



Solvent Extraction and Spectrophotometric Determination of Trace Amount of Ni(II) Metal Ion by using 3-(2-HydroxyPhenylimino) Indolin-2-One (HPI2O) as an Photometric Reagent

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Abstract:

3-(2-Hydroxyphenylimino) indolin-2-one [HPI2O] is used as a reagent for the extractive spectrophotometric determination of Nickel. The reagent HPI2O gave instantaneous and stable bluish violet coloured complex with Nickel at pH 6.8. The colour reaction in detail has been explored and the possibility of photometric determination of the micro amounts of Nickel is established with necessary conditions. A linear calibration graph over the concentration range 1 ppm to 12 ppm was obtained by applying the spectrophotometric method at wavelength 430 nm. The stoichiometry of the complex is established as 1:1 (M:L) by Job's method of continuous variation and confirmed by mole ratio method. The Sandell's Sensitivity is $0.03960 \mu\text{g cm}^{-2}$ with molar absorptivity $1482.0031 \text{ L mol}^{-1} \text{ cm}^{-1}$. The results of the prescribed procedure applied for the determination of the micro amounts of Ni(II) in various synthetic samples and alloys.

Keywords: Spectrophotometric method, Ni(II) metal, reagent HPI2O, complex, n-butanol etc.

1. INTRODUCTION:-

Nickel is a chemical element with symbol Ni and atomic number 28. Nickel is transition element placed in group VIII B and fourth period of periodic table. Nickel is also known as d-block element. It is a silvery-white lustrous metal with a slight golden tinge¹. Nickel belongs to the transition metals and is hard and ductile. Pure nickel shows a significant chemical activity that can be observed when nickel is powdered to maximize the exposed surface area on which reactions can occur, but larger pieces of the metal are slow to react with air at ambient conditions due to the formation of a protective oxide surface. Nickel is one of the four elements that are ferromagnetic around room temperature. Alnico permanent magnets based partly on nickel are of intermediate strength between iron-based permanent magnets and rare-earth magnets. The metal is chiefly valuable in the modern world for the alloys it forms; about 60% of world production is used in nickel-steels (particularly stainless steel). Other common alloys, as well as some new superalloys, make up most of the remainder of world nickel use, with chemical uses for nickel compounds consuming less than 3% of production (Davis, J. RASM, 2000).

2. EXPERIMENTAL

The pH measurements were made using a pH meter Elico, Model LI-129, India in conjunction with a combined glass and calomel electrode. Shimadzu UV-Visible 2100 spectrophotometer with 1.0 cm matched quartz cells were used for all absorbance measurements.

2.1 Reagent and chemicals

0.1% HPI2O reagent is prepared by dissolving the requisite amount of HPI2O in a known volume of ethanol. All chemicals used were of analytical-reagent grade or the highest purity available. Doubly distilled de-ionized water and A.R. grade ethanol, which is used throughout.

2.2 Ni(II) standard solutions

The stock solution of Nickel(II) was prepared by dissolving weighed amount of nickel ammonium sulphate in doubly distilled de-ionized water. More dilute standard solutions were prepared from this stock solution as and when required.

3. PROCEDURE FOR THE EXTRACTION:

1 mL of aqueous solution containing $10 \mu\text{g}$ of Nickel metal and 2 mL of reagent was mixed in a 50 mL beaker (S.P. Janwadkar, 2011). The pH of the solution adjusted to 6.8, it must be noted that the total volume should not exceed 10 mL.

The solution was transferred to 100 mL separatory 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 passed through anhydrous sodium sulphate in order to absorb trace amount of water from organic phase and then collected in 10 mL measuring flask and made up to the mark with organic solvent if required (Vogel, 1957). The amount of Nickel present in the organic phase determined quantitatively by spectrophotometric method by taking absorbance at 430 nm and that in the aqueous phase was determined by EDTA method (Vogel, 1991; C.Y. Patil, 2016).

4. RESULTS AND DISCUSSION:

The results of various studies are discussed below.

4.1 Extraction as a function of pH:

The extraction of Nickel with 3-(2-Hydroxyphenylimino) indolin-2-one has been studied over the pH range 1-10 and was observed that percentage extraction of Nickel is maximum at pH range 6.4 - 6.8. Hence, further extraction and determination carried out at pH 6.8 (Fig I).

4.2 Absorption spectrum:

The absorption spectrum of Nickel:HPI2O in n- Butanol shows the maximum absorption at 430 nm. The absorption due to reagent at this wavelength is nearly negligible. Hence the absorption measurements were carried out at 430 nm (Fig II).

4.3 Influence of diluents:

The suitability of solvent was investigated using various organic solvents and the extraction of Nickel:HPI2O was quantitative in n-Butanol. Hence, n-Butanol was used for further extraction studies as it gave better and quicker phase separation.

4.4 Effect of reagent concentration:

It was found that 2 mL of 0.1% reagent is sufficient for the colour development of the metal Nickel in 10 mL of aqueous solution at pH 6.8.

