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# Separation of Sm-Eu-Gd mixed solutions using bifunctional ionic liquid [A336][P204]

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**Abstract.** This paper studied the use of bifunctional ionic liquid as an extractant to extract and separate Sm-Eu-Gd mixed solutions. The extractant combined the conventional extractant di-(2-ethyl hexyl) phosphate (P204) with ionic liquid Aliquat 336 (A336) through acid/base neutralization method. This work explored the effect of extraction medium (chloride, sulphate and nitrate), acid concentration (1.5-5.0 M) and organic to aqueous phase ratio (1:1, 3:2, 7:3, 4:1, 9:1) to determine the separation factor of each metals. Based on the results, Sm(III) showed the highest separation factor of total solute ( $\beta_{Sm/total-Sm}=2.81$ ) in the nitrate medium with the acid concentration of 3.0M and organic to aqueous phase ratio of 4:1. Sm(III) was chosen to be separated in the first stage of extraction from the Sm-Eu-Gd mixed solution. In the second stage, the optimized condition to separate the remaining Eu-Gd mixed solution into individual metal was also in the nitrate medium with organic to aqueous phase ratio of 3:2, except with lower acid concentration, 2.0M. This work shows that [A336][P204] is an impeccable extractant candidate for an improved and environmental friendly to separate Sm-Eu-Gd in both laboratory and industrial application.

## INTRODUCTION

Rare earth (RE) elements are part of the important strategic resources and have been applied to various areas, such as luminescence, electronics, catalysis, metallurgy, and ceramic industry. Therefore, the selection of suitable extractant to separate those REs become an important task, due to the remarkable chemical similarity of the RE elements. Over the years, an increased number of studies was done on the replacement of conventional extractants, such as the di-(2-ethylhexyl) phosphoric acid (P204) and 2-ethylhexyl phosphoric acid mono 2-ethylhexyl ester (P507) with another extractant that is able to provide a better distribution and separation properties [1-3]. Tributyl phosphate (TBP), for example, has become the most preferred extractant for industrial-scale operations and is proven to be able to separate trivalent rare earth efficiently in nitrate medium. Thorium Ltd. in England and Molycorp processing plant in Colorado used naphthenic acid to purify yttrium and were able to achieve 99.999% of high purity  $Y_2O_3$  from 47%  $Y_2O_3$  concentrates [4].

Nowadays, ionic liquid (IL) is appointed as the most leading technique in RE separation. The research on IL started in 1914 by Paul Walden and the application of IL has extended into the industrial scale [5]. IL possesses numerous benefits, such as low volatility, low combustibility, wide liquid range, thermal stability, adjustable functional group, high conductivity, and wide electrochemical window. In RE extraction, IL not only eliminates the need for saponification process, but it reduces the risk of combustion by removing the diluent from the process [6]. There were many studies published on the numerous combinations of metal-IL, especially for RE extraction [7-9]. Aliquat 336

(A336) is a promising candidate among the ILs in separating the RE due to its low price and less toxicity, as compared to other ILs [10-12].

Apart from formulating a new extractant, researchers also prefer to develop a binary extraction system consisting of two extractants. Over the past decade, many publications on the combination of P204 and P507 have emerged due to the ability of this mixture to overcome the limitations in each individual extractant [13-14]. The combination of these two conventional extractants helps to improve the acidity in the stripping process and provides high extraction efficiency for the extraction of RE. Combining IL with a common extractant has also become a trend as this formula showed high inner synergistic effect towards RE metals. The inner synergistic effect provides an advantage in the extraction system with a larger extraction capacity for both cation and anion in the extractant mixture [15]. Chen et al [16] summarized the work carried out by their group on the development of bifunctional IL extractant for RE separation. Their group explored several extractants, including the A336 with different cation exchange extractants such as P507 and P204. Large separation factors were reported for the separation of heavy REs, which were  $\beta_{Yb/Tm} = 7.92$  and  $\beta_{Lu/Yb} = 8.55$ .

Our previous paper described the interaction and mechanism of the Sm(III)-[A336][P204] [17]. Conventional slope analysis method is used to portray the extraction mechanism of Sm(III) using [A336][P204] as the extractant. The study showed an interaction between 2 molecules of the extractants with Sm(III) when using P204, while for the extraction using the bifunctional IL, 3 molecules of [A336][P204] are needed for each Sm ion. Based on the electroneutrality theory, the neutral extractant ( $[A336]^+[P204]^-$ ) will extract the cation (Sm(III)) and the anion ( $(NO_3)^-$ ) will be co-extracted back into the system due to the electroneutrality. Fig. 1 shows the proposed mechanism coordination for Sm(III)-[A336][P204] that indicates the mechanism involved is ion association.

