



# Organic solvent extraction of uranium from alkaline nuclear waste

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## Abstract

There are proliferation issues with the Plutonium Uranium Redox Extraction process due to the possibility of recovering plutonium. The objective of this research was to evaluate different organic extraction ligands that can remove uranium from the nuclear waste and to determine the most effective organic solvent for extracting uranium only, from alkaline media. The results indicate that Alamine 336 in xylene has zero (0%) extraction capability for surrogate fission products at an optimum extraction time of 15 min. Aliquat 336 in xylene has an extraction percentage of 72% for uranium in 60 min. However, Aliquat 336 in toluene extracted 82% of the uranium from the feed solution after 30 min, decreasing to 76% after 60 min.

**Keywords** Aliquat 336 · Alkaline media · Aqueous solution · Extraction ligand · Organic solvent

## Introduction

A large amount of uranium in the environment is caused by human activities such as mining and milling of other minerals and also from operations of reactors, reprocessing of spent nuclear fuel (SNF) and its disposal [1, 2]. It is also of great commercial interest due to its application in the nuclear industry especially in the use and development of new radiopharmaceuticals and radioisotopes [3].

The uranium waste contains insoluble precipitates formed when the target plates uranium–aluminum alloy are dissolved during the production of Mo-99 [4]. The remaining insoluble residue contains about 90% of enriched uranium that is present in the mixture of various oxidation states.

A process of ammonium carbonate-based dissolution has been developed for uranium recovery from the waste generated by an alkaline dissolution process used for the production of the medical isotope Mo-99 [5]. Uranium recovery from residue was achieved with three successive ammonium

carbonate–peroxide leaches with final decontamination factors from low values of 1–10 Cs-137, Ru-106, and Sb-125 to infinity.

The most commonly used processes for purification and concentration are ion exchange and solvent extraction. Solvent extraction (SX) is one used to recover and separate uranium from sulphuric acid leach liquors [6]. The most well-known process worldwide for extraction of uranium from spent nuclear fuel is the Plutonium Uranium Redox Extraction (PUREX) process, where nitric acid solution is used with Tributyl phosphate (TBP) as an extractant. Smith et al. [7] have studied the dissolution of  $\text{UO}_2$ ,  $\text{U}_3\text{O}_8$  and  $\text{UO}_3$  in carbonate solution with the counter cations  $\text{NH}^+$ ,  $\text{Na}^+$ ,  $\text{K}^+$  and  $\text{Rb}^+$ . In a solution of 1.0 M carbonate and 0.1 M  $\text{H}_2\text{O}_2$ , it was found that the most rapid dissolution occurs in ammonium carbonate. The authors also compared the dissolution rates of the different uranium oxides and found the rate to be decreasing from  $\text{UO}_3 \gg \text{U}_3\text{O}_8 > \text{UO}_2$ . This is expected since the oxidation state of uranium required to be +6 after dissolution.

Kim et al. [8] investigated solvent extraction of uranium using amine based extractants. Amine based extractants such as, Alamine 336, Alamine 308, Alamine 304 and Aliquat 336 in diluent kerosene were investigated. The results showed that Alamine 336 was the best extractant for uranium from sulphate solutions compared to other amine extractants. Extraction percentage of 99.8% was recovered from low grade ore without interference of other metals.

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Zhu et al. [9] stated in his study of solvent extraction of uranium and separation of vanadium that quaternary amine could be used to extract uranium from alkaline solution, but its application is prevented by the formation of a third phase. The results were reported that more than 98% of uranium was extracted using 3% (w/v) of Aliquat 336 and 3% (w/v) isodecanol in Shellsol D70 from carbonate leach solution. The authors reported that over 90% of uranium was stripped from loaded organic solution. Stepanov et al. [10] studied a new technology that can be used for reprocessing of spent nuclear fuel, the so called CARBEX (carbonate extraction) process. The same workers reported that CARBEX process can be more effective and safe than PUREX process. Fourie et al. [11] stated that the recovery of uranium from Mo-99 production waste will reduce the residue volumes that need to be disposed of, while the recuperated enriched uranium can be used during the production of this isotope. This would decrease the cost of production.

