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Development Of Extractive Spectrophotometric Determination Of Nickel (II) Using [N - (O-Methoxy Benzaldehyde)-2-Aminophenol] (NOMBAP) As An Analytical Reagent

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ABSTRACT

Using analytical reagent N - (o - methoxy benzaldehyde) 2-aminophenol (NOMBAP) a sensitive, simple and rapid spectrophotometric method has been developed for the determination of Ni (II). NOMBAP has been synthesized and characterized by elemental analysis. NOMBAP extracts Ni (II) quantitatively (99.35%) into chloroform from an aqueous solution of pH range 7.5 – 8.7. An intense peak at 490 nm (λ max) was observed in the extract of chloroform. Beer's law is obeyed over the concentration range 0.1 – 4.0 µg/ml for Ni (II). The molar absorptivity and sandell's sensitivity for Ni - NOMBAP system is 1396 Lmole^{-1}cm^{-1} and 0.0435µgcm^{-2} respectively. The extracted species gives 1:2 (Ni:NOMBAP) composition by Job's Continuous Variation and Mole Ratio Method. The average of 10 determination of 10 µg of Ni (II) in 10cm^{3} of solution is 9.96µg which is varied between 9.93 µg and 9.99 µg at 95% confidence limit and standard deviation is ±0.04. Study has been done for interference by various ions. The proposed method has been successfully applied for determination of Ni (II) in alloy.

KEYWORDS:[N (O - Methoxy Benzaldehyde) 2 – Aminophenol] (NOMBAP), Extractive Spectrophotometry, Nickel (II), Alloy.

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INTRODUCTION

Nickel is a silvery white lustrous transition element having atomic number 28. Nickel is found naturally in water and food, and is increased by human pollution. Water and soil contamination increases due to nickel plated articles. Mining and smelting is also responsible for dumping of nickel into waste water. Nickel-pigmented dishes may release nickel into food. Humans may absorb nickel directly from tobacco smoke and skin contact with shampoos, detergents, coins and jewelry. Most of the nickel absorbed every day by humans is removed by the kidneys and passed out of the body through urine or is eliminated through the gastrointestinal tract without being absorbed. Nickel is not a cumulative poison, but larger dose or chronic inhalation exposure of nickel is toxic as well as carcinogenic.

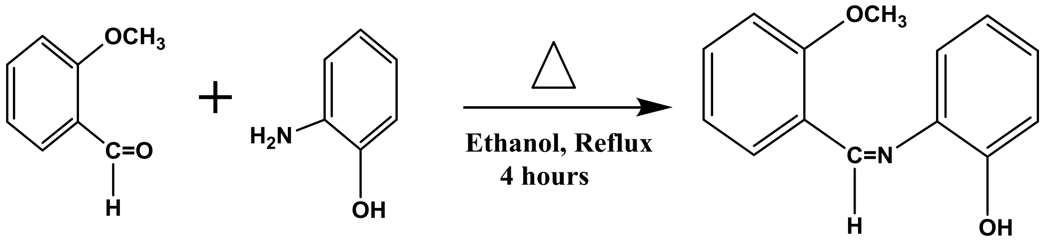
In chemistry solvent extraction plays an important role in separation technique. Solvent extraction technique is used in the separation of metal ions at trace level because of its rapidity, varsity and simplicity.[1,2] Solvent extraction as a separation technique with spectrophotometrically using different organic reagent have significant role in pharmaceutical science.[3] Various reagents are reported in literature review for spectrophotometric determination of nickel (II)[4,5,6,7,8,9,10,11,12,13,14]. In the present communication, we describe the extractive spectrophotometric determination of Ni (II) with N - (o - methoxy benzaldehyde) 2- amino phenol (NOMBAP).

EXPERIMENTAL

The pH measurements were carried out by ELICO - LI 127 pH meter. Absorbance measurement was done by ELICO-SL159 spectrophotometer with optically matched quartz or glass cells of 1cm path length.