In this article, the study focused on the separation of Sm-Eu-Gd from their mixed solution using the mixture of A336 and P204 as the extractant. The research also explores the effect of different acid medium, acid concentration, as well as organic-to-aqueous (O/A) phase ratio. At the end, based on the optimization result, a full solvent extraction system to produce an individual metal of Sm, Eu and Gd concentrate is developed.

## MATERIALS AND METHODS

### Chemicals and Reagents

Aliquat 336, (<99% purity, [A336]Cl) and di-(2-ethylhexyl) phosphate (P204) were purchased from Sigma Aldrich (USA). Nitric acid ( $HNO_3$ ), sulfuric acid ( $H_2SO_4$ ), hydrochloric acid (HCl), sodium hydroxide (NaOH), sodium chloride (NaCl), and sodium nitrate ( $NaNO_3$ ) were purchased from Fischer Scientific. Stock solution of Sm(III), Eu(III) and Gd(III) mixed solution was prepared by dissolving its oxide (99.9%) in concentrated acid. All other chemicals used were of analytical grade.

### Apparatus and Measurement

The pH values were measured using a Mettler Toledo (MP2200) pH meter. The concentration of RE(III) in the aqueous solution was determined by inductively coupled plasma—mass spectrometry (ICP-MS) (Agilent 7500 CX, USA). The concentration of the RE (III) in the organic phase was calculated by mass balance.

### Extraction Procedures

The [A336][P204] was prepared using by acid-base neutralization method as described by Sun et al. [18]. The extractant was dissolved in the n-heptane for extraction. 5 mL of aqueous phase containing RE(III) and 5 mL of organic phase containing the extractant were mixed and shaken for 1 hour at 200 rpm using a mechanical shaker, which was sufficient for equilibrium. The pH of the aqueous is maintained at pH 1–2 using the sodium salt (either NaCl or  $NaNO_3$ ). The mixtures were then centrifuged for 5 min at 8000 rpm to enhance the separation between the aqueous and organic layers. The distribution coefficient ( $D$ ), separation factor of two adjacent RE element ( $\beta_{z/z+1}$ ) and separation factor of targeted metal in a solution with more than 2 elements ( $\beta_{z/total-z}$ ) were calculated by:

$$D = C_{organic} / C_{aqueous} \quad (1)$$

$$\beta_{z/z+1} = D_z/D_{z+1} \quad (2)$$

$$\beta_{z/total-z} = D_z/D_{total-z} \quad (3)$$

where,  $C_{\text{aqueous}}$  is the final concentration of RE(III) in the aqueous solution, and  $C_{\text{organic}}$  is the final concentration of RE(III) in the organic solution,  $D_z$  is the distribution ratio of the element with  $z$  atomic number,  $D_{z+1}$  is the distribution ratio of the adjacent element with  $z+1$ , and  $D_{\text{total}-z}$  is the total distribution ratio of all the elements in the solution, except for the  $z$  element. The experiments were conducted in duplicate under the same conditions, and the relative error between the duplicate was less than 6%. The results were reported as mean values.

The extraction procedures of the mixed solution were done in two stages to achieve the individual separation of the RE. Both stages were studied at different types of acid medium, acid concentration and organic-to-aqueous phase ratio. At the end of every extraction stage, the output was stripped with the 0.5 M  $\text{HNO}_3$  and shaken for 1 hour at 200 rpm in room temperature.

