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# Complexation of Co (II) and Spectrophotometric Determination of the complex using 5-Nitrosalicylaldehyde semicarbazone (NSS) as an Analytical Reagent

Authors
**K. N Patankar Jain*, Irfan R Memon**
Department of Chemistry, B. N. N. College, Bhiwandi, District Thane
*Email: *irfanraza252663@gmail.com, Mobile: 9371241267*

**Abstract**
*5-nitrosalicylaldehyde semicarbazone (NSS) is proposed as a new sensitive reagent for the sensitive extractive spectrophotometric determination of Co (II). NSS reacts with Co (II) in the pH range 8.0 to 8.4 to form a coloured complex, which was well extracted into Iso Amyl Alcohol The absorption spectrum of Co (II) NSS complex in Iso Amyl Alcohol shows maximum absorbance at 417 nm. It was observed that the colour development was instantaneous and stable for 48 hrs. The system obeyed Beer's law up to 0.5 to 3.0 µg / $cm^3$. The molar absorptivity calculated was found to be 2.86 * $10^4$ lit $mol^{-1}$ $cm^{-1}$ and the sensitivity of the method as defined by sandal's was 1.0 * $10^{-4}$ µg/$cm^2$ . 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 Co (II) with present method.*
**Key Words:** *Cobalt, NSS, Semicarbazone, nitrosalicylaldehyde, sandal's sensitivity.*

## Introduction

The cursory look at the above literature survey reveals that Cobalt 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 Co (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 Cobalt leave a scope for as many reagents as can be applied for the extraction and spectrophotometric determination of Cobalt.

The present work describes a spectrophotometric method for quantitative estimation of Co (II) with NSS.

## Materials and Methodology

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 spectra[2] 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 permangnate in 1M sulphuric acid. The observed spectrum was in good agreement with the spectrum reported in the literature.

## Results and Discussion

### Absorbance Maxima

The absorption spectrum of Co (II) NSS complex in Iso Amyl Alcohol shows maximum absorbance at 417 nm. The absorbance due to the reagent at this wavelength was negligible. Hence, 417 nm

was selected for the absorbance measurement[3] in spectro-photometric determination of Co (II) against blank.

**Solvent study**

Various solvents were tried to determine the maximum extraction of Cobalt (II). Iso Amyl Alcohol was found to be most suitable solvent[5] as it showed the highest extraction. The extraction of Cobalt (II) was minimum in solvents Chloform & Carbon Tetra Chloride.

**Effect of reagent concentration**

The effect of variation in concentration of NSS in the range of 0.1 to 4.0 $cm^3$ of 0.02% NSS on the extraction and the color development was tried. It was observed that 2.2 $cm^3$ of 0.02% NSS was sufficient for complete extraction and color development[6].

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

Different sorting out agents such as sulphates, chlorides, carbonates and nitrates of Sodium, Potassium, Barium, Magnesium, Ammonium and Calcium were used in the extraction of Co (II) (1$cm^3$ of 100 ppm solution). It was observed that there was no effect on absorbance.

**pH study**

The extraction of Cobalt (II) with NSS was carried out over pH range of 1 – 11, 1 $cm^3$ of aqueous solution of 100 ppm Cobalt (II) stock solution and 0.5 $cm^3$ of 0.02% solution of the reagent[4] were used. It reveals that 99% of metal is extracted into organic phase in the pH range between 8.0 to 8.4. It was found that complex does not form below pH 5. The analytical work for estimation of Cobalt (II) was carried out at pH 8.2. (Figure 1)

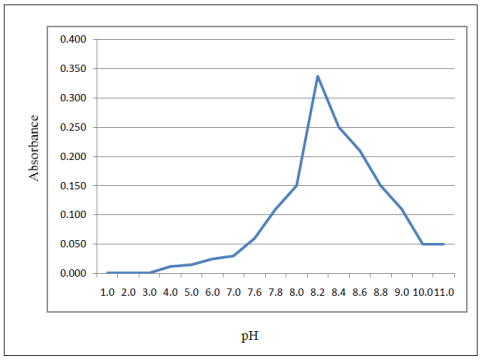


**Figure 1** Effect of pH on the extraction of Cobalt (II): NSS Complex

**Stability of complex with time**

For this study, 1$cm^3$ of 100 ppm solution was extracted with reagent in Iso Amyl Alcohol and absorbance of complex in Iso Amyl Alcohol 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 Cobalt (II) form 0.5 – 3.0 µg /$cm^3$ were extracted quantitatively under optimum experimental conditions and the Iso Amyl Alcohol extract was measured at 417 nm against reagent blank. The plot of absorbance against concentration of Cobalt (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 2.86 * $10^4$ $dm^3$ $mol^{-1}$ $cm^{-1}$ and the sensitivity of the method as defined by Sandal's was 1.0 * $10^{-4}$ µg / $cm^2$.