General procedure for synthesis of N-(o-methoxy benzaldehyde) 2-aminophenol (NOMBAP):

An equimolar mixture of ethanolic solution of o-methoxy benzaldehyde and 2-aminophenol was refluxed for 6 hours for the synthesis of reagent NOMBAP as shown in fig.1. The reaction mixture was cooled to separate out sharp yellow crystal product (yield 80%, m.p.87^0-88^0C) which was collected by filtration[15]. The recrystallized for NOMBAP was done by using aqueous ethanol as per reported procedure recommended by Vogel[16].



o-methoxy benzaldehyde 2-aminophenol N-(o-methoxybenzaldehyde)-2-aminophenol

Figure 1. Synthesis Of N-(O-Methoxy Benzaldehyde) 2-Aminophenol (NOMBAP)

The characterization of the product was done by spectral and elemental analysis. Its solution was prepared in dimethylformamide (DMF).

Preparation of Stock Solution:-

Nickel sulphate was dissolved in water containing dilute sulphuric for preparation of stock solution of Ni (II) and it was standardized by dimethylglyoxime method[3]. Suitable dilutions was carried out to prepare working solutions of NI (II). All the solutions were prepared in doubly distilled water and reagents used were of AR grade.

Procedure for the extractive spectrophotometric determination of Ni (II):

To an aliquot of aqueous solution containing 1.0- 40 µg of Ni (II), 3ml of sodium bicarbonate buffer solution of pH 8.3 and 2 ml of 2% solution of NOMBAP prepared in DMF were added. With the help of distilled water, volume of solution was made up to 10 ml. The resulting solution was first digested on a boiling water bath for 15 minutes, cool and then transferred into 125 ml separatory funnel. The beaker was then washed twice with 5 ml portion of organic solvent and each washing was added to the solution in the separatory funnel. The two phases were equilibrated for 1 minute and allowed to separate. The chloroform extract was collected in a 10 ml standard measuring flask and made up to mark with chloroform, if necessary. The absorbance of chloroform extract was measured at 490 nm against a reagent blank prepared under identical conditions. The measured absorbance was used to compute the amount of Ni (II) present in the sample solution from predetermined 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.

Procedure for the determination of Nickel (II) in alloy sample: (Nickel-Aluminum based alloy)

10 ml of aquaregia was used for dissolving 0.1 to 0.2 g sample of nickel. The solution was evaporated to dryness and the residue was dissolved in 10 ml of 1N HCl and then filter if required

and resulting solution dilution was made up to 100 ml with distilled water. 1ml aliquot of this solution was used for Ni (II) analysis by the procedure as described earlier.

RESULTS AND DISCUSSION:

Ni (II) could be extracted quantitatively (99.35%) by NOMBAP into Chloroform from an aqueous solution of pH 7.5 to 8.7 in presence of 3ml of sodium bicarbonate buffer. Organic solvents used for extraction of n Ni (II) can be arranged on the basis of their extraction coefficient values as chloroform > Ethyl acetate> carbon tetrachloride > n-butanol > Chlorobenzene > Bromobenzene > benzene > Xylene as described in fig.2. Chloroform was selected for the extraction throughout the work because it was found to be the best extracting solvent. An intense peak at 490 nm as shown in fig.3 was observed for chloroform extract of Ni: NOMBAP complex. The molar absorptivity of the extracted complex on the basis of Ni (II) content was calculated and observed to be 1396 L mol^{-1} cm^{-1}. The absorbance due to the reagent is negligible at this wavelength, so the absorption measurements were taken at this wavelength. It was found that 2 ml of 2.0% DMF solution of NOMBAP was sufficient to extract 40µg of Ni (II). The result shows that the system confirmed to Beer’s law at this wavelength over a Ni (II) concentration range of 0.1 to 4.0 µg/ml as shown in fig.4. At room temperature color of the chloroform extract was found to be stable for at least 24 hrs.

