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Selective Separation of Scandium(III) and Yttrium(III) from other Rare Earth Elements using Cyanex302 as an Extractant

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Selective Separation of Scandium(III) and Yttrium(III) from other Rare Earth Elements using Cyanex302 as an Extractant

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Abstract: Liquid-liquid extraction and selective separation of scandium(III) and yttrium(III) with Cyanex302 (bis(2,4,4-trimethylpentyl)monothiophosphinic acid) has been carried out by controlling the aqueous phase pH. Scandium(III) and yttrium(III) were completely recovered from the organic phase using 5.0 M and 4.0 M nitric acid respectively and determined spectrophotometrically as their complexes with Arsenazo(III). The influence of extractant concentration, equilibration time, nature of diluents, stripping agents, and diverse ions on the extraction of scandium(III) and yttrium(III) was investigated. The extractability of scandium(III), yttrium(III), and other rare earth elements was exploited for sequential separation of scandium(III)-yttrium(III)-lanthanum(III) and other rare earth elements viz. lanthanum(III), cerium(IV), praseodymium(III), neodymium(III), gadolinium(III), dysprosium(III), and ytterbium(III) in binary mixtures. The method presented is simple and rapid for isolation of scandium(III) and yttrium(III) from associated elements and has been successfully applied for their selective separation from complex matrices of USGS standard soil GXR-2 and Japanese standard stream sediment sample Jsd-3.

Keywords: Liquid-liquid extraction, scandium(III), yttrium(III), Cyanex302

INTRODUCTION

The demand for rare earth elements (REEs) is ever increasing due to their wide range of applications involving superconductors, optoelectronic materials,

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special alloys, catalysts, and radio therapeutic reagents. The separation of rare earth elements individually or as a group is challenging and has always attracted the attention of researchers owing to the similarities in their chemical properties. Liquid-liquid extraction has been commonly used for their isolation. In the view of the use of scandium(III) and yttrium(III) in high tech industries and nuclear energy programs, their purification has much significance and various extractants have been reported for their extraction (1–5). Over the years much interest has been generated in the use of Cyanex272 (bis(2,4,4-trimethylpentyl)phosphinic acid); Cyanex302 and Cyanex301 which are the monothio and dithio analogs of Cyanex272 for extraction of various REEs (6–15).

A number of reports on the application of bis(2-ethylhexyl)phosphoric acid (HDEHP) for scandium(III) recovery exist (16, 17). The high extraction and stripping acidity with HDEHP was the limiting factor. On the basis of the extraction and stripping behavior of scandium(III) with Cyanex272 and Cyanex302, they were proposed for purification of scandium(III) at an industrial scale (13). Scandium(III) was separated from other associated elements with Cyanex272 (18). The extractant Cyanex302 has poor aqueous solubility, complete miscibility with common organic diluents and resistance to hydrolysis making it better suitable for industrial applications (19). The commercially available Cyanex302 consists of bis(2,4,4-trimethylpentyl)monothiophosphinic acid (i.e., purified Cyanex302: 78–80%), tris-alkylphosphine oxide (R_3PO: 10–12%), bis(2,4,4-trimethylpentyl)phosphinic acid (i.e., Cyanex272: 2–3%), bis(2,4,4-trimetylpentyl)dithiphosphinic acid (i.e., Cyanex301: 1–2%), and other unknown components (8%). It was reported that purified Cyanex302 had lower extraction for Sc(III), Y(III), La(III), and Gd(III), than the commercially available Cyanex302 (20).

Although, various studies have been reported for extraction of scandium(III) and yttrium(III) with Cyanex302 (12–14, 20), its potential for group separation of scandium, yttrium and lanthanum and their isolation from complex matrices has not been explored. The extractability of organophosphinic acid extractants for a metal ion depends upon the nature and concentration of metal ion and also the aqueous phase acidity. Hence by controlling the aqueous phase conditions, selective separation of different metal ions can be achieved. In the present work the difference in the extraction behavior of scandium(III) and yttrium(III) and various REEs was exploited to achieve their separation with high separation factors. Various parameters influencing the extraction of scandium(III) and yttrium(III) such as extractant concentration, equilibration time, nature of diluents, stripping agents and diverse ions were studied. Based on the extraction data, the sequential separation of scandium(III), yttrium(III) and lanthanum(III) is proposed. The method developed was applied for the recovery of scandium(III) and yttrium(III) from USGS standard soil GXR-2 and Japanese standard stream sample Jsd-3.

