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LIQUID LIQUID EXTRACTION AND SEPARATION OF ZINC (II) WITH N-n-HEXYLANILINE

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ABSTRACT

The solvent extraction of Zinc (II) from thiocyanate and sulphuric acid media using N-n-hexylaniline using xylene is described . The effect of acid concentration, extractant, diluent and aqueous to organic phase ratio on the extraction have been investigated .The nature of the probable extracted species has been determined. The effect of several foreign ions on the extraction of Zn(II) has been investigated .

Keywords: Zinc sulphate , Solvent extraction ,N-n-hexylaniline , Diluent, Thorium nitrate , EDTA, Xylenol orange

INTRODUCTION

As zinc is present in pharmaceutical samples, galvanising industry ,paints, pigments, several insecticides, diecasting alloys, light metal alloys, wet-batteries . The extraction of the element from aqueous phase is one of the important fields of environmental analysis.The high molecular weight amines such as N-n-octylaniline has been used in this laboratory for the selective extraction of many metal ions from haloacid media[2,3,4,5,6] .

N-n–hexylaniline is synthesized by using method similar to N-n-octylaniline by Gardlund's methods[1] , N-n-hexylaniline in xylene could be used for the anion exchange extraction of Zn(II) from KSCN and H_2SO_4 .The metal ion in the organic phase is stripped and determined titrimetrically[7].

The proposed method is relatively simple, rapid and does not require long extraction period.The method finds a wide range of applications in the analysis of commercial drug samples.

EXPERIMENTAL

Zinc Solution :Stock solution of Zn (II) was prepared by dissolving suitable amount of $ZnSo_4.7H_2o$ (Lobo chemie) in 4cm^3 of nitric acid and diluting to 100cm^3 with water.The solution was standardized complexometrically[7].

N-n-hexylaniline solution : Solution (% ,v/v) were prepared by using xylene as the diluent .

Acetate buffer solution: It was prepared by dissolving suitable amount of sodium acetate trihydrate in 400cm^3 water , adding 17cm^3 of glacial acetic acid and diluting to 1 dm^3.

Thorium nitrate solution :A 0.01 M solution was prepared by dissolving suitable amount of thorium nitrate tetrahydrate (BDH) in water and diluting to 1 dm^3.

EDTA solution : A 0.01 M solution was prepared by dissolving suitable amount of disodium salt of EDTA in water and diluting to 1 dm^3.

All chemical used were of analytical grade .

General procedure:

To an aliquot of solutions containing up to 0.5mg/cm^3 of Zinc(II) add sufficient quantity of sulphuric acid and potassium thiocyanate to make the concentrations 0.05-3M and 0.05-3M respectively in a volume of 10 cm^3 .Transfer the solution to 125cm^3 seperating funnel .Shake it for 2 min with 10 cm^3 of 1.5% of N-n-hexylaniline in xylene. Swirl the separating funnel slightly. Separate the two layers and strip zinc from

the xylene layer by shaking with acetate buffer (2x 25 cm^3) for 2min. To the aqueous layer add sufficient 0.002 EDTA., 7 drops of 0.1% xylenol orange indicator and titrate excess of EDTA with 0.002 M thorium nitrate.The end point is yellow to red.

RESULTS AND DISCUSSION

Effect of acidity and reagent concentration

The concentration of KSCN was varied from 0.05-3M and that of H_2SO_4 from 0.05-3M keeping the concentration of one constant and other varying .The concentration of N-n-hexylaniline was varied from 0.5-1.5% . It was observed that zinc was quantatively extracted when the concentration of KSCN was 0.3-0.7M and that of H_2SO_4 was 0.1-3M with equal volume of 1.5% N-n-hexylaniline in xylene { Table- 1,2 , Figure-1,2}

Nature of extracted species and extraction mechanism

Log-log plot of distribution ratio versus N-n-hexylaniline concentration at 0.05 and 0.1M thiocyanate gave slopes of 0.8 and 0.9 respectively indicating that metal to amine ratio in extracted species is 1:1 while at 1 and 2M thiocyanate the slope were 1.7and 1.8 respectively indicating that metal to ion ratio in the extracted species is 1:2 {Figure no-3} Hence ,the probable extracted species is $[RR'NH_2^+]_2\ Zn(SCN)_4^{2-}$ at lower thiocyanate concentration.

The extraction mechanism at lower thiocyanate concentration appears to be

$$Zn^{2+} + 4SCN^- = [Zn(SCN)_4]^{2-} \quad (1)$$

$$[RR'NH]_{Org} + HSCN = [RR'NH_2^+ (SCN)^-]_{Org} \quad (2)$$

$$2[RR'NH_2^+ (SCN)^-]_{Org} + [Zn(SCN)_4]^{-2} = [(RR'NH_2^+)_2 Zn(SCN)_4^{-2}] + 2\ (SCN)^- \quad (3)$$

Where $R = C_6H_{13}$ and $R' = C_6H_5$

Effect of Equilibrium Time

Variation of shaking time from 15 seconds to 5min showed that a minimum of 1min shaking is needed for complete extraction of Zinc (II).To ensure the quantitative extraction of zinc 2min shaking time is recommended in general procedure, however the prolonged shaking had no adverse effect on the extraction.

