Role of [N - (O-Hydroxy Benzylidene) Pyridine -2-Amine] (NOHBPA) in Extractive Spectrophotometric Determination of Nickel (II)

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Abstract

Determination of Ni (II) using NOHBPA reagent with the help of very simple, sensitive and rapid spectrophotometric method has been developed. Elemental analysis technique was used for characterization of NOHBPA. Carbon tetrachloride was used for quantitative (99.4%) extraction of Ni (II) with the help of NOHBPA at pH range 7.4 to 8.6 from aqueous solution. An intense peak of λ max (500 nm) was observed from carbon tetrachloride extract. Concentration range for Beers law was observed between 0.1 to 0.5 µg/ml for Ni (II).

For Ni, NOHBPA complex molar absorptivity and Sandell's sensitivity were found to be 1294 Lmole^{-1}cm^{-1} and 0.00435µgcm^{-2} respectively. Job's Continuous Variation and Mole Ratio Method confirm complex nature 1:2 (Ni: NOHBPA). During present study interference of various ions was also discussed. Determination of Ni (II) in alloy was successfully carried out in present method.

Keywords: NOHBPA, Nickel (II), Spectrophotometry, Solvent Extraction, Alloy.

Introduction

Occurrence of Ni in food and water is increased by the human pollution[1]. Along with mining and smelting, nickel plate article, nickel pigmented dishes are especially responsible for water and soil contamination[2]. Tobacco, shampoos, detergents, jewelry and coins are the direct source of absorption of Ni in human body through skin contact[3]. Nickel can be eliminated through body by urine, but larger dose of nickel is carcinogenic as well as toxic[4].

Solvent extraction is one of the important techniques of separation. Because of simple procedures, solvent extraction is used for separation of metal ions at trace level[5,6]. Solvent extraction technique along with spectrophotometer plays an important role in the pharmaceutical science[7]. Spectrophotometric determination of Ni (II) can be done by various reported methods[8-18]. In the present study, we report the spectrophotometric determination of Ni (II) by using [N-(O-Hydroxy Benzylidene) pyridine -2-Amine] (NOHBPA)

Material and Methods

Synthesis of [N - (o - hydroxy benzylidene) pyridine - 2-amine] (NOHBPA): NOHBPA was prepared as shown in figure 1 by refluxing an equimolar quantities of ethanolic solution of 2-aminopyridine and o-hydroxy benzaldehyde for 6 hrs. Yellow crystal was separated out after cooling of reaction mixture (yield 82%, m.p.60^0-62^{0}C)[19]. Aqueous ethanol was used for purification of NOHBPA by recrystallization as per reported method[20]. Spectral and elemental analysis were used for the characterization of the product.

Stock solution preparation: Stock solution of Ni (II) was prepared by dissolving nickel sulphate in water having dil. H_2SO_4. Standardization was done by reported dimethylglyoxime method[7]. Working solution of Ni (II) was prepared by dilution methods. Double distilled water and AR grade reagents were used for preparations of all solutions.

Procedure for the extractive spectrophotometric determination of Ni (II): 3.0 ml of sodium bicarbonate buffer (pH 8.2) and 2% of 2.0 ml DMF solution of NOHBPA were added in to 1.0 to 40 µg of Ni (II) aqueous solution aliquot. Distilled water was used to make up volume up to 10 ml. Resulting solution was then digested for 15 min. on boiling water bath, it was cooled and transferred to 125 ml separating funnel.

Two phases were then separated. Organic layer of chloroform were then diluted upto 10 ml with the help of carbon tetrachloride if necessary.

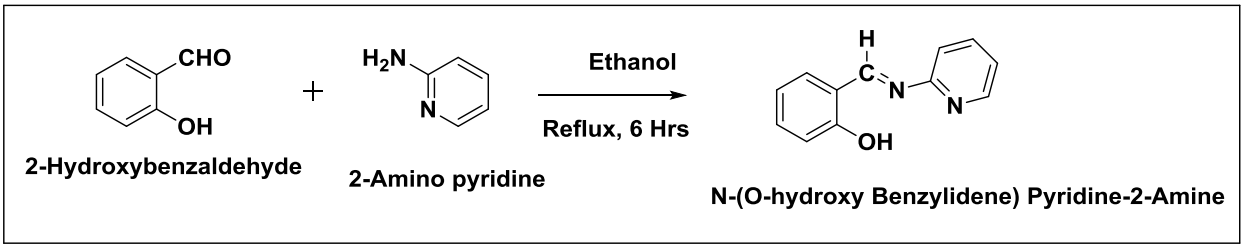


Figure 1: Synthesis of N-(o-hydroxy benzylidene) 2-aminopyridine (NOHBPA)

Absorbance was recorded at 500 nm by using blank solution. With the help of absorbance, amount of Ni (II) was determined. Foreign ions study was carried out by adjustment of pH before extraction.

