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Research Paper

Liquid-Liquid Extraction and Spectrophotometric Determination of Ni (II) using 5-nitrosalicylaldehyde semicarbazone (NSS) as an Analytical Reagent

Ram L.S.¹, *Kalpana P.J.N.², Irfan M.R.²

¹Department of Chemistry, Jaipur National University, Jaipur, Rajasthan, INDIA

²Department of Chemistry, B. N. N.College, Bhiwandi, District Thane, INDIA

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Abstract: 5-nitrosalicylaldehyde semicarbazone (NSS) is proposed as a new sensitive reagent for the sensitive extractive spectrophotometric determination of Ni (II). NSS reacts with Ni (II) in the pH range 4.0 to 4.4 to form a coloured complex, which was well extracted into Ethyl acetate. The absorption spectrum of Ni (II) NSS complex in Ethyl acetate shows maximum absorbance at 420 nm. It was observed that the colour development was instantaneous and stable for 48 hrs. The system obeyed Beer's law up to 1-6 $\mu\text{g} / \text{cm}^3$. The molar absorptivity calculated was found to be $2.5016 * 10^4 \text{ lit mol}^{-1} \text{ cm}^{-1}$ and the sensitivity of the method as defined by sandal's was $1.067 * 10^{-2} \mu\text{g}/\text{cm}^2$. The composition of the extracted species was determined by Job's Continuous variation method and Mole ratio method was found to be 1:4. It may be satisfactorily applied for the determination of Ni (II) with present method.

Keywords: Ethyl acetate, Solvent Study, Calibration curve, sandal's sensitivity.

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Introduction

The cursory look at the above literature survey reveals that Nickel forms chelate complexes with many organic reagents which are bonded through N, O & / S Items. Out of the reagents used for the extraction and spectrophotometric determination of Ni (II), many reagents suffer limitations as of, interference by other ions, lengthy and tedious procedure for reagent preparation, requirements of surfactants, the stability of complex with time, lack of appropriate solvent for extraction. Survey of innumerable applications of Nickel leave a scope for as many reagents as can be applied for the extraction and spectrophotometric determination of Nickel. The present work describes a spectrophotometric method for quantitative estimation of Ni (II) with NSS.

Material and Methods

Apparatus: Glassware used in the present investigations was all made up of Borosil. The burettes pipettes and standard flasks were calibrated in accordance with method described in Vogel^[1]. An analytical balance of 0.001g sensitivity of Contech-120 (modelCA-123) used for weighing the samples. All measurements of absorption spectra² were made on one cm silica cells or 1 cm glass

cells. The spectrophotometer used was Jasco (UV/VIS/NIR)Model-630. The calibration of the spectrophotometer was checked measuring absorption spectrum of 0.004% solution of potassium chromate in 0.05 M potassium hydroxide solution and also with 0.0058% solution of potassium permanganate in 1M sulphuric acid. The observed spectrum was in good agreement with the spectrum reported in the literature^[2].

Results and Discussion

Absorbance Maxima

The absorption spectrum of Ni (II) NSS complex in Ethyl acetate shows maximum absorbance at 420 nm. The absorbance due to the reagent at this wavelength was negligible. Hence, 420 nm was selected for the absorbance measurement^[3] in spectro-photometric determination of Ni (II) against blank.

Solvent study

Various solvents were tried to determine the maximum extraction of Nickel (II). Ethyl acetate was found to be most suitable solvent^[4] as it showed the highest extraction. The extraction of Nickel (II) was minimum in solvents Chloroform & Carbon Tetra Chloride.

Effect of reagent concentration

The effect of variation in concentration of NSS in the range of 0.1 to 4.0 cm³ of 0.02% NSS on the extraction and the color development was tried. It was observed that 2.2 cm³ of 0.02% NSS was sufficient for complete extraction and color development^[5].

