

Solvent Extraction and Spectrophotometric Determination of zinc (II) with N,N'-Bis (salicylaldehyde) ethylenediamine [salen].

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ABSTRACT

A method for solvent extraction and spectrophotometric determination of zinc (II) with N, N'-Bis(salicylaldehyde) ethylenediamine (salen) has been studied .The method is based on formation of Zn-N,N'-Bis(salicylaldehyde) ethylenediamine (salen) complex. Here Zn act as metal and N,N'-Bis(salicylaldehyde) ethylenediamine (salen) act as ligand .The absorption spectra of Zn(II)-salen complex extracted at different pH and it was found to be maximum at pH 7 and measured against reagent blank .The light yellow Zn(II)- N,N'-Bis(salicylaldehyde) ethylenediamine (salen) complex solution show maximum absorption at 410 nm .

Keywords: Solvent extraction, Spectrophotometer, Zinc, N, N'-Bis(salicylaldehyde) ethylenediamine (salen) , pH .

INTRODUCTION

Introduction and historical aspects of Solvent Extraction: Solvent or liquid-liquid extraction is based on the principle that a solute can distribute itself in a certain ratio between two immiscible solvents, one of which is usually water and the other an organic solvent such as benzene, carbon tetrachloride or chloroform. In certain cases the solute can be more or less completely transferred into the organic phase. The technique can be used for purpose of preparation, purification, enrichment, separation and analysis, on all scales of working, from microanalysis to production processes.

In analytical chemistry, solvent extraction has come to the forefront in recent years as a popular separation technique because of its elegance, simplicity, speed and applicability to both tracer and macro amounts of metal ions. The aspects and basic principles of solvent extraction are very well explained [3, 4]. The solvent extraction method has made great strides in last four decades. A number of monographs have been published [4,8] dealing with various aspects of solvent extraction. Some of them put emphasis on the theory of chelates extraction [5,8] while others have dwelt on rigorous treatment of extraction equilibria [7,9] or on the mechanism of extraction. Few of them have ramification with aspects of solvent extraction [4, 5]. The chemical engineering aspects pertaining to design and development have been covered in worthwhile monographs [10,12]. The handbook [13] of solvent extraction is an excellent source of information on all aspects of the extraction. The modern chemistry of supramolecules in solvent extraction is very well covered in recent research monographs [14] by the author. An account of various separation methods [15,19] like chromatography, reversed osmosis, electrophoresis, and dialysis are available in the monographs [20]. They indicate how an excellent instrumental method of analysis can be used if supplemented by an efficient separation technique.

Theory of Spectrophotometry and Colorimetry:

Theory of Spectrophotometry which is helpful in the quantitative analysis of sample is very well explained [20]. When light (monochromatic or heterogeneous) is incident upon a homogeneous medium, a part of the incident light is reflected, a part is absorbed within the medium, and the remainder is transmitted. If the intensity of the incident light is expressed by I_0 , that of the absorbed light (I_a), the transmitted light (I_t), and the reflected light (I_r), then:

$$I_0 = I_a + I_t + I_r \quad \dots\dots\dots (1)$$

For air-glass interface, consequent upon the use of glass cells, it may be stated that about 4 per cent of the incident light is reflected. I_r is usually eliminated by the use of a control, such as a comparison cell, hence:

$$I_0 = I_a + I_t \quad \dots\dots\dots (2)$$

Lambert (1760) investigated the relation between I_0 and I_t . Beer (1852) extended to solutions. Spectrophotometry and colorimetry are based upon Lambert's and Beer's laws.

Basic Principles of Solvent Extraction-

The solvent extraction methods are based on the four basic principles

- (a) Gibb's phase rule
- (b) Distribution ratio or extraction coefficient
- (c) Partition coefficient, P
- (d) Percentage extraction

Classification of Solvent Extraction systems :

Extraction can be classified on the basis of

- I] Nature of extracted species
- II] Process of extraction

I] There are two types of extraction depending on the basis of the nature of extracted species,

- a. Chelate extraction
- b. Ion association

II] Classification based on the basis of process of extraction

- (a) Extraction by chelation or Chelate formation
- (b) Extraction by Ion pair formation
- (c) Extraction by salvation
- (d) Synergistic extraction

Now a day's extraction based on the process of extraction is widely used and is explained as follows:

METHODOLOGY

All the chemicals are AR grades of more than 98% purity.

Separating funnels for batch extraction, special glass apparatus. The instruments required for the whole solvent extraction and determination process are very simple like pH meter, spectrophotometer etc.

Preparation of stock solution :-

For 0.01M $ZnSO_4$ Solution : 0.287 g of Zinc sulphate (purity more than 98%) was dissolved in minimum quantity of dil. H_2SO_4 and finally volume was made

by distilled water up to till mark in 100 ml volumetric flask .

