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Separation and purification of ^{106}Ru from effluent streams of ion-exchange cycle used for Pu purification in PUREX process

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ABSTRACT

This paper describes the separation and purification of ^{106}Ru from ion-exchange effluent streams of PUREX process using combinations of solvent extraction, extraction chromatography, and ion-exchange. The effluent stream from ion-exchange cycle contains ~100 mCi/L ^{106}Ru activity along with contaminants like ^{95}Nb, uranium, and plutonium at an acidity of ~7.00 M HNO_3. In first step, the feed solution is contacted with 30% Tri-n-butyl phosphate in *n*-dodecane which not only lowers the acidity of the feed solution but also reduces the Pu, U, and Nb concentration significantly. The raffinate from this step is subjected to extraction chromatography wherein uranium and plutonium are completely sorbed on a column containing 2-ethylhexyl 2-ethylhexyl phosphonic acid (KSM-17) sorbed on an inert polymeric support XAD-4. In these steps, Ru activity is found to retain in the aqueous raffinate/effluent phase without any loss. The effluent from the second step is neutralized to an acidity of ~0.50 M using NaOH solution and passed through a silica gel column for complete sorption of Nb activity. The effluent obtained from the silica gel column shows ^{106}Ru, free from all the radioactive contaminants. The recovery of Ru separation is found to be >90%. Based on results, a flow sheet is proposed and used for Ru separation at mCi levels.

Keywords: PUREX process; Anion-exchange resin; TBP; KSM-17; Silica gel; ^{106}Ru; ^{95}Nb

1. Introduction

PUREX process is the most developed and widely used liquid–liquid extraction method for spent fuel reprocessing [1]. In this process, uranium and plutonium are co-extracted in 30% Tri-n-butyl phosphate (TBP) in *n*-dodecane from dissolved spent fuel in nitric acid solution under optimum conditions to remove bulk of fission products in aqueous raffinate phase. Organic phase after scrubbing with nitric acid is subjected to partitioning step in which uranium and plutonium are separated mutually. Aqueous streams containing uranium and plutonium are further purified to the desired level using solvent extraction/ion-exchange processes. The process is

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operated in multicycles as the decontamination from certain fission products *viz.* ruthenium, zirconium, and niobium becomes difficult due to their complex extraction chemistry under process conditions.

Among the troublesome fission products, isotopes of Ru, i.e. ^{106}Ru and ^{103}Ru having $T_{1/2} = 1$ y and 40 days, respectively, are more concerned to the PUREX process. They along with their daughter isotopes, ^{106}Rh ($T_{1/2} = 30$ s) and ^{103}Rh ($T_{1/2} = 54$ min) contribute a large fraction of the total radioactivity of the process solutions [2] when short cooled fuel is processed. Although most of the ruthenium is essentially non-extractable into PUREX solvent, a small fraction shows a relatively high distribution ratio under process conditions. This small fraction is shown to consist of several species exhibiting various extraction and oxidation characteristics [3]. During reductive partitioning of plutonium from uranium using uranous nitrate, major fraction of ^{106}Ru is found to follow plutonium stream along with small concentration of uranium and niobium. To purify Pu from these contaminants either solvent extraction or ion-exchange method are employed. In ion-exchange method of purification, Dowex (1 × 4) anion exchanger in nitrate form is used. Pu forms hexa-nitrato complex $[Pu(NO_3)_6^{2-}]$ when the concentration of nitric acid in the feed is kept at ~7.0 M and is selectively sorbed on the column. After washing the column with ~7.0 M HNO_3, it is eluted using 0.5 M HNO_3. During loading operation, effluent streams from ion-exchange cycle of PUREX process is found to contain mCi/L-level activity of ^{106}Ru beside ^{95}Nb, uranium, and plutonium at an acidity of ~7 M HNO_3.

^{106}Ru finds many applications in nuclear medicine [4–6] especially in ophthalmology. It is used as an external irradiator [7–9] for the treatment of benign conditions of eye such as pterygia, traumatic corneal ulceration, corneal scars, vernal conjunctivitis, hemangioma of eyelid, vascularization of cornea and in preparation for a corneal transplant. Radionuclidic purity is the most important criteria for any radionuclide to be considered for nuclear medicine. Several studies have been carried out in the past to separate Ru activity using some of the PUREX process streams and in most of the attempts high-level liquid waste have been used as feed [10–12]. In most of these studies, ruthenium could be efficiently separated from other fission products by oxidation to RuO_4 with oxidants, e.g. $HClO_4$, $KMnO_4$, $K_2Cr_2O_7$, $Ce(SO_4)_2$, $(NH_4)S_2O_8$ and PbO_2. El-Absy et al. [13] separated ^{103}Ru from a ^{131}I-free fission product solution of 40% H_2SO_4 containing $KMnO_4$, by boiling for 40 min. Ruthenium was eliminated by electrovolatization from a 3 M HNO_3 solution of high-level waste, as RuO_4, in the presence of $AgNO_3$ at 60 ℃ [14]. Motoki et al. [15] separated ^{106}Ru from liquid waste onto zinc-electrodeposited charcoal column of 30–80% zinc concentration within the acidic region of pH < 3. Gandon et al. [16] separated ruthenium by a co-precipitation method in which a ferrocyanide complex ion was added to the solution to form a ruthenocyanide complex ion which was subsequently co-precipitated as copper ruthenoferrocyanide at pH 5 ± 1. Literature survey carried out revealed that no attempts have been made so far to recover Ru from secondary streams of PUREX process such as effluent stream of anion-exchange column used for tail end purification of plutonium. The effluent stream is generally found to contain Ru activity in mCi/L levels along with small impurities of U, Pu, and Nb. If short cooled spent fuel is reprocessed the stream may contain higher ruthenium activity. Considering large effluent volumes that are generated in purification cycle, it was found suitable for Ru recovery. Uranium, plutonium and a part of niobium from this feed can be depleted using solvents TBP/KSM-17 either in solvent extraction or in extraction chromatography mode, whereas entire Nb activity can sorbed on silica gel column which is well established in PUREX process [2]. The final effluent should contain Ru activity in purest form. Studies carried out in this paper describe about the separation and purification of ^{106}Ru from ion-exchange effluent streams of PUREX process using combinations of solvent extraction, extraction chromatography and ion exchange. Except solvent, KSM-17, all the chemicals used are compatible to the PUREX process generating minimum waste. Using actual effluent solution from Plant, a flow sheet is proposed and used for Ru activity in mCi levels.

