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Extractive Spectrophotometric Estimation of Zinc from food and pharmaceutical samples using 1-phenyl-1-hydrazonyl-2-oximino propane –1, 2 –dione reagent

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ABSTRACT

An accurate, inexpensive and less laborious liquid–liquid extractive spectrophotometric procedure for the determination of Zinc in aqueous media has been developed. 1-phenyl-1-hydrazonyl-2-oximino propane – 1,2 – dione (HPHOPD) reagent was used for extractive spectrophotometric determination of Zinc (II). HPHOPD reagent formed a complex with Zinc (II) in n-butanol. Comprehensive study was carried out for maximum extraction conditions. Maximum extraction was obtained at pH 8.5. The extracted species has absorption maxima at 415 nm and obeyed Beer's law over the range 1 – 20 ppm of Zinc. The molar absorptivity at this wave length is 0.156 × 10^3 Lit $Mol^{-1}cm^{-1}$. The proposed method is selective for Zinc (II) and was satisfactorily applied to the determination of the total Zinc in food and pharmaceutical samples.

Keywords: Zinc (II), extractive spectrophotometric, HPHOPD, food and pharmaceutical samples

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INTRODUCTION

In biology, Zinc is as common as iron [1]. It is an important trace element and plays a vital role in normal growth and development [2 - 6] (José 1995, María 2002). Many biological functions like cellular integrity, protein synthesis and nucleic acid metabolism require Zinc. It plays significant role in brain development and also as an antioxidant [7 – 10]. Zinc is an essential micronutrient [11 - 12].

The studies have been carried out about the Zinc deficiency [13] and its association with diseases like HIV infection [14], Diabetics [15] and cancer risk [16] etc. Zinc deficiency during pregnancy can have adverse effect on both mother and foetus [8]. Hence Zinc in nutrition is indispensable for health.

An extractive spectrophotometric technique is a separation method [17] which allows the determination of a metal in organic phase without using stripping solvent. It is simple and economical method [18 – 20]. Current endeavor accomplished the maximum extraction of zinc by varying different parameters of extraction. The method was successfully carried out for determination of Zinc in food samples and pharmaceutical product by extractive spectrophotometric method.

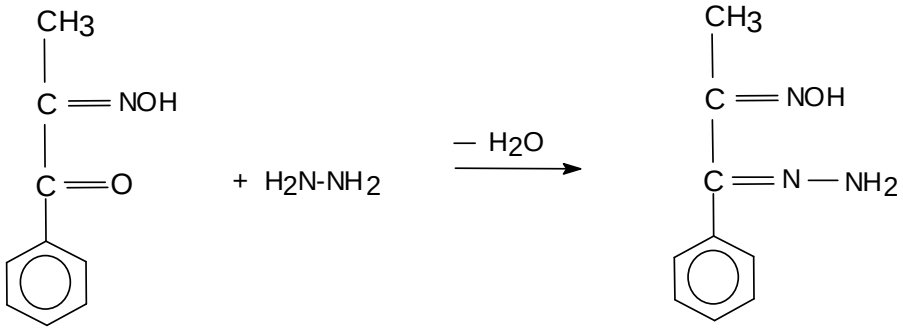
MATERIALS AND METHODS

The stock solution of Zn (II) were prepared from Zinc chloride (Loba Chemie). The stock solution was standardized gravimetrically by Zn pyrophosphte.

A digital pH meter, (Elico Private Ltd, India) with a combined glass and calomel electrode (Toshniwal - Mollar, India) and UV 2100 spectrophotometer (Shimadzu) with glass cells of path length 1 cm was used.

Synthesis of 1-phenyl-1-hydrazonyl-2-oximino propane – 1,2 – dione (HPHOPD) reagent:
The reagent HPHOPD was synthesized [18] by carrying out a reaction between *iso*-nitrosopropiophenone and 85 % hydrazine hydrate. The purity of the product was checked by melting point and GC-MS technique.

