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**Solvent Extraction Separation and Determination of Yttrium with Cyanex272**

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A simple and selective method for the liquid – liquid extraction of yttrium with Cyanex272 is presented. The influence of the pH of aqueous phase, concentration of extractant, nature of diluent, extraction time, type of stripping agent and stripping time were evaluated to optimize the conditions for quantitative extraction of yttrium. The effect of certain foreign ions on the extraction of yttrium was studied. Yttrium was quantitatively extracted at pH = 7.0 with $5 \times 10^{-4}$ M Cyanex272 in xylene. It was stripped from the organic phase with 4M nitric acid and determined spectrophotometrically with Arsenazo (III) at 650nm.

INTRODUCTION

Yttrium has applications in various fields ranging from metallurgy, catalyst to electronics. Liquid – liquid extraction has been the most commonly used technique for isolation of yttrium. The extraction of yttrium with various alkyl phosphoric acids such as mono - 2 - ethyl hexyl phosphoric acid [1] and bis - (2 - ethylhexyl) phosphoric acid (HDEHP) [2] have been well established. Its extraction with other organo phosphorus compounds like, 2 - ethylhexyl phosphonic acid mono - 2 - ethylhexyl ester [3] and mono-octyl phosphinic acid [4] was also studied. The addition of Cyanex923 (a mixture of four trialkyl phosphine oxides) to Cyanex301 [ bis (2,4,4 – trimethylpentyldithiophosphinic acid [5] or Cyanex272 [ bis(2,4,4–trimethylpentyl)phosphinic acid]) [6] improved the extraction efficiency of yttrium. Its extraction was influenced by the aqueous phase acidity, concentration of extractant and nature of diluent using acidic organo phosphorus compounds as extractants [1, 2]. The selective recovery of yttrium from associated elements was therefore achieved by the process of selective extraction. Due to the ever increasing demand for yttrium in industries, its separation and isolation for reuse has importance. Hence the recovery of yttrium needs to be explored for better extraction and stripping characteristics, using versatile extractants particularly for the practical applicability. Cyanex272 was selected as an extractant for yttrium due to its low water solubility and relatively lower acid strength (pKa = 6.4) [7] as the acidity of the extractant influences its extractability [4]. Also the systematic studies of yttrium using Cyanex272 are lacking. Various parameters such as pH of aqueous phase, nature of diluents, extractant concentration and the equilibration time were studied in detail to optimize the conditions for the quantitative extraction of yttrium. The influence of different mineral acids as stripping agents for yttrium was studied to enable its easy recovery from the organic phase. The effect of diverse ions on yttrium extraction was also investigated.
EXPERIMENTAL

Materials

A Digispec 110 D spectrometer (Feedback, India Ltd.) with matched 10mm glass cuvettes, a digital pH meter (Elico Private Ltd., India) with a combined glass and calomel electrode (Toshniwal – Moller, India) and a Wrist Action Flask Shaker (General Trading Corporation, India) were used.

The stock solution of yttrium(III) was prepared by dissolving 0.2252 g of yttrium carbonate in 1.0 cm$^3$ nitric acid, evaporated completely and then treated with 10 cm$^3$ nitric acid and finally diluted to 100 cm$^3$ with distilled water. It was standardized complexometrically [8] and was found to contain 1.0010 mg/cm$^3$ yttrium. A solution containing 30 µg/cm$^3$ of yttrium was prepared by appropriate dilution. Cyanex272 [CYTEC Canada Inc.] received as a gift sample was used without further purification. A 0.1 % aqueous solution of Arsenazo(III) [Loba Chemie, A.R.] was used for spectrophotometric determination.

General Procedure

An aliquot of solution containing yttrium (30 µg) was adjusted to pH = 7.0 with 0.1 M sulphuric acid or 0.1 M ammonium hydroxide and then equilibrated with 10 cm$^3$ of 5.0 x 10$^{-4}$ M Cyanex272 in xylene. The phase volume ratio of aqueous to organic phase was maintained at 1:1. The equilibration was done on a wrist action flask shaker and the period of equilibration was 20 minutes. After allowing the two phases to settle and separate out yttrium from the organic phase was stripped with 10 cm$^3$ of 4.0 M nitric acid. Yttrium was estimated in the aqueous phase spectrophotometrically with Arsenazo(III) at 650 nm [9]. The amount of yttrium was computed from the calibration curve. The distribution ratio (D) of yttrium was calculated from the ratio of percent of yttrium extracted in the organic phase to the aqueous phase. The extraction of yttrium carried out as per the general procedure for six replicate determinations had a relative standard deviation of 0.15.

