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# Extractive Spectrophotometric Determination Of Co (Ii) Using 5-Nitrosalicylaldehyde Thiosemicarbazone

# Lokhande R. S<sup>1.</sup>, Jain N.G.<sup>2\*</sup>, Jain K. N<sup>2</sup>., and Bhiwandkar P.R<sup>3</sup>.

## <sup>1</sup>Department of Chemistry, Jaipur National University, Jaipur, Rajasthan,India. <sup>2</sup>Department of Chemistry, B. N. N.Collage, Bhiwandi, District Thane,MH,Indai. <sup>3</sup>Unichem Lab. Ltd,India.

## \*Corres.author: ngjc1957@gmail.com, Mobile no: 9967598578, 8806864000

**Abstract:** 5-Nitrosalicylaldehyde thiosemicarbazone (NSTS) has been used for the effective separation through solvent extraction followed by spectrophotometric determination of Co (II) at trace level as well as for the studies of transition metals in complex form. Solvent extraction one of the separation technique can be advantageously employed for the determination of metals at low concentration in microgram by judicious use of spectrophotometric determination. In the present paper solvent extraction technique has been used to develop methods for the separation and spectrophotometric determination of Co(II) with 5-Nitrosalicylaldehyde thiosemicarbazone (NSTS) synthesized as per procedure. Complex formed was extracted into Ethyl acetate from the aqueous solution in the pH range 4.4 to 4.8. The absorption maxima was also tried for the extracted species and Beer's law was also studied for its applicability. During this study, interference from the foreign ion has also been examined.

**KeyWords:** Cobalt, Nitrosalicyl aldehyde thiosemicarbazone, Beer'slaw, Extractive spectrophotometric determination etc.

#### **INTRODUCTION**

A survey of literature <sup>(1-3)</sup> reveals that 5-Nitrosalicylaldehyde Thiosemicarbazone(NSTS) has not been investigated in details as an analytical reagent. NSTS is found to react with many metal ions to give the colour reactions of analytical importance. The present paper describes a sensitive and selective method for the photometric determination of Co (II) with (NSTS). Various methods have been reported from literature for the extraction and spectrophotometric determination of Co (II). The newly developed method is less time consuming.

#### EXPERIMENTAL

A standard solution of Co (II) (100 ppm) was prepared by dissolving required quantity of cobalt sulphate heptahydrate (A.R. grade) in double distilled water. 0.02% NSTS in methanol is prepared for investigation<sup>(4)</sup>.

Spectrophotometer Jasco (UV/VIS/NIR) model-630 was used for all the measurements of absorption spectra. Analytical balance Contech-120 (modelCA-123) with 0.001 g sensitivity was used for weighing. An Equiptronics (modelEQ-621) micro controller based pH meter used for pH measurements.

### PROCEDURE

To an aliquot containing Co (II) in the range 1.0 to 3.0 ppm, methanolic solution of  $NSTS^{(5,6,7)}(0.02\%)$  was added. The mixture was adjusted to pH 4.6 and extracted with 10 cm<sup>3</sup> (4+4+2) of ethyl acetate. The coloured organic layer was separated which was passed through anhydrous sodium sulphate to absorb moisture if any.

The volume was  $10.0 \text{ cm}^3$ . The absorbance of ethyl acetate extract was measured at 452 nm, against blank. The amount of Co (II) was found from the calibration curve.

#### **RESULT AND DISCUSSION**

#### **ABSORPTION SPECTRA**

Absorption spectrum of Co(II) complex was taken in wavelength region 400 to 800 nm containing 1.0 ppm Co (II). The wavelength maxima of the complex was found to be 452 nm. At this wavelength reagent blank shows negligible absorbance measurements.

#### EFFECT OF pH

The effect of variation of pH was studied in absorbance Co(II) complex at 452 nm. The result shows that pH 4.4 to 4.8 is suitable for complete extraction of Co(II).

#### EFFECT OF REAGENT CONCENTRATION

The reagent concentration on the absorbance Co(II) complex was studied by changing reagent concentration from 0.01 to 0.1%. 1.0 cm<sup>3</sup> of 0.02% concentration is required for colour development for 1.0 ppm Co(II).

#### **EFFECT OF EQUILIBRIUM TIME**

To determine the minimum equilibrium time for the extraction of Co(II): NSTS complex, various solutions containing 1.0 ppm of Co(II) and 1.0 cm<sup>3</sup> of 0.02% NSTS were equilibrated for different time intervals from 0.5 min to 15 mins keeping all other parameters constant.

Equilibration time for Co(II) : NSTS complex is 1.5 mins.

#### **CALIBRATION CURVE FOR Co(II)**

pH: 4.6

Aqueous phase: 0.5 to 3.0 ppm Co(II) solution +1.0 cm<sup>3</sup> of 0.02% NSTS in methanol.

Organic phase: Ethyl acetate.

Wavelength: 452 nm.

The plotting of absorbance against concentration of Co(II) gave a straight line indicating that the Beer's law is obeyed over the concentration range of 0.1 to  $3.0 \,\mu\text{g/cm}^3$  (Table-1).

#### DETERMINATION OF Co(II) USING NSTS

#### TABLE-1

Sr No	Concentration of Co (II) in µg / ml	Absorbance
1	0.5	0.149
2	1.0	0.291
3	1.5	0.457
4	2.0	0.629
5	2.5	0.809
6	3.0	0.953

Molar absoptivity =9.07 x10<sup>4</sup> dm<sup>3</sup>/mol/cm. Sandells sensitivity =  $3.13 \times 10^{-3} \mu g/cm^{-3}$ 

#### **EFFECT OF DIVERSE METAL IONS**

Various cations and anions were investigated in order to assess their tolerance in the extraction of Co(II)

The tolerance limit for the variation was fixed at  $\pm 2\%$  absorbance. For  $10\mu$ g/mL working solution of Co(II), it was found that bromide, chlorate, iodate, sulphate, nitrate, chloride, acetate,oxalate, tartarate, citrate, thorium(IV), manganese(II), cerium(IV), aluminium(III), zirconium(IV), mangnesium(II) did not interfere.

Thiourea, EDTA, nickel(II), selenium(VI), cadmium(II), mercury(II) and tin(II) interfere seriously; copper interference was masked with thiosulphate. (Table-2)

#### MASKING AGENTS REQUIRED TO SUPPRESS THE INTERFERENCE BY OTHER BY IONS

Aqueous phase:  $10.0 \text{ cm}^3$  containing 1.0 ppm of Co(II)+1.0 cm<sup>3</sup> of NSTS, interfering ion and masking agent.

Organic phase: 10.0 cm<sup>3</sup> ethyl acetate. Wavelength: 452 nm

#### TABLE-2

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 <sub>3</sub>

#### NATURE OF THE EXTRACTED SPECIES:

Job's variation method<sup>(8,9)</sup> indicates ratio as 1:1.5 which was further confirmed by mole ratio method.

#### **APPLICATION OF THE METHOD**

The method has been successfully applied for the determination of Co(II) in alloys, vegetable oil, synthetic mixtures and industrial effluent.

The results are then compared with the result obtained by known standard methods. It is that theresults obtained in the present method are in good agreement with the result from standard methods, confirming the validity of the developed method.(Table-3)

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

#### TABLE-3

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