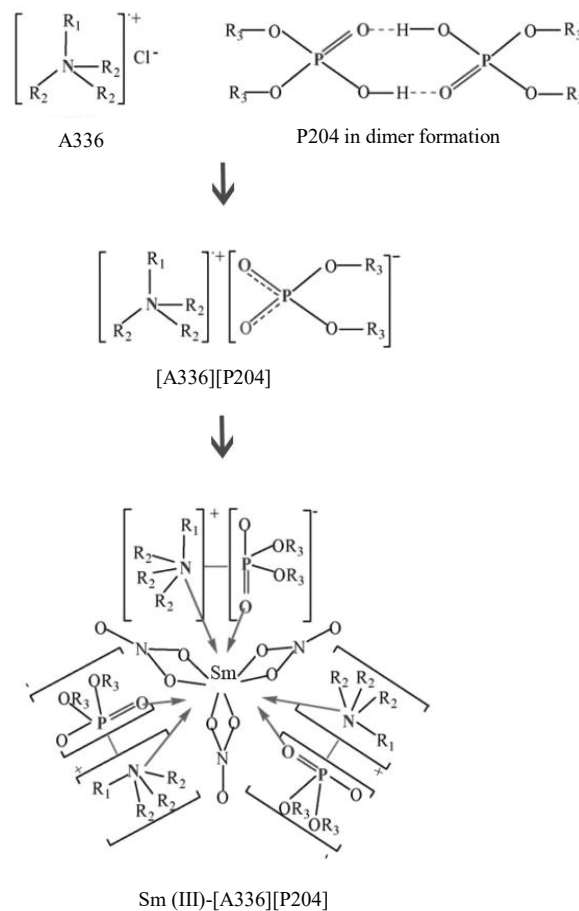


FIGURE 1. Proposed mechanism of the Sm(III)-[A336][P204] complex [17].

## RESULTS AND DISCUSSION

### The Separation of Sm-Eu-Gd Mixed Solution

#### *Effect of Different Types of Acid Medium and Concentration towards Sm-Eu-Gd Mixed Solution*

One of the issues in RE study is the separation of the RE elements, as it is known as a notorious task due to the chemical similarity of the RE that is very identical to one another [19]. The separation factor ( $\beta$ ) is defined as the distribution ratio for two elements at identical conditions. The  $\beta$  indicates relative difficulty level of separation between the two elements. Small value of  $\beta$  implies that the separation process between the two elements of interest is difficult and requires a greater number of extraction stages to get the concentrated metal product. The increased number of extraction stages had led to the increment of the process cost due to the higher chemical consumption [3].

The effect of HNO<sub>3</sub>, HCl and H<sub>2</sub>SO<sub>4</sub> on Sm(III), Eu(III) and Gd(III) was explored with [A336][P204] as the extractant, as shown in Table 1. The results were shown as  $\beta_{z/z+1}$  and  $\beta_{z/\text{total-z}}$  value. The number of  $\beta_{z/\text{total-z}}$  showed the capability of the extractant to attract the targeted element and maintain the other elements in the aqueous form. From the table, the values of  $\beta_{z/\text{total-z}}$  were the highest in the extraction of Sm(III) in the 3.0 M HNO<sub>3</sub> medium, followed by Sm(III) in the 2.0 M HCl, and Sm(III) in the 1.5 M HNO<sub>3</sub>. H<sub>2</sub>SO<sub>4</sub> showed poor performance for the separation of Sm from other REs compared to HNO<sub>3</sub> and HCl; however, this medium was the highest for the separation of Eu and Gd for all range of concentration. In the  $\beta_{\text{Sm/Eu}}$  perspective, 4.0 M HCl portrayed the highest separation capability with  $\beta_{\text{Sm/Eu}} = 2.20$ . Although the 4.0 M HCl showed good potential as the separation medium, the separation factor value of the Sm/Gd in the same condition was so close to the  $\beta_{\text{Sm/Gd}} = 2.27$ , creating high possibility for the Gd to interrupt the separation of the Sm/Eu.

Sun, X. et al. [20] proposed that the main extraction mechanism of [A336][P204] in HNO<sub>3</sub> medium is a neutral dissociation. In his paper, the distribution ratio of the Eu(III) in HNO<sub>3</sub> is 11 times higher than in the HCl ( $D_{\text{HNO}_3} = 21.7$ ,  $D_{\text{HCl}} = 1.97$ ), while in this study, the highest distribution ratio for HNO<sub>3</sub> compared to HCl is only 2.9 higher times when using the same extractant. The changes in the extraction capabilities in a different medium were the effect from the property of the anion in the system (NO<sub>3</sub><sup>-</sup>, Cl<sup>-</sup> and SO<sub>4</sub><sup>2-</sup>), which regulates the stability and selectivity of the extractant in the system [21]. Although it can be confirmed the HNO<sub>3</sub> is the best medium for the extraction, the detailed understanding of this mechanism is far from complete.

