



RESEARCH ARTICLE

Extractive Spectrophotometric Determination of Nickel (II) Using 2-(5- Bromo-2- Oxindolin-3-Ylidene) Hydrazine Carbothioamide as an Analytical Reagent

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ABSTRACT

2-(5- bromo-2- oxindolin-3-ylidenehydrazine carbothioamide), [5-Bromoisatin thiosemicarbazone (HBITSC)] extracts Nickel (II) quantitatively (99.83%) into n-amyl alcohol from an aqueous solution of pH 7.2 – 8.8. The n-amyl alcohol extract shows an intense peak at 510nm (λ max). Beer's law is obeyed over the Ni (II) concentration range 1.0–6.0ug/ml. The molar absorptivity is 4412 L mole⁻¹cm⁻¹ at 510 nm. And Sandel's sensitivity is 13.3 ng/cm². The composition of extracted species is found to be 1:2 (Ni: HBITSC) by Job's continuous variation and Mole ratio method. Interference by various ions has been studied. The proposed method has been applied for determination of Ni (II) in alloy samples.

KEYWORDS

HBITSC, Nickel, Spectrophotometry

INTRODUCTION

Various reagents¹⁻¹¹ are available for the spectrophotometric determination of Nickel (II). In the present communication, we describe the extractive spectrophotometric determination of Ni (II) with 2-(5- bromo-2- oxindolin-3-ylidenehydrazine carbothioamide), [5-Bromoisatin thiosemicarbazone (HBITSC)].

Its solution was prepared in Dimethyl formamide (DMF). A stock solution of Ni (II) was prepared by dissolving nickel sulphate in water containing dilute sulphuric acid and it was standardized by dimethylglyoxime method³. Working solutions of Ni (II) were made by suitable dilutions. All other reagents used were of AR grade and all the solutions were prepared in doubly distilled water.

MATERIAL AND METHODS

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 HBITSC was synthesized by condensations of 5-Bromoisatin and Thiosemicarbazide procedure recommended by Vogel² and characterized by elemental and spectral analysis.

Extractive Spectrophotometric Determination of Ni (II)

To an aliquot of aqueous solution containing 10-60µg of Ni (II), 3ml of Sodium bicarbonate buffer solution of pH 8.3 and 1ml of 0.6% solution of HBITSC prepared in DMF added. The volume of solution was made up to 10 ml with distilled water and then equilibrated for 1 min with 10 ml of n-amyl alcohol and the phases were allowed to separate. The n-amyl alcohol extract was collected in a 10 ml measuring flask and made up to mark with n-amyl alcohol. The absorbance of n-amyl alcohol extract was measured at 510 nm against a reagent blank

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prepared under identical conditions. The Nickel 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 Samples

(Nickel-Aluminum based alloy)

0.1 to 0.2 gm. sample of nickel was dissolved in 10 ml of aquaregia. The solution was evaporated to dryness and the residue was dissolved in 10 ml of 1N HCl filter, if required and resulting solution was diluted to 100 ml with water. 1ml aliquot of this solution was analyzed for Ni (II) by the procedure as described earlier.

RESULTS AND DISCUSSION

Nickel (II) could be extracted quantitatively (99.83%) by HBITSC into n-amyl alcohol from an aqueous solution of pH 7.2 to 8.8. Organic solvents used for extraction of Ni (II) can be arranged on the basis of their extraction coefficient values as n-amyl alcohol > n-butanol > ethyl acetate > benzyl alcohol > chloroform > carbon tetrachloride > xylene > nitrobenzene > toluene > chlorobenzene.

N-amyl alcohol was found to be the best extracting solvent; hence, it was selected for extraction throughout the work. The n-amyl alcohol extract of Ni- HBITSC complex showed intense peak at 510 nm. (Figure 1). 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 1.0 to 6.0ug/ml.

The molar absorptivity of the extracted complex on the basis of Ni (II) content was calculated to be $4412 \text{ L mol}^{-1} \text{ cm}^{-1}$. The Sandel's sensitivity was found to be 13.3 ng/cm^2 . It was found that 1 ml of 0.6% DMF solution of HBITSC was sufficient to extract 60 ug of Ni (II). The colour of the n-amyl alcohol extract was found to be stable at least 48 hrs at room temperature.

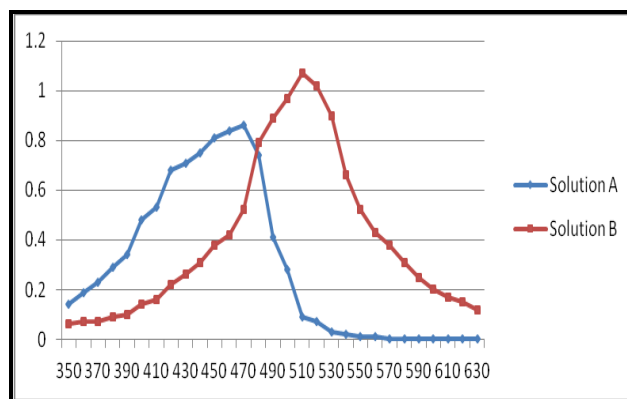


Figure 1: Solution A: Absorbance spectra of HBITSC

Solution B: Absorbance spectra of Ni – HBITSC Complex

Effect of Other Ions

Ni (II) (30 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) (30 μg) : 10 mg each of Ca(II), Mg(II), Ba(II), Li(I), 5mg each of Hg(II), As(III), Bi(III), Sb(III) and V(V).1mg each of Ti(II), Fe(II), Fe(III), Cu(II), Cd(II), Pb(II), Ce(IV), Th(IV), Zr(IV), Pt(IV), Ru(III), U(VI), Mo(IV) and W(IV), 0.5gm each of Cr(III) , Mn(II), Co(II), Zn(II), Ru(III) and Rh(III). 0.1mg each of Pd (II), Os (IV) and Ir(IV).

20 mg each of chloride, fluoride, sulphate, acetate, nitrate, nitrite, persulphate, thiourea, acetate and thiosulphate. 5 mg each of iodide and bromide. Interference due to E.D.T.A. was removed by boiling the solution with concentrated nitric acid before the adjustment of pH.

Composition of the Extracted Complex

The composition of the extracted complex was found to be 1:2(Ni:HBITSC) by Job's continuous variation and Mole ratio method

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 20 µg of Ni (II) in 10 cm³ solutions was 19.95µg, which is varied between 19.686µg and 20.214µg at 95% confidence limit and standard deviation was 0.369. 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 method³ for Ni (II) (Table 1).

Table 1: 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

*Average of three determinations

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