Effect of salting out agents on absorbance of Ni (II) NSS complex

Different salting out agents such as sulphates, chlorides, carbonates and nitrates of Sodium, Potassium, Barium, Magnesium, Ammonium and Calcium were used in the extraction of Ni (II) (1cm³ of 100 ppm solution). It was observed that there was no effect on absorbance.

pH study

The extraction of Nickel (II) with NSS was carried out over pH range of 1 – 12, 1 cm³ of aqueous solution of 100 ppm Nickel (II) stock solution and 0.5 cm³ of 0.02% solution of the reagent^[6] were used. It reveals that 99% of metal is extracted into organic phase in the pH range between 4.0 to 4.4. It was found that complex does not form below pH 2. The analytical work for estimation of Nickel (II) was carried out at pH 4.2. (Figure 1)

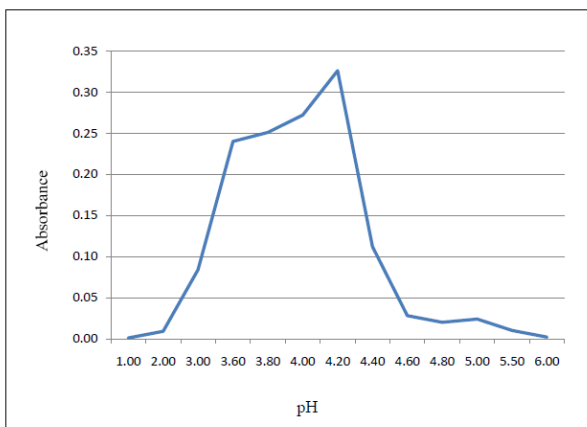


Figure 1: Effect of pH on the extraction of Nickel (II): NSS Complex

Stability of complex with time

For this study, 1cm³ of 100 ppm solution was extracted with reagent in Ethyl acetate and absorbance of complex in Ethyl acetate was measured at different intervals of time. The study of stability of complex with variation in time showed that the complex was stable up to 48 hours, after which absorbance decreased slowly.

Calibration Curve

Different amounts of nickel (II) from 1 – 6 µg / cm³ were extracted quantitatively under optimum experimental conditions and the Ethyl acetate extract was measured at 420 nm against reagent blank. The plot of absorbance against concentration of Nickel (II) gave a straight line (Figure 2) indicating that Beer's Law is obeyed in this range. The molar absorptivity calculated was found to be

2.5016 * 10⁴ dm³ mol⁻¹ cm⁻¹ and the sensitivity of the method as defined by Sandal's was 1.067 * 10⁻² µg / cm².

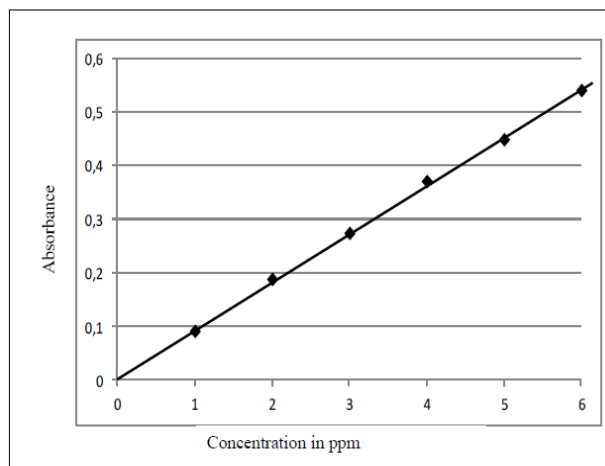


Figure 2: Calibration Curve for Nickel (II)

Effect of masking agents

The ions which interfere in the spectro-photometry determination of Copper (II) were masked by using appropriate masking agents (Table 1).

Table 1

S.No.	Interfering Ion	Masking Agent Added
1	Ag (I)	Potassium Iodide
2	Cd (II)	Potassium Iodide
3	Pb (II)	Sodium Thiosulphate
4	Mn (II)	Sodium Fluoride
5	Ce (IV)	Sodium Fluoride
6	Cr (II)	Ammonium Acetate
7	Citrate	Sodium Molybdate
8	Tartarate	Sodium Molybdate
9	EDTA	Boiling with Conc.HNO ₃
10	CN ⁻	Boiling with Conc.HNO ₃ and formaldehyde
11	Fe (III)	Thiourea
12	Zr (IV)	Sodium Fluoride
13	Cu (II)	Sodium Cyanide
14	Pd (II)	Sodium Thiosulphate

Interference Study

The effect of diverse ions on the Nickel (II) determination was studied in presence of a definite amount of Foreign Ion. Various cations and anions were investigated in order to find the tolerance limit of these foreign ions in the extraction of Nickel (II). The tolerance limit of the foreign ion was taken as the amount required causing an error of not more than +2% in the recovery of Nickel (II).

