Available online www.jocpr.com

Journal of Chemical and Pharmaceutical Research, 2015, 7(4):1069-1073

Research Article

ISSN : 0975-7384
CODEN(USA) : JCPRC5

Development of extractive spectrophotometric method for the determination of nickel (II) with Schiff base 2-[(2-hydroxyphenylimino)methyl]-4-nitrophenol

Datta B. Mandhare and Vasant D. Barhate *

V. E. S. College of Art, Science and Commerce, Sindhi Society, Chembur, Mumbai, India

---

ABSTRACT

A simple spectrophotometric method has been developed for the determination of Nickel (II) by using Schiff base 2-[(2-hydroxyphenylimino) methyl]-4-nitrophenol [HPIMNP]. HPIMNP extracts Ni (II) quantitatively (99.92%) into n-Butyl alcohol from an aqueous solution of pH range 7.8 - 8.8 in the presence of 2 ml of 5 M solution of Ammonium chloride. The n- Butyl alcohol extract shows maximum absorption at 480 nm (λ max). Beer's law is obeyed over the Ni (II) concentration range 0.2-20.0 µg/ml. The molar absorptivity and sandell's sensitivity of Nickel- HPIMNP system is 882.35 L mole-^{1} cm^{-1} and 0.067 µg.cm-^{2} respectively. The composition of extracted species is found to be 1:2 (Ni: HPIMNP) by Job's continuous variation and Mole ratio method. Interference by various ions has been studied. The proposed method had been applied for determination of Ni (II) in alloy samples.

Key words: Extractive Spectrophotometry, Schiff base, Nickel (II), 2-[(2-hydroxyphenylimino) methyl]-4-nitrophenol [HPIMNP], Alloy samples

---

INTRODUCTION

Various reagents [1] are available for the spectrophotometric determination of Nickel (II) of which Oximes, Schiff bases and it derivatives constitutes an important class [2] [3]. Synthesis and Antimicrobial Activity of Schiff base 2-[(2-hydroxyphenylimino) methyl]-4-nitrophenol [HPIMNP] has been reported [4]. However Analytical application of HPIMNP was not studied. In the present communication, we describe the extractive spectrophotometric determination of Nickel (II) with 2-[(2-hydroxyphenylimino) methyl]-4-nitrophenol [HPIMNP].

EXPERIMENTAL SECTION

ELICO - SL 159 spectrophotometer with optically matched quartz or glass cells of 1cm path length were used for absorbance measurement. An ELICO LI 127 pH meter was employed for pH measurements. The reagent HPIMNP was synthesized by condensation of 5-Nitro salicylaldehyde with 2 - amino phenol as per reported procedure [4]. The resulting product was recrystallized by using ethanol [5] and characterized by elemental and spectral analysis. Its 0.5 % solution was prepared in dimethylformamide (DMF). A stock solution of Ni (II) was prepared by dissolving Nickel sulphate in double distilled water containing dilute sulphuric acid. It was standardized by Dimethyl glyoxime method [6]. Working solutions of Ni (II) were made by suitable dilution. All other reagents used were of AR grade and all the solutions were prepared in doubly distilled water.

Extraction and separation of Nickel (II)

To an aliquot of aqueous solution containing 500 µg of Ni (II) and 2ml of 0.5 % solution of HPIMNP prepared in DMF were mixed in 25 ml beaker. The pH of solution was adjusted to desired value with dilute solution of HCl/ NaOH, Keeping the total volume to 10 ml with distilled water. The resulting solution was then transferred into 125 ml separatory funnel. The beaker was then washed with 5 ml portion of organic solvent twice and each washing was added to the solution in separatory funnel. The two phases were equilibrated for 30 seconds and allowed to separate. After the separation of two phases, pH of the equilibrated aqueous phase was measured and Nickel content in each phase was determined by Dimethyl glyoxime method [6]. The extraction was carried out with different solvents to find out the best extracting solvent. On the basis of Nickel content in aqueous and organic phase, extraction coefficient and percent extraction was calculated.