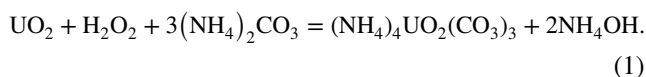
The uranium waste consists of an insoluble precipitate formed when the target plates containing uranium–aluminum alloy are dissolved during the production of Mo-99 [4]. This solid residue contains uranium and most fission products, like molybdenum (Mo-99), cesium (Cs-137), strontium (Sr-90), barium (Ba-140), antimony (Sb-125), tellurium (Te-132), iodine (I-131) and a portion of the ruthenium (Ru-106) and zirconium (Zr-95).

U is an element that presents various oxidation states ( $U^{2+}$ ,  $U^{3+}$ ,  $U^{4+}$ ,  $U^{5+}$  and  $U^{6+}$ ), of which uranous ( $U^{4+}$ ) and uranyl ( $U^{6+}$ ) are the most important [3]. A dissolution study for the recovery of uranium waste produced from Mo-99 was first conducted at Kernforschungszentrum Karlsruhe (KFK) [12]. This work was concentrated on using carbonate/bicarbonate mixtures as the dissolution medium with different oxidizing reagents e.g. hydrogen peroxide. Stepanov et al. [10] stated that carbonate solutions differ from acid leach solutions because they do not show oxidative activity when there are no oxidants. Therefore, oxidants are needed for an effective dissolution of uranium dioxide in carbonate solution. Most researchers use hydrogen peroxide as an oxidant to convert U(IV) to U(VI). Uranyl ion  $UO_2^{2+}$  forms strong complexes with different carbonates and natural organic matter ligands depending on the pH [1].

When extracting uranium, whether through acid or alkaline leaching, it must be oxidized to the hexavalent state U(VI), before it is dissolved [13]. An advantage of using alkaline leaching is high selectivity of uranium over impurities because some of the impurities do not dissolve in alkaline solution [9]. Furthermore, it causes no corrosive activity in the equipment and carbonates are nontoxic to biological objects. The formation of uranyl carbonate–peroxide complex ions has been observed in sodium carbonate and hydrogen peroxide medium. It was proven in the alkaline leaching, that hydrogen peroxide plays two roles, firstly as an oxidant that accelerates the rate

of dissolution, and secondly as a strong ligand forming mixed carbonate–peroxide complex when reacting with uranium [14].

The use of anodic dissolution of simulated spent nuclear fuel containing  $UO_2$  and fission products in alkaline aqueous solution using sodium carbonate–sodium bicarbonate [15] and ammonium carbonate solutions [16] has been performed. In these studies, uranyl ions were produced anodically as stable carbonate complexes, and at the same time, simulated fission products were precipitated as hydroxo or carbonate compounds. Uranyl ions were recovered as hydroxo compounds by adding sodium hydroxide to the solution after removing the Fission products precipitates. During the dissolution experiments, precipitates of the simulated fission products were observed on the pellet and in  $(NH_4)_2CO_3$  solution used as the electrolytic solution. Analysis of the electrolytic solution revealed that most of the simulated fission products, i.e. alkaline earth and rare earth elements, are precipitated in high ratios [16]. The aim of this work was to investigate the extraction of Uranium from irradiated target waste residue plates for Mo-99 production using alkaline extraction technology. This is therefore a new application of an existing process. In alkaline leaching, oxygen is used as a strong oxidizing agent. However, in alkaline leaching, ammonium carbonate is used instead of sodium carbonate because reagents are recoverable for re-use, ammonium carbonate is a milder alkali than sodium carbonate leading to reduced chance of uranium precipitation. Uranium oxidizes and forms carbonate–peroxide complexes when uranium dioxide is oxidatively dissolved in carbonate solution containing hydrogen peroxide, which eventually converts to the soluble uranyl carbonate anion  $UO_2(CO_3)_3^{4-}$  [17]. Equation 1 shows the main reaction that take place:



## Theory

The percentage amount of uranium extracted during the extraction process can be calculated using the following equations:

$$D = \frac{[U_i] - [U_f]}{[U_f]}, \quad (2)$$

where D is distribution ratio,  $U_i$  is initial concentration before extraction and  $U_f$  is final concentration after extraction

$$\%E = \frac{100D}{D + \frac{V_{aq}}{V_{org}}}, \quad (3)$$

where %E is the extraction percentage,  $V_{aq}$  is the volume of the aqueous phase and  $V_{org}$  is the volume of the organic phase.

