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Development of Method for Extractive Spectrophotometric Determination of Zn (II) with of 2-[2-(4-CHLORO BENZOTHIOZOLE) IMINO]-5- NITRO PHENOL as an Analytical Reagent

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Abstract: ***A spectrophotometric method has been developed for the determination of Zn (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*** $_{p}^{H}$ ***8.8. The method obeys Beer's law over a range from 1 to 10 ppm. The maximum wavelength λmax for the reagent is 388 nm. The Molar absorptivity and Sandell's sensitivity calculated are*** 0.2795×10^{4} ***L*** mol^{-1} cm^{-1} ***and 0.1028 µg*** cm^{-2} ***respectively. The proposed method is very sensitive and selective. The method has been successfully applied to synthetic and commercial samples.***

Keywords: Zinc, Spectrophotometric determination, n-Butanol, 2-[2- (4-Chloro Benzothiozole) imino] -5-Nitro Phenol.

I. INTRODUCTION

Zinc is an important element for human being including animal at trace level. It is an industrially important element as a biological nutrient, epidemiological preventive, toxicant and environmental pollutant [1-6]. It plays an essential role in human blood distributed 75-85% in erythrocytes, 12 to 22% in plasma and 3% in leukocytes. Zinc is present in many complexes of histidine and cysteine and many enzyme systems [7-10]. One-third of present zinc production is used in galvanizing ferrous metals. Brass alloy has been converted in many chemical compounds. Zinc deficiency leads to impaired DNA synthesis, delayed wound healing and decrease in collagen synthesis. Deficiency of zinc leads to retarded growth, lower feed efficiency, inhibits the general well being, causes ulcers, scaling of the skin, besides affecting the bones and joints.

Spectroscopy is an important technique for a trace analysis of element and it is one of the most powerful tools in chemical analysis. There are numerous methods for the spectrophotometric determination of zinc at trace level [11-16]. 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 zinc (II) at trace levels.

II. EXPERIMENTAL

The reagent 2- [2- (4- Chloro Benzothiozole) imino] -5 – Nitro Phenol (CBZTINP) was prepared by the given procedure. The stock solution of Zinc (II) was prepared by dissolving a weight amount of its sulphate in double distilled water containing dilute sulphuric acid, which was diluted to the desired volume with double distilled water and standardized by 8-hydroxyquinaldehyde method. 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.

A. Procedure for the extraction

1.0 mL of aqueous solution containing 0.1 mg of Zinc 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 Zn (II) in each phase was determined by 8-hydroxyquinaldehyde.

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III. RESULTS AND DISCUSSION

The reagent CBZTINP forms yellow coloured complex with Zn (II), which was extracted into organic phase. The extraction of Zn (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.

A. Extraction as a Function of p^H

The extraction of zinc with 2- [2- (4- Chloro Benzothiozole) imino] -5 – Nitro Phenol has been studied over the $_pH$ range 1- 10 and was observed that percentage extraction of Zn (II) is maximum at $_pH$ 8.8.

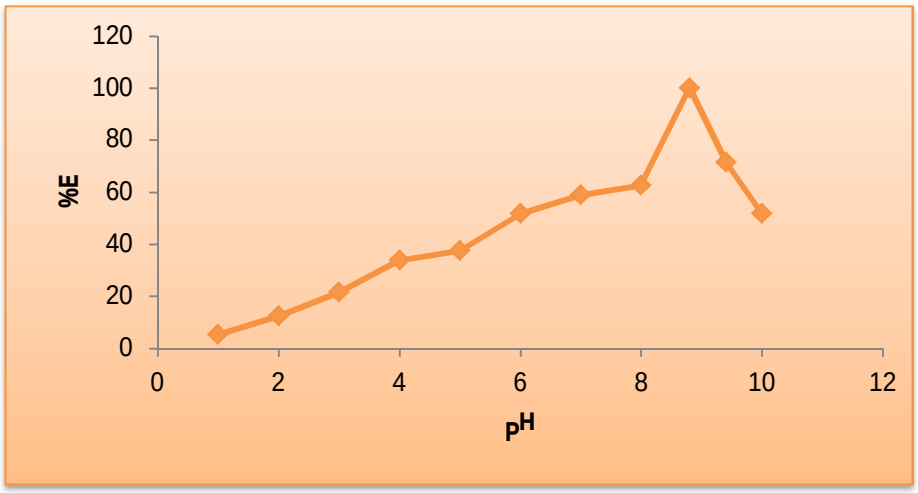


Fig 1: Percentage Extraction as a function of $_pH$

B. Absorption Spectrum

The absorption spectrum of Zn (II): 2- [2- (4- Chloro Benzothiozole) imino] -5 – Nitro Phenol in n-butanol shows the maximum absorption at 388 nm. The absorption due to reagent at this wavelength is nearly negligible. Hence the absorption measurements were carried out at 388 nm.

