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Extraction of Yttrium (III) from Sodium Acetate and Sodium Succinate Mediums using Cyanex-923

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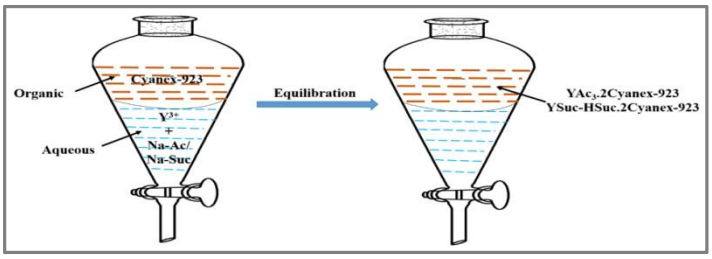
ABSTRACT

The present work deals with solvent extraction of Y(III)using commercial extractant Cyanex-923 in toluene from a solution containing salts of weak organic acids such as sodium acetate and sodium succinate. Effect of pH, effect of extractant concentration, effect of sodium acetate, sodium succinate concentration, effect of diluents, time and stripping agent, effect of temperature, effect of aqueous to organic volume ratio and effect of metal loading capacity has been studied. Slope analysis method and FTIR study was used to determine the probable composition of the extracted complex in the organic phase. The proposed method was extended to separation of Y(III) from synthetic binary mixtures.

Graphical Abstract



Keywords: Yttrium, Acetate, Succinate, Cyanex-923, Stripping.

INTRODUCTION

Yttrium is a one of the important rare earth element possessing [1] pseudo lanthanoid behavior due to its similar chemical and physical properties with lanthanoids [2]. Yttrium of high purity is mostly required for its application in metallurgical industries and photo electronic devices like color TV tubes fluorescent lamp. Yttrium is used in manufacturing of superconductors, radio therapeutic agent, alloys and catalysts [3]. Solvent extraction technique is most widely used industrially for separation of Yttrium from other rare earth elements [4]. Different extractants that have been previously used as a prominent solvent for extraction of yttrium are tributyl phosphine oxide [5], tris(2-ethylhexyl)phosphate [6], Cyanex-301[7], di(2-ethylhexyl)phosphoric acid [8, 9],Cyanex-272 [10],

sec-nonylphenoxy acetic acid (CA-100) [**11, 12**], primene-JMT (tri-alkyl methylamine) [**13**], 2-ethylhexylphosphonic acid mono-2-ethyl hexyl ester P_{507} [**14**], TBP [**15**], diisodecylphosphoric acid [**16**], 2-ethylhexyl phosphonic acid mono-(2-ethylhexyl) ester and bis(2,4,4-trimethylpentyl) phosphinic acid (Cyanex272) [**17**].

Extraction of metals has been done using different extractants in different mediums like, hydrochloric [**7, 8**], phosphoric acid [**18**], sulphuric acid [**13**], nitric acid [**19**] and perchloric acid [**15**]. These mediums are mostly strong acids and are corrosive to skin and are not eco-friendly. Hence there is need of using alternative medium for extraction of metal ions. There are reported works on extraction of metal ion using other medium such acetate for the extraction of Zn(II) with D2HEPA [**20**] and extraction of Pb(II) [**21**], Tl(III) [**22**] with 2-octylaminopyridine in sodium succinate medium. In the present work, we have proposed the extraction of Y(III) using weak organic acid mediums such as sodium acetate and sodium succinate by trialkyl phosphine oxide, Cyanex-923 extractant. This method was then applied for extraction of Y(III) from different synthetic mixture of metals.

MATERIALS AND METHODS

Organo-phosphorus extractant Cyanex-923 supplied by Cytec Industries Inc. Canada was used for extraction without further purification. A known quantity of Y_2O_3 was dissolved in minimum amount of hydrochloric acid and final solution of known concentration was prepared using double distilled water. Sodium acetate, sodium succinate, solvents, acids, metal salts, Arsenazo I indicator of AR grade were used. Elico model LI 120 pH meter was used to adjust pH, Lab UV Nexgen spectrophotometer was used for absorbance study and FTIR study was done using 3000 Hyperion Microscope with vertex 80 FTIR system.