## Experimental

### Phase 1: Evaluation of the organic solvent using surrogates

#### Sample preparation

A total of 90 samples of organic solvents were prepared using three different extractants (Alamine 336 and Aliquat 336) and three different diluents (kerosene (viscosity, 1.30 at 40 °C and dielectric constant of 1.8 at 20 °C), Xylene (viscosity of 0.81 and dielectric constant of 2.57 at 20 °C), and benzene (viscosity: 0.65 20 °C and dielectric constant: 2.3 at 20 °C) at varying extraction time. A measure of 30 ml of each of the extractants; Aliquat 336 and Alamine 336 were mixed with 70 ml of each of the diluents. A 2 M solution of ammonium carbonate was prepared in 1000 ml of deionized water. Surrogate impurities (Sr, Sb, Ce, Fe, Nd, and Mg) were dissolved in the ammonium carbonate solution.

#### Extraction procedure

20 ml of surrogate solution and 20 ml of each organic solution were added together in falcon tubes and rotated using a Stuart SB3 (Bibby Scientific, United States) rotator for varying times from 3 to 30 min. The solution was then transferred into a separatory funnel and was left for an hour to allow the aqueous and organic phases to separate. The organic phase was eluted and the aqueous phase was analyzed by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS). This procedure was repeated for each organic solution.

#### Aqueous sample for ICP-MS analysis

5 g of aqueous sample was weighed in a crucible. 5 ml of 65% nitric acid and 1 ml of 32% hydrochloric acid were added to the sample. The samples were placed in an Anton Par 3000 Microwave digester and left overnight to digest. An aliquot of 5 ml from each sample was diluted to 100 ml using deionized water and analyzed using a calibrated Agilent 7500 CE ICP-MS. Each sample was analyzed in duplicates.

### Phase 2: Evaluation of best diluent using uranium samples

#### Sample preparation

The uranyl solution used for the extraction was generated from the dissolution of Uranium Ore Concentrate (UOC) with ammonium carbonate and hydrogen peroxide solution. A 0.01 M solution of uranium in 1 M of ammonium carbonate

was prepared by dissolving 0.70 g of  $U_3O_8$  in 250 ml of carbonate solution. 30% hydrogen peroxide was added as an oxidant to a final concentration of 0.9 M in the mixture, which was heated for 60 min at 60 °C. After an hour, the reaction was complete, heating was stopped and the solution was cooled down. The solution was then filtered using a 541 Whatman filter paper. The filter paper was washed with a small amount of deionized water. The final volume of the solution was recorded.

#### Pre-equilibrium

During SX, there is a decrease in feedstock solution volumes and inaccurate concentration values after extraction due to the occurrence of co-extraction of the feed solution into the organic solution. To prevent this, the organic phase has to be pre-equilibrated with feed solution that does not contain any solute using the following steps. (1) An equal amount of aqueous feed phase without solute (without U) and organic extractant phase was added in 50 ml falcon tubes. (2) The falcon tubes were inserted in a rotator at 7 rpm for 60 min. (3) After 60 min, the solutions in the tubes were transferred into a separatory funnel and left for an hour to allow the two phases to separate. (4) The pre-equilibrated organic phase was poured into a separate container for further use.

#### Extraction procedure

The  $U_3O_8$  dissolved in  $(NH_4)_2CO_3$  was used as the aqueous feedstock for the extraction step. Xylene, Kerosene and Toluene (viscosity: 0.59 at 20 °C and dielectric constant: 2.4 at 25 °C) were used as organic phase extracting agents. The aqueous solution obtained from  $U_3O_8$  dissolution and pre-equilibrated organic solution were added together in a falcon tube at a ratio of 1:1. Step 2–4 in pre-equilibration process was repeated. Following phase contact and reaching equilibrium in step 3, the solution was left for 60 min to ensure that mass transfer equilibrium is reached. The aqueous and organic phases were separated by separation funnel and the aqueous phase was used to prepare the solution used for UV VIS analysis. An aliquot of uranium from the aqueous solution was transferred into a 25 ml volumetric flask. 3 ml of sodium carbonate and 1 ml of hydrogen peroxide was added to the flask and diluted with deionized water. Then the sample was analyzed at 450 nm using a CARY 100 VARIAN UV–VIS Spectrophotometer, after setting up of a calibration curve in the region 50–200 ppm. The extraction was performed for various time periods of 15, 30, 45 and 60 min.

