

Spectrophotometric Determination of Zinc in Water Samples Using 3-Hydroxybenzylaminobenzoic Acid

K. KIRAN

Department of Environmental Sciences, Sri Venkateswara University,
Tirupati-517502, India

kiran.sairam@gmail.com

Received 9 June 2012 / Accepted 7 July 2012

Abstract: 3-Hydroxybenzylaminobenzoic acid synthesized in the laboratory as a reagent for the determination of zinc in various water samples. Zinc(II) reacts with 3-hydroxybenzylaminobenzoic acid and forms a light yellow colored complex at pH 5.0. The complex has a maximum absorbance at 460 nm. The complex is stable for more than twelve hours. Hence, a detailed study of the extraction of zinc(II) with 3-hydroxybenzylaminobenzoic acid has been studied. Other parameters like effect of pH, choice of solvent, interference of electrolytes has also been investigated.

Keywords: 3-Hydroxybenzylaminobenzoic acid, Zn(II), Water samples

Introduction

Zinc is an essential element and it plays an important physiological role in human beings. Zinc is present in many alloys and is also found in a number of pharmaceutical samples and in airborne particulates, causing environmental pollution. Concentration of zinc greater than 5.0 ppm affects the potable nature in alkaline waters. The determination of zinc in water is also of considerable interest to investigate further. Zinc also has industrial significance. About one-third of the present zinc production is used in galvanizing ferrous metals. Brass alloys consume another one-third of the world zinc production, while the remaining zinc is converted into a number of chemical products. It is clear that zinc is an essential element and has significant importance, both biologically and industrially. When the quantity is more than what is required, zinc produces toxic effects. Hence, separation and determination of zinc(II) from its associated metal ions is indispensable.

Organic reagents like Victoria blue¹, Semi xylenol orange², Methyl thymol blue³ and Semi methyl thymol blue⁴ Xylenol orange⁵ are some of the organic reagents used for the spectrophotometric determination of zinc(II). 1-(Phenyl-2-pyridyl)-carbilidime-5-resorcili-dinethiocarbohydrazone for the spectrophotometric determination of zinc(II) was reported by Gonazalez and Perez⁶. Several authors used 2,2'-bipyridyl-2-pyridylhydrazone⁷, for the determination of zinc(II) in various samples. 2,3'-(Sulphobenzoyl) pyridine-4-phenyl-3-

thiosemicarbazone is used for the spectrophotometric determination of zinc(II) by Rodriguez and Pavon⁸. Wasey *et al*⁹ reported 2-oximinodimedonedithiosemicarbazone for the extractive spectrophotometric determination of zinc(II). Many reported methods¹⁰⁻¹² have been employed for the determination of Zn(II) in various environmental samples. In the present work, the author used 3-hydroxy benzyl amino benzoic acid synthesized in the laboratory as a reagent for the determination of zinc in various water samples.

Experimental

All reagents were purchased from local market. The stock and standard solutions were prepared freshly throughout the experiment. Under recommended conditions, Zn(II) metal was determined in various water samples following UV-VIS Spectrophotometer. An Elico pH meter was used for the pH adjustments.

General procedure for the preparation of ligand

1.0 g of 3-Hydroxybenzaldehyde was dissolved in 25 mL of double distilled water and mixed in a flask with 1.0 g of 4-aminobenzoic acid and refluxed for 3 h. A pale yellow colored crystal product was formed. After filtering the product, it was dried at room temperature. Finally the product was recrystallized by using ethanol. The resulting product has melting point of 165 °C and the yield was 80-90%. The above reaction is shown in Figure 1.