#### *Effect of O/A Phase Ratio towards Sm-Eu-Gd Mixed Solution*

Based on the result above, the optimization study for the Sm(III) separation from Sm-Eu-Gd mixed solution extended for the effect of the organic-to-aqueous phase ratio using the HNO<sub>3</sub> as the separation medium. As presented in Table 2, the effectiveness of the separation process increases with the amount of the extractant in the system until 4:1, and then reduced, as the extractant concentration rises. The order of the separation difficulty based on the O/A phase ratio is in an increasing order, as follows: Sm(III) < Eu(III) < Gd(III). It is clear that the best separation of Sm(III) could be performed using the O/A phase ratio of 4:1, with  $\beta_{\text{Sm/Eu}} = 2.26$  and  $\beta_{z/\text{total-z}} = 2.81$  selected as the best conditions for further extraction process.

### The Separation of Eu-Gd Mixed Solution

#### *Effect of Different Type of Acid Medium and Concentration towards Eu-Gd Mixed Solution*

The extraction experiment of Eu-Gd mixed solution with fixed O/A phase ratio was carried out at room temperature with different acid media with the concentration range from 1.5 M to 5.0 M. The results were displayed as in Table 3; it was seen that in HNO<sub>3</sub> medium, the  $\beta_{\text{Eu/Gd}}$  increased until it reached its maximum separation at 2.0 M, and decreased afterward. However, the trend for HCl and H<sub>2</sub>SO<sub>4</sub> showed the maximum separation factor took place at 3.0 M with  $\beta_{\text{Eu/Gd}} = 1.86$  for HCl and  $\beta_{\text{Eu/Gd}} = 1.17$ . Similar outcome was reported by Guo, L. et al. [21] showing high separation factor of RE(III) in the nitrate medium compared to chloride medium for light and heavy RE. They also stated that the separation value is higher in the bifunctional IL compared to conventional extractant such as P204, P507, TBP, and P350. Furthermore, conventional extractant required saponification process, which led to a high concentration of Ca<sup>2+</sup> and Mg<sup>2+</sup> in the wastewater; employing the [A336][P204] as the extractant can avoid this issue. The reason for the

high separation factor of RE in the nitrate medium is probably because of the anion hydration energy ( $\Delta G_{\text{NO}_3^-} = -314$  kJ/mol,  $\Delta G_{\text{Cl}^-} = -340$  KJ/mol and  $\Delta G_{\text{SO}_4^{2-}} = -1103$  kJ/mol) [16], which depicted that the characteristic of  $\text{Cl}^-$  and  $\text{SO}_4^{2-}$  are more hydrated than  $\text{NO}_3^-$ .

**TABLE 1.** Separation factor of Sm-Eu-Gd mixed solution in different types of acid medium.

<b>HNO<sub>3</sub></b>									
Conc.	RE(III)	Eu	Gd	$\beta_{z/\text{total-z}}$	Conc.	RE(III)	Eu	Gd	$\beta_{z/\text{total-z}}$
<b>1.5 M</b>	Sm	1.77	2.92	2.25	<b>4.0 M</b>	Sm	1.88	2.62	2.21
	Eu		1.65	0.99		Eu		1.39	0.85
	Gd			0.47		Gd			0.53
<b>2.0 M</b>	Sm	1.90	2.62	2.19	<b>5.0 M</b>	Sm	1.76	2.90	2.21
	Eu		1.38	0.86		Eu		1.64	0.92
	Gd			0.55		Gd			0.46
<b>3.0 M</b>	Sm	2.01	2.85	2.35					
	Eu		1.41	0.88					
	Gd			0.53					
<b>HCl</b>									
Conc.	RE(III)	Eu	Gd	$\beta_{z/\text{total-z}}$	Conc.	RE(III)	Eu	Gd	$\beta_{z/\text{total-z}}$
<b>1.5 M</b>	Sm	2.05	2.24	2.15	<b>4.0 M</b>	Sm	2.20	2.27	2.24
	Eu		1.08	0.74		Eu		1.03	0.70
	Gd			0.63		Gd			0.63
<b>2.0 M</b>	Sm	2.18	2.37	2.27	<b>5.0 M</b>	Sm	2.09	2.33	2.21
	Eu		1.09	0.70		Eu		1.11	0.75
	Gd			0.61		Gd			0.64
<b>3.0 M</b>	Sm	2.04	2.41	2.21					
	Eu		1.18	0.73					
	Gd			0.59					
<b>H<sub>2</sub>SO<sub>4</sub></b>									
Conc.	RE(III)	Eu	Gd	$\beta_{z/\text{total-z}}$	Conc.	RE(III)	Eu	Gd	$\beta_{z/\text{total-z}}$
<b>1.5 M</b>	Sm	1.01	1.27	1.14	<b>4.0 M</b>	Sm	1.22	1.44	1.33
	Eu		1.26	1.14		Eu		1.18	1.01
	Gd			0.79		Gd			0.77
<b>2.0 M</b>	Sm	0.96	1.35	1.12	<b>5.0 M</b>	Sm	1.14	1.31	1.22
	Eu		1.41	1.23		Eu		1.15	1.04
	Gd			0.72		Gd			0.82
<b>3.0 M</b>	Sm	0.89	1.34	1.07					
	Eu		1.51	1.32					
	Gd			0.70					

