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NEW ANALYTICAL TECHNIQUE FOR DETERMINATION OF TRACE AMOUNT OF NI (II) BY USING UV-VISIBLE SPECTROPHOTOMETRIC METHOD WITH PHOTOMETRIC REAGENT

Vaibhava S. More*, R. S. Lokhande and S. P. Janwadkar

S.D.Arts,V.S.Apte Commerce and M.H.Mehta Science college, Palghar,Dist- Palghar (M.S.).

***Corresponding Author: Vaibhava S. More**
S.D.Arts,V.S.Apte Commerce and M.H.Mehta Science college, Palghar,Dist- Palghar (M.S.).

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ABSTRACT

3-nitrosalicylaldehyde thiosemicarbazone (3-NSTS) is proposed as a new photometric reagent for the extractive spectrophotometric determination of Ni (II). (3-NSTS) reacts with Ni (II) and form a stable coloured complex in the pH range 3.0 to 4.0.This was well extracted in n-butanol. The absorption spectrum of Ni (II) (3-NSTS) complex in n-butanol shows maximum absorbance at 440 nm. The system obeyed Beer's law up to 2-6 µg / cm^3. The molar extinction coefficient was found to be 5.035 x 10^3 lit $mol^{-1}cm^{-1}$ and the sensitivity of the method as defined by Sandell's sensitivity was 1.162 x 10^{-2} µg/cm^2. The composition of the extracted species was determined by Job's Continuous variation method, Mole ratio method and slope ratio method and it was found to be 1:3. The proposed reagent is satisfactorily applied for the determination of trace amount of Ni (II) from industrial waste water as well as synthetic and commercial samples.

KEYWORDS: Nickel,n-butanol,3-Nitro-salicylaldehyde thiosemicarbazone derivative(3-NSTS) etc.

INTRODUCTION

Nickel is a chemical element with the chemical symbol Ni and atomic number 28. It is a silvery-white lustrous metal with a slight golden tinge.[1] On Earth, such native nickel is always found in combination with iron, a reflection of those elements' origin as major end products of supernova nucleosynthesis.[2] Major production sites include Sudbury region in Canada.[3] 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.[4]

On Earth, nickel occurs most often in combination with sulfur and iron in pentlandite, with sulfur in millerite, with arsenic in the mineral nickeline and with arsenic and sulfur in nickel galena.[5] Nickel plays important roles in the biology of microorganisms and plants. In fact, urease (an enzyme that assists in the hydrolysis of urea) contains nickel. The minimum risk level of nickel and its compounds is set to 0.2 µg/m^3 for inhalation during 15–364 days. Nickel sulfide fume and dust are believed to be carcinogenic, and various other nickel compounds may be as well.

EXPERIMENTAL

Procedure for the Extraction

An aliquot of solution containing 1mL of 100ppm of Nickel was taken. To this 1mL of (3-NSTS) reagent is mixed. The pH of the solution adjusted to 3.0, & noted that the total volume should not exceed than 10mL.The solution was transfer to the 125mL of separating funnel & equilibrated with 10mL of n-butanol solution. The separating funnel was shaken vigorously and allowed to stand for some time to separate the two phases. The aqueous phase is separated and the organic phase is passed through anhydrous sodium sulphate in order to absorb water and then collected in 10mL volumetric flask and dilute up to the mark with n-butanol. The absorbance was measured at λmax = 440nm on a Shimadzu UV-Visible 2100 Spectrophotometer with 1cm quarts cells.

RESULT AND DISCUSSION

The results of various studies were discussed as given below

Effect of solvent on extraction

n-Butanol is chosen as solvent, since it was found that the metal complex Ni (II) (3-NSTS) complex in n-butanol shows maximum absorbance at 440nm.

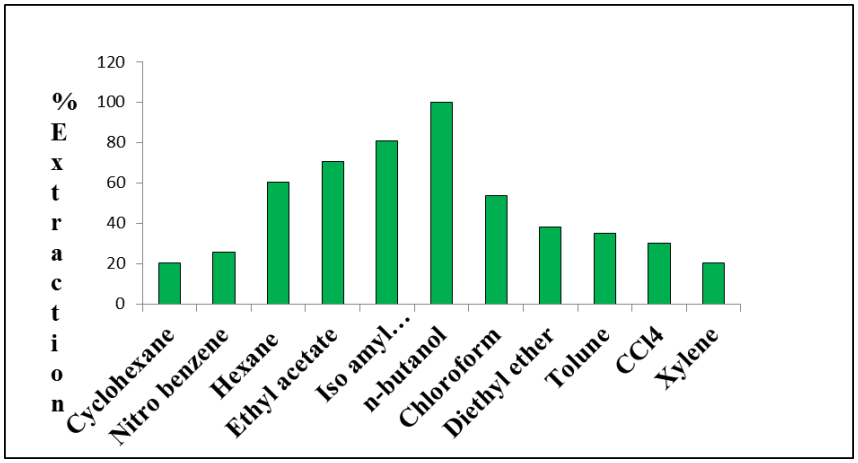

Fig. 1: Effect of solvent on extraction.