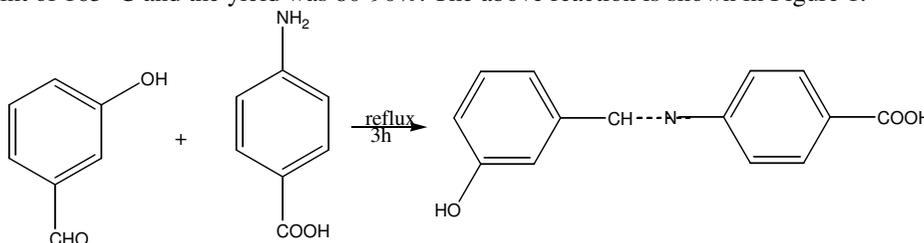


Figure 1. Schematic diagram of 3-hydroxybenzylaminobenzoic acid

Results and Discussion

Zinc(II) reacts with 3-hydroxybenzylaminobenzoic acid and forms a light yellow colored complex at pH 5.0. The complex has a maximum absorbance at 460 nm. The complex is stable for more than twelve hours. Hence, a detailed study of the extraction of zinc(II) with 3-hydroxy benzyl amino benzoic acid has been studied.

Absorption spectrum of the reagent complex

1.0 mL of zinc(II) solution was transferred into a 25 mL standard flask and to it, 3.0 mL of buffer (pH 5.0), 2.0 mL of 3-hydroxybenzylaminobenzoic acid solutions were added and the volume of the aqueous phase was brought upto 10.0 mL with double distilled water. The absorption spectrum of the complex was recorded. The absorption spectrum of the reagent was also recorded, using the solvent as a blank. The absorption spectrum of the reagent complex is depicted in Figure 2. The reagent metal complex gave maximum absorbance at 460 nm.

Choice of solvent

In this method various organic solvents such as MIBK, *n*-butanol, benzene, chloroform, carbontetrachloride, ethyl acetate *etc.*, were used for the extraction of 3-hydroxy benzyl amino benzoic acid - zinc complex. Maximum absorbance was obtained in MIBK. Hence, MIBK solvent was chosen in further investigations.

Effect of pH

The effect of pH on the formation of Zn(II)- 3-hydroxybenzylaminobenzoic acid complex was studied to find out the optimum pH for Zn(II) determination. The studies were carried out keeping the 0.5 mL of zinc(II) solution and 1.0 mL of 3-hydroxy benzyl amino benzoic acid solution constant and varying the pH values of aqueous phases from 2.0 to 8.0 using suitable buffer solutions. The volume of each aqueous phase was adjusted to 10.0 mL with double distilled water. The plot between pH and the absorbance is shown in Figure 3. From the graph, it is observed that the extraction of the metal ion increases with increase of pH from 3.0 to 6.0. At pH 5.0 Zn(II)- 3-hydroxy benzyl amino benzoic acid complex gives maximum absorbance values. Hence, pH 5.0 was chosen for further investigations.

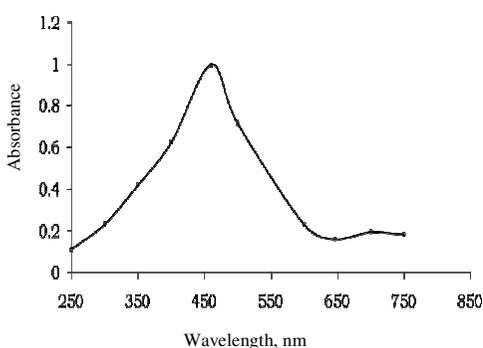


Figure 2. Absorption spectrum of 3-hydroxy benzyl amino benzoic acid-zinc complex

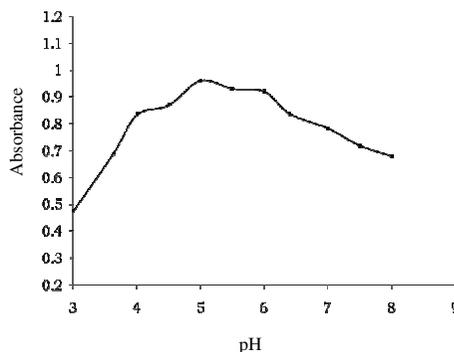


Figure 3. variation of absorbance with pH for 3-hydroxy benzyl amino benzoic acid-zinc complex