Procedure for the determination of Nickel (II) in alloy sample- (Nickel-Aluminum based alloy): 0.1 to 0.2 g of nickel sample were dissolved in 10 ml of aquaregia. Residue obtained upon dryness of the solution was dissolved in 1N HCl. After filtration resulting solution was diluted upto 100 ml with distilled water. Ni (II) analysis was performed by using 1ml aliquot of the solution as described in the earlier procedure.

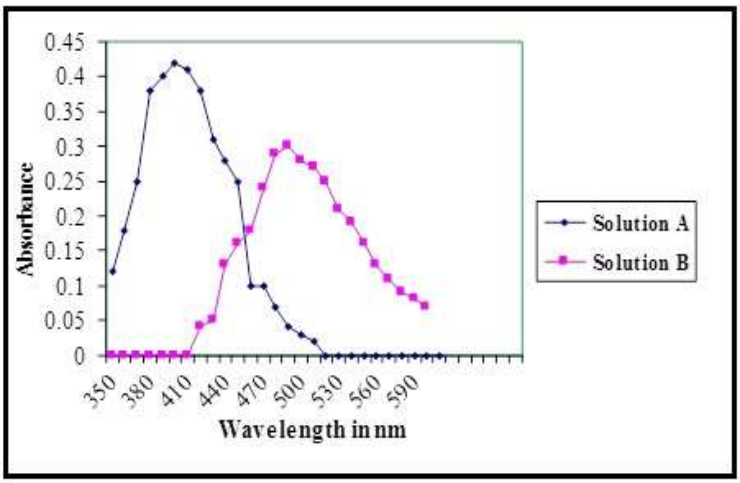
Results and Discussion

NOHBPA was able to extract Ni (II) quantitatively (99.35%) into carbon tetrachloride by using sodium bicarbonate buffer extraction from an aqueous solution of pH 7.4 to 8.6.

Extraction of Ni(II) was done in organic solvents. The values of extraction coefficient were in the order carbon tetrachloride > ethyl acetate> chloroform > n-butanol > chlorobenzene > bromobenzene > benzene > xylene as described in table 1. Due to the highest extraction coefficient, carbon tetrachloride was the choice of solvent for extraction.

Carbon tetrachloride extract of Ni: NOHBPA complex shows an intense peak at 500 nm as shown in figure 2. The absorbance due to the reagent is negligible at this wavelength, (wavelength for reagent = 380). Hence the entire experiment was done at wavelength 500 nm.

Calibration curve was plotted as shown in figure 3 between absorbance values and concentration of Ni -complex. Calibration curve is linear over concentration range of 0.5 to 4.5 µg/ml. The molar absorptivity was observed to be 1294 L mol^{-1} cm^{-1}. 2.0% DMF solution (2 ml) of NOHBPA was sufficient to extract 50µg of Ni (II). It was found that color of the chloroform extract stable for at least 24 hrs at room temperature.



Solution A: Absorbance spectra of NOHBPA
Solution B: Absorbance spectra of Ni–NOHBPA Complex
Figure 2: Absorbance spectra of NOHBPA and Ni–NOHBPA complex

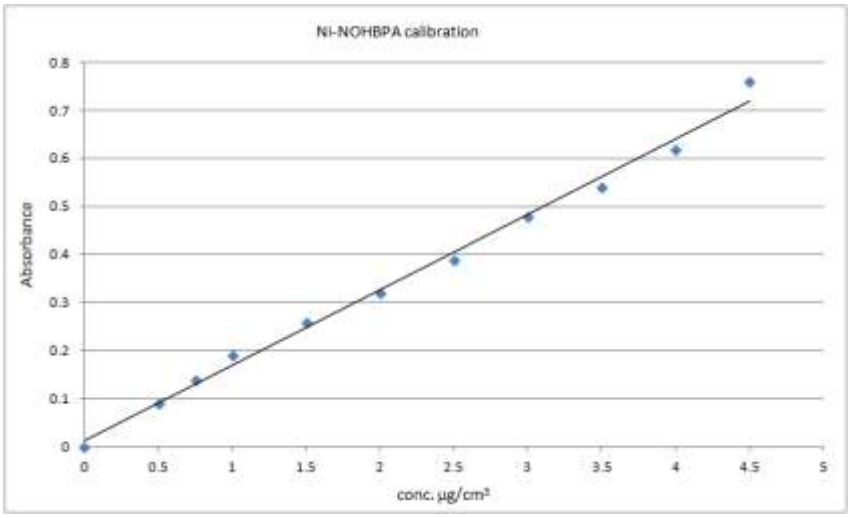


Figure 3: Calibration curve of Ni–NOHBPA complex

Table 1
List of solvents used for solvent extraction

Solvent	Percentage of extraction coefficient
Carbon tetrachloride	99.4
Ethyl acetate	95.1
Chloroform	93.8
n-butanol	79.5
Chlorobenzene	73.5
Bromobenzene	70.3
Benzene	44.4
Xylene	39.4

Effect of other ions: Study of interference of ions was done. Ni (II) (20µg) was used. It was found that 10 mg each of Li(I), Ba(II), Hg(II), Sn(II), Sr(II), Zr(II), Ca(II), Zn(II), V(V), Mg(II), Cd(II) and Mo(IV), 5 mg each of Th (IV), Ce (IV) and W (VI) and 0.1mg each of Ru (III), Rh (III) and Pt (IV) did not interfere in Ni (II) study. 20 mg each of sulphate, sulphide, nitrate, nitrite, chloride, bromide, iodide, fluoride, phosphate, citrate, triethanol amine, thiocynate, acetate and 5-sulphosalicylic acid also did not interfere.

