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Spectrophotometric Determination of Copper Piperazine Dithiocarbamate

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ABSTRACT

Spectrophotometric method for determination of cu(II) in the trace quantities was developed by using piperazine dithiocarbamate as ligand in ammonium chloride-ammonium hydroxide medium at pH -9.5. The procedure developed was applied for the estimation of cu(II) in microgram quantities in the water samples and the method developed was found to be simple and sensitive. Spectrophotometry is one of the instrumental methods introduced in Analytical Chemistry which has its own significance even today inspite of various other sophisticated instruments developed and has been in common use in trace analysis[1].

Keywords: Spectrophotometry, copper (II), piperazine dithiocarbamate.

INTRODUCTION

Copper is one of the several metals that play an important role in the biological systems. Copper occurs naturally in many vegetables, meat and grains. Increased copper levels in drinking water may also cause increase in lead levels which in turn may be associated with liver damage and kidney diseases. Rogachev[2] and his team have reported the stability constants of copper dithiocarbamates through copper complexation and its spectrophotometric determination

In 1929 Callan and Henderson[3] determined copper with diethyl dithiocarbamate spectrophotometrically. The dithiocarbamates class of chemical compounds occupy a unique place because of their applications in all fields of science[4]. In view of the wide range applications of the reagent (PDTC) in various instrumental methods reported earlier[5,6] the present work was taken up for determination of trace cu(II) in the presence of piperazine dithiocarbamate as reagent in ammonium chloride-ammonium hydroxide medium at pH-9.5.

MATERIALS AND METHODS

Copper reacts with (PDTC) forming a brownish yellow colored complex in ammonium chloride buffer medium of pH-9.5 and shows maximum absorption at 451nm. Since the Cu-PDTC has maximum absorption at 451nm the experiment was carried out at the same wavelength.

SL 191, double beam U.V- Visible spectrophotometer was used for recording absorption spectrum of the solutions. Digital pH meter ELICO L1 120 provided with temperature control knob was used throughout the work. Varian spectra AA-220 atomic absorption spectrophotometer was used for the analysis of samples. Analytical grade chemicals and double distilled water have been used.

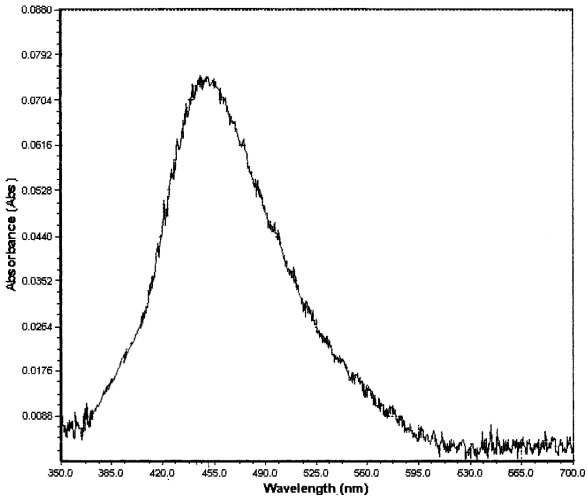

Fig.1 Absorption Spectrum of Cu-PDTC Complex

RESULTS AND DISCUSSION

The experimental conditions for quantitative results have been developed by studying the effect of various parameters that follow.

Effect of pH: The experiments were conducted by mixing volumes of 1ml of 0.001M cu, 1ml of 2M ammonium chloride buffer, 2ml of 0.005M PDTC and ammonium chloride solution of varying pH of 8.0-10.0. It was observed that absorbance values of the metal complex increases with increase in pH from 8 to 9.5 and decreases thereafter. Since the complex formation is quantitative at pH 9.5, all the experiments were carried out at this pH only (Fig.2)

