DEVELOPMENT OF METHOD FOR EXTRACTIVE SPECTROPHOTOMETRIC DETERMINATION OF Co (II) WITH OF 2-[2-(4-CHLORO BENZOTHIOZOLE) IMINO]-5- NITRO PHENOL AS AN ANALYTICALREAGENT

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ABSTRACT

A spectrophotometric method has been developed for the determination of Co (II) using 2-[2-(4-Chloro Benzothiozole) imino]-5- nitro phenol as an extractive reagent. The reagent forms a coloured complex, which has been quantitatively extracted into n-butanol at $_{P}^{H}$ *9.4. The method obeys Beer's law over a range from 1 to 10 ppm. The maximum wavelength λmax for the reagent is 415nm. The Molar absorptivity and Sandell's sensitivity calculated are 0.1712 × 10*^{4} *L mol*$^{-1}$ *cm*$^{-1}$ *and 0.1455 µg cm*$^{-2}$ *respectively. The proposed method is very sensitive and selective. The method has been successfully applied to synthetic and commercial samples.*

Keywords*:* Cobalt, Spectrophotometric determination, n-Butanol,

2-[2- (4-Chloro Benzothiozole) imino] -5-Nitro Phenol.

1. INTRODUCTION

Cobalt and its compounds have great importance in metallurgical and biological field[1-2]. Cobalt compounds are used as catalyst also. These compounds are used as catalysts in hydrogenation, dehydrogenation, and hydrogenolysis including hydro nitrification and hydro sulfurization, hydro treatment of petroleum products, ammonoxidation, oxidation, hydroformylation, polymerization and ammonia synthesis. Most of the cobalt production is used in Mossbauer spectroscopy. It has great importance in alloy formation also. The extraction of cobalt[3-10] is of great importance at trace level. For the extractive separation of cobalt from aqueous solution either oxime or Organophosphorous based extraction are often employed[11-15.] The various Organophosphorous extractants have been preferred for cobalt. Due to great importance of cobalt, the

investigation methods for cobalt grew rapidly and got to be employed for its utilize and trace content in environment sample which incorporate spectrophotometry, AAS, Flow injection, Polarography, Chemiluminescence, HPLC, ICP-MS. In the present investigation 2- [2- (4- Chloro Benzothiozole) imino] -5 – Nitro Phenol (CBZTINP) is used for the first time as an analytical reagent for the spectrophotometric determination of cobalt (II) at trace levels.

2. EXPERIMENTAL

The reagent 2- [2- (4- Chloro Benzothiozole) imino] -5 – Nitro Phenol (CBZTINP) was prepared by the given procedure. The stock solution of Cobalt (II) was prepared by dissolving a weight amount of its acetate in double distilled water containing dilute acetic acid, which was diluted to the desired volume with double distilled water and standardized by tetrathiocyanatomercurate (II). Absorbance and p^H measurement were carried out on a Shimadzu UV- Visible 2100 spectrophotometer with 1cm quartz cells and digital p^H meter with combined glass electrode respectively.

2.1. Procedure for the extraction

1.0 mL of aqueous solution containing 0.1 mg of Cobalt metal and 1 mL of reagent were mixed in 50 mL beaker. The p^H of the solution adjusted to 8.8 with 0.2M boric acid and potassium chloride, keeping the volume 10 mL. The solution was transferred to 100 mL separatory funnel. The beaker was washed twice with n-butanol and transferred to the same funnel. The two phases were shaken for two minutes and allowed to separate. The organic phase was collected in 10 mL measuring flask and made up to the mark with organic solvent, if required. After separation of the two phases, the p^H of the aqueous phase was measured and the Co (II) in each phase was determined by tetrathiocyanatomercurate (II).

3. RESULTS AND DISCUSSION

The reagent CBZTINP forms orange coloured complex with Co (II), which was extracted into organic phase. The extraction of Co (II) froms an aqueous phase by CBZTINP in n-butanol is studied over a wide range experimental condition. The results of various studies are discussed below.

3.1. Extraction as a function of p^H

The extraction of cobalt with 2- [2- (4- Chloro Benzothiozole) imino] -5 – Nitro Phenol (CBZTINP) has been studied over the p^H range 1- 10 and was observed that percentage extraction of Co (II) is maximum at p^H 9.4.

