SPECTROPHOTOMERIC DETERMINATION OF COPPER (II) BY ADSORPTION OF ITS N-p-IODOPHENYL THIOBENZOHYDROXAMIC ACID COMPLEX ON MICROCRYSTALLINE NAPHTHALENE.

ATUL KUMAR AND HARSUKH RAM CHHARANG
SBRM GOVT. COLLEGE, NAGAUR, RAJASTHAN

ABSTRACT

A reliable and Sensitive photometric method for the determination of copper (II) with Iodophenyl Thiobenzohydroxamic Acid (IPTBHA) has been described. A stable water insoluble complex formed from copper (II) and IPTPHA cannot be directly extracted into benzene and chloroform, but can be easily absorbed on microcrystalline naphthalene at room temperature. The adsorbed complex is dissolved in DMF. The maximum absorbance has been found at 395 nm. Its absorption obeys Beer's law over the range 3 80 µg of copper (II) in 10 ml of DMF. The molar absorptivity is found to be 6.7×10^4 $Lmol^{-1}cm^{-1}$ and the sensitivity being 0.018 µg cm^{-2} of copper (II) for the absorbance of 0.001.

Key Words – N-p-IODOPHENYL THIOBENZOHYDROXAMIC ACID(IPTBHA)**,** Copper (II), Absorptivity, Absorbance, Beer's law, DMF

INTRODUCTION

Thiohydroxamic acids have been widely investigated as chelating agents[1-4] due to their biocidal activity. Survey of the literature reveals that analytical aspects of thiohydroxamic acids have not been studied[5-6] in detail.

The present communication describes a sensitive and selective photometric method for the determination of copper (II) with N-p-Iodophenyl thiobenzohydroxamic Acid.

The usual liquid - liquid extraction method cannot be used directly for the extraction of metal ions that form complexes with the complexing reagent at a high temperature. This difficulty can be avoided with the method of solid-liquid separation after liquid-liquid extraction developed by Fujinaga and co-workers using naphthalene as an extractant[7]. This method has many advantages over the usual liquid-liquid extraction[8-9].

In order to over come draw-backs of liquid-liquid extraction method a new method i.e. solid liquid separation after adsorption of metal chelate on microcrystalline naphthalene was developed[10-15]. The method is very convenient and less time consuming.

EXPERIMENTAL

Materials and Methods

Standard Copper (II) Solution

To prepare 1000 ppm, standard stock solution of copper (II), 3.926 gm copper sulphate penta hydrate were dissolved in distilled were water and volume was made to one litre.

5 ml of stock solution of copper (II) were diluted to 1000 ml with distilled water in order to prepare 5 ppm working standard solution of copper (II) copper contents were determined gravimetrically.

Reagent Solution

A 0.2% solution of reagent was prepared by dissolving 0.2 gm of IPTBHA in 100 ml of ethanol.

A 20% solution of naphthalene was prepared by dissolving 20 gm of naphthalene in 100 ml of acetone.

Buffer solutions of pH range of 3-6, were prepared by mixing 1 M acetic acid and 1 M ammonium acetate solution and PH range of 8-10, 1 M aqueous ammonia and 1 M ammonium acetate solution were mixed.

All reagents used were of analytical reagent grade.

APPARATUS

A spectrophotometer Ec model (GS-5701) was used for all absorbance measurements.

The PH measurements were made with Toshniwal model (CL-43) equipped with glass and calomel electrode.

PROCEDURE

An aliquot of standard copper (II) solution containing 40 µg of copper, was taken in tightly stoppered Erlenmeyer flask and diluted to 50 ml with distilled water. To it, 3.0 ml of acetate buffer solution were added to adjust the Ph of the solution to 3.0 and 3.5 ml of 0.2% IPTBHA solution were mixed The solution was digested for 40 minutes at 40°C. Then 2.5 ml of 20% naphthalene solution were added and shake vigorously for 60 seconds. Thus copper (II) complex was adsorbed on microcrystalline naphthalene. The mixture was filtered through a whatman-42 filter paper, washed thoroughly with distilled water and dried in oven at 50°C. The solid was dissolved in 10 ml of DMF. The absorbance of the solution was measured at 395 nm against the reagent blank which was prepared in the similar manner.