EXPERIMENTAL

Reagents

Cyanex302 supplied by Cytec Canada as a gift sample was used without further purification. The diluent used was m-xylene (Extra pure, E-Merck). The stock solution of scandium(III) was prepared from scandium oxide and yttrium(III) was prepared from yttrium carbonate and standardized complexometrically (21). A working solution containing 30 mg dm^{-3} of scandium(III) and yttrium(III) was prepared by appropriate dilution. Standard stock solutions (1 mg cm^{-3}) of other REEs were used to prepare working standards. Arsenazo(III) (Loba Chemie) was used as a 0.1% aqueous solution. All the reagents used were of A.R. grade.

Instrumentation

A Digispec 110D spectrophotometer (Feedback, India Ltd) with matched 10mm glass cuvettes, digital pH meter (Elico Private Ltd, India) with a combined glass and calomel electrode (Toshniwal - Mollar, India) and a wrist action flask shaker (General Trading Corporation, India) were used. An inductively coupled plasma atomic emission spectrophotometer, ICP-AES GBC 8440 plasma lab was also used.

General Extraction Procedure

Equal volumes of aqueous phase containing scandium(III)/yttrium(III) (3.0 mg dm^{-3}) and 5×10^{-4} M Cyanex302 in xylene were equilibrated on a wrist action flask shaker for 15.0 min. After phase separation, scandium(III) and yttrium(III) were stripped from the organic phase with 10 cm^3 of 5.0 M and 4.0 M nitric acid respectively and determined spectrophotometrically in the aqueous phase with Arsenazo(III) at 675 nm and 650 nm respectively (22). The distribution ratio (D) was calculated from the percentage of scandium(III) and yttrium(III) extracted in the organic phase.

RESULTS AND DISCUSSION

Effect of pH and Cyanex302 Concentration

The effect of pH on the extraction of scandium(III)/yttrium(III) with 5×10^{-4} M Cyanex302 in xylene was studied (Fig. 1). Cyanex302 being a liquid cation exchanger, the cation exchange mechanism is responsible for the extraction of scandium(III)/yttrium(III) and is thus influenced by the pH

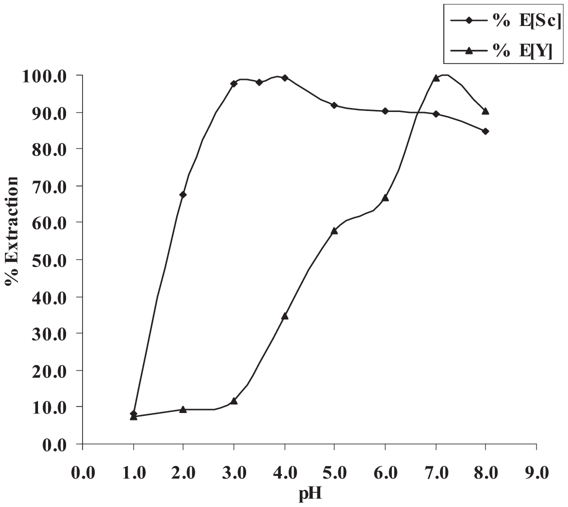

Figure 1. Effect of pH on extraction of scandium(III) and yttrium(III); Organic phase = 5×10^{-4} M Cyanex302 in xylene; Equilibration time = 15 minutes.

of the aqueous phase. The extraction of scandium(III) increased with the increase in pH of the aqueous phase up to pH = 4.0 and decreased beyond pH = 5.0. Similar observations were seen for yttrium(III) and its extraction was quantitative (99.4%) at pH = 7.0. Hence, pH = 4.0 and 7.0 were selected as the optimum pH for quantitative extraction of scandium(III) and yttrium(III) respectively.

The outcome of studies on extractant concentration is of prime importance to evaluate the nature of extracted species of the metal ion in the aqueous phase. Using $(0.5 - 5.0) \times 10^{-4}$ M of Cyanex302 in xylene, the extraction dependencies of scandium(III) and yttrium(III) were investigated. The extraction increased with increase in the concentration of the extractant as shown in a graph of log[D] vs. log[Cyanex302] (Fig. 2).