Figure 1 shows the experimental setup for the separation of aqueous phase and organic phase. After allowing the two phases to separate, the organic phase is eluted, and the aqueous phase is analyzed. During the extraction process, it was found that a third phase appeared when extraction of uranium using Aliquat 336 with kerosene was performed.



**Fig. 1** Experimental setup for the separation of aqueous phase and organic phase

Due to the appearance of a third phase, the diluent kerosene was eliminated from the experiment

From the alkaline solution containing 2 M of ammonium carbonate and 0.9 M hydrogen peroxide, the extraction of surrogates Mg and Fe (representative of activation products), and Sr, Sb, Ce, Nd (representative of fission products) was carried out using Aliquat-336 and Alamine-336 as extracting agents dissolved in each of kerosene, xylene and benzene respectively.

### Evaluation of the best diluent

Furthermore in this study, evaluation of the best diluent for optimal uranium extraction was investigated. From the alkaline solution containing 1 M of ammonium carbonate and 0.9 M of hydrogen peroxide, the extraction of uranium was

carried out using Aliquat 336 which was dissolved in each of kerosene, xylene and toluene respectively.

## Results and discussion

### Phase 1: Evaluation of the organic solvent using surrogates

The evaluation of different extractants and diluents were investigated at different times. The experimental data of extraction of surrogates at various time (3, 10, 15, 20 and 30 min) using different organic solvents are reported in Tables 1, 2, 3, 4 and 5. These results show the best combination of extractants and diluents at various times. However, these studies were performed at 1 M ammonium carbonate, which is not optimal for U extraction, and the influence of ammonium carbonate concentration on the extraction of the fission product surrogates were therefore investigated in the next section.

The negative extraction percentages indicate that precipitation has occurred. This is a positive indication that separation of uranium from these products may be possible by extracting uranium only using the organic extractants in prototype experiments. Table 6 shows that the optimum extraction time was found to be 15 min with Alamine 336 in Xylene.

Aliquat 336 in xylene, kerosene and toluene was selected for the extraction of uranium from alkaline solutions as it is a quaternary amine reagent shown to be a successful extractant from carbonate media by Stepanov et al. [10]. It is an ionic liquid reagent similar to Alamine 336. Aliquat 336 extracts metal ions higher than other extractants. The increase in metal extractions by Aliquat 336 is due to the increase in basic property of alkyl group which improves the cation–anion ion pair

**Table 1** Extraction percentages for surrogates at 3 min rotating time

Element	Alamine 336			Aliquat 336		
	Kerosene	Xylene	Benzene	Kerosene	Xylene	Benzene
Mg	–125.0	–36.4	–127.3	–25.0	–22.7	–25.0
Fe	–95.4	–20.7	–129.9	–20.7	–14.9	–60.9
Sr	–1,049,900.0	–354,900.0	–550,180.0	–450.0	–150.0	–435.0
Ce	–80.8	–7.7	–23.1	29.6	30.4	18.8
Nd	–12.3	23.2	8.2	40.9	55.0	34.1

**Table 2** Extraction percentages for surrogates at 10 min rotating time

Element	Alamine 336			Aliquat 336		
	Kerosene	Xylene	Benzene	Kerosene	Xylene	Benzene
Mg	–104.5	–25.0	–13.6	–77.3	–79.5	–88.6
Fe	–83.9	–10.9	–60.9	–78.2	–66.7	–106.9
Sr	–33,900.0	–544,900.0	–245.0	–190.0	–49.0	–20.0
Ce	–69.2	–7.7	19.2	11.5	7.7	–7.7
Nd	–18.2	34.1	9.1	37.7	29.1	0.0