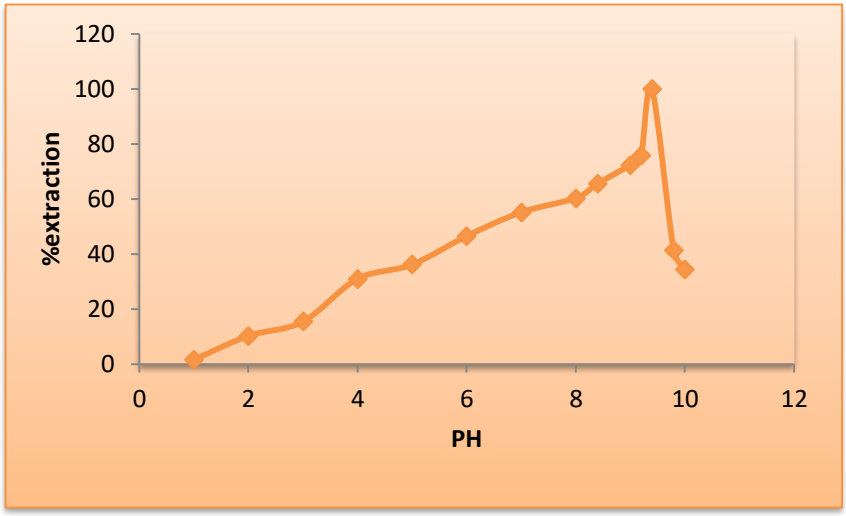

Fig 1: Percentage Extraction as a function of $_{P}{}^{H}$

3.2. Absorption spectrum

The absorption spectrum of Co (II): 2- [2- (4- Chloro Benzothiozole) imino] -5 – Nitro Phenol in n-butanol shows the maximum absorption at 415 nm. The absorption due to reagent at this wavelength is nearly negligible. Hence the absorption measurements were carried out at 415 nm.

3.3. Influence of diluents

The suitability of diluents was investigated using organic solvents such as chloroform, ethyl acetate, isoamyl alcohol, xylene, hexane, toluene, n-butanol, carbon tetra chloride. The extraction of Co (II) was quantitative with CBZTINP in n-butanol. Hence, n-butanol was used for further extraction studies as it gave better and quicker phase separation.

Sr. No.	Solvent	% Extraction	D
1	**n- butanol**	**99.99**	**9999**
2	Toluene	59.18	1.449
3	Chloroform	18.38	0.2249
4	Xylene	40.82	0.6896
5	Pentane	30.62	0.4411
6	Hexane	36.73	0.5806
7	Diethyl ether	22.45	0.2894
8	Ethyl acetate	22.12	0.2794
9	Ketone	26.53	0.3610

Table1: Influence of dilution

3.4. Effect of reagent concentration

Various volumes of 0.1% reagent solution were added to the sample solution containing 50µg of cobalt at respective $_{P}{}^{H}$ values. The absorbance remained nearly constant when the volume of the reagent solution used was 0.8 mL. Therefore, 1 mL of 0.1 % reagent was chosen for the quantitative determination of the metal.