Nature of Extracted Species

Attempts were made to ascertain the nature of extracted species of scandium(III)/yttrium(III) with the extractant using slope analysis. The distribution ratio (D) of scandium(III)/yttrium(III) evaluated at different concentrations of Cyanex302 was used to plot a graph of log[D] vs. log[Cyanex302] (Fig. 2). The plots were straight line graph with a slope of 2.55 for scandium(III) and 1.52 for yttrium(III). This indicates a mole ratio of scandium(III) and yttrium(III) to Cyanex302 as 1:2.5 and 1:1.5 respectively.

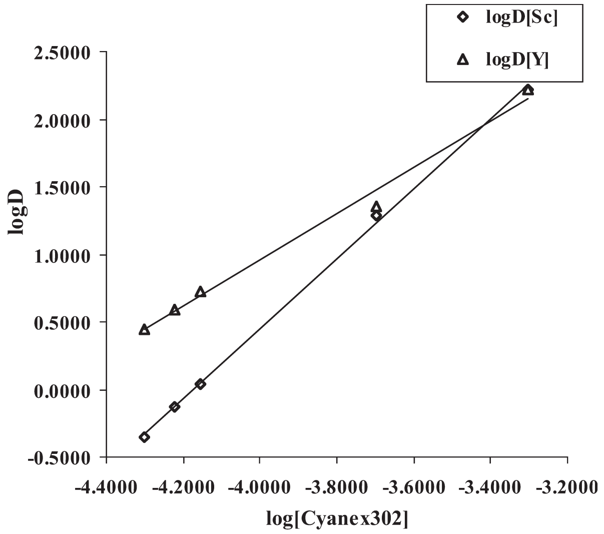

Figure 2. Effect of Cyanex302 concentration on extraction of scandium(III) and yttrium(III); Scandium(III) 3.0 mg dm^{-3} at pH = 4.0, yttrium(III) 3.0 mg dm^{-3} at pH = 7.0; Equilibration time = 15 minutes.

Effect of Diluents

Extraction with an acidic organophosphorus extractant depends upon the nature of the diluent. In order to study the role of diluents during extraction of scandium(III) and yttrium(III), various organic solvents such as benzene, toluene, xylene, cyclohexane, chloroform, and carbon tetrachloride were examined as diluents for 5.0×10^{-4} M solution of Cyanex302. The % extraction of scandium(III) and yttrium(III) was 73.3, 63.5, 99.4, 94.0, 58.6, 59.3 and 64.0, 45.7, 99.4, 62.3, 42.3, 42.3 respectively indicating that non-polar organic solvents with a low dielectric constant had better extractability for scandium(III) and yttrium(III). However, no particular reason can be given for the comparatively lower 'D' values for scandium(III) and yttrium(III) using toluene (dielectric constant $(\varepsilon) = 2.3$) as diluent. Xylene was used as a diluent for Cyanex302 throughout the studies as it was found to be most efficient for extraction of scandium(III) and yttrium(III).

Period of Equilibration

Scandium(III) (3.0 mg dm^{-3}) and yttrium(III) (3.0 mg dm^{-3}) were extracted with 5.0×10^{-4} M Cyanex302 in xylene for 5.0, 10.0, and 15.0 minutes. The corresponding percentage extraction was 97.7, 98.4 and 99.4 respectively for scandium(III) where as it was 96.7, 98.3, and

99.4 respectively for yttrium(III). Indicating that 15.0 minutes is required for highest distribution ratio.

Influence of Stripping Agents on Recovery of Scandium(III) and Yttrium(III)

In order to choose the most effective stripping agent for scandium(III)/yttrium(III), different concentrations (0.5 M – 8.0 M) of nitric acid, hydrochloric acid and sulphuric acid were studied. The results are summarized in Table 1. From the results, it is clear that quantitative recovery (>99.0%) of scandium(III) and yttrium(III) was achieved when 5.0 M and 4.0 M nitric acid were used as stripping agents respectively.

Extraction Behavior of REEs

The mutual separation of REEs from scandium(III) and yttrium(III) is important. Hence the extraction of various REEs was studied as a function of aqueous phase pH using 5×10^{-4} M Cyanex302. The extraction conditions were same as that mentioned in the general procedure. The stripping of REEs was done with 10 cm^3 of 5.0 M nitric acid followed by their spectrophotometric determination in aqueous phase using Arsenazo(III) (22). The extractability of the REEs increased with the increase in aqueous phase pH (Fig. 3).