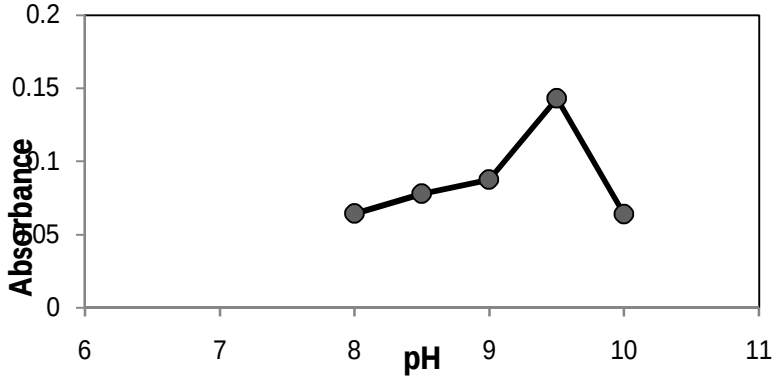

Fig.2.Effect of pH

Effect of ammonium chloride: By keeping all the factors constant including pH, the volume of ammonium chloride solution was changed from 0.5ml to 2ml and the subsequent effect was noted. It was observed that 1ml of 2M ammonium chloride solution shows maximum absorbance, followed by a decrease in the same.

Effect of reagent concentration: The effect of reagent concentration on Cu-PDTC complex was studied by maintaining the metal ion concentration of 1ml of 0.001M , 1ml of 2M NH_4Cl of pH 9.5 and by varying the reagent concentration between 1.0-3.5ml of 0.005M PDTC. It was observed that a minimum of 2ml of the reagent is sufficient for complete complexation of cu(II) with PDTC and hence 2ml of 0.005M reagent was selected for all studies conducted.

Applicability of Beer's law: To the solution containing different amounts of copper of 0.001 M, 1ml of 2M NH_4Cl at pH 9.5 and 2 ml of 0.005 M PDTC were added .The total volume was made up to 10ml by adding double distilled water. The plot between the concentration of copper and absorbance values is linear, passing through the origin ,obeying Beer's law in the concentration range of 1.27-25.41 ppm of copper ion(Fig.3)

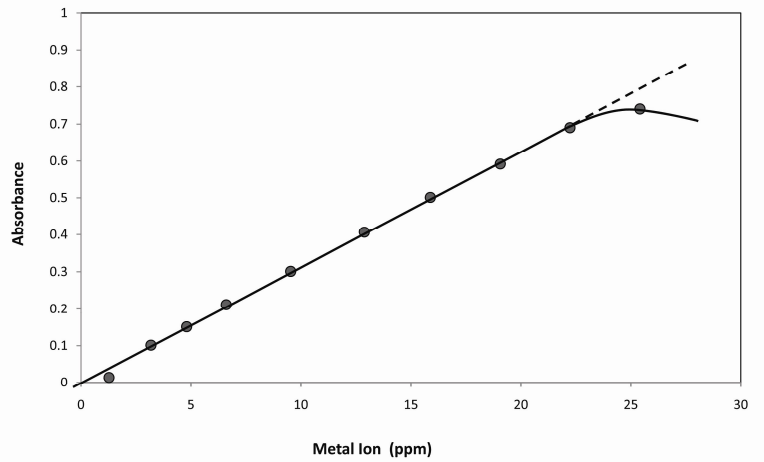

Fig.3. Applicability of Beer's Law

Composition of the complex:

Job's method of continuous variation: Equimolar solution of cu (II) and PDTC (0.001M) were prepared and the metal and the reagent solutions were mixed in different proportions keeping the total volume at 4ml. In each case 1ml of 2M NH_4Cl buffer of pH 9.5 was added and the total volume was maintained at10ml. A graph was plotted between absorption and mole fraction of the ligand. From the graph it is observed that one mole of the metal ion reacts with two moles of the ligand indicating that the composition of the complex as 1:2 in Cu-PDTC complex.

Mole ratio method: 1ml of copper solution of 0.001M was treated with different known volumes of ligand of 0.5 to 5ml of the 0.001M in the presence of 1ml of 2M NH_4Cl buffer at a pH 9.5. The mixture was diluted to 10ml and absorbance values were noted at 451nm and a graph was plotted. From the graph it is observed that 1mole of copper chelates with 2 moles of PDTC graph it is observed that 1mole of copper chelates with 2 moles of PDTC (Fig.4).

Asmus method: The experimental part for Asmus method is same as performed in mole ratio method. A linear plot is obtained between 1/m and $1/v^2$ confirming the ratio of metal to ligand as 1:2.The instability constant of the copper complex calculated is found to be 1.1904×10^{-6}.

