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Solvent Extraction and Spectrophotometric Determination of Cobalt (Ii) with N, N'-Bis (Salicylaldehyde) Ethylenediamine [Salen]

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ABSTRACT

A method for solvent extraction and spectrophotometric determination of Cobalt (II) with N,N'-Bis(salicylaldehyde) ethylenediamine (salen) has been studied .The method is based on formation of Co - N,N'-Bis(salicylaldehyde) ethylenediamine (salen) complex. Here Co act as metal and N,N'-Bis(salicylaldehyde) ethylenediamine (salen) act as ligand .The absorption spectra of Co(II)-salen complex extracted at different pH and it was found to be maximum at pH 6 and measured against reagent blank .The light yellow Co(II)- N,N'-Bis(salicylaldehyde) ethylenediamine (salen) complex solution show maximum absorption at 410 nm .

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

I. INTRODUCTION

In its simplest form, extraction refers to the transfer of a solute from one liquid phase to another. The most common case is the extraction of an aqueous solution with an organic solvent. Diethyl ether, benzene, and other hydrocarbons are common solvent that are less dense than water and form a phase that sits on top of the aqueous phase. Chloroform, dichloromethane, and carbon tetrachloride are common solvents that are immiscible with and denser than water [4]. Solvent extraction is another name for liquid-liquid distribution that is distribution of a solute between two liquids that must not be completely mutually miscible. This method makes use of an organic compound capable of extracting the metal ion of interest, or a complex of it, from the aqueous phase into an immiscible organic solution.

Theory of Spectrophotometry and Colorimetry:-

Theory of Spectrophotometry which is helpful in the quantitative analysis of sample is very well explained [5]. 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 Io, that of the absorbed light (Ia), the transmitted light (It), and the reflected light (Ir), then:

$$I_o = I_a + I_t + I_r \text{ ………………. } (1)$$

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. Ir is usually eliminated by the use of a control, such as a comparison cell, hence:

Io = Ia + It ………………….. (2)

Lambert (1760) investigated the relation between Io and It. 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

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

a. Chelate extraction b. Ion association
IV] 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

II. MATERIALS AND METHODS

All chemicals used were of A.R.grade. Separating funnels for batch extraction, 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 0.01 M $CoSO_4$ Solution : Cobalt sulphate solution was prepared by dissolving 0.2811 g of $CoSO_4.5H_2O$ in slightly acidic media and volume was made by distilled water till mark in 100 ml volumetric flask.

For 0.001 M $CoSO_4$ Solution : 10 ml of 0.01 M cobalt sulphate solution was taken in 100 ml volumetric flask and volume made up to the mark by adding distilled water.

For 0.0001 M $CoSO_4$ Solution : 10 ml of 0.001 M cobalt sulphate solution was diluted in 100 ml volumetric flask by adding distilled water.
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.

III. RESULT AND DISCUSSION

Systems were prepared, having different pH of solution with constant metal ion concentration 5.89 µg (in 1 ml). pH was adjusted with ammonium hydroxide and hydrochloric acid. Volume of aqueous phase was kept constant to 25 ml. Solution was then transferred to separating funnel, 5 ml of salen solution was added.

Each system was shaken for 5 minute allowed to equilibrate for 5 minute. Co (II) from aqueous phase was extracted into organic phase. Absorbance of organic phase was measured at 410 nm against reagent blank.
To determine λmax of a solution:

Wavelength(nm)	Absorbance
400	0.349
410	0.366
420	0.333
430	0.305
440	0.280

System No.	pH	0.0001M $CoSO_4$ Solution (ml)	Amount of Co (II) taken in (µg)	0.1 % Salen Reagent in chloroform (ml)	Distilled water (ml)	Absorbance
1	1	1	5.89	5	19	0.028
2	2	1	5.89	5	19	0.036
3	3	1	5.89	5	19	0.306
4	4	1	5.89	5	19	0.382
5	5	1	5.89	5	19	0.406
6	**6**	**1**	**5.89**	**5**	**19**	**0.467**
7	7	1	5.89	5	19	0.463
8	8	1	5.89	5	19	0.455
9	9	1	5.89	5	19	0.451
10	10	1	5.89	5	19	0.435

λ_{max} = 410 nm

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

Absorbance of system at different pH :

From the above table it is cleared that, maximum absorbance shown at pH = 6.0, Hence the different systems were prepared at pH = 6.0 for determination of calibration graph and absorbance were measured.