C. 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 Zn (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	30.52	0.4392
3	Chloroform	40.81	0.6890
4	Xylene	24.48	0.3240
5	Pentane	34.69	0.531
6	Hexane	36.73	0.581
7	Diethyl ether	18.37	0.225
8	Ethyl acetate	20.40	0.256
9	Ketone	32.65	0.485

Table 1: Influence of dilution

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D. Effect Of Reagent Concentration

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

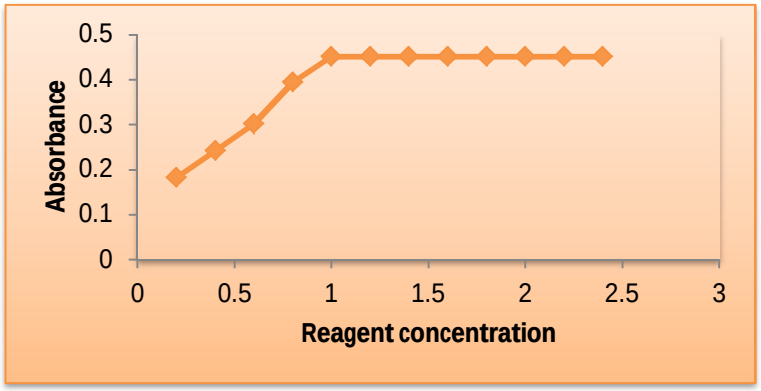


Fig. 2: Effect of reagent concentration

E. 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 zinc. The study of stability of colour of the Zn (II): CBZTZNP complex with respect to time shows that the absorbance due to extracted species is stable up to 30 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 Zinc.

Sr. No.	Time (Hours)	Absorbance
1	1	0.452
2	2	0.452
3	5	0.452
4	6	0.452
5	10	0.452
6	24	0.452
7	30	0.452

F. Calibration Plot

A calibration plot of absorbance against varying zinc concentration and fixed CBZTINP concentration gives linear and reproducible graph in the concentration range 1 to 10 ppm of zinc. This shows that the Beer's law is obeyed in this range. The Molar absorptivity and Sandell sensitivity were calculated to be is 0.2795×10^4 L mol^{-1} cm^{-1} and 0.1028 µg $/cm^{-2}$ respectively.

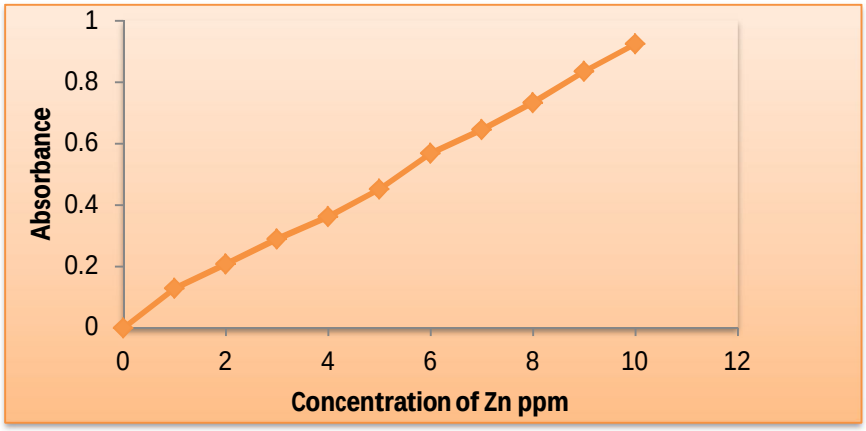


Fig. 3: Calibration plot of Zn: CBZTINP complex

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G. 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 Zn (II): CBZTINP complex is 1:1.