Experiments were done in room temperature for 1 hour using [A336][P204] as the extractant, n-heptane as the diluent with organic-to-aqueous ratio 1:1 and pH maintain at pH 1–2.

**TABLE 2.** Separation factor of Sm(III), Eu(III) and Gd(III) mixed solution in different O/A phase ratio.

O:A	RE(III)	Eu	Gd	$\beta_{z/\text{total-z}}$	O:A	RE(III)	Eu	Gd	$\beta_{z/\text{total-z}}$
<b>1:1</b>	Sm	1.52	2.85	1.97	<b>4:1</b>	Sm	2.26	4.00	2.81
	Eu		1.87	1.17		Eu		1.77	0.92
	Gd			0.46		Gd			0.42
<b>3:2</b>	Sm	1.78	3.05	2.15	<b>9:1</b>	Sm	1.98	4.04	2.54
	Eu		1.71	0.99		Eu		2.03	1.00
	Gd			0.46		Gd			0.38
<b>7:3</b>	Sm	1.72	2.58	1.98					
	Eu		1.50	0.89					
	Gd			0.54					

Experiments were done in room temperature for 1 hour using [A336][P204] as the extractant, n-heptane as the diluent in 3.0 M of  $\text{HNO}_3$  and the pH maintain at pH 1-2.

**TABLE 3.** Separation factor of Eu(III) and Gd(III) ( $\beta_{Eu/Gd}$ ) in different types of acid media and concentrations

Acid concentration	1.5 M	2.0 M	3.0 M	4.0 M	5.0 M
HNO <sub>3</sub>	2.25	2.44	2.15	2.12	2.12
HCl	1.79	1.79	1.86	1.75	1.61
H <sub>2</sub> SO <sub>4</sub>	1.08	1.12	1.17	1.05	1.04

Experiments were done in room temperature for 1 hour using [A336][P204] as the extractant, n-heptane as the diluent with organic-to-aqueous ratio 1:1 and pH maintain at pH 1–2.

#### *Effect of O/A Phase Ratio towards Eu-Gd Mixed Solution*

It is important to improve the mass transfer and extraction efficiency of the separation of RE(III) by investigating the organic to aqueous phase ratio. At the optimum O/A phase ratio, the rate of coalescence and redispersion of the dispersed phase are enhanced. To find the suitable phase ratio, the extraction of Eu/Gd with [A33][P204] was carried out using a range of O/A phase ratio of 1:1, 3:2, 7:3, 4:1, and 9:1. The result in Table 4 showed that the extraction was optimized at the phase ratio of 4:1 with  $\beta_{Eu/Gd}$  of 4.34. At the higher amount of extractant, the separation abilities fall down to  $\beta_{Eu/Gd}$  = 2.95. The amount of [A336][P204] must be high enough to ensure high separation ability, but not as high as to compromising the viscosity of the medium [22].

**TABLE 4.** Separation factor of Eu-Gd mixed solution in different O/A phase ratio.

Organic to acid ratio	1:1	3:2	7:3	4:1	9:1
$\beta_{Eu/Gd}$	2.44	3.62	4.02	4.34	2.95

Experiments were done in room temperature for 1 hour using [A336][P204] as the extractant, n-heptane as the diluent in 2.0 M of HNO<sub>3</sub> and pH maintain at pH 1-2.