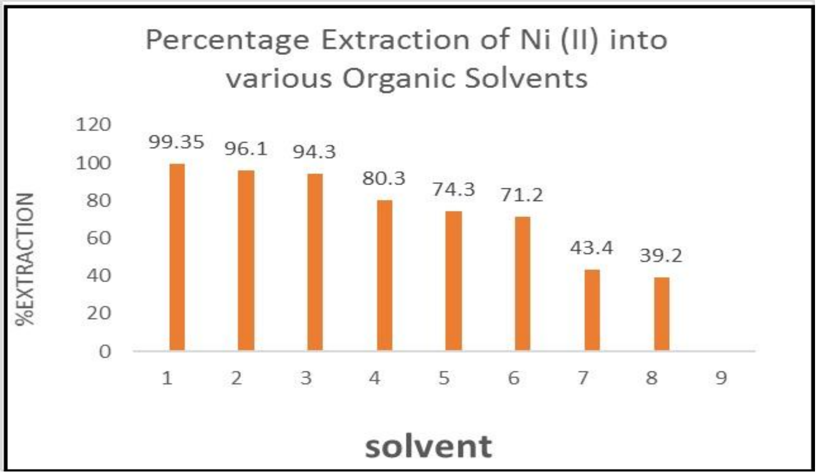
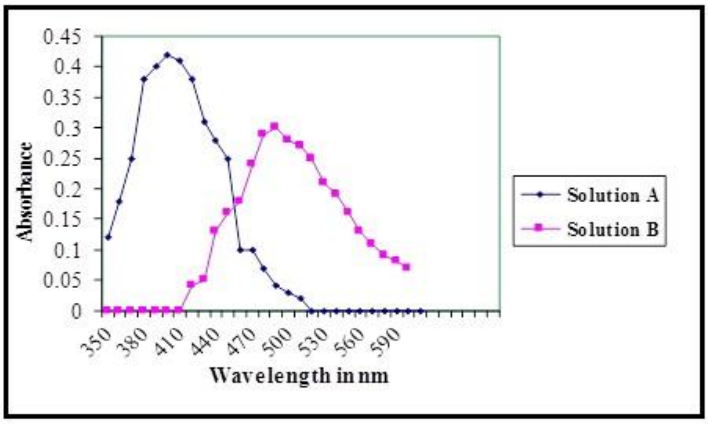


Figure 2. Percentage Extraction of Ni (II) Into Various Organic Solvents.



Solution A: Absorbance spectra of NOMBAP

Solution B: Absorbance spectra of Ni–NOMBAP Complex

Figure 3. Absorbance Spectra of NOMBAP And Ni–NOMBAP Complex

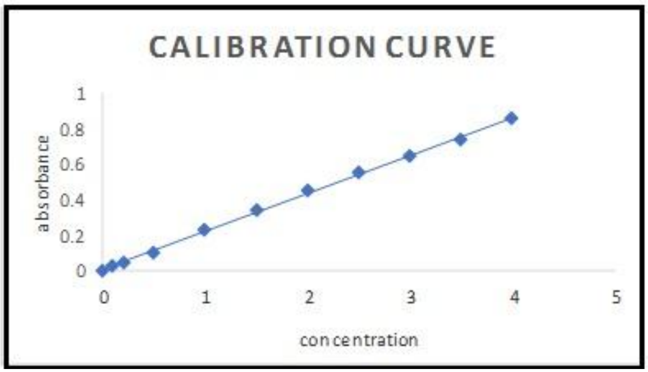


Figure 4. Calibration Curve of Ni–NOMBAP Complex

Effect of other ion:

Ni (II) (20µg) was determined in the presence of various ions. The following ions in the amount indicated, they did not interfere in the spectrophotometric determination of Ni (II):

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).

0.1mg each of Ru (III), Rh (III) and Pt (IV).

20 mg each of sulphate, sulphide, nitrate, nitrite, chloride, bromide, iodide, fluoride, phosphate, citrate, triethanol amine, thiocynate, acetate and 5-sulphosalicylic acid.

Interference by the various ions were removed by using appropriate masking agent as shown in Table - 1.

Table – 1: Interference by Various Ions

Sr. No.	Amount of interfering ions added in mg	Masking agent added 1ml of 0.5 M solution	Absorbance
1.	10 mg of Cr(III)	Sodium fluoride	0.45
2.	10 mg of Mn(II)	Sodium Fluoride	0.45
3.	10 mg of Ag(I)	Pottasium thiocynate	0.45
4.	10 mg of Cu(II)	Sodium dihydrogen phosphate	0.45
5.	10 mg of Fe(II) & Fe(III)	Triethanol amine	0.45
6.	10 mg of Oxalate	Sodium Molybdate	0.45

Composition of the Extracted Complex:

The composition of the extracted complex was found to be 1:2 (Ni: NOMBAP) by Job's continuous variation as shown in fig. 5 and Mole ratio methods in fig. 6.