Effect of pH on extraction

The absorbance the complex Ni (II) (3-NSTS) was measured as a function of pH of the aqueous phase. The complexation of Ni (II) was carried out at pH 1-10. From which pH range 3.0-4.0 is selected. The data obtained shows maximum absorbance at pH 3.0.

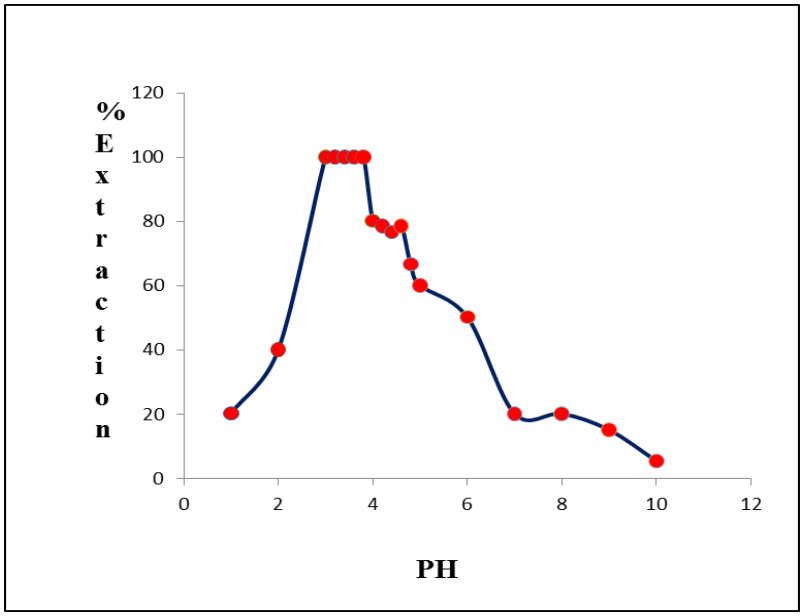

Fig. 2: Effect of pH on extraction.

Effect of Reagent concentration

The results obtained from the plot of absorbance versus concentration of (3-NSTS) shows that 1mL of 8.45 $x10^{-4}$ M reagent sufficient for the quantitative extraction and spectrophotometric determination of100ppm of Ni (II) (3-NSTS) in n-butanol. Addition of excess reagent did not interfere with complexation and extraction of complex, so further study was carried out by using 1mL of 2.08 x 10^{-3} M reagent.

Effect of equilibration time and stability of the complex

The equilibration time of 1 minute was sufficient for the quantitative extraction of Nickel and the complex Ni (II) (3-NSTS) is stable for 48 hours, after which slight decrease in absorbance is observed.

Calibration plot

The system obeys Beer's law in the concentration range up to 2-6 µg / cm^3 at 440nm.The molar absorptivity and Sandell's sensitivity were calculated and found to be 5.035 x 10^3 lit $mol^{-1}cm^{-1}$ and 1.162 x 10^{-2}µg/cm^2 respectively.(Fig.3).

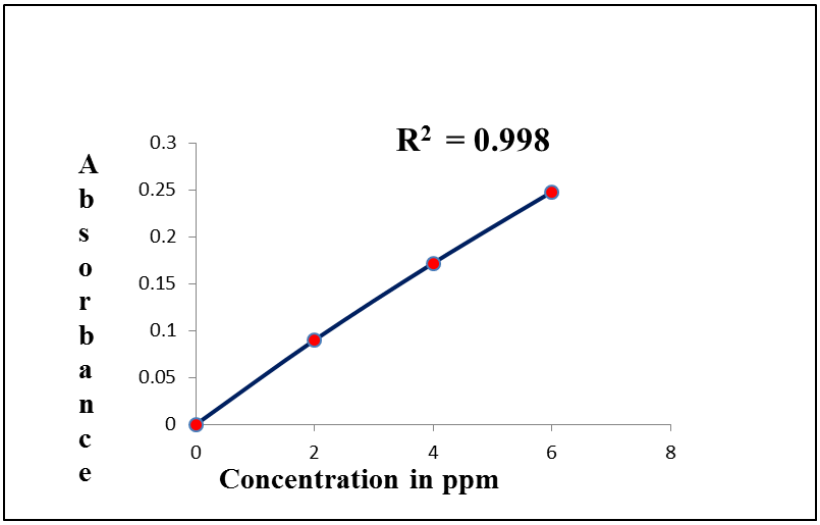

Fig. 3: Calibration Curve.