RESULTS AND DISCUSSION

Effect of pH

Yttrium was extracted from pH = 1.0 – 8.0 with 5.0 x 10$^{-4}$ M Cyanex272 in xylene. The extraction was quantitative at pH =7.0 with 5.0 x 10$^{-4}$ M Cyanex272 in xylene as shown in Fig. 1.0. Hence further studies were carried out by maintaining the pH of aqueous phase at pH = 7.0.

Effect of Diluents on Extraction

Various organic solvents were used as diluents for 5.0 x 10$^{-4}$ M Cyanex272 (Table 1). The liquid cation exchangers undergo polymerization in non polar organic solvents. The extent of aggregation and hence the extractability is influenced by the nature of the organic solvent. Low dielectric constant solvents favour extraction as the extraction proceeds by ion pair formation. The increase in the aromatic character of solvent results in the decrease of extraction The interaction of the diluent with the extractant may inhibit extraction [10]. The incomplete extraction of yttrium with chloroform may be due to its high dielectric constant. The observed trends in extraction using toluene, benzene and cyclohexane may be attributed to their aromatic character. However a particular physical property of the organic solvent cannot be correlated with the extent of extraction. Hence it was observed that yttrium was quantitatively extracted when xylene was used as a diluent for Cyanex272. Hence the organic phase used was 5.0 x 10$^{-4}$ M Cyanex272 in xylene throughout the studies.
**Figure 1. Plot of pH Vs % Extraction**

![Plot of pH Vs % Extraction](image)

**Table 1 Effect of Diluents on Extraction**

<table>
<thead>
<tr>
<th>Diluents</th>
<th>Dielectric Constant</th>
<th>Percent Extraction</th>
<th>Distribution Ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>Benzene</td>
<td>2.20</td>
<td>85.0</td>
<td>5.7</td>
</tr>
<tr>
<td>Toluene</td>
<td>2.24</td>
<td>74.5</td>
<td>2.9</td>
</tr>
<tr>
<td>Xylene</td>
<td>2.28</td>
<td>99.4</td>
<td>165.7</td>
</tr>
<tr>
<td>Cyclohexane</td>
<td>2.02</td>
<td>88.4</td>
<td>7.6</td>
</tr>
<tr>
<td>Chloroform</td>
<td>4.90</td>
<td>76.8</td>
<td>3.3</td>
</tr>
<tr>
<td>Carbon</td>
<td>2.20</td>
<td>80.1</td>
<td>4.0</td>
</tr>
<tr>
<td>Tetrachloride</td>
<td>2.20</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Effect of Extractant Concentration**

Yttrium was extracted from \((0.9 - 5.0) \times 10^{-4}\) M Cyanex272 in xylene. It was quantitatively extracted at \(5.0 \times 10^{-4}\) M Cyanex272 (Table 2). Hence further studies were carried out with \(5.0 \times 10^{-4}\) M Cyanex272 in xylene.

**Table 2 Effect of Cyanex272 Concentration**

<table>
<thead>
<tr>
<th>Cyanex272 1 X 10^{-4} M</th>
<th>Percent Extraction</th>
<th>Distribution Ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.9</td>
<td>82.3</td>
<td>4.6</td>
</tr>
<tr>
<td>1.0</td>
<td>88.3</td>
<td>7.5</td>
</tr>
<tr>
<td>2.0</td>
<td>94.0</td>
<td>15.7</td>
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<tr>
<td>3.0</td>
<td>95.0</td>
<td>19.0</td>
</tr>
<tr>
<td>4.0</td>
<td>98.3</td>
<td>57.8</td>
</tr>
<tr>
<td>5.0</td>
<td>99.4</td>
<td>165.7</td>
</tr>
</tbody>
</table>

**Effect of Stripping Agents**

The influence of stripping agents was studied by stripping yttrium from the organic phase with \((1 - 8)\) M hydrochloric acid, \((1 - 8)\) M nitric acid or \((0.5 - 4)\) M sulphuric acid (Table 3). The recovery of yttrium was incomplete with hydrochloric acid and sulphuric acid. However,
it was quantitatively stripped with 4 M nitric acid. Hence the stripping agent used for all studies used was 4 M nitric acid.