Job's Continuous Variation Method

A series of solutions were prepared by mixing 0.0 to 10.0 cm³ of 5.0 x 10⁻⁴ M Nickel (II) solution with 10.0 to 0.0

cm³ of 5.0 x 10⁻⁴ M NSS solution, such that the volume of each mixture was 10.0 cm³ each solution was treated at optimum pH as per procedure and the absorbance was then measured against blank. The absorbance values were plotted against mole ratio of Ni (II) to NSS. It shows sharp maxima at 0.2 Mole Fraction of Ni (II), indicating that the colored complex extracted into Ethyl acetate was formed by the reaction of Ni (II) and NSS in the ratio 1:4.(Figure 3)

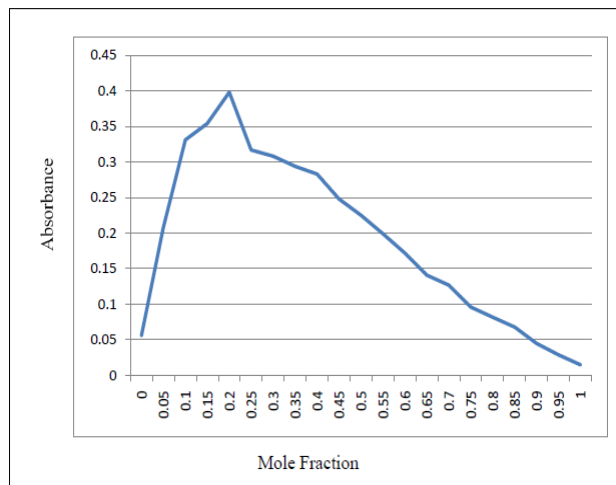


Figure 3: Job's Continuous Variation Method for Ni (II) with NSS in aqueous medium

Mole Ratio Method

A series of solutions containing 1 cm³ of 5.0 * 10⁻⁴ Ni (II) increasing amounts of 5.0 * 10⁻⁴ molar NSS were added. Each mixture was treated similarly and absorbance was measured using blank. The absorbance values were plotted against the mole ratio of NSS to Nickel (II)^[7]. It shows a sharp break corresponding to mole ratio 1:4 which supports the composition of Ni (II): NSS Complex.

Nature of the extracted species

The absorption spectrum of the Ethyl acetate extracts of the aqueous solution containing Ni(II):NSS in molar concentration ration 1: 1, 1: 2, 1: 3 & 1: 4 indicated that under the experimental conditions, the nature of absorption spectrum remains identical irrespective of the reagent concentration with no change in the value of wavelength maxima. However, the absorbance increases as the molar reagent concentration increases with respect to the metal concentration. The composition of the extracted species was determined by Job's Continuous Variation Method and verified by Mole Ratio Method^[8].

Equilibration Time

The absorbance of Ni (II) – NSS Complex was checked by varying the time of equilibration from 30 seconds to 15 minutes. It was observed that equilibration time of 1 minute was found to be optimum for complete extraction of Nickel (II).

Applications of method

The present method was applied for the determination of amount of Nickel (II) in various samples as alloys, milk samples, synthetic mixtures, beverages and industrial waste water. The results obtained were well in agreement with those of standard methods. (Table 2)

Table2: Determination of Ni (II) using NSS from Different Samples

S. No.	Sample	Amount of Ni (II)	
		Standard Method	Present Method
Nickel Alloys			
1	Nichrome	23.30%	23.21%
2	Steel	30.20%	30.19%
3	Nickel Cast iron	11.40%	11.41%
	Vegetable Oil	0.0015%	0.0014%
Synthetic Mixture			
1	Ni (10 mg) + Zn (10 mg)	9.99mg	9.98mg
2	Ni (10 mg) + Mg (10 mg)	9.99mg	9.97mg
	Industrial Waste Water at Sarawali – Kalyan	2.5ppm	2.4ppm

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