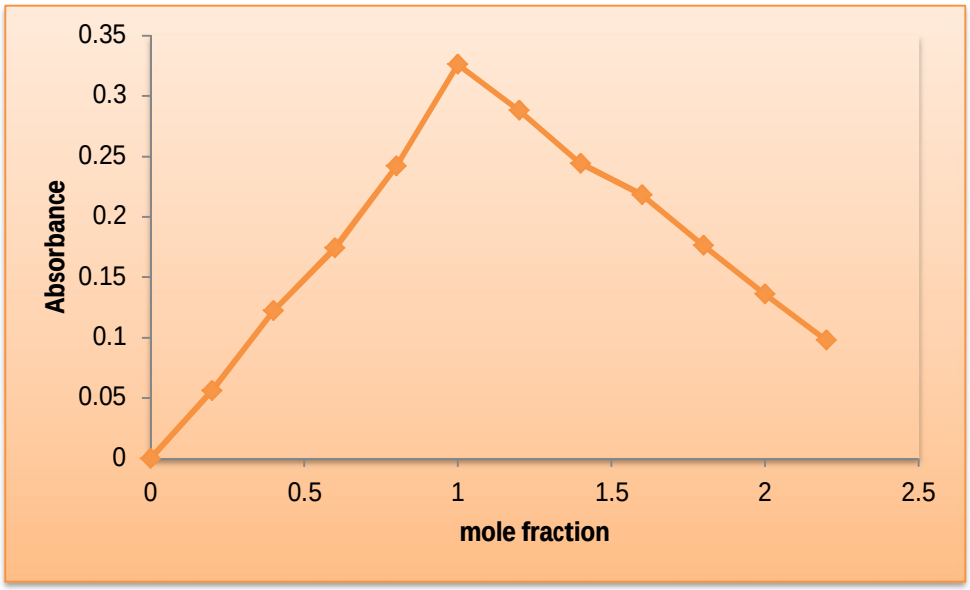


Fig. 4: Job's Continuous variation method for Zn (II): CBZTINP complex

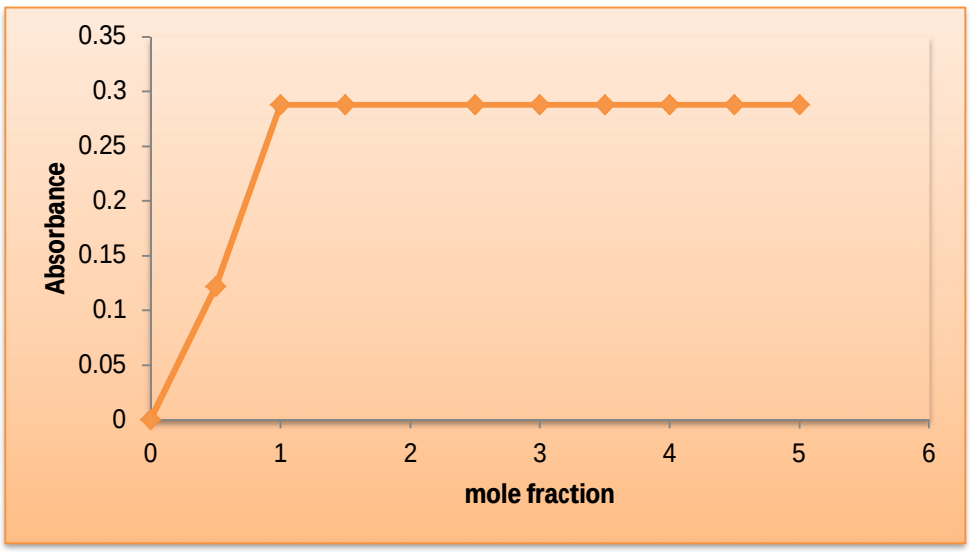


Fig.5: Mole Ratio of Zn (II): CBZTINP complex

H. Precision and Accuracy

The precision and accuracy of the developed spectrophotometric method has been studied by analyzing five solutions each containing 50 µg of zinc in the aqueous phase. The average of five determinations was 50.0 and variation from mean at 95% confidence limit was ± 0.5368.

I. Applications

Different commercial samples and synthetic mixtures containing Zn (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.

