 60 sec. are sufficient for the quantitative extraction of nickel. The study of stability of colour of the Ni (II): BPIHMB complex with respect to time shows that the absorbance due to extracted species is stable up to 48 hours, after which slight decrease in absorbance is observed. Throughout the experimental work, for practical convenience, the measurements have been carried out within one hour of extraction of nickel.

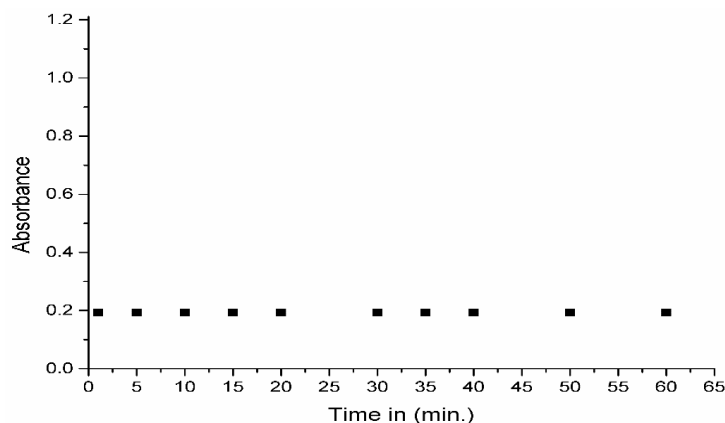


Fig.2 Effect of time

G. Calibration plot

A calibration plot of absorbance against varying nickel concentration and fixed BPIHMB concentration gives linear and reproducible graph in the concentration range 1 to 10 ppm of nickel. This shows that the Beer's law is obeyed in this range. The Molar absorptivity and Sandell sensitivity were calculated to be $0.1186 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$ and $0.22163 \mu\text{g}/\text{cm}^{-2}$ respectively.

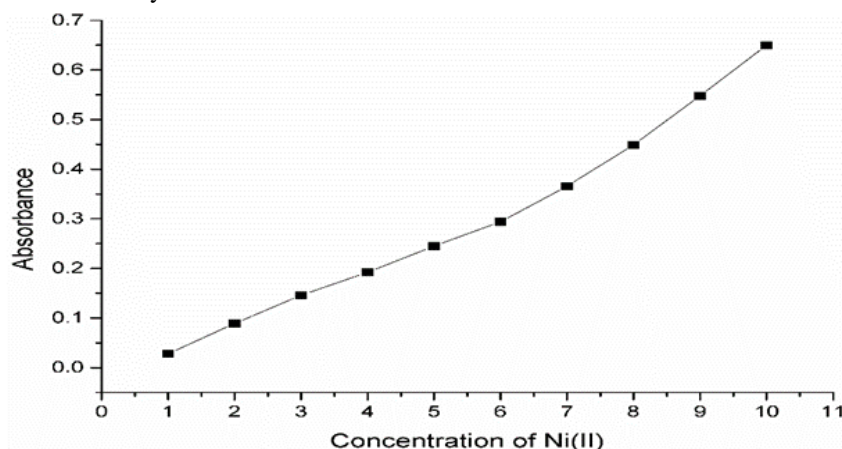


Fig.3 Calibration plot of Ni (II) with BPIHMB

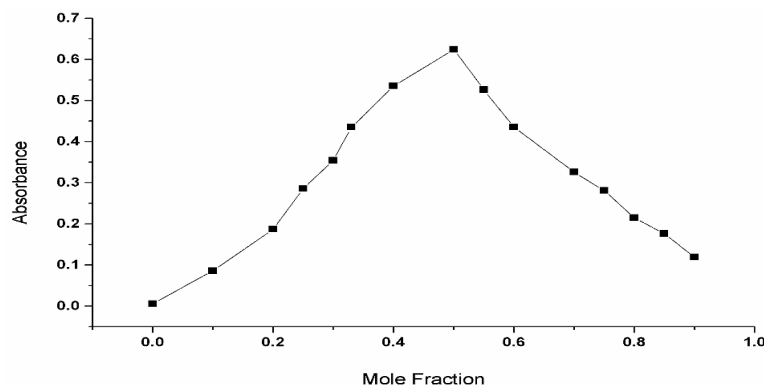


Fig.4 Job's continuous variation method

H. Nature of Extracted Species

The composition of extracted species has been determined by Job's continuous variation method, Slope ratio method and Mole ratio method. It shows that the composition of Ni (II): BPIHMB complex is 1:2.

I. Effect of Divalent ions and Foreign Ions

The effect of other ions presents in various amount indicated no interference in the spectrophotometric determination of 50 µg of nickel. The ions which show interference in the spectrophotometric determination of nickel were overcome by using appropriate masking agents (Table 1).

TABLE I
EFFECT OF DIVALENT IONS AND FOREIGN IONS

Sr. No.	Ion	Amount added in µg	Absorbance
1	Zn ²⁺	10.0	0.215
2	Ba ²⁺	9.0	0.215
3	Ca ²⁺	8.0	0.215
4	Ni ²⁺	12.0	0.215
5	Fe ²⁺	13.0	0.215
6	Pb ²⁺	16.0	0.215
7	K ²⁺	20.0	0.215
8	Mg ²⁺	14.0	0.215
9	Co ²⁺	10.0	0.215
10	Na ⁺	14.0	0.215
11	Mo(II)	16.0	0.215
12	Mn(II)	9.0	0.215

IV. PRECISION AND ACCURACY

The precision and accuracy of the developed spectrophotometric method has been studied by analysing five solutions each containing 60 µg of nickel in the aqueous phase. The average of five determinations was 60.04 and variation from mean at 60 confidence limits was ± 0.13345.

V. APPLICATIONS

The proposed method was successfully applied for the determination of nickel from various commercial mixture and synthetic mixtures. The results found to be in good agreement with those obtained by the standard known method. (Table 2).

TABLE II
DETERMINATION OF Ni (II) USING BPIHMB FROM DIFFERENT SAMPLES

Sr. No.	Sample	Amount of Ni (II)	
		Standard method	Present method
1	SYNTHETIC MIXTURE		
	Ni (5) + Fe (5)	4.99	4.90
	Cu (5) + Ni (5)	4.95	4.90
	Ni (4) + Zn (4)	3.97	3.95
2	Alloy sample		
	Copper-nickel alloy	5.15	5.12
	Nickel base super alloy	54.9	54.1
	Magnesium alloy	0.051	0.049

VI. CONCLUSIONS

The proposed method is highly sensitive and selective than the other reported methods for extractive spectrophotometric determination of microgram amounts of nickel. It offers advantages like reliability and reproducibility in addition to its simplicity, instant colour development and suffers from less interference. It has been successfully applied to the determination of nickel at trace level in synthetic mixtures and commercial mixture.

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