Extractive Spectrophotometric Determination of Ni (II):

To an aliquot of aqueous solution containing 2-200 µg of Ni (II), 2 ml of ammonium acetate and ammonium hydroxide buffer solution of pH 8.3, 2ml of 5M Ammonium chloride and 1ml of 0.5 % solution of HPIMNP prepared in DMF were added. The volume of solution was made up to 10 ml with distilled water. The solution was equilibrated for 30 seconds with 10 ml of n- butyl alcohol and the phases were allowed to separate. The n- butyl alcohol extract was collected in a 10 ml measuring flask and made up to mark with n- butyl alcohol. The absorbance of n- butyl alcohol extract was measured at 480 nm against a reagent blank prepared under identical conditions. The Ni (II) content of the sample solution was determined from calibration curve. To study the effect of other ions, the respective foreign ions were added to aqueous phase before the extraction and adjustment of pH.

Determination of Ni (II) in alloy sample

Accurately Weighed 0.2 - 0.4 gm alloy sample was dissolved in boiling with 10 ml aquaregia. The resulting solution was evaporated to almost dryness. The residue was then dissolved in 10 ml of 1 M HCl, filter if required and resulting solution was diluted to 100 ml with doubly distilled water. To an aliquot of this solution 1 ml was analyzed for Ni (II) by the procedure as described earlier.

RESULTS AND DISCUSSION

Nickel (II) could be extracted quantitatively (99.92%) by HPIMNP into n- butyl alcohol from an aqueous solution at pH 8.3 in the presence of 2ml of 5M ammonium chloride solution. Organic solvents used for extraction of Ni (II) can be arranged on the basis of their extraction coefficient values as n- butyl alcohol > n-amyl alcohol > benzyl alcohol > benzene > nitro benzene > xylene > toluene > ethyl acetate > chloroform > carbon tetrachloride. N-butyl alcohol was found to be the best extracting solvent; hence, it was selected for extraction throughout the work.

The n-butyl alcohol extract of Ni- HPIMNP complex showed an intense peak at 480 nm. The absorbance due to the reagent is negligible at this wavelength, so the absorption measurements were taken at this wavelength. The result shows that the system confirmed to Beer's law at this wavelength over a Nickel concentration range 0.2 to 20 µg/ml (Fig- 1). The molar absorptivity and sandell's sensitivity of the extracted complex on the basis of Ni (II) content were calculated to be 882.35 L mol $^{-1}$ cm^{-1} and 0.067 µg. cm^{-2} respectively. It was found that 1 ml of 0.5% DMF solution of HPIMNP was sufficient to extract 200 µg of Ni (II). The colour of the n-butyl alcohol extract was found to be stable at least 24 hrs at room temperature.

EFFECT OF OTHER IONS

Ni (II) (100 µg) was determined in the presence of various ions. The following ions in the amount indicated, did not interfere in the spectrophotometric determination of Ni (II) (100 µg) : 10 mg each of, Li (I), Be (II), Ba (II), Ca (II), Sr (II), Al (III), Ti (III), V (V), Mo (VI), U (VI), Ru (III), Pt (IV) and Rh (III). And 20 mg each of chloride, bromide, iodide, fluoride, chlorate, bromate, iodate, sulphide, phosphates, tartrate, acetate, citrate and thiosulphate, thiocyanide, triethanol amine, ascorbic acid. Interference due to iron removed before the extraction by conventional method [6]. Interference by various ions was removed by using appropriate masking agent (Table 1).