Effect of Various Diverse Ions

The effect of various diverse ions on extraction of scandium(III)/yttrium(III) with Cyanex302 in xylene was studied. The tolerance limit was set as the

Table 1. Influence of stripping agents on recovery of scandium(III) and yttrium(III); Scandium(III) 3.0 mg dm^{-3} at pH = 4.0, yttrium(III) 3.0 mg dm^{-3} at pH = 7.0; Organic phase = 5×10^{-4} M Cyanex302 in xylene; Equilibration time = 15 minutes

	Concentration of stripping agents [M]								
Stripping agents	0.5	1.0	2.0	3.0	4.0	5.0	6.0	7.0	8.0
% Recovery of scandium(III)									
HCl	—	80.5	94.5	92.8	92.8	95.1	98.4	99.3	98.4
HNO_3	—	92.8	92.8	98.4	97.7	99.4	98.4	97.7	97.4
H_2SO_4	78.2	89.6	99.3	97.7	86.9	—	—	—	—
% Recovery of yttrium(III)									
HCl	—	72.3	75.0	75.7	79.0	75.0	68.3	66.7	65.7
HNO_3	—	65.2	71.9	88.4	99.4	89.4	59.6	66.7	65.7
H_2SO_4	61.9	76.2	83.4	90.8	78.7	—	—	—	—

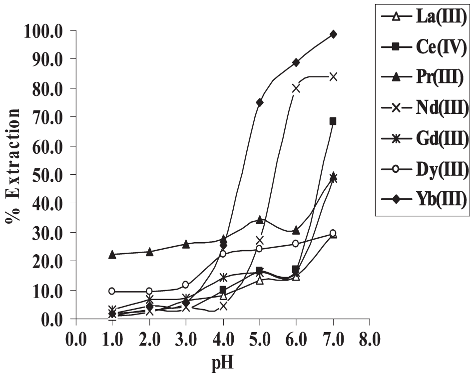

Figure 3. Extraction behavior of REEs; REEs = 3.0 mg dm^{-3}; Organic phase = 5×10^{-4} M Cyanex302 in xylene; Equilibration time = 15 minutes.

amount of foreign ion causing $\pm 2\%$ error in the recovery of scandium(III)/yttrium(III) (Table 2).

Binary Separations from REEs

Scandium(III),yttrium(III) and some REEs were extracted using 5×10^{-4} M Cyanex302 in xylene in binary mixtures. The distribution ratio was calculated from the ratio of concentration of metal in organic phase to concentration of metal in aqueous phase ($D = [M]_{Org.}: [M]_{Aq.}$) and the separation factor β was calculated as the ratio of distribution ratio of scandium(III)/yttrium(III) and the other REEs. The results presented in Table 3 indicate very high separation factors.

Separation of Scandium(III) and Yttrium(III) from Multicomponent Mixtures

The use of Cyanex302 for isolation and determination of scandium(III) at trace levels from complex mixtures was checked. Some synthetic mixtures were prepared for recovery of scandium(III) and yttrium(III). The mixture containing scandium(III), yttrium(III), and lanthanum(III) and the other containing scandium(III), yttrium(III), and magnesium(II) were resolved by first extracting scandium(III) into the organic phase as per the general procedure, yttrium(III), lanthanum(III), and magnesium(II) remained in the aqueous phase. Yttrium(III) was selectively extracted at pH = 7.0 with 5×10^{-4} M Cyanex302 while lanthanum(III) and magnesium(II) remained unextracted were determined as mentioned in Table 4.

Table 2. Effect of various diverse ions; Scandium(III) 3.0 mg dm^{-3} at pH = 4.0, yttrium(III) 3.0 mg dm^{-3} at pH = 7.0; Organic phase = 5×10^{-4} M Cyanex302 in xylene; Equilibration time = 15 minutes

	Tolerance limit (mg)	
Metal ions	Scandium(III)	Yttrium(III)
Na^+, K^+, Mg^{2+}, Ca^{2+}, Sr^{2+}, Ti^{4+}	5.0	5.0
Cr^{6+}	0.5	0.25
Mn^{2+}	0.5	1.0
Fe^{3+}	0.05	—
Co^{2+}	1.0	2.0
Ni^{2+}	1.0	1.0
Cd^{2+}	1.0	[a]
Zn^{2+}	1.0	[a]
Zr^{4+}	0.1	0.1
Mo^{6+}	0.25	2.0
Sc^{3+}	—	0.08
Y^{3+}	0.5	—
La^{3+}	0.5	0.05
Pr^{3+}, Gd^{3+}, Dy^{3+}, Nd^{3+}, Yb^{3+}	0.03	0.03
Ce^{4+}	0.03	0.1
Th^{4+}	0.03	[a]
U^{6+}	0.03	[a]
Al^{3+}	0.25	[a]
Cl^-, NO_3^-, SO_4^{2-}	5.0	5.0

[a] Interference due to co-extraction.