Effect of reagent concentration

The effect of reagent concentration has been studied by keeping 1.0 mL of zinc(II) solution and 3.0 mL of pH 5.0 buffer constant. The concentration of 3-hydroxybenzylaminobenzoic acid was varied between 1.0×10^{-3} M and 11.0×10^{-3} M to obtain maximum color formation. The total volume of aqueous phases was brought to 10.0 mL with double distilled water. The aqueous phases were shaken with 10.0 mL of *n*-butanol in each case; the organic phases collected in 25 mL standard flasks and made upto 25 mL with *n*-butanol. The absorbance of these phases was measured at 460 nm, against their corresponding reagent blanks. The experiment revealed that six folds of reagent to that of metal ion are sufficient for maximum color development of the complex. Hence, a six-fold molar excess of the reagent is maintained for all further studies.

Effect of foreign ions

In order to assess possible analytical applications of the method, the effect of diverse ions on the extraction and spectrophotometric determination of zinc were studied. The results are presented in Table 1. The other tested metallic and anionic species had no adverse effect on the analytical signal(s) of zinc in water samples.

Sensitive and molar absorptivity of Zn(II)-3-hydroxybenzylaminobenzoic acid complex

The molar absorptivity of the complex is calculated and noted as 1.2×10^4 L mol⁻¹ cm⁻¹ and the Sandell's sensitivity of the complex is 3.875×10^{-3} cm² μg.

Table 1. Tolerent limit of electrolytes

Species	Limiting concentration (molar ratio)
KNO ₃	<960
NaCl	<715
Na ₂ SO ₄	<450
Al(NO ₃) ₃	<900
NaH ₂ PO ₄	<960
NaNO ₂	<400
CaCl ₂	<1000
NaHCO ₃	<800
CuSO ₄	<730
K ₃ PO ₄	<1000
KH ₂ PO ₄	<900

Stability of the color reaction

The absorbance values of Zn(II)- 3-hydroxybenzylaminobenzoic acid complex were noted at different intervals of time at 460 nm. It was observed that the absorbance remained constant up to ten hours. This indicates that the color of the complex is stable for at least ten hours making it suitable for accurate estimation of zinc.

Applications of the developed method

The proposed method was applied for the determination of zinc(II) in water and industrial effluent samples.

Analysis of water samples

Samples were collected from industrial areas in and around Tirupati. Pre-treatment of waste water samples was done as per the procedure described in the literature and analysed for zinc(II) by the present analytical procedure. The values determined with the present reagent are compared with the values estimated with another standard reagent leuco methylene green and the results are shown in Table 2. The results are indicative that the present reagent is very well suitable for estimation of zinc in water samples.

Table 2. Determination of Zn in various water samples

Sample	Standard method ^a , µg mL ⁻¹	Present Method, µg mL ⁻¹	Recovery, %
Drinking water ^b	0.33	0.29±0.06	88.22
Bore well water ^c	0.90	0.88± 0.04	97.72
River water ^d	0.94	0.92±0.02	97.90
Dam water ^e	0.91	0.91± 0.01	100.00
Polluted water ^f	1.02	1,00±0.04	99.00

^aUsing Leuco Methylene Green procured from local market, ^bTelugu Ganga water, Tirupati, ^cCollected from 4km around Tirupati, ^dSwarnamukhi river, Srikalahasti, ^eKalyani water dam, Chandragiri, ^fIndustrial waste water, Gajulamandyam, Tirupati

Conclusion

The present investigations were carried out with a view to test the potentially of 3-hydroxybenzylaminobenzoic acid as a complexing agent for Zn(II) present in various environmental

samples especially in water samples and its subsequent determination by extractive spectrophotometry. The method has shown good sensitivity, reliability and easy preparation of the reagent compared with other existing extractive spectrophotometric determination methods.

Acknowledgement

This work was carried out as a part of my Research on determination of selected metals using novel reagents in various environmental samples. I thank P. Nagaraju for his participation in sample collection. The author is also grateful to Professor K. Janardhanam to support me to carry out the experiment.

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