**Table 3** Extraction percentages for surrogates at 15 min rotating time

Element	Alamine 336			Aliquat 336		
	Kerosene	Xylene	Benzene	Kerosene	Xylene	Benzene
Mg	−104.5	−68.2	−27.3	−140.9	−40.9	−31.8
Fe	−66.7	1.0	−89.7	−55.2	−31.0	−72.4
Sr	−264,900.0	0.0	−525.0	−295,035.0	−270.0	ND
Ce	−53.8	0.0	9.6	−3.8	28.8	11.5
Nd	8.2	0.0	30.0	38.6	51.8	−31.8

**Table 4** Extraction percentages for surrogates at 20 min rotating time

Element	Alamine 336			Aliquat 336		
	Kerosene	Xylene	Benzene	Kerosene	Xylene	Benzene
Mg	−59.2	6.7	104.6	−177.3	−229.5	−118.2
Fe	−20.8	25.2	144.1	−7.5	−43.7	−124.1
Sr	−1,099,908.0	−125,300.0	267.2	−289,900.0	−549,900.0	−1,499,900.0
Ce	−11.6	42.3	73.6	7.7	−19.2	−19.2
Nd	31.8	54.5	68.3	59.1	42.7	−27.3

**Table 5** Extraction percentages for surrogates at 30 min rotating time

Element	Alamine 336			Aliquat 336		
	Kerosene	Xylene	Benzene	Kerosene	Xylene	Benzene
Mg	−131.8	9.0	−229.5	−86.4	−150.0	−140.9
Fe	−83.9	25.2	−175.9	−72.4	−32.2	−101.1
Sr	−2,999,900.0	−180,700.0	−449,900.0	ND	−75,900.0	ND
Ce	−61.5	34.5	−50.0	11.5	15.4	−15.4
Nd	−4.5	40.9	−31.8	10.0	20.5	−9.1

**Table 6** Effects of surrogate's extraction at the best various extractants

Extracting time	3 Min	10 Min	15 Min	20 Min	30 Min
Extractants	Aliquat 336	Aliquat 336	Alamine 336	Aliquat 336	Aliquat 336
Element	Xylene	Xylene	Xylene	Kerosene	Kerosene
Mg	−22.7	−79.5	−68.2	−177.3	−86.4
Fe	−14.9	−66.7	1.0	−7.5	−72.4
Sr	−150.0	−49.0	0.0	−289,900.0	ND
Ce	30.4	7.7	0.0	7.7	11.5
Nd	55.0	29.1	0.0	59.1	10.0

and increases the stability of the complex formed in extraction process [18].

## Phase 2: Evaluation of uranium extraction using Aliquat 336 in different diluents

### Extraction of uranium with 5% Aliquat 336 diluted in 95% kerosene, toluene and xylene

The extraction of uranium with Aliquat 336 in kerosene, toluene and xylene was investigated. There was no formation

of a third phase observed during the extraction of uranium with xylene and toluene, as was the case with kerosene. Due to the formation of the third phase, and inconsistency at 30 and 60 min (Table 7), Kerosene was eliminated from the experiment. These results indicate that the uranyl ion present in ammonium carbonate solution does not complex easily with the extractant when diluted with kerosene. The extractant composition was 5% Aliquat 336 mixed with xylene to a total of 100 ml in a volumetric flask. A ratio of 1:1 volume of aqueous/organic solution was used for the extraction process and the samples were prepared in triplicates. The same

**Table 7** Results of uranium extracted with 5% Aliquat 336 and 95% Kerosene

Extraction time (min)	D	%E
0	0	0
15	0.09	8.19
30	0.01	0.54
45	0.03	2.58
60	0.01	0.76

procedure was used for Aliquat 336 in toluene. The graph in Fig. 2 shows the extraction percentage of uranium with Aliquat 336 in xylene at various times. For Aliquat 336 in xylene, the extraction percentage was found to be 71% at 15 min which decreased at 30 min and then increased again to 72% at 60 min. Extraction of uranium using Aliquat 336 in toluene was also investigated. Uranium extraction percentage increases with time from 0 to 30 min and slightly decreases thereafter. This implies that for toluene the optimum rotation time for the mixture of aqueous and organic solutions is 30 min (Fig. 3). At 30 min an extraction percentage of 82% for uranium in toluene was reached and as time increased from 45 to 60 min, the extraction percentage decreased from 78 to 76%.