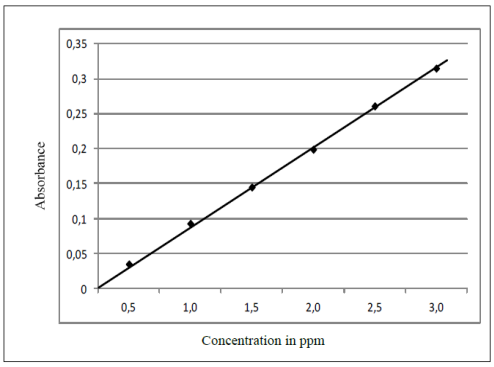


**Figure 2** Calibration Curve for Cobalt (II)

**Interference Study**

The effect of diverse ions on the Cobalt (II) determination was studies 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 Cobalt (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 Cobalt (II)[9].

**Effect of masking agents**

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

**Table: 1**

| Sr 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 Cynide |
| 14 | Pd (II) | Sodium Thiosulphate |

**Mole Ratio Method**

A series of solutions containing 1 $cm^3$ of 5.0 * $10^{-4}$ Co (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 Co (II)[8]. It shows a sharp break corresponding to mole ratio 1:4 which supports the composition of Co (II): NSS Complex.

**Job's Continuous Variation Method**

A series of solutions were prepared by mixing 0.0 to 10.0 $cm^3$ of 5.0 x $10^{-4}$ M Cobalt (II) solution with 10.0 to 0.0 $cm^3$ of 5.0 x 10-4 M NSS solution, such that the volume of each mixture was 10.0 $cm^3$ 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 Co (II) to NSS. It shows sharp maxima at 0.2 Mole Fraction of Co (II), indicating that the colored complex extracted into Ethyl acetate was formed by the reaction of Co (II) and NSS in the ratio 1:4.(Figure:3)

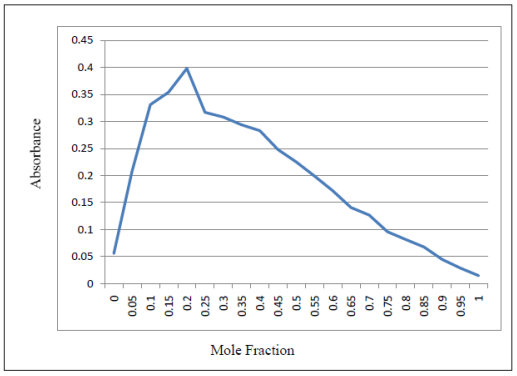


**Figure: 3** Job's Continuous Variation Method for Co (II) with NSS in aqueous medium.

**Nature of the extracted species**

The absorption spectrum of the Iso Amyl Alcohol extracts of the aqueous solution containing Co (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 w.r.t the metal concentration. The composition of the extracted species was determined by Job's Continuous Variation Method and verified by Mole Ratio Method[7].

**Equilibration Time**

The absorbance of Co (II) – NSS Complex was checked by varying the time of equilibration from 30 seconds to 15 minutes. It was observed that equilibration time of 1.5 minute was found to be optimum for complete extraction of Co (II).

**Applications of method**

The present method was applied for the determination of amount of Cobalt (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)

**Table: 2** Determination of Co (II) using NSS from Different Samples

| Sr No | Sample | Amount of Co (II) | |
|---|---|---|---|
| | | Standard Method | Present Method |
| Cobalt Alloys | | | |
| | | | |
| 1 | Steel | 9.66% | 9.65% |
| | | | |
| 1 | Vegetable Oil | 0.0020% | 0.0019% |
| | | | |
| Synthetic Mixture | | | |
| 1 | Co (10 mg) + Zn (10 mg) | 9.98mg | 9.98mg |
| 2 | Co (10 mg) + Mo (10 mg) | 9.99mg | 9.98mg |
| | Industrial Waste Water at Sarawali – Kalyan | 0.2ppm | 0.2ppm |

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