Effect of divalent ions and foreign ions

The effect of diverse ions on the Ni (II) was studied, in the presence of foreign ions. The ions which show interference in the spectrophotometric determination of Iron were overcome by using appropriate masking agent as given in table 1.

Table 1:

Sr.no	Interfering ions	Masking agent
1	Co(II)	Ammonium Sulphite
2	Cu(II)	Sodium Thiosulphate
3	Fe(II)	Thiourea
4	CN^{-1}	Boiled with concentrated HNO_3 and formaldehyde.
5	EDTA	Boiled with concentrated HNO_3

Precision and Accuracy

The precision and accuracy of the spectrophotometric method were tested by analyzing ten solutions containing 3.0 µg of Nickel in 10 cm^3.The average of ten determinations was 2.0067µg which is varies between 2.0067µg to 0.04148µg at 95% confidence limit.

Limit of detection

Standard deviation of blank solution and slope of calibration curve is used for calculating limit of detection, which found to be 0.179 µg / mL.

Nature of extracted species

The composition of extracted Ni (II) (3-NSTS)complex has been determined by Job's continuous variation method, Slope method and Mole ratio method. It shows that the composition of Ni (II) (3-NSTS)complex is 1:3 (Fig.4).

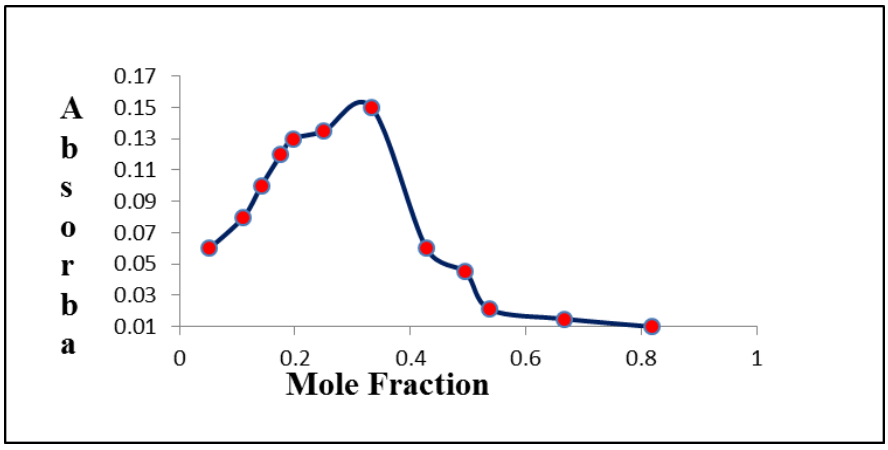

Fig. 4: Job Continuous Variation.

Application

The proposed method was successfully applied for the determination of Nickel from various synthetic mixtures, Qindustrial waste and alloys and commercial samples etc. The results obtained were found to be in good agreement with those obtained by the standard method as given in below table.

Sr. No.	Sample	Certified value	Present method
Nickel alloys			
1	**Nichrome Alloy**	**51.48%**	**51.41%**
2	**Cu-Ni Alloy**	**47.87%**	**47.82%**
Synthetic mixture			
1	**Fe+ Zn+Ni**	**5.5 ppm**	**5.4 ppm**
2	**Cu+ Ni+ Cd**	**5.0 ppm**	**4.9 ppm**
3	**Co +Ni +Mn**	**3.98ppm**	**3.96 ppm**

Industrial effluents are collected from the following the following industries

1.Cameline fine chemical 2.Aarti drugs 3.Lupin ltd 4.Macload pharma Industrial effluents collected Bimonthly Analysis.

Table 7.22

Sr. No	Industrial effluent from	January February March	April May June	July August September	October November December
1	**Camelin fine chemical**	9.31 ppm	10.95 ppm	9.22 ppm	9.39 ppm
2	**Aarti drugs**	8.95 ppm	9.18 ppm	8.15 ppm	8.31ppm
3	**Lupin ltd**	14.34 ppm	14.88 ppm	13.85 ppm	14.16 ppm
4	**Macload pharma**	7.5 ppm	8.58 ppm	7.11 ppm	10.45 ppm
5	**Unibias pharma**	9.58 ppm	10.95 ppm	9.28 ppm	9.41 ppm
6	**Suyog chemical**	11.15 ppm	12.95 ppm	10.22 ppm	10.39 ppm

1) Each result is average of three independent determinations.
2) Compared with EDTA method.

CONCLUSIONS

The method required simple apparatus which have low cost. This method offer several silent features such as rapidity, selectivity and simplicity. The other associated elements do not interfere in the determination. Hence the proposed method is recommended for the determination of Ni (II) (3-NSTS) by spectrophotometric method, at trace level analysis of various alloys, synthetic mixture and industrial waste.

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