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STUDIES OF DIGESTION TIME, SOLVENT EFFECT AND EFFECT OF DIVERSE IONS IN THE SPECTROPHOTOMETRIC DETERMINATION OF SILVER (I) BY ITS3-ALLYL-1-(5-CHLORO-2-PYRIDYL) THIOUREA COMPLEX ON MICRO CRYSTALLINE NAPHTHALENE

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ABSTRTACT

The effect of digestion time, shaking time and effect of diverse ions in the spectrophotometric determination of silver (I) in visible region is discussed. The method is based on analysis of metals by solid-liquid separation after liquid-liquid extraction[1]. The silver(I) formed a stable water insoluble complex with 3-allyl-1-(5-chloro-2-pyridyl)thiourea which was adsorbed on microcrystalline naphthalene. The adsorbed complex is dissolved in dimethylformamide and its absorbance was measured at 430nm against a reagent blank. Themlolar absorptivity was found to be 3.18x $10^4$1mol^{-1} $cm^{-1}$and sensitivity being 1.591x10^{-2} µg cm^{-2} of silver for the absorbance of 0.001. The optimum conditions of the present study were investigated by examining the effect of parameters like shaking time, digestion time, effect of solvent and diverse ions on absorbance measurements.

KEYWORDS: Shaking Time, Digestion Time, Diverse Ions

INTRODUCTION

Pyridyl Thiourea and its derivatives (S P Mathur, 1977) reported to have myriad analytical applications (Sukhveer Singh *et al.*, 1986) for the determination of many metals (K Rahmatullaev *et al.*, 1989, S P Mathur *et al.*, 1988). Keeping this in mind, pyridyl substituted thioureas (S K Hawa *et al.*, 1986) viz.3-allyl-1-(5-chloro-2-pyridyl) thiourea has been synthesized (George Young and William Eyrer, 1901) and its application as an analytical reagent (U R Ranga Rao and V R Shrinivasan, 1970) for the determination of silver (I), (Qi Wenbi and Luo Hongshan, 1988) has been studied using an effective method called "analysis of metals by Solid-Liquid separation after Liquid-Liquid" extraction (M Satake and Y Takagi, 1969). In the present study, the effect of digestion time, shaking time, effect pf solvent and diverse ions using 3-allyl-1-(5-chloro-2-pyridyl) thiourea as an organic reagent for the photometric determination of trace amount of silver has been carried out.

EXPERIMENTAL

Standard silver(I)Solution

A stock standard solution (1000ppm of silver(I) was prepared by dissolving requisite amount of silver nitrate in distilled water.A10ppm solution was prepared by diluting 10ml of the stock solution to 1000ml with distilled water. Amount of silver present in the sample solution was determined gravimetrically.

3-Allyl-1-(5-chloro-2-pyridyl)thiourea Solution

A 0.2% solution of 3-allyl-1-(5-chloro-2-pyridyl)thiourea was prepared by dissolving 0.2g of this reagent in 100ml ethanol.

Naphthalene-acetone solution

A 20% naphthalene solution was prepared by dissolving 0.2 g of naphthalene in 100ml of acetone.

Buffer Solution

Buffer solution of different pH values were prepared by mixing 1M acetic acid and 1M ammonium acetate solution for pH range 3-6 and 1M ammonium acetate solution for pH range 8-11.

All the reagents used were of analytical grade.

Apparatus

A Toshniwal spectrophotometer (model CL-10) was used for all absorbance measurements and all pH measurements were taken with Toshniwal pH meter model (CL-43) equipped with glass and calomel electrodes.

Procedure

An aliquot of standard sample solutions of silver (I) containing 10-120 µg of silver, was taken in a dry, clean, tightly stoppered Erlenmeyer flask. To it 3ml of acetate buffer solution was added to adjust the pH of the solution to 6.0 and then 3.0 ml of 0.2% 1-ally1-3-(5-chloro-2-pyridyl)thiourea solution was mixed. The contents of the flask were kept standing in hot waterbath (50^0-60^0C) for 25 minutes. Then, 3 ml of 20% naphthalene solution were added to the solution of silver

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(I) complex and shaken vigorously for four minutes. The silver (I) complex of 1-allyl1-3-(5-chloro-2-pyridy1) thiourea was adsorbed on microcrystalline naphthalene. It was filtered off, washed with water and dried in an oven at 50^0-60^0C. This dried solid was dissolved in dimethylformamide and diluted to 10 ml. The absorbance measurements of silver (I) complex were taken at 30 nm wavelength against the reagent blank which was prepared similarly.