RESULT AND DISCUSSION

Absorption spectra

The copper (II) complex of IPTBHA, obtained from the sample solution containing 40 µg of copper at PH 3.0, was adsorbed on microcrystalline naphthalene as given in the recommended procedure. The mixture of copper (II) complex and naphthalene was dissolved in DMF and the absorbance of the solution was measured at different wave lengths between 360 nm-650 nm. The maximum absorbance due to copper (II) complex obtained at 395 nm, where as the absorbance due to reagent blank was negligible at this wave length. Therefore, 395 nm wave length was selected as a max, at which all the absorbance measurements of the present study were determined.

Effect of IPTBHA Concentration

The effect of IPTBHA concentration on the absorbance was investigated by adding the varying amount of IPTBHA solution into solution containing 40 µg of copper at pH 3.0. Absorbance increased slowly up to addition of 2.0 ml of reagent solution and practically remained constant in the range 2.0-4.5 ml. Above 4.5 ml of reagent solution, absorbance started decreasing gradually. Therefore 3.5 ml of 0.2% reagent solution was added for the absorbance measurements.

Effect of Digestion Time

Effect of digestion time on the absorbance measurements of copper (II) complex of IPTBHA was studied. Sample solution containing 40 µg of copper at pH 3.0 and 3.5 ml of 0.2% IPTBHA, was digested for different period of time at 40°C. The absorbance after each varied digestion time was determined according to the given procedure. The effect of digestion time on the absorption of copper (II) complex was studied at 395 nm. The digestion of copper (II) complex up to 20 minutes, gave the absorbance measurements in the increasing order. Absorption remained almost the same in the range 25-45 minutes digestion time. Therefore, the absorbance was measured after digesting the copper (II) complex for 40 minutes.

Effect of Naphthalene Concentration

To study the effect of naphthalene concentration on the extraction of copper (II) complex of IPTBHA by adsorption, the different amounts of naphthalene solution were added to the solution containing copper (II) complex and then absorption studies were carried out at 395 nm. Absorbance increased with the increasing amount of naphthalene solution up to 2.5 ml. Then become almost content in the range 2.5-5.0 ml and further decreased above 5.0 ml. Therefore,

2.5 ml of 20% naphthalene solution were used for the complete extraction of copper (II) complex from aqueous solution.

Effect of Diverse ions

Possible interference due to the presence of various metal ions was examined into the solution of copper (II) complex of IPTBHA containing 40 µg of copper. Results are presented in Table -1

Calibration Curve for Copper (II)

Under the optimum conditions as described in recommended; procedure,

The calibration curve was obtained for copper determination. A serice of sample solutions of copper (II) complex in D.M.F. were prepared, which contained copper in the range 3-80 µg and then, the absorbance of each solution was measured. The data of absorbance was plotted against the respective concentration of copper. The curve was found to be linear and obeys beer's law over the range of 3-80 µg copper.

The molar absorptivity was found to be 6.7×10^{4} $Lmol^{-1}cm^{-1}$, the sensitivity being 0.01838 µg cm^{-2} of copper for the absorbance of 0.001.

TABLE - 1

Effect of diverse metal ions

Diverse metal **lons**	Amount added (mg)	Copper (II) found (µg)
Co (II)	40	40.4
	80	40.6
Cd (II)	30	40.2
	90	40.4
Ca (II)	35	40.2
	100	40.1
Pb (II)	50	39.8
	110	39.9
Ni (II)	45	40.5
	100	40.2
Hg (II)	30	39.8
	80	40.2
Mn (II)	50	40.5
	150	40.8
Cr (III)	45	40.4
	110	40.7
Fe (III)	45	40.6
	120	40.9
Bi (III)	40	39.5
	80	39.7
Pt (IV)	30	39.8
	100	40.1
V (V)	50	38.6
	120	40.4
Mo (VI)	50	40.8
	100	40.4

Copper (II) : 40 µg ; pH : 3.0 ; Naphthalene : 0.5 gm

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