Chemical reaction:



iso-nitrosopropiophenone 1-phenyl-1-hydrazonyl-2-oximinopropane–1,2–dione

Extraction Procedure:
An aqueous solution (10.0 cm^3) containing 0.1 mg Zn (II) metal and 0.005 M of 1-phenyl-1-hydrazonyl-2-oximino propane -1,2- dione reagent in n-butanol, after adjusting the pH = 8.5 was equilibrated with 10.0 cm^3 of n-butanol for 1 min. After separation of the phases, the absorbance of the Zn (II) : HPHOPD complex in organic phase was directly measured at 415 nm.

Preparation of Pharmaceutical samples :
For determination of Zn (II) from Samples
To a 20.0 cm^3 of injectible liquid or 40.0 –50.0 cm^3 Multivitamin Syrup or a 5 gm of tablet powder, 1.0 cm^3 of concentrated HCl : HNO_3 (1:1) was added and evaporated to dryness. It was treated with 5.0 cm^3 of 30 % H_2O_2 until solution became colorless. The colourless solution was then treated with dil. HCl and evaporated to dryness. The residue was dissolved in 10.0 cm^3 of distilled water and an aliquot of this was used for further analysis.

RESULTS AND DISCUSSION

Absorption spectrum
Zn : HPHOPD complex after extraction from aqueous phase into organic phase was scanned from 300 nm to 600 nm against reagent blank (Figure 1). Maximum Absorbance value was observed at 415 nm. Therefore, 415 nm was selected for the absorbance measurement throughout the experiments.

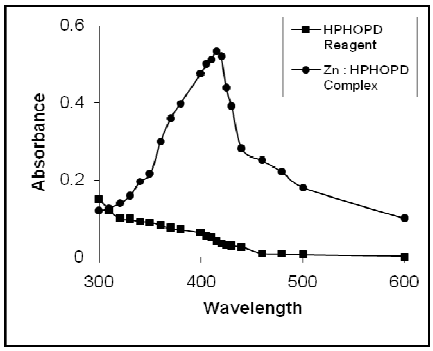


Figure 1. Absorption Spectrum of Zn (II) : HPHOPD Complex

Systematic Study of extraction of Zn (II):
A systematic study of the extraction of Zn (II) was carried to obtain the conditions where maximum extraction was obtained. Different parameters were studied which are as follows -

Effect of pH
The extraction of Zinc was carried out over the pH range 1.0 -10.0 (Figure 2). It was observed that the extraction was increased upto 8.5 pH and decreased beyond it. Therefore, pH 8.5 was used further for all experiments.

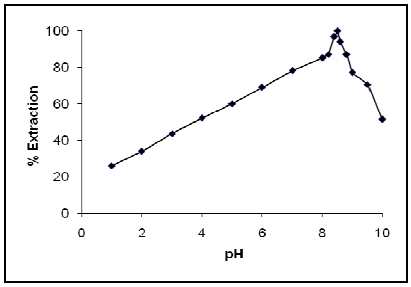


Figure 2. Effect of pH on extraction of Zn (II) : HPHOPD Complex

Effect of different solvents

Different organic solvents were used for the extraction of Zn (II) : HPHOPD complex. When n-Butanol was used as organic phase, maximum extraction and quick phase separation was obtained. Therefore, n-Butanol was used as organic phase for all experiments.

Effect of HPHOPD reagent concentration

The concentration of HPHOPD reagent was varied from 0.001 M to 0.005 M (Figure 3). It was observed that as the reagent concentration increased percent extraction increased. Maximum extraction was observed at 0.005 M concentration of HPHOPD reagent. Therefore, 0.005 M HPHOPD was used for extraction.