\textbf{Table 3 Effect of Stripping Agents}

<table>
<thead>
<tr>
<th>Stripping Agent</th>
<th>0.1</th>
<th>0.5</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
</tr>
</thead>
<tbody>
<tr>
<td>M</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>% Recovery</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>HNO\textsubscript{3}</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>80.1</td>
<td>82.3</td>
<td>84.0</td>
<td>99.4</td>
<td>98.3</td>
<td>88.3</td>
<td>84.0</td>
<td>78.3</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>HCl</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>80.7</td>
<td>82.3</td>
<td>83.3</td>
<td>85.0</td>
<td>87.3</td>
<td>89.0</td>
<td>90.7</td>
<td>80.7</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>H\textsubscript{2}SO\textsubscript{4}</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>22.2</td>
<td>67.9</td>
<td>76.2</td>
<td>96.7</td>
<td>85.3</td>
<td>--</td>
<td>--</td>
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<td>--</td>
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<td></td>
</tr>
</tbody>
</table>

\textbf{Period of Equilibration and Stripping}

The extraction of yttrium was studied for different periods of equilibration and stripping. The extraction was studied for 5.0, 10.0, 15.0 and 20.0 minutes and the corresponding percentage extraction was 95.7, 97.3, 98.3 and 99.4 respectively. The stripping of yttrium was studied using different time periods. The percent recovery with 5.0, 10.0, 15.0 and 20.0 minutes of stripping with 4M nitric acid as stripping agent was 85.7, 90.0, 93.3 and 99.3 respectively. Hence the period of equilibration and stripping throughout the studies was 20 minutes.

\textbf{Nature of Extracted Species}

The nature of extracted species of yttrium with the extractant was ascertained by slope analysis [10]. A graph of Log [Distribution ratio] versus Log [Cyanex272] at fixed pH = 7.0 (Fig 2.) was a straight-line plot with slope = 1.7. Hence the probable stoichiometric ratio of yttrium with the extractant was 1:2.

\textbf{Effect of Various Diverse Ions}

The extraction of yttrium was studied in the presence of various diverse ions with 5.0 x 10\textsuperscript{-4} M Cyanex272 in xylene (Table 4). The tolerance limit was set as the amount of foreign ion which will cause \pm 2% error in the recovery of yttrium. The alkali and alkaline earth metals were tolerated in ratios exceeding 1:150. Chromium(VI), manganese(II) and zinc(II) were tolerated in the ratio 1:16 while cobalt(II) and cadmium(II) were tolerated in the ratio 1:30. Various ions like nickel(II), zirconium(IV), scandium(III) and lanthanum(III) were tolerated at the ratios 1:1.6, 1:3.3, 1:2.7 and 1:1 respectively. Cadmium(II) was tolerated in the ratio 1:33. Elements like vanadium(IV), vanadium(V), aluminium(III), thorium(IV) and some lanthanides interfered by co-extraction. The anions chloride, nitrate and sulphate were tolerated in ratios exceeding 1:150.

The separation of yttrium(III) from sodium(I), potassium(I), magnesium(II), calcium(II) and strontium(II) in binary mixtures was possible as these elements remained unextracted in the aqueous phase under the extraction conditions of yttrium. It was similarly separated from cerium(IV), lanthanum(III), zirconium(IV), cobalt(II) and nickel(II) as these elements were not extracted under the condition for extraction of yttrium. The elements after separation were determined spectrophotometrically with a suitable chromogenic ligand [9].

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Elements | Added As | Tolerance Limit μg
--- | --- | ---
Na⁺ | NaCl | 5000
K⁺ | KCl | 5000
Mg²⁺ | MgSO₄·7H₂O | 5000
Ca²⁺ | CaCl₂ | 5000
Sr²⁺ | Sr(NO₃)₂ | 5000
La³⁺ | La₂(CO₃)₃ | 30
Cr⁶⁺ | Cr(NO₃)₆ | 500
Mn²⁺ | MnSO₄·7H₂O | 500
Co³⁺ | Co(NO₃)₂·6H₂O | 1000
Ni²⁺ | (NH₄)₂SO₄·NiSO₄·6H₂O | 50
Zn²⁺ | ZnSO₄·7H₂O | 500
Cd²⁺ | 3CdSO₄·8H₂O | 1000
Ce⁴⁺ | Ce(SO₄)₂·4H₂O | 100
Zr⁴⁺ | Zr(NO₃)₄·2H₂O | 100
Mo⁶⁺ | (NH₄)₆Mo₇O₂₄·4H₂O | 500
Sc³⁺ | Sc(NO₃)₃ | 80
Cl⁻ | NaCl | 5000
NO₃⁻ | KNO₃ | 5000
SO₄²⁻ | K₂SO₄ | 5000

**Conclusion**

The method developed for the solvent extraction separation of yttrium is simple, rapid and reproducible. The concentration of extractant required for the quantitative extraction is very
low and the diluent used is non toxic in nature. Complete recovery of yttrium by nitric acid in a single stage will have potential for practical applicability. The method is selective for yttrium as most of the common elements are tolerated in high ratios during extraction of yttrium.

Reference

7. American Cyanamid Company, Cyanex272, Cyanex302 and Cyanex301 Technical Brochures