Preparation of Calibration Curve at pH = 6.0

In this case different concentration of metal ion was used and reagent concentration was kept constant.

Each system was shaken for 5 minute. After equilibration organic layer was extracted and absorbance was measured for each system.

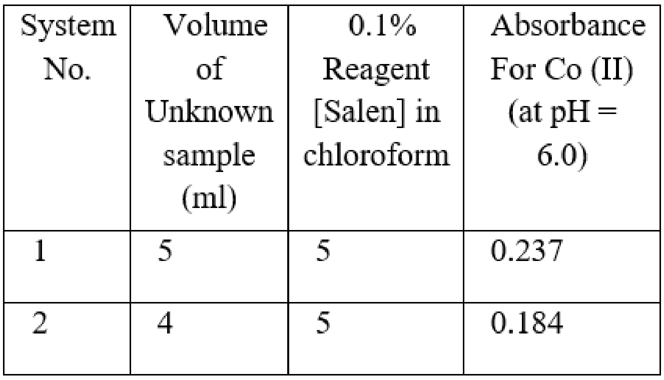
System No.	Volume of 0.0001M $CoSO_4$ (ml)	Amount of Co(II) taken in µg	Reagent 0.1% Salen in chloroform (ml)	pH water (ml) [pH =6]	Absorbance
1	1	5.89	5	19	0.050
2	2	11.78	5	18	0.102
3	3	17.67	5	17	0.186
4	4	23.57	5	16	0.215
5	5	29.46	5	15	0.252
6	6	35.35	5	14	0.291
7	7	41.25	5	13	0.342
8	8	47.14	5	12	0.420
9	9	53.03	5	11	0.430
10	10	58.93	5	10	0.446
Blank			5	20	

Graphs:

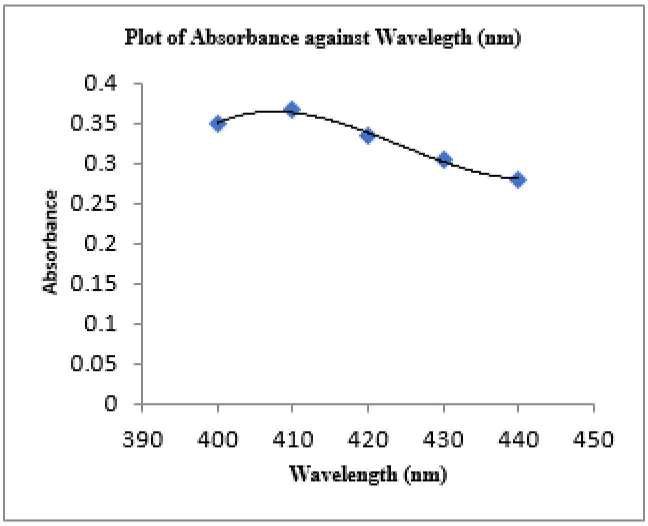
For determination of λ_{max}

To determine the absorbance of unknown sample:-

Following systems of unknown sample were prepared and absorbance was measured.

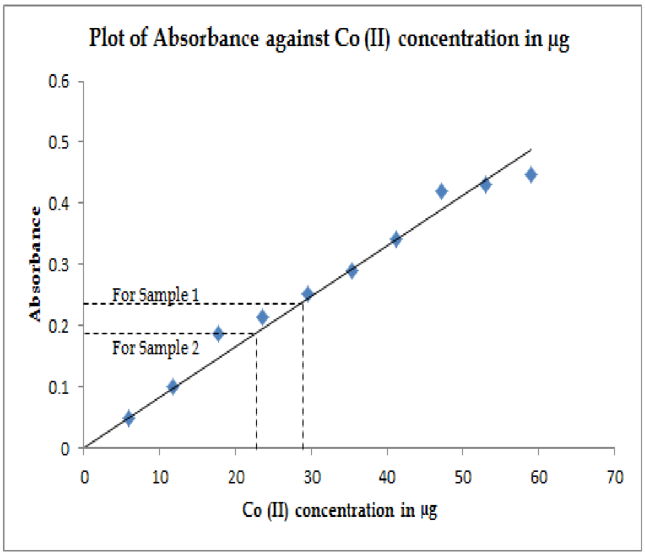


System No.	Volume of Unknown sample (ml)	0.1% Reagent [Salen] in chloroform	Absorbance For Co (II) (at pH = 6.0)
1	5	5	0.237
2	4	5	0.184