Extraction Procedure: 50 µg of Y(III) solution containing salts of weak organic acid sodium acetate/sodium succinate was prepared. Using dilute HCl and NH_4OH solutions, pH of the Y(III) solution was maintained at 9.0, diluted to 10 mL using double distilled water and transferred into separating funnel containing 10 mL of Cyanex-923 diluted in toluene. The two immiscible phases were hand shaken for about 10 mins and then allowed to separate for 5 mins. The two phases obtained after extraction were allowed to separate for some time and then the organic phase was used to strip back the extracted metal into the aqueous phase. 5M HCl and 3M H_2SO_4 was used to strip Y(III) from metal loaded organic phase of sodium acetate and sodium succinate respectively. The amount of yttrium was then determined by Arsenazo I method at 580nm using UV-Visible spectrophotometer [**23**].

RESULTS AND DISCUSSION

Effect of pH and concentration of Cyanex-923: The effect of pH on extraction efficiency of Y(III) was studied in the range of pH 1-9. The percentage extraction increases with increase in pH (Figure 1) and the extraction in both the mediums was found to be quantitative at pH 9.0 hence further study was performed at pH 9.0 in both the mediums using Cyanex-923 in toluene keeping parameters like concentration of sodium acetate and sodium succinate, time of equilibration, diluent and temperature constant. At pH 9.0, the effect of extractant concentration on the extraction efficiency in both the medium was studied. In sodium acetate medium, the concentration of Cyanex-923 was varied from $7.8x10^{-5}$ M to $5.0x10^{-3}$ M and extraction was found to be quantitative at $2.5x10^{-3}$ M whereas, in sodium succinate medium the concentration of cyanex-923 was varied from $1.56x10^{-4}$ M to $5.0x10^{-3}$ M and the extraction was found to be quantitative at $5.0x10^{-3}$ M.

Effect of sodium acetate and sodium succinate concentration: The effect of medium i.e. sodium acetate and sodium succinate concentration on extraction efficiency of Y(III) was studied. At pH 9.0 extraction of Y(III) was done at $2.5x10^{-3}$ M of Cyanex-923 in toluene, on varying the concentration of

sodium acetate from $1.56x10^{-4}$ M to $1.5x10^{-2}$ M, the extraction was found to be quantitative at $1.0x10^{-2}$ M. Similarly, at pH 9.0 extraction of Y(III) was done at $5.0x10^{-3}$ M of Cyanex-923 in toluene, on varying the concentration of sodium succinate from $1.56x10^{-4}$ M to $1.5x10^{-2}$ M, the extraction was found to be quantitative at $1.0x10^{-2}$ M.

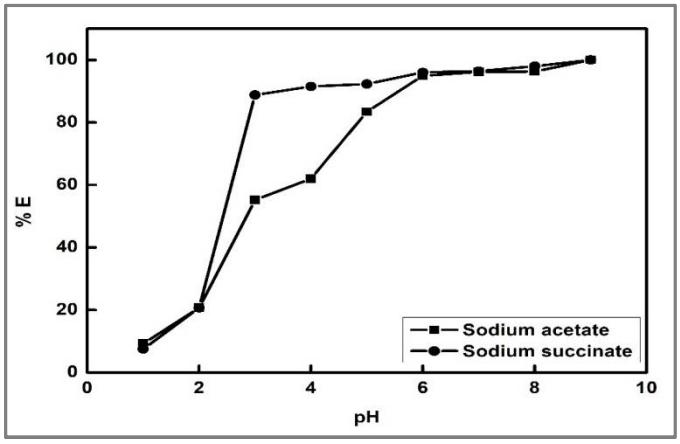


Figure 1. Effect of pH on % extraction of Y(III) in weak organic mediums.