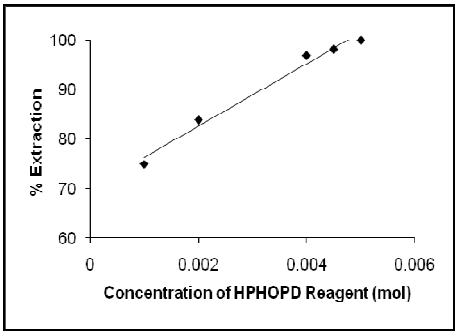


Figure 3. Effect of HPHOPD reagent concentration

Effect of equilibration time

Equilibration time for the extraction of Zn (II) : HPHOPD complex from aqueous phase to organic phase was varied from 0.5, 1 and 2 minutes. It was observed that 1.0 minute was sufficient equilibration time for maximum extraction. Therefore, 1 minute was used as equilibration time for all experiments.

Calibration plot

A calibration plot of absorbance against concentration of Zn (II) : HPHOPD complex gave a linear and reproducible graph in the concentration range of 1- 20 ppm (Figure 4). The Beer's law is obeyed in this range. The molar absorptivity was 0.156 X 10 ^{3} Lit Mol^{-1}cm^{-1}.

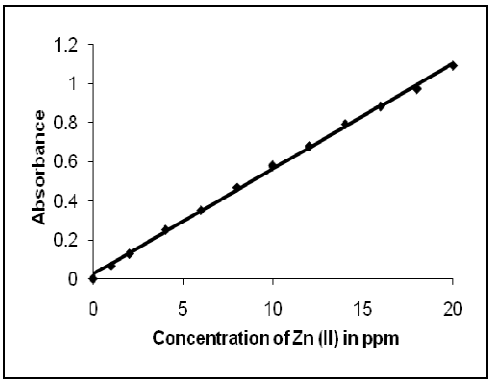


Figure 4. Calibration plot

Effect of diverse ion concentration

Extraction of Zn (II) was carried out in presence of different metals ions (Table 1). The tolerance limit was set to that amount of foreign ion causing ± 2% error in recovery of Zn (II). It was observed that metal ions like Ni^{+2}, Mg^{+2}, Ca^{+2}, Pb^{+2}, Mn^{+2}, Al^{+3} and Fe^{+3} showed high tolerance limit. Therefore the optimized parameters can use for maximum extraction of Zn (II) even in presence of diverse ions.

Table 1. Effect of diverse ion concentration Zn (II) concentration: 10 ppm

Sr. No.	Ion	Tolerance limit (ppm)
1.	Ni^{+2}	15.0
2.	Mg^{+2}	18.0
3.	Ca^{+2}	16.0
4.	Pb^{+2}	19.2
5.	Mn^{+2}	17.8
6.	Al^{+3}	20.0
7.	Fe^{+3}	20.0

Quantitative determination of Zn (II) from Samples

Food Samples

Juices (from local Juice centre) like Orange Juice, Pineapple Juice and Mix Fruit Juice were analyzed for Zinc contents (Table 2). The percent extraction was in the 90 – 95 % range.

Table 2. Determination of Zinc (II) from Food Samples

Sr. No.	Sample	% Recovery of Zn (II)*
1	Orange Juice	95.2
2.	Pineapple Juice	92.3
3.	Mixed Fruit Juice	91.0

***The values of percent extraction of Zinc are mean of three readings.*

Pharmaceutical Samples

Extraction of Zinc was carried out from Injectable liquid, Multivitamin Syrup and tablet (Branded). The results are included in Table 3.

Table 3. Determination of Zinc (II) from Pharmaceutical Samples

Sr. No.	Sample	% Recovery of Zn (II)*
1.	Injectable liquid	99.2
2.	Multivitamin Syrup	98.6
3.	Multivitamin Tablet	95.2

***The values of percent extraction of Zinc are mean of three readings.*

CONCLUSION

Zinc formed a complex with HPHOPD which was extracted in n-butanol at pH = 8.5 quantitatively. The interference of various ions was studied and optimum conditions were developed for the determination of Zinc metal in food and Pharmaceutical samples. The advantage of this process is the ease, rapid reliable and economical.

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