Calibration graph for Co(II) at pH=6

[Plot of Absorbance Vs concentration of Co(II) in ppm]



Calculation

- Calculation for Unknown Sample No. 1 :-

Theoretical Value of Co(II) – 1000 ml of 1 M $CoSO_4$ Solution = 58.93 g of Co(II)

∴ 1 ml of 0.0001 M $CoSO_4$ Solution = X g of Co(II)

$$\therefore X = \frac{(1 \times 0.0001 \times 58.93)}{1000}$$

$X = 5.893 \times 10^{-6}$ g of Co(II)

= 5.89 µg of Co(II)

But in Unknown Sample, 5 ml of $CoSO_4$ Solution was taken.

∴5 ml of $CoSO_4$ Solution = 5 × 5.89

= 29.46 µg of Co(II).

Experimental Value of Co(II) -

From calibration graph of Co(II), In unknown sample, Co(II) was extracted by solvent extraction method shows absorbance 0.237 which corresponds to 28.5 µg.

Hence experimental value = 28.5 µg

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

$$\% \text{ Error} = \frac{(29.465 - 28.5)}{29.465} \times 100 = 3.27\%$$

- Calculation for Unknown Sample No. 2 :-

Theoretical Value of Co(II) –

1000 ml of 1 M $CoSO_4$ Solution = 58.93 g of Co(II)

∴ 1 ml of 0.0001 M $CoSO_4$ Solution = X g of Co(II)

$$\therefore X = \frac{(1 \times 0.0001 \times 58.93)}{1000}$$

∴ $X = 5.893 \times 10^{-6}$ g of Co(II)

∴ X= 5.89 µg of Co(II)

But in Unknown Sample, 4 ml of $CoSO_4$ Solution was taken.

∴4 ml of $CoSO_4$ Solution = 4 × 5.89

= 23.57 µg of Co(II).

Experimental Value of Co(II) -

From calibration graph of Co(II), In unknown sample, Co(II) was extracted by solvent extraction method shows absorbance 0.184 which corresponds to 22.3 µg.

Hence experimental value = 22.3 µg

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

$$\% \text{ Error} = \frac{(23.572 - 22.3)}{23.572} \times 100 = 5.39\%$$

Sample No	Metal	Theoretical Value	Experimental Value	% Error
1	Co(II)	29.46	28.5	3.27
2	Co(II)	23.57	22.3	5.39

The % Error was found to be

Absorption Spectra -

The absorption spectra of Co(II) salen complex extracted at different pH and it was found to be maximum at pH = 6.0 and measured against reagent as blank. The light yellow colour of Co(II) salen complex solution show maximum absorption at 410 nm. Hence all absorbance measured at 410 nm.

Effect of Various Solvent -

Various organic solvent such as alcohol, n-butanol, ethyl alcohol, carbon tetrachloride were tried as a solvent but chloroform was found to be most effective.

Effect of pH and Metal Concentration -

Solvent extraction behavior of Co(II) salen complex was studied in pH range 1.0 to 10.0 and varying metal concentration i.e. 0.001 M, 0.0005 M, 0.0001 M. It was observed that extraction was quantitative at the pH = 6.0 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 1.0 to 2.0, it was almost colourless but from pH = 3.0 to 10.0 it developed into pale yellow colour.

IV. CONCLUSION

The present investigation had resulted in offering a convenient liquid-liquid extraction method for separation of Cobalt (II). Cobalt (II) can be extracted by salen dissolved in chloroform.

This method has number of advantages over existing method such as easy phase separation, less equilibration time etc. Salen extracted Cobalt (II) very rapidly, equilibrium was reached within 5 minutes.

On the basis of my practical work and Literature review, it can be concluded that this technique is prominent to execute for extraction of various metal from the mixture of metals, alloys etc and above data can be used in industries.

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