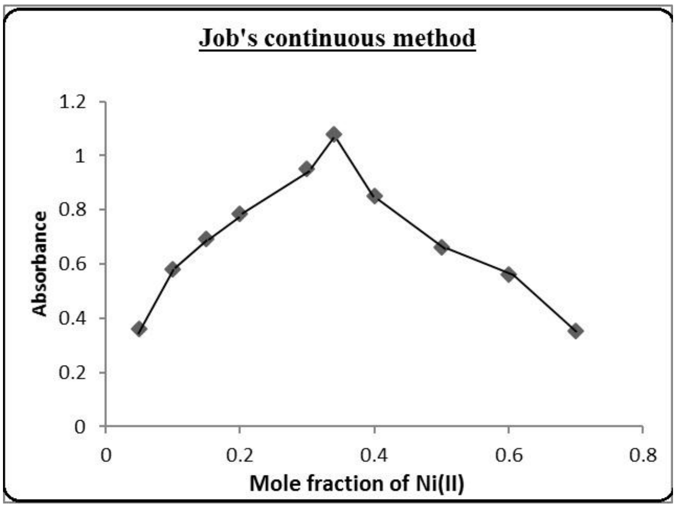


Figure 5. Composition of (Ni: NOMBAP) Complex by Job's continuous Method.

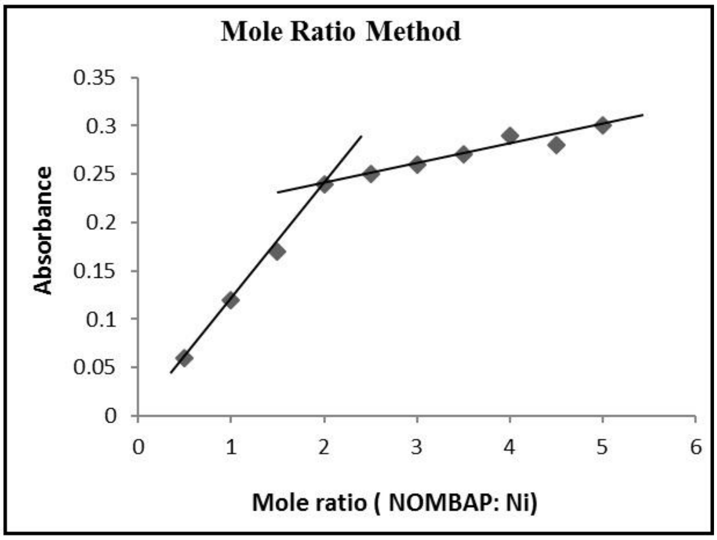


Figure 6. Composition Of (Ni: NOMBAP) Complex by Mole Ratio Methods.

Accuracy, Precision, Sensitivity and Applications of Method

The accuracy and precision of the method were tested by analyzing the solution containing a known amount of Ni (II) by using the recommended procedure. The average of 10 determination of 10 µg of Ni (II) in 10 cm^3 solutions was 9.96 µg, which is varied between 9.93 and 9.99 at 95% confidence limit. Sandell's sensitivity and standard deviation of the extracted species is found to be 0.0425 $\mu g cm^{-2}$ and ±0.04 respectively.

The proposed method has been applied for the determination of Ni (II) in alloy samples.

The results of the analysis of the samples were comparable with those obtained by the dimethyl glyoxime method[3] for Ni (II) as shown in 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%)	1.92	1.94
BAS 85 (Nickel -0.91%)	0.90	0.91

* Average of three determinations

CONCLUSIONS

The extractive spectrophotometric determination of Ni (II) was successfully carried out. The reagent NOMBAP formed complex with nickel (II) which were easily extracted into organic phase. The method is simple and reliable. Experimental conditions for maximum extraction was determined and applied for the analysis of samples.

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