Effect of Diluents, Time and Stripping agent: Different diluents influence the percentage extraction of Y(III) using Cyanex-923 in toluene. Hence various aliphatic and aromatic solvents were used as diluents for the extraction of Y(III) in sodium acetate and sodium succinate medium. As seen in (Table 1) sodium acetate and sodium succinate medium, toluene showed quantitative extraction whereas in other solvents the extraction was not quantitative, hence in both the mediums extraction was carried out using toluene. The effect of time of shaking of separating funnel for quantitative extraction was studied from 1-15 mins. It was found that for quantitative extraction, shaking time required is 10 mins (Table 2). The Y(III) loaded organic phase was stripped back in the aqueous phase using different acids like HCl, HNO_3, H_2SO_4, CH_3COOH, $HClO_4$, HCOOH of different molarities. In Sodium Acetate medium, 5M HCl was used to strip Y(III) quantitatively from organic phase whereas in sodium succinate medium, 3M $H_2SO_4$was sufficient for quantitative stripping (Table 3).

Table 1. Effect of diluent on the percentage extraction of Y(III) in sodium acetate and sodium succinate medium using Cyanex-923

Diluent	Dielectric constant	% E of Y(III)	
		Sodium Acetate Medium	Sodium Succinate Medium
Toluene	2.3	99.9	99.9
Xylene	2.2	99.0	98.4
Chloroform	5.1	96.6	94.2
n- Hexane	1.7	98.4	89.6
Carbon tetrachloride	2.2	99.0	97.2
Cyclohexane	2.0	95.6	92.1
Dichloromethane	8.9	91.8	90.9
Methyl Isobutyl Ketone	13.1	99.0	98.2

Composition of the Extracted Species: The data obtained on plotting the graph of log D v/s log R (R= Cyanex-923) in sodium acetate and sodium succinate medium maintaining 9.0 pH, the probable structure of the extracted species was determined using slope analysis method. The slope obtained in sodium acetate medium from the graph is 2.1 (Figure 2) and in sodium succinate medium the slope found is 2.0 (Figure 3) which shows that two molecules of Cyanex-923 react with one molecule of Y(III) ion in both the mediums. Therefore, the possible stoichiometric ratio for Y(III) to Cyanex-923

Table 2. Effect of period of equilibration on percentage of extraction of Y(III) in sodium acetate and sodium succinate medium.

Time (mins)	% E of Y(III)	
	Sodium Acetate Medium	Sodium Succinate Medium
1	94.9	96.3
3	98.5	97.9
5	99.7	99.1
10	99.9	99.9
15	99.9	99.9

Table 3. Effect of stripping agent on percentage recovery of Y(III) from metal loaded organic phase in sodium acetate/sodium succinate with Cyanex-923 in toluene

Acid	% Recovery of Y(III)from metal loaded organic phase									
	Sodium Acetate Medium					Sodium Succinate Medium				
	1M	2M	3M	4M	5M	1M	2M	3M	4M	5M
HCl	30.3	52.6	60.3	86.7	99.9	34.0	42.2	66.2	99.8	--
HNO_3	99.7	99.8	99.8	--	--	85.8	92.9	98.2	99.7	--
H_2SO_4	85.2	89.0	99.8	--	--	79.2	85.5	99.9	--	--
CH_3COOH	5.1	25.4	59.7	99.3	--	4.4	52.5	97.6	99.2	99.8
$HClO_4$	96.1	99.4	99.8	--	--	77.7	99.6	99.8	--	--
HCOOH	2.4	10.9	31.1	62.5	76.8	1.1	5.3	22.6	82.8	98.0

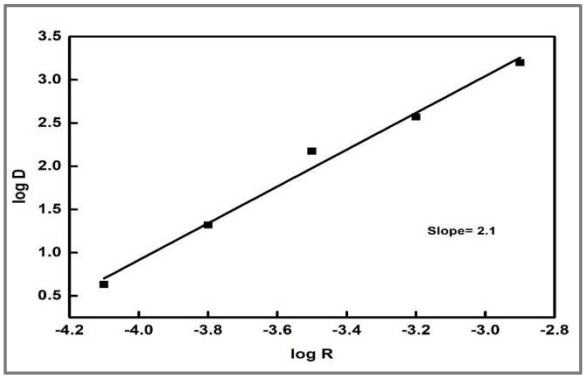


Figure 2. Effect of Cyanex-923 concentration on distribution ratio of Y(III) in sodium acetate medium.