Effect of Diluents

Various other solvents such as xylene, benzene ,toluene and nitrobenzene, chloroform were also tried as diluents .The extraction of zinc was quantitative with xylene, benzene, toluene nitrobenzene as diluents .Xylene was selected as the diluent as it gives clear-cut separation of the phases {Table no-3}

The effect Of Aq : Org.Vol.ratio on % Extraction :

Zinc(II) was extracted from aqueous (1cm^3 to 10cm^3) of 0.5M OF KSCN and 1M H_2SO_4 with 10cm^3 of 1.5% N-n-hexylaniline in xylene .Zn(II) was stripped and determined as described in the general procedure .It was found that extraction of Zn(II) was quantitative when aq:org volume ratio was only 1:1 and it decreased beyond it this may be attribute to the less stability of ion pair formed under conditions{Table no-4)

Effect of foreign ions

An interference study showed that a large of cations and anions offers no interference (as shown by less than 1% error in analysis recovery) the effect of foreign ions and their tolerance limits in the extraction Of Zinc are reported in(Table 5)

Analysis of pharmaceuticals samples

A tablet was dissolved in perchloric acid and solution was evaporated to near dryness.The residue was taken up in the minimum amount of perchloric acid and solution was evaporated to dryness again .The residue was then leached with water and diluted to 100cm^3 with water .An aliquot was taken for the extraction and estimation of zinc by the recommended procedure .The mean of six results is reported in (Table no- 6).

CONCULSION

The method is used for the separation and determination of Zinc(II) in pharmaceutical preparations . The results are reproducible and accurate to ±0.4% .

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Table-1: Percentage extraction of Zn(II) with N-n-hexylaniline in xylene (Conditions as in the general procedure except KSCN Conc ;Zn(II) taken 0.5mg , H_2SO_4 Conc 3M)

Reagent %	KSCN M	Extraction %	D
0.5%	0.05	50.00	0.00
	0.1	66.66	1.99
	0.3	86.66	6.49
	0.5	86.66	6.49
	0.7	86.66	6.49
	1.0	60.00	1.50
	2.0	31.25	0.45
	3.0	7.82	0.08
1%	0.05	63.07	1.70
	0.1	78.46	3.64
	0.3	95.38	20.64
	0.5	95.38	20.64
	0.7	95.38	20.64
	1.0	83.07	4.90
	2.0	61.66	1.60
	3.0	28.33	0.39
	0.05	71.87	2.55
	0.1	85.00	5.66

1.5%	0.3	100.00	∞
	0.5	100.00	∞
	0.7	100.00	∞
	1.0	92.18	11.78
	2.0	78.12	3.57
	3.0	48.43	0.93

Table-2:Percentage extraction of Zn(II) with N-n-hexylaniline in xylene (Conditions as in the general procedure except conc of Sulphuric acid Zn(II) taken 0.5mg , KSCN Conc 0.5M)

Reagent %	Sulphuric acid M	Extraction %	D
0.5%	0.05	64.06	1.78
	0.1	75.00	3.00
	0.3	81.25	4.33
	0.5	81.25	4.33
	0.7	81.25	4.33
	1.0	81.25	4.33
	2.0	81.25	4.33
	3.0	81.25	4.33
1%	0.05	90.47	9.49
	0.1	95.00	19.00
	0.3	95.00	19.00
	0.5	95.00	19.00
	0.7	95.00	19.00
	1.0	95.00	19.00
	2.0	95.00	19.00
	3.0	95.00	19.00
1.5%	0.05	95.23	19.96
	0.1	100.00	∞
	0.3	100.00	∞
	0.5	100.00	∞
	0.7	100.00	∞
	1.0	100.00	∞
	2.0	100.00	∞
	3.0	100.00	∞

Table– 3: Effects of diluents

Diluents used	Dielectric constant	Extraction %
Xylene	2.30	100.00
Nitrobenzene	34.8	100.00
Benzene	2.28	100.00
Toluene	2.38	100.00
chloroform	4.81	83.60

Table-4: Enrichment study of Zinc(II)

Aqueous to organic phase	Extraction %
1:1	100.00
2:1	89.23
3:1	74.57
4:1	52.54
5:1	32.20
10:1	0.000

Table-5: Zn(II) taken 0.5mg/cm^3, 1.5% N-n- hexylaniline , KSCN-0.5M , H_2SO_4- 1M; Aqueous to organic ratio 1:1