Recovery of Scandium(III) and Yttrium(III) from Certified Reference Materials

The applicability of Cyanex302 for recovery of scandium(III) and yttrium(III) from associated elements was exploited for analysis of standard reference materials USGS standard soil GXR-2 and Japanese stream sediment Jsd-3.

A USGS standard soil GXR-2 and a Japanese stream sediment Jsd-3, (0.5 g of each) were treated with concentrated HF and H_2SO_4 in 5:1 ratio at 150°C on a hot plate. The residue was cooled and fused with 2.0 g of $KHSO_4$ for 45 min. The melt was extracted with 6.0 M hydrochloric acid and made to a suitable volume.

An aliquot was extracted with diethyl ether for removal of Fe(III) (23). Zirconium(IV) was extracted as described earlier (24). Aqueous phase containing scandium(III), yttrium(III), other REEs and other base metals were extracted with Cyanex302 at pH = 4.0. Scandium(III) was quantitatively extracted in the organic phase leaving the other elements in the aqueous

Table 3. Separation of scandium(III) and yttrium(III) in binary mixtures; Scandium(III) 3.0 mg dm^{-3} at pH = 4.0, yttrium(III) 3.0 mg dm^{-3} at pH = 7.0; REEs= 3.0 mg dm^{-3}; Organic phase = 5 × 10^{-4} M Cyanex302 in xylene; Equilibration time = 15 minutes

Binary mixture	$D_{Sc(III)}$	D_M	Separation factor (β)	Binary mixture	$D_{Y(III)}$	D_M	Separation factor (β)
La(III): Sc(III)	165.7	0.010	1.66×10^4	La(III): Y(III)	165.7	0.012	1.38×10^4
Nd(III): Sc(III)	141.9	0.012	1.18×10^3	Nd(III): Y(III)	89.9	1.84	4.90×10
Pr(III): Sc(III)	332.3	0.011	3.02×10^4	Pr(III): Y(III)	110.1	0.27	4.07×10^2
Ce(IV): Sc(III)	249.0	0.010	2.49×10^4	Ce(IV): Y(III)	75.9	0.55	1.38×10^2
Dy(III): Sc(III)	332.3	0.007	4.75×10^4	Dy(III): Y(III)	165.7	0.18	9.20×10^2
Gd(III): Sc(III)	165.7	0.006	2.76×10^4	Gd(III): Y(III)	124	0.43	2.88×10^2
Yb(III): Sc(III)	141.9	0.037	3.84×10^3	Yb(III): Y(III)	82.3	3.59	2.30×10

Table 4. Separation of scandium(III) and yttrium(III) from multicomponent mixtures; Organic phase = 5×10^{-4} M Cyanex302 in xylene; Equilibration time = 15 minutes