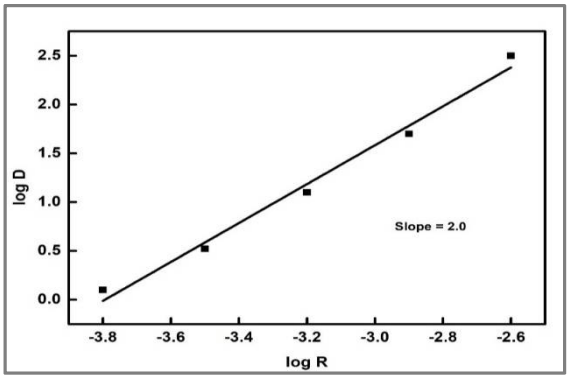


Figure 3. Effect of Cyanex-923 concentration on distribution ratio of Y(III) in sodium succinate medium.

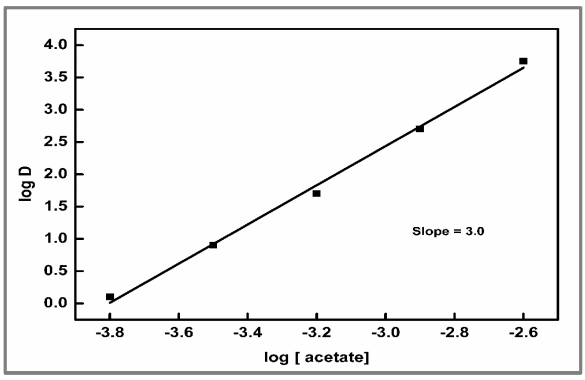


Figure 4. Effect of sodium acetate concentration on distribution ratio of Y(III) using 2.5 x 10^{-3}M cyanex-923.

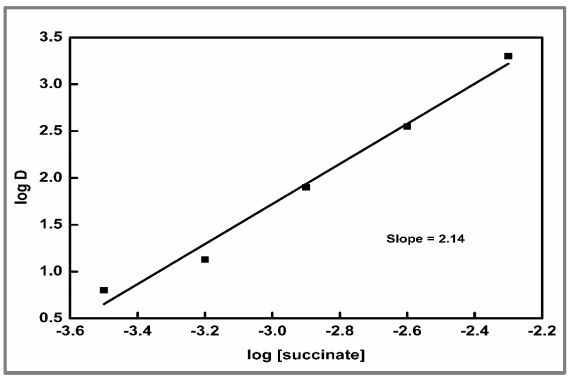


Figure 5. Effect of sodium succinate concentration on distribution ratio of Y(III) using 5.0 x 10^{-3}M cyanex-923.

in both mediums is1:2. Also a graph of log D v/s log [acetate ions] was plotted which gives slope 3.0 (Figure 4) indicating a stoichiometric ratio 1:3 of yttrium with respect to sodium acetate medium and

the graph of log D v/s log [succinate ions] gave slope of 2.0 (Figure 5) indicating a stoichiometric ratio 1:2 of yttrium with respect to sodium succinate medium. Therefore, in sodium acetate medium the probable extracted species Y(III) in organic phase is $[Y(Ac)_3].2Cyanex\text{-}923$ [24] and in sodium succinate medium is [YSuc-HSuc].2Cyanex923. (where Suc = Succinate ion).

$$Y^{3+}{}_{(aq)} + 3Ac^{-}{}_{(aq)} + 2Cyanex\text{-}923_{(org)} \rightleftharpoons YAc_3.2Cyanex\text{-}923_{(org)} \quad \ldots (1)$$

$$Y^{3+}{}_{(aq)} + Suc^{2-}{}_{(aq)} \rightleftharpoons YSuc^{+}{}_{(aq)} \quad \ldots (2)$$

$$YSuc^{+}{}_{(aq)} + HSuc^{-}{}_{(aq)} \rightleftharpoons YSuc\text{-}HSuc_{(aq)} \quad \ldots(3)$$

$$YSuc\text{-}HSuc_{(aq)} + 2Cyanex\text{-}923_{(org)} \rightleftharpoons YSuc\text{-}HSuc.\ 2Cyanex\text{-}923_{(org)} \quad \ldots(4)$$

