Spectrophotometric determination of Cadmium (II) by adsorption of its N-p-Iodophenyl Thiobenzohydroxamic Acid complex on Microcrystalline Naphthalene

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ABSTRACT

A reliable and sensitive photometric method for the determination of cadmium (II) with N-p-Iodophenyl thiobenzohydroxamic acid (IPTBHA) has been discussed in present study. A stable water insoluble complex formed by cadmium (II) and IPTPHA cannot be directly extracted by benzene and chloroform but can be easily absorbed on microcrystalline naphthalene at room temperature. The adsorbed complex was dissolved in DMF and the maximum absorbance was found at 620 nm. Its absorption obeys Beer's law over the range 3-75 µg of cadmium (II) in 10 ml of DMF. The molar absorptivity was found 6.75×10^4 L $mol^{-1}cm^{-1}$ and the sensitivity being 0.015 µg cm^{-2} of cadmium (II) for the absorbance of 0.001.

Key Words – N-p-Iodophenyl thiobenzohydroxamic acid (IPTBHA), Cadmium (II), Absorptivity, Absorbance, Beer's law, DMF

INTRODUCTION

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

The present study describes a sensitive and selective photometric method for the determination of cadmium (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[10-17]. The method is very convenient and less time consuming.

EXPERIMENTAL

Materials and Methods

Standard Cadmium (II) Solution

To prepare 1000 ppm, standard stock solution of cadmium (II), 1.630 gm cadmium chloride was dissolved in distilled water and volume was made to one litre.

5 ml of stock solution of cadmium (II) was diluted to 1000 ml with distilled water in order to prepare 5 ppm working standard solution of cadmium (II).

Reagent Solutions

0.20% solution of IPTBHA reagent was prepared by dissolving 0.2 gm of IPTBHA in 100 ml of ethanol.

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 buffer solutions of pH range of 8-10 were prepared by mixing 1 M aqueous ammonia and 1 M ammonium acetate solutions.

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 cadmium (II) solution containing 40 µg of cadmium was taken in a tightly stoppered Erlenmeyer flask and diluted to 50 ml with distilled water. To it, 3.0 ml of acetate buffer solution was added to adjust the pH of the solution to 3.0 and to this 3.5 ml of 0.2% IPTBHA solution was mixed. The contents were digested for 40 minutes at 40°C. Then 2.5 ml of 20% naphthalene solution was added and shaken vigorously for 60 seconds. Thus cadmium (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 620 nm against the reagent blank which was prepared in the similar manner.

RESULT AND DISCUSSION

Absorption spectra

The cadmium (II) complex of IPTBHA, obtained from the sample solution containing 40 µg of cadmium at pH 3.0, was adsorbed on microcrystalline naphthalene as given in the recommended procedure. The mixture of cadmium (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 cadmium (II) complex obtained at 620 nm, where as the absorbance due to reagent blank was negligible at this wave length. Therefore, 620 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 the solution containing 40 µg of cadmium 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 cadmium (II) complex of IPTBHA was studied. Sample solution containing 40 µg of cadmium at pH 3.0 and 3.5 ml of 0.2% IPTBHA, were 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 cadmium (II) complex was studied at 620 nm. The digestion of cadmium (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 cadmium (II) complex for 40 minutes.

Effect of Naphthalene Concentration

To study the effect of naphthalene concentration on the extraction of cadmium (II) complex of IPTBHA by adsorption, the different amounts of naphthalene solutions were added to the solution containing cadmium (II) complex and then absorption studies were carried out at 620 nm. Absorbance increased with the increasing amount of naphthalene solution up to 2.5 ml. Then it become almost constant in the range 2.5-5.0 ml and further decreased above 5.0 ml. Therefore, 2.5 ml of 20% naphthalene solution was used for the complete extraction of cadmium (II) complex from aqueous solution.

Effect of Diverse ions

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

Calibration Curve for Cadmium (II)

Under the optimum conditions as described in recommended procedure, the calibration curve was obtained for cadmium determination. A series of sample solutions of cadmium (II) complex in DMF were prepared, which contained cadmium in the range 3-75 µg and then the absorbance of each solution was measured. The data of absorbance was plotted against the respective concentration of cadmium. The curve was found to be linear and obeys beer's law over the range of 3-75 µg cadmium.

The molar absorptivity was found 6.75×10^{4} L $mol^{-1}cm^{-1}$ and the sensitivity being 0.015 µg cm^{-2} of cadmium (II) for the absorbance of 0.001.

TABLE - 1

Effect of diverse metal ion

Diverse metal ion	Amount added (mg)	Cadmium (II) found (µg)
Co (II)	40	40.6
	80	40.8
Cu (II)	30	40.4
	90	40.6
Ca (II)	35	40.4
	100	40.3
Pb (II)	50	40.0
	110	40.1
Ni (II)	45	40.7

	100	40.4
Hg (II)	30	40.0
	80	40.4
Mn (II)	50	40.7
	150	41.0
Cr (III)	45	40.6
	110	40.9
Fe (III)	45	40.8
	120	41.1
Bi (III)	40	39.7
	80	39.9
Mo (VI)	50	41.0
	100	40.6

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

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