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Sr. No.	Name of Sample	Composition certified value mg/tablet	Amount of Zn (II) found mg/tablet	
			Standard method (FAAS method)	Present method
1	Antoxid	Zinc sulphate monohydrate,27.45 (equivalent to element zinc,9.99); Selenium oxide,70µg	9.99	9.56
2	Becozine	Zinc sulphate monohydrate, 54.93mg (equivalent to elemental zinc, 199.99 mg); Niacinamide, 50mg; calcium panttothenate,2.5mg; folic acid,1mg	19.99	19.91
			Conc.(µg mL^{-})	Present method
1	A	Zn^{+2} +Ni^{+2} +SO_4^{-2} +EDTA	3.00	2.98
2	B	Zn^{+2} +Ni^{+2} +Mg^{+2} +SO_4^{-2} +EDTA	3.00	2.97

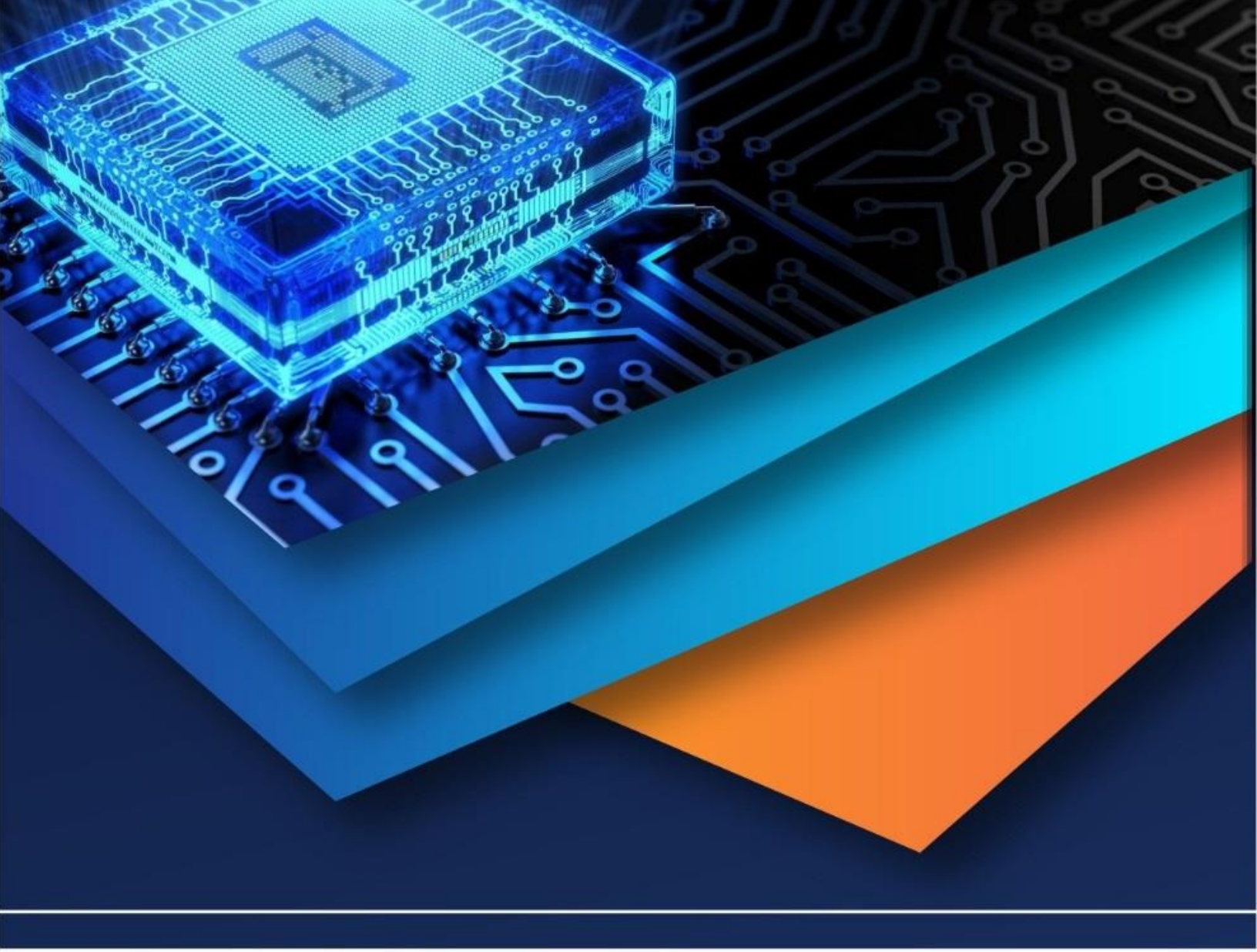
IV. CONCLUSION

The proposed method is highly sensitive and selective methods for the extractive spectrophotometric determination of microgram amounts of zinc. 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 Zn (II) from various mixture and real samples.

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