For 0.001M ZnSO₄ Solution :

10 ml of 0.01M Zinc sulphate solution was taken in 100 ml volumetric flask and volume was made by distilled water up to till mark.

For 0.0001M ZnSO₄ Solution :

10 ml of 0.001M Zinc sulphate solution was taken out in 100 ml volumetric flask and volume was made by distilled water up to till mark.

Preparation of 0.1% reagent :

0.1g of N,N'-Bis(salicylaldehyde) ethylenediamine (salen) (purity more than 98%) was dissolved in chloroform in 100 ml volumetric flask and volume made up to mark .

RESULTS AND DISCUSSION

Following system (Table no-2) were prepared by using 0.0001 M Zinc sulphate solution and 0.1% of salen in chloroform .Then pH maintained as given below (Table no-2) and all of the system shaken for 5 minutes in separatory funnel and the lower organic

Sr. no.	Unknown sample (ml)	Reagent 0.1% Salen in chloroform (ml)	Absorbance of Zn(II) at pH = 7
1	10	5	0.131
2	10	5	0.185

layers were separated out , from one of the system absorbance was measured. And λ_{\max} was determined.

Selection of λ_{\max} ;

Wavelength (nm)	Absorbance
380	0.333
390	0.341
400	0.370
410	0.398
420	0.345
430	0.331

$$\lambda_{\max} = 410 \text{ nm}$$

At this λ_{\max} , the absorbance of each system were measured

From the above table it is cleared that, maximum absorbance shown at pH =7, Hence the different systems were prepared at pH =7 for calibration curve, and absorbance were measured.

To determine the absorbance of system at pH =7

In this case different concentration of metal ion was used and reagent concentration was kept constant. A systems were prepared as follows and absorbance were measured

Calibration graph for Zn(II) at pH = 7 [Plot of Absorbance Vs concentration of Zn(II) in ppm
To determine the absorbance of unknown sample
Following systems of unknown sample were prepared and absorbance was measured.

Calculation for Zn(II) :-

For unknown sample no. (1)

Theoretical value :

$$1000 \text{ ml of } 1 \text{ M ZnSO}_4 \text{ Solution} = 65.37 \text{ g of Zn(II)}$$

$$\therefore 1 \text{ ml of } 0.0001 \text{ M ZnSO}_4 \text{ Solution} = X \text{ g of Zn(II)}$$

$$\therefore X = \frac{(1 \times 0.0001 \times 65.37)}{1000}$$

$$\therefore x = 6.537 \times 10^{-6} \text{ g of Zn(II)}$$

$$= 6.537 \text{ ppm of Zn(II)}$$

But in sample we have taken 5 ml of ZnSO₄ Solution

$$\therefore 5 \text{ ml of ZnSO}_4 \text{ Solution} = 5 \times 6.537 = 32.685 \text{ ppm of Zn(II) .}$$

Experimental value: From calibration graph ,In unknown sample Zn(II) extracted by solvent extraction show absorbance 0.131 which corresponds to 31.00 ppm

$$\text{Hence experimental value} = 31.00 \text{ ppm}$$

$$\% \text{ Error} = \frac{\text{Theoretical value} - \text{Experimental value}}{\text{Theoretical value}} \times 100$$

$$= \frac{(32.685 - 31.00)}{32.685} \times 100 = 5.15 \%$$

For unknown sample no. (2)

Theoretical value :

In sample no.(2), we have taken 7 ml of ZnSO₄ Solution

∴ 7 ml of ZnSO₄ Solution = 7 × 6.537 = 45.759 ppm of Zn(II).

Experimental value: From calibration graph, In unknown sample, Zn(II) extracted by solvent extraction show absorbance 0.185 which corresponds to 44.00 ppm

Hence experimental value = 44.00 ppm

$$\% \text{ Error} = \frac{\text{Theoretical value} - \text{Experimental value}}{\text{Theoretical value}} \times 100$$