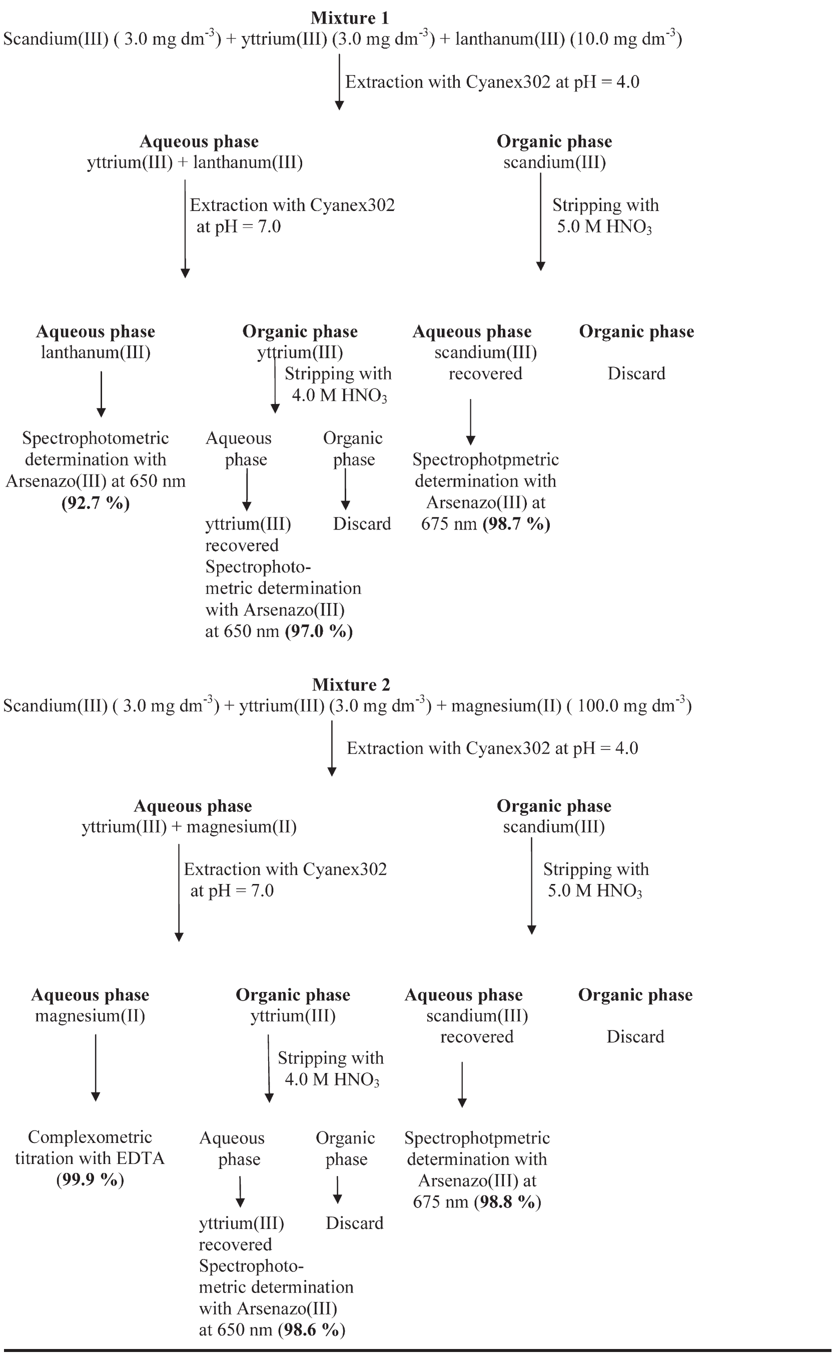

Table 5. Recovery of scandium(III) and yttrium(III) from certified reference materials; Organic phase = 5×10^{-4} M Cyanex302 in xylene; Equilibration time = 15 minutes

Samples	Certified value (ppm)	Amount recovered (ppm, ICP-AES)	% Recovery (ICP-AES)	% Recovery spectrophotometry$^{\pm a}$
USGS standard soil sample GXR-2				
Scandium(III)	6.88	6.8	98.8	99.2 ± 0.4
Yttrium(III)	17.0	16.3	96	99.1 ± 0.3
Japanese stream sediment sample Jsd-3				
Scandium(III)	10.5	10.46	99.6	99.4 ± 0.2
Yttrium(III)	14.9	14.8	99.3	98.9 ± 0.2

$^{\pm a}$= % R. S. D. of triplicate analysis.

phase which was extracted with Cyanex302 at pH = 7.0 for extraction of yttrium(III). The other REEs were quantitatively detected in the aqueous phase by ICP-AES. The results obtained for scandium(III) and yttrium(III) when compared with ICP-AES indicate that scandium(III) and yttrium(III) can be isolated from complex materials (Table 5).

CONCLUSION

Scandium(III) was extracted quantitatively (99.4%) at pH = 4.0 while very little yttrium(III) was extracted so as to permit their mutual separation. Trace level scandium(III) and yttrium(III) extracted using low concentration of Cyanex302 were completely recovered using 5.0 M and 4.0 M nitric acid respectively. Differences in the extraction behavior of scandium(III), yttrium(III) and other REEs were exploited to achieve high separation factors in binary mixtures. Sequential separation of scandium(III) - yttrium(III) - lanthanum(III) and scandium(III) - yttrium(III) - magnesium(II) was possible. The practical applicability of the method developed with Cyanex302 was checked for isolation of scandium and yttrium from standard reference materials.

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