RESULT AND DISCUSSION

Absorption Spectra

A sample solution containing 90 µg of silver (I) 3.0 ml of 0.2% 3-allyl-1-(5-chloro-2-pyridyl)thiourea solution and 3.0 ml of acetate buffer solution, pH 6.0 was treated according to the recommended procedure. The silver (I) complex, so formed was adsorbed on microcrystalline naphthalene on vigorous shaking for 4 minutes. The solid mixture of naphthalene and silver (I) complex was dissolved in dimethylformamide and the absorbance of the solution was measured at wavelength between 380-600nm. The data of absorbance was plotted against the wavelengths and absorption spectra of silver (I) complex solution was obtained against the reagent blank as shown in fig. 3.0. The silver (I) complex had the maximum absorption at 485 nm wavelength whereas the reagent blank had negligible absorption at this wavelength. Therefore, all absorbance measurements were carried out at 485 nm wavelength (γ max).

Effect of Digestion Time

The solution of silver (I) complex of 3-allyl-1-(5-chloro-2-pyridyl)thiourea containing 90µg of silver was digested for different time periods at 50-60oC and the absorbance were measured at 480nm to investigate the effect of digestion time on the absorbance. The results are given in the Table 1. It was observed that absorbance increased slowly up to 10 minutes, digestion time, in the range of 15-45 minutes the absorbance remained constant and above 45 minutes, it increased slowly. Hence digestion time of 30 minutes was opted for the absorbance measurements.

Table 1: Effect of digestion time

Digestion Time	Absorbance 485 nm
2	0.385
5	0.452
8	0.510
10	0.548
15	0.562
20	0.560
25	0.563
30	0.565
35	0.567
40	0.564
45	0.566
50	0.550
55	0.528
60	0.516
65	0.486
70	0.454
Silver(I) :90 mg; pH : 6.0; 0.2% reagent 3ml	

Effect of Shaking Time

To study the effect of shaking time on the absorbance, 3.0ml of 20 % naphthalene solution was added to the silver (I) complex solution containing 90 ug at pH 6.0 and shaken vigorously for different periods are given in Table 2. Absorbance remained practically constant up to 7minutes shaking time. Therefore, shaking time of 4 minutes was chosen for the absorbance measurements.

Table 2: Effect of Shaking time

Shaking Time (sec)	Absorbance 485 nm
10	0.385
15	0.561
20	0.560
25	0.563
30	0.564
35	0.568
40	0.564
45	0.565
50	0.562
60	0.564
70	0.558
80	0552
90	0.537
100	0.528
110	0.506
120	0.476
130	0.451
140	0.418
Silver(I) : 90 mg; pH : 6.0; 0.2% reagent 3ml	

Effect of Volume of Aqueousphase

The effect of volume of aqueous phase of silver (I) complex on the absorbance was investigated in the range 100-1000 ml. The results are tabulated in the Table 3. The absorbance was almost constant in the range 100-700 ml, but decreased gradually with increasing volume.

Choice of Solvent

Tests were made with various organic solvents to dissolve the mixture of the silver (I) complex and naphthalene. The mixture is easily soluble in acetonitrile and dimethylformamide at room temperature. Therefore, dimethylformamide was chosen as the solvent.

Effect of Diverse Ions

The possible interference due to the presence of alkali metal salts and metal ions are summarized in Table 3 and 4 respectively.

PRECISION

The precision of the of the proposed method was estimated with ten samples of silver (I) complex solution containing 90µg of silver. The mean absorbance of 0.566 with a standard deviation of 0.26%.

Table 3 Effect of Diverse Alkali metal Ions

Alkali Metal Salts	Amount Added (mg)	Found (mg)
NaCl	50	90.6
	100	91.1
	150	91.3
NH_4Cl	75	89.8
	100	90.1
	120	90.2
NaCl	50	89.8
	75	89.9
	150	90.6
Na_2CO_3	75	90.5
	150	90.9
	200	90.2
Na_2HPO4	100	90.4
	150	90.7
	200	90.9
$(NH_4)_2SO_4$	50	90.8
	150	90.9
	250	91.4
	Silver(I) : 90 mg; pH : 6.0; Naphthalene:0.6gm	

Table 4: Effect of Diverse Metal Ions metal Ions

Metal Ions	Amount of Ion Added (mg)	Found (mg)
Fe(III)	30	90.4
	150	90.7
Ni(II)	50	91.1
	120	90.2
Cu(II)	40	91.3
	150	90.6
Zn(II)	60	90.2
	120	90.7
Co(II)	50	91.1
	100	91.4
Mn(II)	70	90.3
	150	90.6
Cd(II)	50	90.9
	150	91.2
Mg(II)	90	92.3
	150	92.7
Bi(III)	50	91.3
	100	91.8
	Silver(I) : 90 mg; pH : 6.0; Naphthalene:0.6gm	

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