### **Solvent Extraction System for the separation of Sm(III), Eu(III) and Gd(III)**

Fig. 2 showed the development of a solvent extraction system with the bifunctional IL [A336][P204] as extractant in the n-heptane for the separation of Sm(III), Eu(III) and Gd(III). The Sm(III) separation from the mixture took place in the first part of the extraction system due to the high separation factor in the 3.0 M HNO<sub>3</sub> medium with O/A phase ratio 4:1. The extracted Sm(III) had undergone the stripping process with 0.5 M HNO<sub>3</sub> to obtain the Sm concentrate. In the next stage of the extraction, the Eu(III) separation occurred for the remaining Eu and Gd in the mixture with the extraction parameter of 2.0 M HNO<sub>3</sub>, O/A = 3:2 at room temperature for the duration of one hour. The Eu(III) was attracted to the extractant and stripped off in the next stage, while Gd remained in the aqueous phase. Although the separation was performed successfully, the extraction percentage for each metal was quite low, which was between 35–50%. A further study will explore on the number of cycles required for each extraction and stripping stages to increase the yield of the metal concentrate in the final product.

### **CONCLUSION**

In this paper, a systematic investigation for the extraction of Sm(III), Eu(III) and Gd(III) in HNO<sub>3</sub>, H<sub>2</sub>SO<sub>4</sub> and HCl media and various O/A phase ratio by [A336][P204] was carried out. The results indicated that the Sm(III) was easier to separate from the Sm-Eu-Gd mixed solution based on the  $\beta_{z/total-z}$  value in the first stage of the extraction, and Eu(III) for the second stage of the extraction. HNO<sub>3</sub> gave higher separation capabilities compared to H<sub>2</sub>SO<sub>4</sub> and HCl media. For both cases in HNO<sub>3</sub>, the separation factor increases until it reached 4.0 M for the separation of Sm(III) and 2.0 M for the separation of Eu(III). The separation factor was then reduced at higher concentration. The effect of O/A phase ratio gave similar trend, which it escalated until its maximum level, and is reduced afterward. This study provides an evidence to the capability of the bifunctional IL to separate the Sm(III), Eu(III) and Gd(III), thus reducing the problem in the conventional extractant.

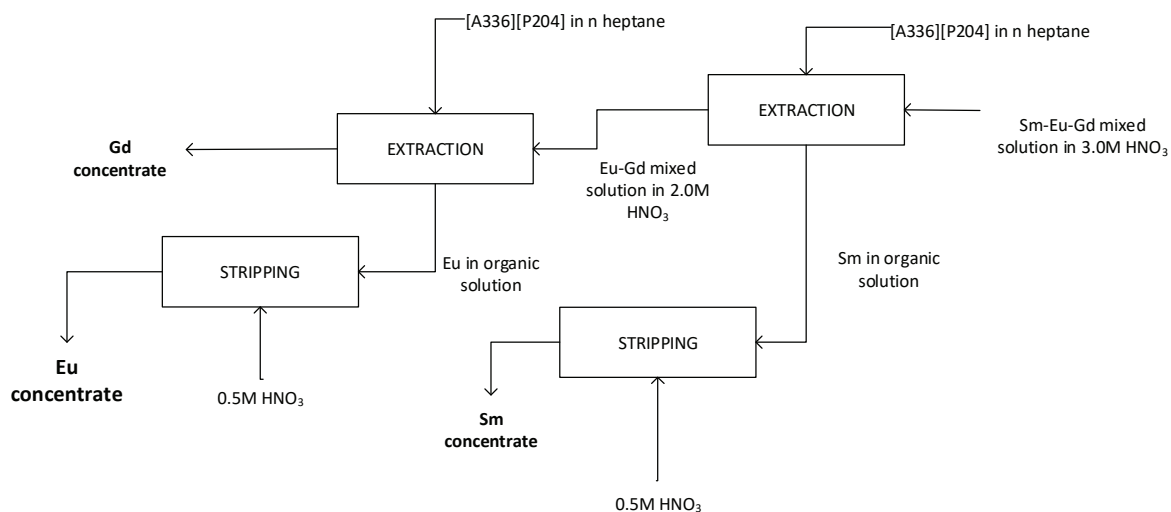


FIGURE 2. Solvent extraction system for separation of Sm-Eu-Gd mixed solution using [A336][P204]

## ACKNOWLEDGEMENT

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