The equilibrium time was optimized as 30 min for uranium extraction in all further experiments, and toluene was chosen as the best diluent due to higher extraction of uranium Fig. 3.

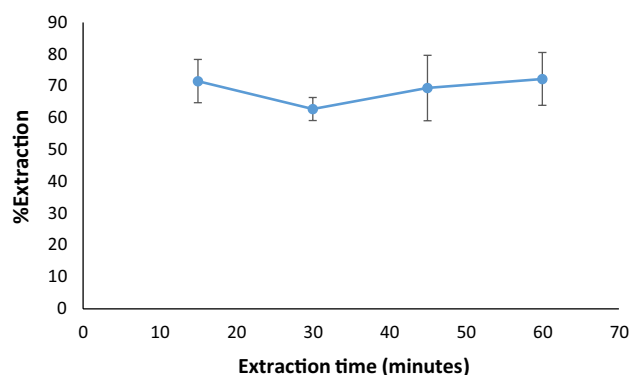
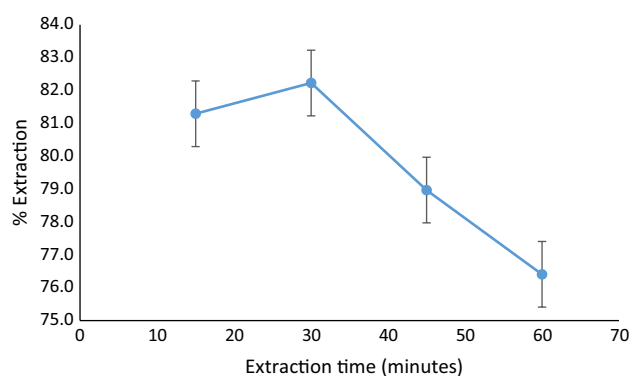
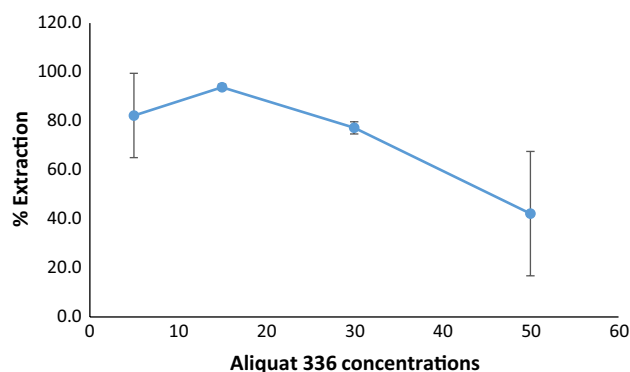
### Evaluation of the best concentration of Aliquat 336

The extraction percentage of uranium using Aliquat 336 concentrations of 5%, 15%, 30% and 50% with toluene was investigated. In Fig. 4, it can be seen that the extraction percentage of uranium increases from 80 to 90% as the concentration of Aliquat 336 increases from 5 to 15%, but decreased at higher Aliquat 336 concentrations. This implies the optimum concentration percentage of Aliquat 336 is 15%.

### Conclusion

The experimental investigation reported here studied extraction of surrogates from alkaline solutions. From Table 6, it was concluded that the optimum extraction time was 15 min with Alamine 336 in xylene.

The study of uranium extraction from alkaline solutions was also investigated. An amine based extractant Aliquat 336 was proposed as a promising extractant for the uranium extraction without extracting other elements. kerosene, xylene and toluene were proposed diluents for the

**Fig. 2** Extraction percentage of uranium in 1 M ammonium carbonate in 5% Aliquat 336 in Xylene**Fig. 3** Extraction percentage of uranium in 1 M ammonium carbonate in 5% Aliquat 336 in Toluene**Fig. 4** The effect of Aliquat 336 concentration with constant 0.9 M  $H_2O_2$ , 1 M  $(NH_4)_2CO_3$  and U

experiment. In the results above, it is shown that kerosene forms a third phase. Due to this third phase, kerosene was eliminated from the experiment. Toluene was found to be the best diluent with the optimum extraction time of 30 min. Evaluation of different concentrations of Aliquat 336 showed

that at a concentration of 15% of Aliquat 336, the extraction percentage was found to be 93%, therefore it is concluded that 15% of Aliquat 336 is the optimum concentration.

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