$$= \frac{(45.759 - 44.00)}{45.759} \times 100 = 3.84 \%$$

The % Error was found to be ,

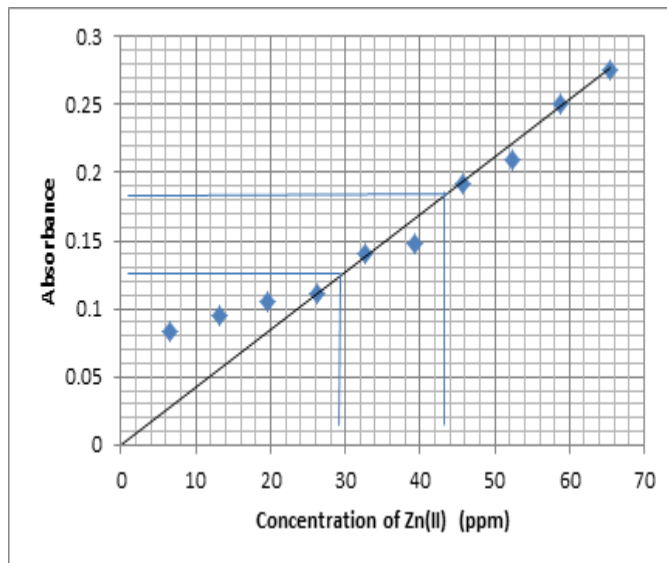
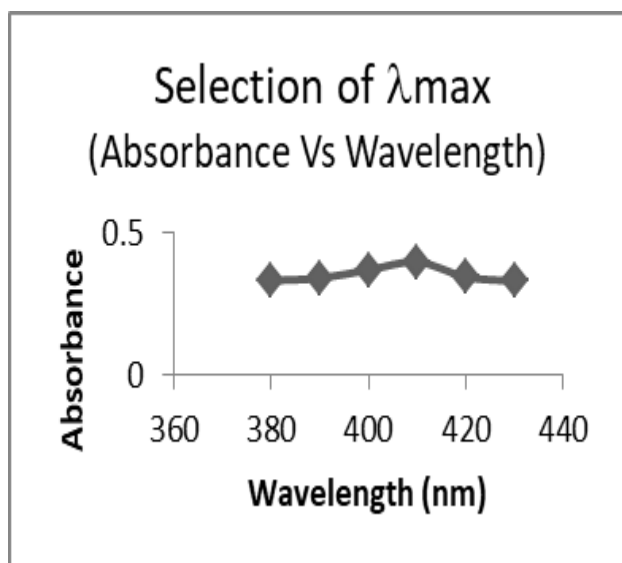
Sample No	Metal	Theoretical Value	Experimental Value	% Error
1	Zn (II)	32.685	31.00	5.15
2	Zn (II)	45.759	44.00	3.84

Absorbance of system at different pH :

System no.	pH	ZnSO ₄ Solution 0.0001M (ml)	Reagent 0.1% Salen in chloroform(ml)	Distilled water (ml)	Absorbance
1	1	1	5	9	0.053
2	2	1	5	9	0.099
3	3	1	5	9	0.354
4	4	1	5	9	0.390
5	5	1	5	9	0.397
6	6	1	5	9	0.418
7	7	1	5	9	0.426
8	8	1	5	9	0.416
9	9	1	5	9	0.415
10	10	1	5	9	0.418

System no.	ZnSO ₄ Solution 0.0001M (ml)	Zn(II) in ZnSO ₄ Solution (ppm)	Reagent 0.1% Salen in chloroform (ml)	pH water (ml) To maintain, pH = 7	Absorbance
1	1	6.537	5	19	0.084
2	2	13.074	5	18	0.095
3	3	19.611	5	17	0.105
4	4	26.148	5	16	0.111
5	5	32.685	5	15	0.140
6	6	39.222	5	14	0.148
7	7	45.759	5	13	0.192
8	8	52.296	5	12	0.209
9	9	58.833	5	11	0.251
10	10	65.37	5	10	0.276
Blank (Reagent)	-----		5	20	

GRAPH :-



Absorption spectra: The absorption spectra of Zn(II) salen complex extracted at different pH and it was found to be maximum at pH 7 and measured against reagent as blank. The light yellow Zn(II) salen complex solution show maximum absorption at 410 nm hence all absorbance measured at 410 nm .

Effect of various solvent : Various solvent such as alcohol, n- butanol, ethyl alcohol were tried as a solvent but chloroform was found to be most effective.

Effect of pH and reagent concentration: Solvent extraction behavior of Zn(II) salen complex was studied in pH range 1 to 10 and varying reagent concentration i.e. 0.001 M, 0.0005M , 0.0001M .It was observed that extraction was quantitative at the pH =7 and at the concentration 0.0001 M.

Colour of the species: Colour of the species at different pH was found to be slightly different. At pH 1 to 2 it was almost colourless but from pH 3 to 10 it developed into pale yellow colour.

CONCLUSION

From the experiment and result ,following conclusion can be drawn,

- Solvent Extraction is a proven technique for extraction and separation of different metal from various complex solution and industrial effluent using different organic extractants. .A suitable

organic extractant can be used for selective extraction of desired metal or impurities depending on presence of anion and cation and their concentration in solution.

- From experiment, Zinc (II) can be quantitatively extracted with salen dissolved in chloroform at the pH 7 respectively.
- The Zinc extraction system is pH dependant that is there is change in the absorbance of metal-salen complex decreasing and increasing the pH. It is found that the advantages to extract Zinc (II) ion from the aqueous phase at particular pH. Accordingly they can be extracted from more acidic solution away from their hydrolysis region

Conflicts of interest: The authors stated that no conflicts of interest.

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