4.5 Effect of equilibration time and stability of the complex:

The equilibration time of 1 minute is sufficient for the quantitative extraction of Nickel. The stability of colour of the Nickel:HPI2O complex with respect to time shows that the absorbance due to extracted species is stable up to 45 hours, after which slight decrease in absorbance is observed.

4.6 Calibration plot:

The Beer's law is obeyed from 1 to 12 ppm. The molar absorptivity and sandell's sensitivity were calculated to be $1482.0031 \text{ Lmol}^{-1}\text{cm}^{-1}$ and $0.03960 \mu\text{g cm}^{-2}$ respectively (Fig III).

4.7 LOD:

LOD^{10} (Limit Of Detection) of the present method was calculate at 98.3 % confidence level, it was 0.4344 ppm.

4.8 Effect of divalent ions and foreign ions:

The effect of other ions present in various amount indicated no interference in the spectrophotometric determination of 10 ppm of Nickel. The ions which show interference in the spectrophotometric determination of Nickel were overcome by using appropriate masking agents. (Table I)

4.9 Precision and accuracy:

The precision and accuracy of the developed spectrophotometric method have been studied by analyzing six solutions each containing 4 μg of Nickel in the aqueous phase. The average of ten determinations was 3.994 and variation from mean at 95% confidence limit was 3.994 ± 0.02332 .

4.10 Nature of extracted species:

The composition of extracted Nickel:HPI2O complex has been determined by Job's continuous variation method, Slope ratio method and Mole ratio method. It shows that the composition of Nickel:HPI2O complex is 1:1 (Fig IV).

5. APPLICATION:

The present method was applied for determination of amount nickel in various samples as alloys, synthetic mixture and in pharmaceutical sample. The result obtained was well in agreement with those of standard methods. (Table II)

Procedure for determination of nickel in analytical sample:

1) Alloys

Weigh accurately about 0.4 g of alloy and dissolve in conc. HNO_3 . Evaporate to dryness to boil off excess of acid. Extract the metal ion in dilute HCl and boil for 5 minutes. Dilute the extract to 250 cm^3 in volumetric flask. Use aliquot for further analysis.

Synthetic mixture:- The separation of Nickel(II) from synthetic mixture of associated metals containing Cu(II), Zn(II), Co(II), Cd(II), Mn(II) and Zn(II) with varying combination was carried out. A definite aliquot of this solution was taken and after the adjustment of the aqueous solution to pH 6.8 and addition of 2 ml of 0.1% HPI2O solution, the Nickel complex formed was extracted into 10 ml of n-Butanol. The amount Nickel present was computed using the calibration curve method. The result obtained is compared with those obtained by standard method.

6. CONCLUSION

The results obtained show that the newly developed method in which the reagent HPI2O was used, can be effectively used for quantitative extraction and estimation of Nickel from aqueous media. The proposed method is quick and requires less volume of organic solvent. The results show good agreement with the standard method. The method is very precise, faster and simpler than other methods. The method is accurate, less time consuming and easily employed anywhere, even in small laboratories as it requires only uv – visible spectrophotometer and not much sophisticated and costly measurement devices or instrumentation.