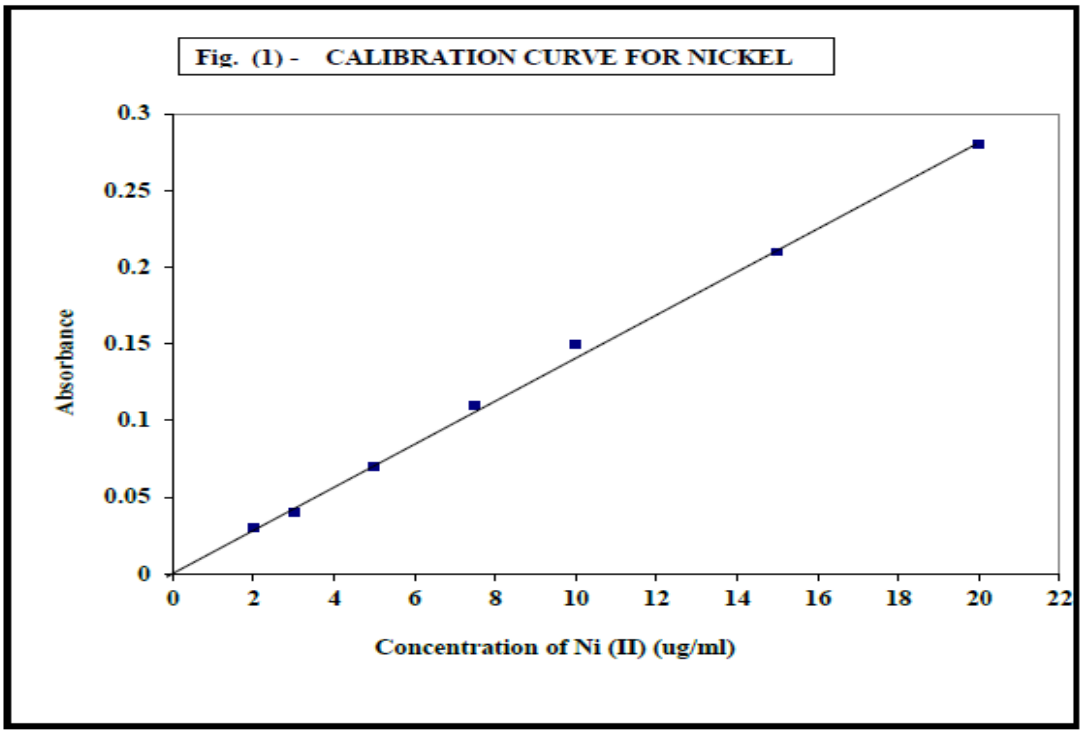
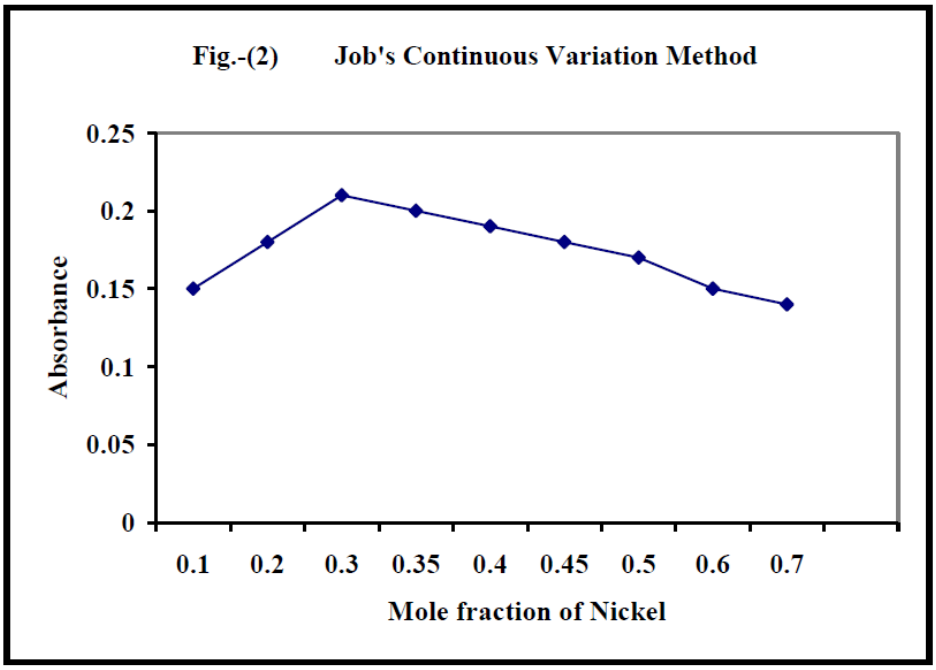