Appropriate masking agent was used to remove the interference by the various ions as shown in table 2.

Composition of the extracted complex: Carbon tetrachloride extract of Ni: NOHBPA complex was subjected to Job's continuous variation and Mole ratio method. It was found that composition of the extracted complex was 1:2 (Ni: NOHBPA).as shown in figure 4 and 5. Hence formula of the complex was Ni $(NOHBPA)_2$.

Accuracy, precision, sensitivity and applications of method: In order to determine the accuracy and precision of the proposed method, the experiment was repeated ten times. 20 µg of Ni (II) in 10 cm^3 solutions was taken. The average of 10 determination of 20 µg of Ni (II) in 10 cm^3 solutions was 19.89 µg, which varied between 19.72 and 20.19 at 95% confidence limit.

Sandell's sensitivity and standard deviation are 0.0425 $\mu g cm^{-2}$ and ±0.368 respectively. Ni (II) determination in alloy samples is carried out and the results were compared with dimethyl glyoxime method[3] for Ni (II) as shown in table 3.

Table 2
Interference by various ions

S.N.	Amount of interfering ions added in mg	Masking agent added 1ml of 0.5 M solution
1.	10 mg of Cr(III)	Sodium fluoride
2.	10 mg of Ag(I)	Pottasium thiocynate
3	10 mg of Cu(II)	Sodium dihydrogen phosphate
4.	10 mg of Fe(II) & Fe(III)	Triethanol amine

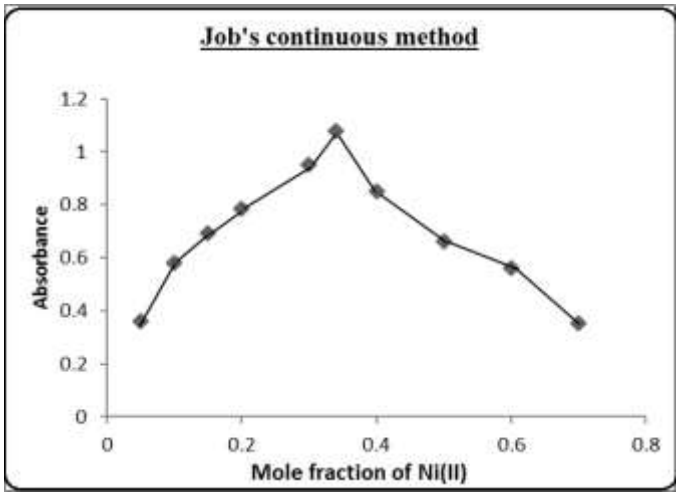


Figure 4: Composition of (Ni: NOHBPA) complex by Job's continuous method.

Table 3
Determination of Ni (II) in alloy sample

Percentage of Ni (II)	Alloy Sample (Nickel-Aluminum based alloy)	
	BAS 20 (Nickel -1.90%)	BAS 85 (Nickel -0.92%)
Proposed method	1.90*	1.91
Dimethyl glyoxime method	0.91*	0.93

***** Average of three determinations

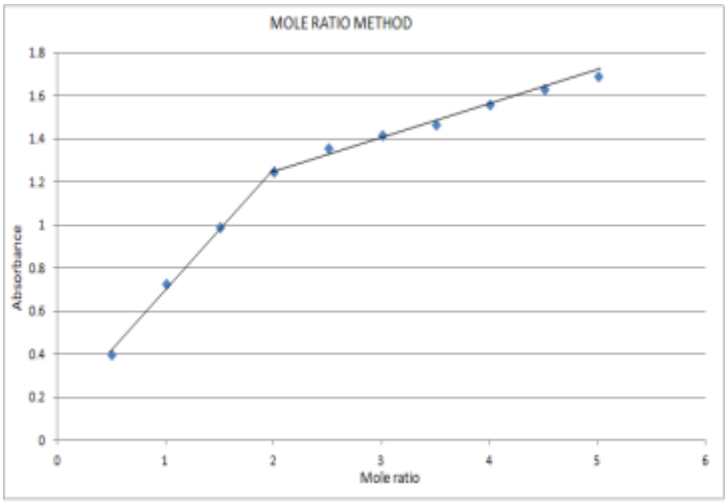


Figure 5: Composition of (Ni: NOHBPA) complex by Mole Ratio methods.

Conclusion

The method was successfully utilized for extractive spectrophotometric determination of Ni (II). Extraction of Ni (II) in aqueous phase was easily carried out by forming complex with NOHBPA. This method is reliable and simple. Experimental conditions for maximum extraction were determined and applied for the analysis of samples.

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