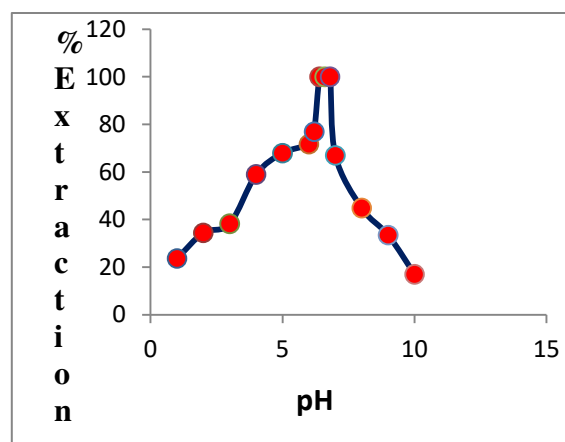


Figure.1. Extraction as a function of pH

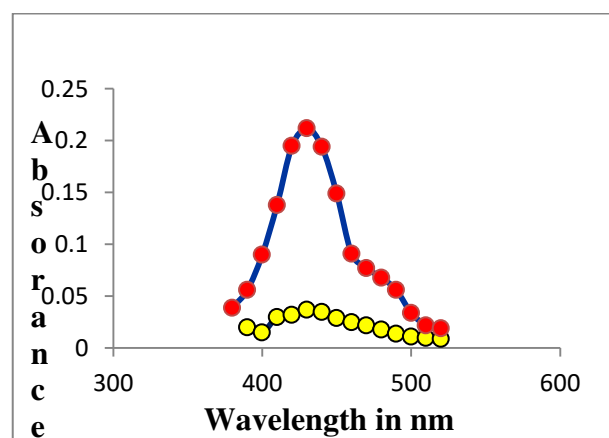


Figure.2. Absorption spectrum

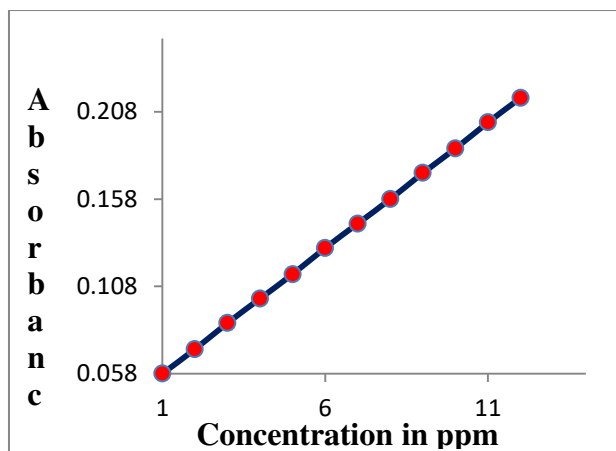


Figure.3. Calibration plot

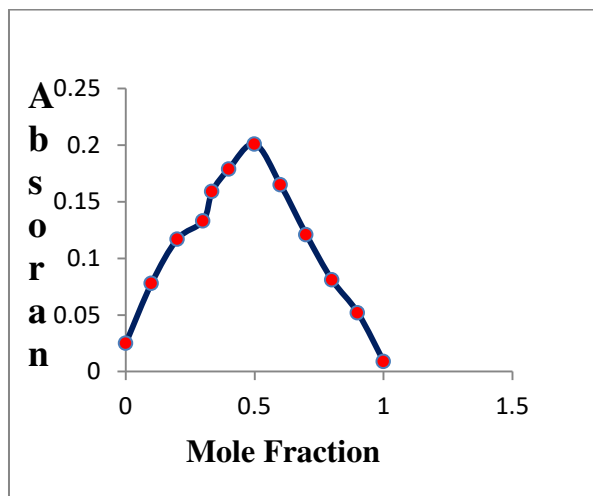


Figure.4. Job continuous variation Nature of extracted species

Table.I. Use of masking agent

Sr. No.	Interfering Ion	Masking agent
1	Fe(III)	Thiourea
2	Ce(IV)	Sodium fluoride.
3	Cu(II)	Sodium Thiosulphate
4	Th (IV)	Sodium fluoride.
5	CN ⁻¹	Boiled with concentrated HNO ₃ and formaldehyde.
6	EDTA	Boiled with concentrated HNO ₃

Table.2. Applications

Sr.No.	Sample	Certified value	Present method
Nickel alloys			
1	Nichrome Alloy	51.48	51.43
2	Cu-Ni Alloy	47.87	47.81
Synthetic mixture:			
1	Fe+ Zn+Ni	6.5	6.45
2	Cu+ Ni+ Cd	5.0	4.91
3	Co +Ni +Mn	4.99	4.97

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8. REFERENCES:-

- [1]. Skoog D A, Fundamentals of analytical chemistry, 8th ed., Thomson, (2004), chap.8.
- [2]. Suhas Janwadkar, Ph.D. Thesis, University of Mumbai, (2011), chapter 9.
- [3]. Vogel A I, Textbook of Organic synthesis, 3rd ed., London, ELBS, (1957).
- [4]. Davis, J.R.ASM Specialty Hand book: Nickel, Cobalt, and Their Alloys, ASM International, (2000).
- [5]. Vogel A I, Textbook of Quantitative chemical analysis, 5th ed, London, ELBS, (1991), 690.
- [6]. Lokhande Rama S.¹, Patil SS², Dharap SB³, Shevde Poonam⁴, Patil Jayashree⁴, Liquid-Liquid Extraction and Spectrophotometric Determination of Ni (II) with 2, 4-Dimethyl-3H-1, 5 Benzodizepine Derivative as an Analytical Reagent; An International Journal of Jaipur National University. Year (2013), Vol.2, issue2.
- [7]. B. Ranganath¹, V. Saleem Basha¹, M.R. Jayapal², P. Venkata Ramana; Direct Spectrophotometric Determination of Ni (II) Using Esomeprazole ; International Journal of Pharmacy and Chemistry.(2015), Vol. 1, No. 1, pp-1-7.
- [8]. D. Prem Kumar, A. Praveen Kumar, P. Saifulla Khan and P.Raveendra Reddy* Department of Chemistry, Sri Krishna devaraya; Derivative Simultaneous Spectrophotometric Determination of Iron (III) and Nickel (II) University, Anantapur International Journal of Analytical and Bioanalytical Chemistry(2015); 5(3): 61-64.
- [9]. V. More, Separation And Estimation Of Toxic Metal Ions From Industrial. Waste And Commercial Samples, (2016), Department of Chemistry, Jaipur National University.