Temperature Study: In sodium acetate and sodium succinate medium the extraction of Y(III) were done at temperature range from 303K to 343K at pH 3.0 respectively and it was found that with increase in temperature the extraction increased. The graph of log D v/s (1/T) x 1000 was plotted to obtain slopes in both the mediums. These slopes were then used to calculate the enthalpy variation, ΔH of the reaction using Van't Hoff equation,

The Van't Hoff equation is,

$$\log D = -(\Delta H/2.303\,RT) + C \quad \ldots(5)$$

D is the distribution ratio, ΔH is the enthalpy change for the reaction and C is the constant.

$$\text{Slope} = -\Delta H/2.303\,R \quad \ldots(6)$$

The slope obtained from the graph of log D v/s 1/T x 1000 in sodium acetate medium is -3.98 and in sodium succinate medium is -3.1 (Figure 6).

$$\Delta G = -RT\ln D = -2.303RT\log D \quad \ldots(7)$$

$$\Delta S = \frac{\Delta H - \Delta G}{T} \quad \ldots(8)$$

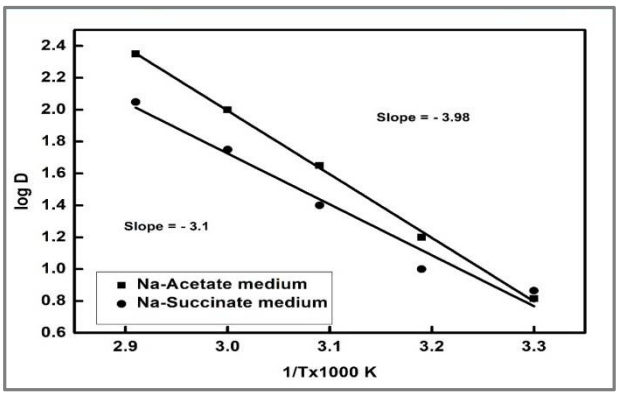


Figure 6. Effect of temperature on distribution ratio of Y(III) in acetate and succinate medium.

Using the slopes obtained from graph 6, the thermodynamic parameters i.e. ΔH, ΔG, ΔS have been calculated by using equations 6, 7 and 8 (Table 4). The ΔH value in sodium acetate medium is 76.20 kJ mol^{-1} and that in sodium succinate medium is 59.35 kJ mol^{-1}, which signifies that the reaction in both the mediums is endothermic [25].

Table 4. Thermodynamic parameters of the extraction of Y(III) from Sodium Acetate and Sodium Succinate medium by Cyanex-923 in toluene solution.

Temp (K)	Sodium Acetate Medium				Sodium Succinate Medium			
	Log D	-ΔG kJ mol^{-1}	ΔS J mol^{-1}	ΔH kJ mol^{-1}	Log D	-ΔG kJ mol^{-1}	ΔS J mol^{-1}	ΔH kJ mol^{-1}
303	0.81	4.72	267.0		0.87	5.01	212.4	
313	1.20	7.18	266.4		0.97	5.78	208.0	
323	1.51	9.33	264.8	76.20	1.03	6.36	203.4	59.4
333	2.00	12.70	267.1		1.26	8.00	202.2	
343	2.35	15.40	267.1		2.05	13.4	212.2	

The negative ΔG value confirms that extraction of Y(III) in acetate and succinate medium favours thermodynamically and the extraction reaction is spontaneous. ΔS value obtained is positive which shows that extraction of Y(III) in sodium acetate and sodium succinate medium using Cyanex-923 in toluene is entropy driven process.