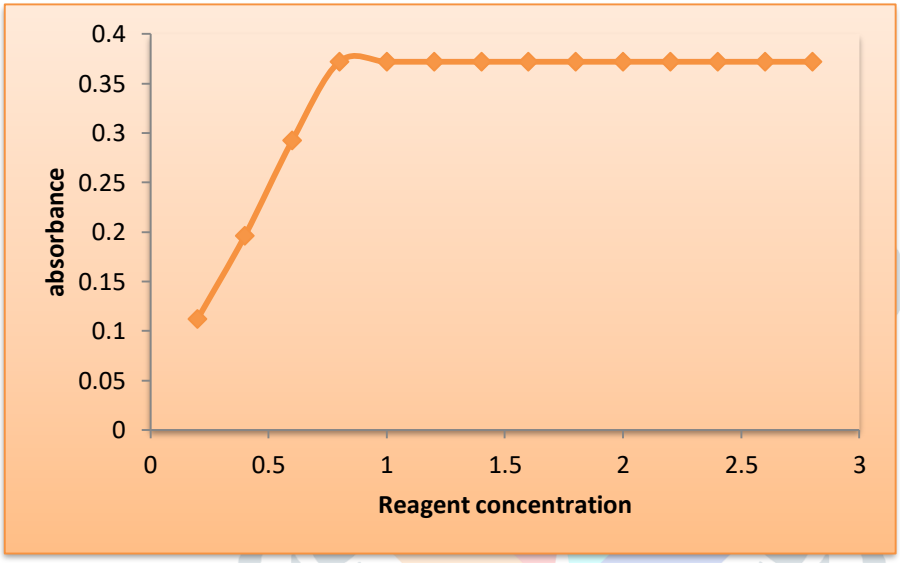

Fig. 2: Effect of reagent concentration

3.5 Effect of equilibrium time and stability of the complex

The study of change in absorbance with variation in equilibrium time extraction of the complex into organic solvent shows that equilibrium time of 60 second are sufficient for the quantitative extraction of cobalt. The study of stability of colour of the Co (II): CBZTZNP complex with respect to time shows that the absorbance due to extracted species is stable up to 40 hours, after which slight decrease in absorbance is observed. Throughout the experimental work, for practical convenience, the measurements have been carried out within one hour of extraction of cobalt.

Sr. No.	Time (Hours)	Absorbance
1	1	0.372
2	2	0.372
3	5	0.372
4	6	0.372
5	10	0.372
6	24	0.372
7	40	0.372

3.6. Calibration plot

A calibration plot of absorbance against varying cobalt concentration and fixed CBZTINP concentration gives linear and reproducible graph in the concentration range 1 to 10 ppm of cobalt. This shows that the Beer's law is obeyed in this range. The Molar absorptivity and Sandell sensitivity were calculated to be is 0.1712×10^4 L mol^{-1} cm^{-1} and 0.1455 µg $/cm^{-2}$ respectively.

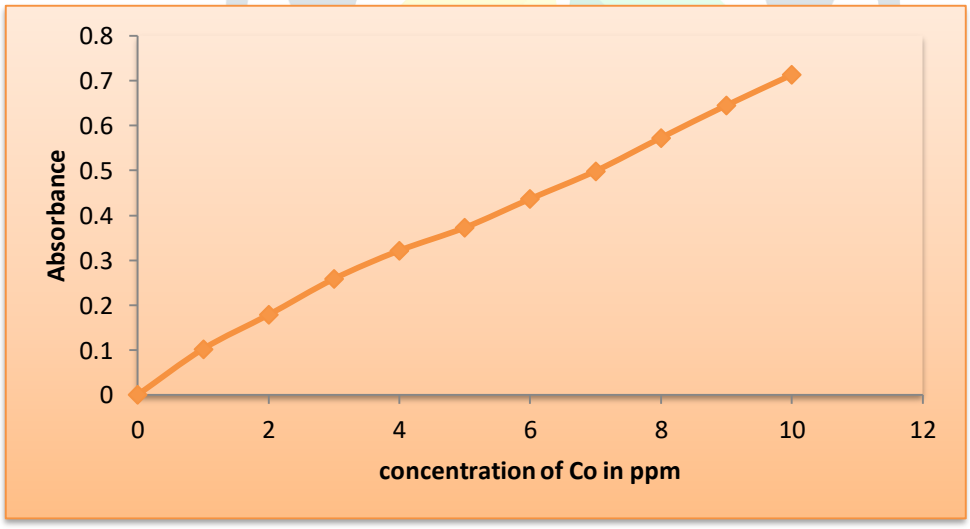

Fig. 3: Calibration plot of Co: CBZTINP complex

3.7. Nature of extracted species

The composition of extracted species has been determined by Job's continuous variation method, Slope ratio method and Mole ratio method. It shows that the composition of Co (II): CBZTINP complex is 1:2.