Sr.No	Foreign ions	Tolerance limit , mg
1.	Ba (II)	30
2.	Mg(II)	30
3.	Mn (II)	30
4.	Ca (II)	20
5.	U(IV)	10
6.	Th(IV)	10
7.	Ni (II)[b]	5
8.	Bi(III)[a]	5
9.	Sn (II)[a]	5
10.	Cr(III)[a]	5
11.	Cu(II)[a]	5
12.	Cd(II)[b]	5
13.	Sr (II)[a]	5
14.	Fe(III)[b]	5
15.	Ascorbate	200
16.	Thiourea	200
17.	Succinate	200
18.	Tartarate	200
19.	Oxalate	200
20.	Acetate	200
21.	Citrate	None

(a) Masked with Thiourea
(b) Masked with Ascorbic acid

Table-6: Analysis of Pharmaceutical samples

Sample	Manufacture	Composition	Amount of zinc Certified	Amount of zinc by Proposed method
Levozen-Z	Alme Formulation Pvt Ltd ,Roper Road ,Nalagarh	Zinc Sulphate monohydrate 61.8mg	22.5mg	22.1mg
Folinz	Apex Labs Pvt Ltd ,B-23 ,Sidco pharmaceutical complex,latur-603110	Zinc Sulphate monohydrate 55mg	20.03mg	19.5mg

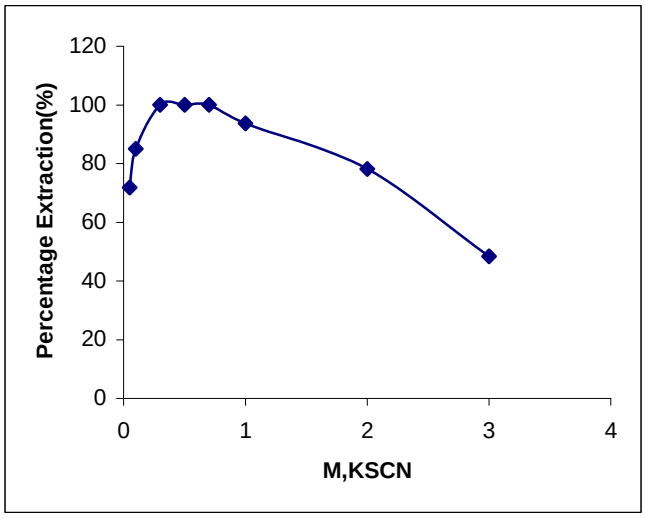

Fig.-1 :- The extraction of Zn(II) with N-n-hexylaniline (1.5%) in xylene as a function of thiocyanate concentration at 3.0 M H_2SO_4

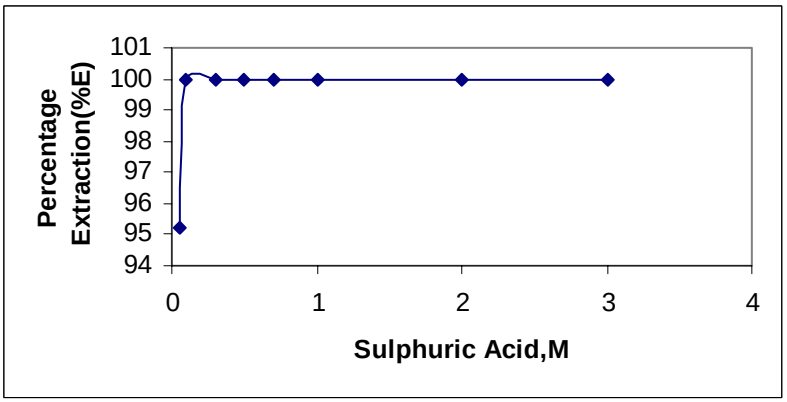

Fig.-2:The extraction of Zn(II) with 1.5 % N-n-hexylaniline in xylene as a function of H_2SO_4 concentration at 0.5 M KSCN

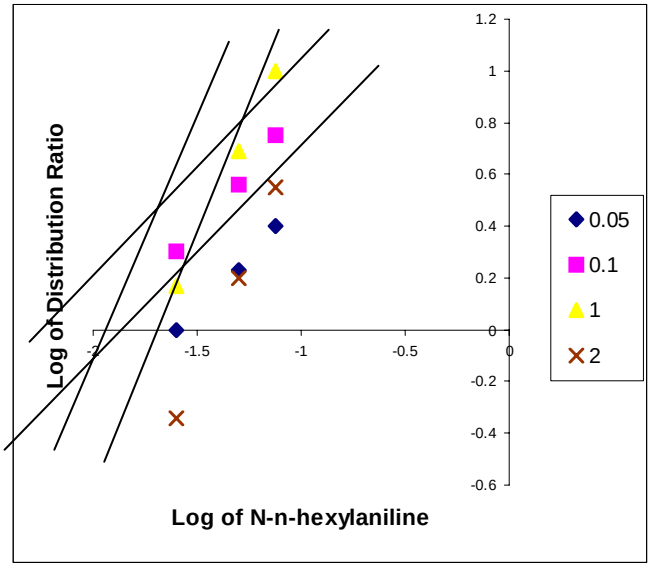

Fig.-3: Distribution ratio of Zn(II) as a function of N-n-hexylaniline conc at 0.05,0.1, 1 and 2M thiocyanate concentration

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