Metal Loading Capacity of Cyanex-923: The metal loading capacity of Cyanex-923 in sodium acetate and sodium succinate medium was studied by changing the concentration of metal Y(III) from 10 to 100 µg. It was observed that the extraction of Y(III) in both the mediums is quantitative till 50 µg, further increasing the amount of Y(III), the percent extraction decreases. Hence, the quantitative extraction of Y(III) in sodium acetate and sodium succinate medium using Cyanex-923 in toluene the loading capacity is 50 µg for 10 mL of Cyanex-923 in toluene (Table 5).

Table 5. Metal Loading Capacity

Amount (µg)	Sodium Acetate	Sodium Succinate
	% E	% E
10	100	100
20	100	100
30	100	100
40	100	100
50	100	100
60	98.2	99.8
70	96.4	99.3
80	95.3	95.4
90	94.7	93.7
100	94.1	93.4

Effect of aqueous to organic volume ratio: The study on the effect of various aqueous to organic volume ratios on extraction of Y(III) in sodium acetate medium and sodium succinate medium using Cyanex-923 in toluene was carried out where the volume of organic phase was kept constant and the volume of aqueous phase containing Y(III)was varied from 10 to 300 mL. It was observed that in both the mediums there was increase in percentage extraction on varying the aqueous to organic volume phase ratio from 30:1 to 1:1. In sodium acetate medium the quantitative extraction was observed from 10:1 to 1:1whereas in sodium succinate medium the extraction was found to be quantitative from 6:1 to 1:1. Hence, to study all the other parameters in the extraction of Y(III) in both mediums using Cyanex-923, the phase ratio was kept maintained at 1:1 (Table 6).

FTIR Study: The study of IR spectra (Figure 7) showed that the stretching frequency of P=O in pure Cyanex-923 was obtained at 1640 cm^{-1} where as in YAc_3.2Cyanex-923 and YSuc-HSuc. 2Cyanex-923 complexes there was a shift seen in the P=O stretching frequency i.e.1080 cm^{-1}signifying that there is interaction between the oxygen of the P=O group of the Cyanex-923 and Yttrium in both the complexes [26]. Also, IR spectra of pure Cyanex-923 showed a band at 3419 cm^{-1} due to O-H stretching and this band was found to be missing in both complexes of Yttrium in both mediums [27]. Hence, with these spectral changes it can be concluded that the complex extracted is formed in both

the medium with Cyanex-923 in toluene through the co-ordination of the lone pair on oxygen in the P=O group of Cyanex-923 to the Y(III) metal ion (Table 7).

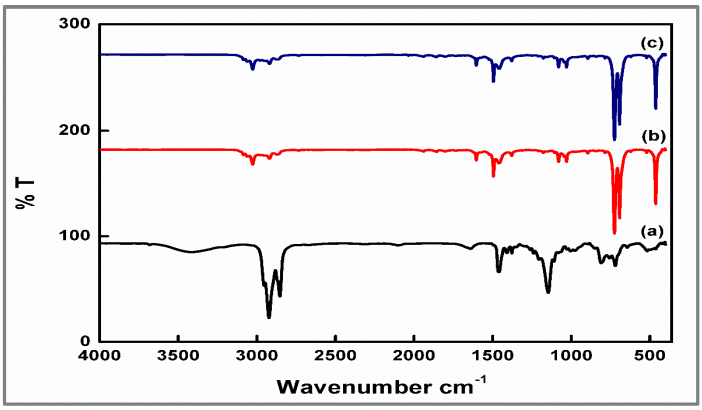


Figure 7. IR spectra: a) Pure Cyanex-923, b) YAc_3. 2Cyanex-923 complex, c) YSuc-HSuc. 2Cyanex-923complex.

Table 6. Effect of aqueous to organic volume ratio

Aq : Org Vol (mL)	Sodium Acetate	Sodium Succinate
	% E	% E
10 : 10	100	100
15 : 10	100	100
25 : 10	100	100
40 : 10	100	100
50 : 10	100	100
60 : 10	100	100
75 : 10	100	98.4
100 : 10	100	96.6
150 : 10	97.0	95.8
200 : 10	95.7	95.4
300 : 10	94.6	95.0

Table 7. IR Characteristics Spectral Data for pure Cyanex-923 and Loaded Organic Solution of Cyanex-923 in n-heptane in sodium acetate and sodium succinate medium.