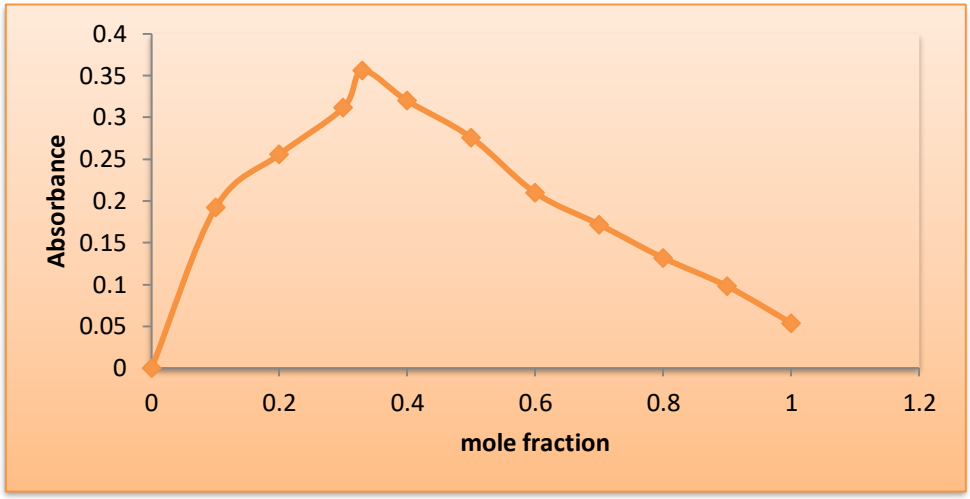

Fig. 4: Job's Continuous variation method for Co (II): CBZTINP complex

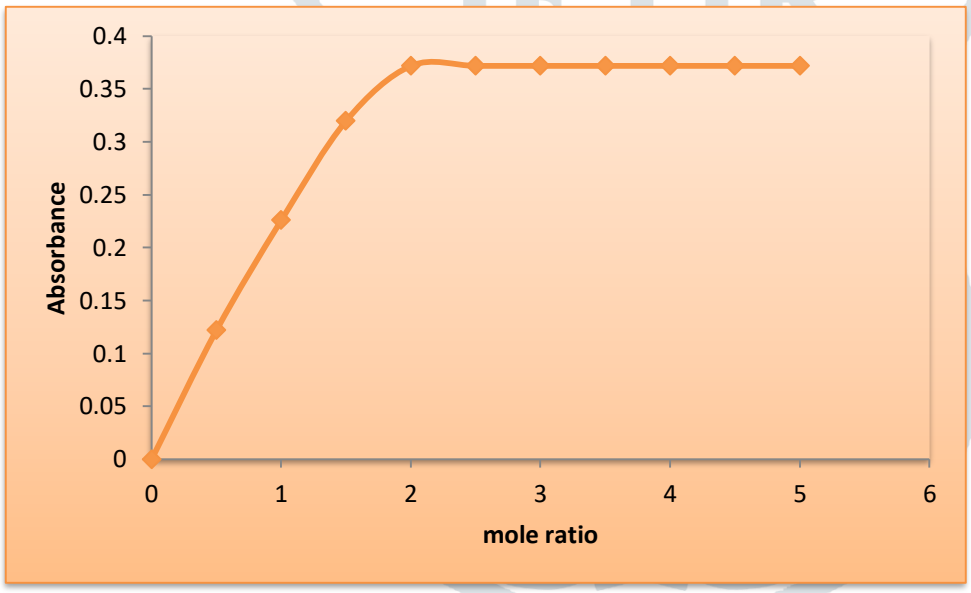

Fig.5: Mole Ratio of Co (II): CBZTINP complex

3.8. Precision and Accuracy

The precision and accuracy of the developed spectrophotometric method has been studied by analyzing five solutions each containing 50 µg of cobalt in the aqueous phase. The average of five determinations was 50.10 and variation from mean at 95% confidence limit was ± 0.4111.

3.9. Applications

Different commercial samples and synthetic mixtures containing Co (II) were prepared and analyzed according to the recommended procedure and the results were compared to those obtained by

standard method. The results found to be in good agreement with those obtained by the standard known method.

Sr. No.	Sample	Amount of Fe (II)	
		Standard method	**Present method**
1	Synthetic Mixture a) Co(s)+Mo(s) b) Co(s)+V(s)	 5.02ppm 5.01ppm	 4.99ppm 4.98ppm
2	Commercial Samples a) Glaxo (Inj.) b) Surbex-T c) Vit-B12	 4.35mg 0.326mg 50.0mg	 4.28mg 0.320mg 49.2mg

4. CONCLUSION

The proposed method is highly sensitive and selective methods for the extractive spectrophotometric determination of microgram amounts of cobalt. It offers advantages like reliability and reproducibility in addition to its simplicity, instant colour development and suffers from less interference. The 2- [2- (4- Chloro Benzothiozole) imino] -5 – Nitro Phenol (CBZTINP) was used for the first time for extraction of Co (II) from various mixture and real samples.

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