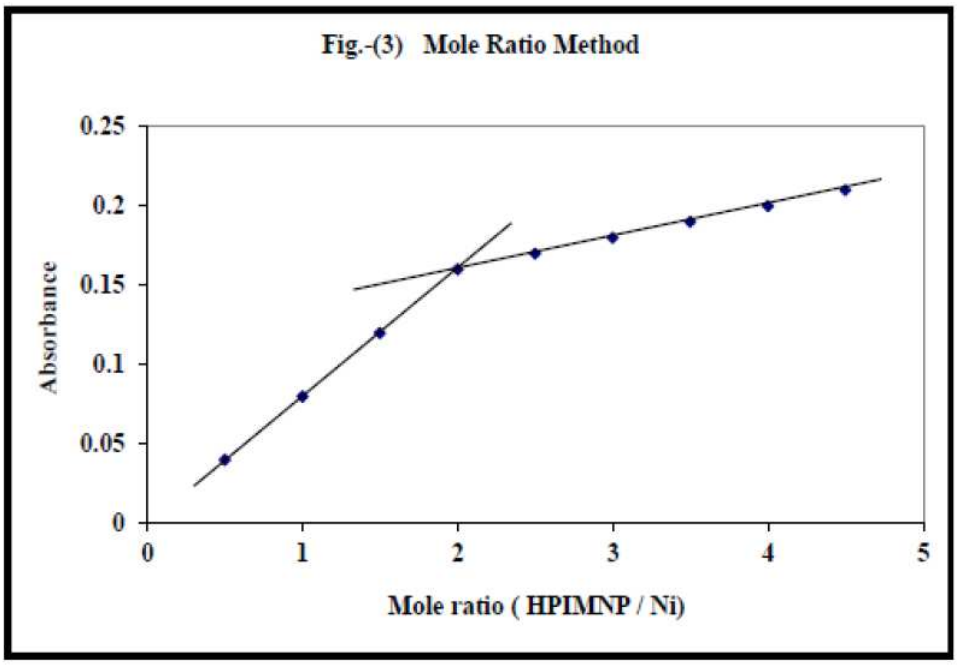
Table – 1 Masking agents required for suppressing the interference by other ions

Interfering ion	Amount added in mg	Masking agent added 1ml of 2M solution
Cr (III)	10	Tri ethanol amine
Co (II)	10	Ascorbic acid
Cu (II)	10	Sodium thiosulphate



COMPOSITION OF THE EXTRACTED COMPLEX:

The composition of the extracted complex was found to be 1:2 (Ni: HPIMNP) by Job's continuous variation and Mole ratio methods **(Fig- 2 & 3).**



PRECISION, ACCURACY, SENSITIVITY AND APPLICATION OF METHOD

The precision and accuracy of the method were tested by analyzing the solution containing a known amount of Ni (II) following the recommended procedure. The average of 10 determination of 100 µg of Ni (II) in 10 cm3 solutions was 100 µg, which is varied between 99.25 and 100.75 at 95% confidence limit and standard deviation is ±1.054. The proposed method has been applied for the determination of Ni (II) in alloy sample. The results of the analysis of the sample were comparable with those obtained by the Dimethyl glyoxime method **(Table 2)**.

Table – 2 Determination of Ni (II) in Alloy sample

Alloy Sample (Nickel - Aluminum based alloy)	Ni (II) found %	
	Present method	Dimethyl glyoxime method
BAS 20 (Nickel -1.93%)	1.93	1.92
BAS 85 (Nickel -0.91%)	0.91	0.90

Results are the average of three independent determinations.

CONCLUSION

From the above discussions, it is found that Schiff base, 2-[(2-hydroxyphenylimino) methyl]-4-nitrophenol [HPIMNP] is a good sensitive reagent for development of rapid and sensitive extractive spectrophotometric method for the determination of Ni (II) and it has been satisfactory applied for determination of Nickel in alloy samples.

Acknowledgement

Authors are thankful to the Principal Dr. (Mrs.) J. K. Phadnis, V.E. S. College of Arts, Science and Commerce, Sindhi Society, Chembur, Mumbai- 400071 for providing necessary research facilities.

REFERENCES

[1] F. Feig "Chemistry of specific, selective and sensitive Reactions". English Ed. By. Oesper R.E., Academic press, New York, (**1949**) 209

[2] R. Nath, S.C. Nantial and H.Singh, Labdev, (**1974**), Part A, 12 A(2), 53-58

[3] W.W. Scott; "Standard methods of chemical Analysis", Von nostrand company, Inc., (**1939**) 182
[4] T. M Fasina, F.N. Ejiah, C.U. Dueke-Eze and N. Idika, *Journal of Sci. Res. Dev.* **2013**: Vol.14, 94-97
[5] A. I. Vogel, "Practical Organic chemistry." 5th Ed. Longman group limited London (**1989**)
[6] Vogel A. I., "Textbook of Quantitative inorganic analysis", 4th Ed. Longman group limited, London (**1978**)