Pure Cyanex-923 (cm^{-1})	YAc_3.2Cyanex-923complex(cm^{-1})	YSuc-HSuc. 2Cyanex-923 complex(cm^{-1})	Probable assignment
3419	--	--	$\nu_{(O-H)}$
2922-2853	2919	2919	$\nu_{s(C-H)}$, $\nu_{as(C-H)}$
1640	1604	1604	$\nu_{(P-OH)}$
1457	1494	1494	$\delta_{as(CH3)}$
1164	1080	1080	P=O
810	895	895	P-C

APPLICATION

Yttrium ion is separated from the other metal ions by considering the difference in their extraction conditions. In $1.0x10^{-2}$ M sodium acetate medium, mixture of 50 µg of Y(III) and 100 µg of U(VI) were taken and the pH of the solution mixture were maintained 9.0. This solution was then extracted with $2.5x10^{-3}$ M of Cyanex-923 in toluene, at this extractant concentration only Y(III) gets easily extracted while U(VI) remains unextracted. The extracted Y(III) in this medium is stripped back in the aqueous phase with 5M HCl. Also, in $1.0x10^{-2}$M sodium succinate medium, mixture of 50 µg of Y(III) and 100 µg of U(VI) were taken and the pH of the solution mixture were maintained 9.0. This

solution was then extracted with $5.0x10^{-3}$ M of Cyanex-923 in toluene. At this condition, only Y(III) gets extracted while U(VI) remains unextracted. The extracted Y(III) in this medium is stripped back in the aqueous phase with 3M H_2SO_4. Similarly, other metal ions like Al(III), Ga(III), Tl(III) are separated from Y(III) metal ion (Table 8).

Table 8. Separation of Y(III) at pH=9 from Multicomponent mixtures in sodium acetate and sodium succinate medium using Cyanex-923 in toluene.

Binary mixture of Metal ions and Amount (µg)	Sodium Acetate medium			Sodium Succinate medium		
	Extractant Cyanex-923 (M)	Stripping agents	Recovery (%)	Extractant Cyanex-923 (M)	Stripping agents	Recovery (%)
Y^{3+} (50) U^{6+} (100)	2.5×10^{-3}	5M HCl 2M HNO_3	99.9 99.5	5.0×10^{-3}	3MH_2SO_4 3M HNO_3	99.9 99.9
Y^{3+}(50) Th^{4+} (100)	2.5×10^{-3}	5M HCl 2M $HClO_4$	99.9 99.8	5.0×10^{-3}	3M H_2SO_4 6M $HClO_4$	99.9 97.8
Y^{3+}(50) Fe^{3+} (50)	2.5×10^{-3}	5M HCl Water	99.9 99.8	5.0×10^{-3}	3M H_2SO_4 Water	99.9 99.8
Y^{3+} (50) Mo^{6+} (50)	2.5×10^{-3}	5M HCl 0.3M KOH	99.9 99.8	5.0×10^{-3}	3M H_2SO_4 1M NaOH	99.9 99.6
Y^{3+} (50) Hg^{2+}(100)	2.5×10^{-3}	5M HCl 2M HNO_3	99.9 99.6	5.0×10^{-3}	3M H_2SO_4 2M HNO_3	99.9 94.7

CONCLUSION

It was concluded that this method of extraction of Y(III) in sodium acetate and sodium succinate medium using extractant Cyanex-923 in toluene is quantitative and the extraction takes place through solvation mechanism. Yttrium forms complexes in the organic phase in both acetate and succinate medium and the proposed composition of the complexes are $[Y(Ac)_3]$.2Cyanex-923 and [YSuc-HSuc].2Cyanex-923. This proposed method of separation is efficient for separation of Y(III) from binary mixtures. The mediums used in this work are weak organic acids which enhance the process of extraction and these mediums are non- corrosive and are non-harmful to environment.

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