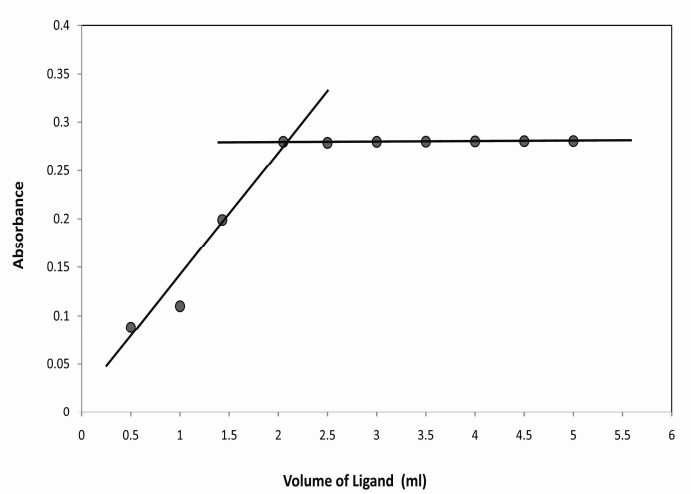

Fig.4. Mole ratio method

APPLICATIONS

Estimation of copper in drinking water samples: 1 liter of the water samples collected from Krishna River, Bore water on hill and from well water of Vijayawada town were pre concentrated to 100ml and 5ml of this solution was taken for analysis.

Leafy Vegetables: 5 grams of the leafy vegetables grown in the nearby villages of Vijayawada town, Krishna District were collected, digested by dry ash method and brought into solution by dissolving in 500ml double distilled water except curry leaves. The curry leaves sample was dissolved in 50ml.The leafy samples are Mentha spicata (Mint leaves), Piper beetle (Beetle leaves) and Murraya Koenigii (Curry Leaves).
Aliquots of the above solutions were taken into beakers and the experimental conditions developed as already mentioned were maintained. The results obtained are further supported by atomic absorption spectrophotometric method. The values are compared with AAS method and the results obtained from Spectrophotometric method are presented in the following tables 1 and 2

Table 1. Determination of copper (II) in water samples of Vijayawada town

Sample	S. No.	Cu(II) added (ppm)	Cu(II) found (ppm)	% recovery	AAS method	
					Cu(II) found (ppm)	% recovery
I	1	0.7	0.70	100.00	0.69	98.57
	2	0.9	0.89	98.88	0.90	100.00
	3	1.2	1.19	99.16	1.19	99.16
	4	1.5	1.49	99.33	1.50	100.00
	5	1.8	1.80	100.00	1.79	99.44
			Average	99.47	Average	99.43
II	1	0.7	0.70	100.00	0.69	98.57
	2	0.9	0.90	100.00	0.89	98.88
	3	1.2	1.19	99.16	1.20	100.00

	4	1.5	1.48	98.66	1.50	100.00
	5	1.8	1.80	100.00	1.80	100.00
			Average	99.56	Average	99.49
III	1	0.7	0.69	98.57	0.70	100.00
	2	0.9	0.89	98.88	0.90	100.00
	3	1.2	1.20	100.00	1.19	99.16
	4	1.5	1.49	99.33	1.48	98.66
	5	1.8	1.80	100.00	1.79	99.44
			Average	99.35	Average	99.45

I. Krishna river water. II. Bore water on hill. III. Well water. Ammonium Chloride: 2MPDTC: 0.005M. pH: 9.5

Table No 2. Determination of Cu (II) in Leafy Vegetables

Sample	S. No	Cu (II) ppm		Cu(II), ppm found AAS Method
		Added	Found	
I	1	0.5	0.72	0.72
	2	0.5	0.66	0.65
	3	0.5	0.67	0.66
II	1	1.0	1.25	1.26
	2	1.0	1.22	1.23
	3	1.0	1.20	1.22
III	1	0.5	0.55	0.58
	2	0.5	0.56	0.55
	3	0.5	0.54	0.52

I. Mentha Spicata (Mint Leaves). II. Piper Beetle (Beetle Leaves). III. Murraya Koenigii Curry Leaves). Ammonium Chloride: 2M. PDTC: 0.005M pH: 9.5

CONCLUSIONS

The results in the table indicate that the water samples analyzed are free from copper content and the percent recovery values obtained are comparable and in good agreement with atomic absorption spectrophotometric data. The Copper content present in leafy vegetables are in agreement with the standard values reported[7].

The method developed for Cu (II) in the presence of ammonium chloride buffer medium using PDTC is found to be sensitive, selective, specific, rapid and may be successfully applied for the determination of low concentration of copper present in water samples and leafy vegetables.

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