2. Experimental

TBP obtained from M/s. Bharat Vijay Chemicals, India and *n*-dodecane (~93% C_{12}) supplied by M/s. Transware Chemia Handelsgeselschaft, Hamburg, Germany were used as such without any further purification. Solvent extraction grade, 2-ethylhexyl 2-ethylhexyl phosphonic acid (KSM-17) synthesized and purified at this research centre using the procedures reported in the literature was used [17,18]. During extraction studies, 30% TBP and 20% KSM-17 in *n*-dodecane are used.

2.1. Feed solution

The feed solutions used is ion-exchange effluent stream collected from a reprocessing plant that contained ^{106}Ru activity along with small contamination

Table 1
Composition of ion-exchange effluent stream

Analyte	Concentration
HNO_3	7.10 M
Plutonium	1.52 mg/L
Uranium	850 mg/L
^{106}Ru	110.50 mCi/L
^{95}Nb	0.29 mCi/L

of ^{95}Nb besides small concentration of uranium and plutonium at an acidity of ~7.0 M HNO_3. Known volumes from this effluent is diluted to the desired acidity and used as feed for studying the extraction/ sorption behavior of relevant radionuclide. The composition of a typical ion-exchange effluent stream is given in Table 1.

2.2. *KSM-XAD*

Extraction chromatographic sorbent, KSM-XAD, was prepared by impregnating indigenously synthesised KSM-17, on an inert polymeric support, XAD-4 supplied by Sigma Chemical Co. (mesh size 20–60) as per the method reported earlier [19]. Impregnation of XAD-4 with KSM-17 (in 1:1 w/w ratio of XAD-4 and KSM-17) was carried out in 100% methanol. The final material was washed with 20% methanol and air dried.

2.3. *Silica gel*

Silica gel (mesh size 100–200) procured from SISCO Research Laboratories Pvt. Limited Mumbai, India was used in all experiments. Prior to using, it was contacted with HNO_3 solution of respective concentration.

2.4. *Analyses*

Plutonium was analyzed by radiometry using ZnS(Ag) alpha scintillation counter. Uranium analysis was carried out by spectrophotometric method using Bromo-PADAP as chromogenic reagent [20]. Individual fission product gamma activities were assayed using a 62 cc HPGe detector coupled to a 4 K multichannel analyzer.

2.5. *Extraction and sorption experiments*

In batch extraction experiments, generally known volume of the ion-exchange effluent solution, adjusted to different acidities (~0.5–7.0 M), is contacted with organic solvents at a fixed organic to aqueous phase ratios for about 10 min which is found sufficient to attain equilibrium for both TBP-HNO_3 as well as KSM-17-HNO_3 solvent systems. The phases are separated and subjected to radiometric assay for desired radionuclide.

In batch studies involving extraction chromatography, known weight of sorbent (KSM-XAD, ~50 mg) is contacted with aqueous phase for about 10 min. After equilibration, the solution is assayed by radiometry/ spectrophotometry. Percentage extraction/retention is calculated using the activity/concentration values in feed and raffinate/effluent.

Sorption behavior of ^{106}Ru and ^{95}Nb is studied in batch mode using weighed quantity (~50 mg) of silica gel from aqueous phase containing different concentration of nitric acid varied in the range of 0.1–1.0 M. For adjusting the acidity in these experiments, a concentrated alkali solution is used.

Experiments are carried out in dynamic column mode using small glass columns packed with ~0.5 g of KSM-XAD for complete sorption of uranium/ plutonium and ~0.50 g silica gel for Nb sorption. The column volumes in both the cases are ~1.00 mL in each case. The acidity of the feed solution passed through KSM-XAD column is ~4.00 M, whereas it is adjusted to ~0.50 M HNO_3 prior to its passing onto the silica gel column. The flow rates during column runs are maintained at about 4–5 bed volume/h. Effluent samples are subjected to analysis using radiometry/ spectrophotometry.

Final Ru separation experiment under optimized conditions is carried out at ~100 mL scale wherein three steps are followed. In first step, major concentration of U, Pu, and Nb are removed from the ion-exchange effluent solution using solvent extraction employing 30% TBP in *n*-dodecane in batch mode at an organic to aqueous phase ratio of 2:1. Raffinate from this step is passed through extraction chromatographic column containing KSM-XAD where entire U, Pu, and significant Nb activity is sorbed leaving entire Ru in the effluent. Finally, the silica gel column is used for Nb sorption leaving effluent stream as pure Ru.

3. Results and discussion

Typical gamma spectrum of an ion-exchange effluent stream collected from PUREX Plant is shown in Fig. 1 and its composition is given in experimental section (Table 1). Considering availability of large volume and simplicity, it forms suitable source for ^{106}Ru separation and purification. Solvent extraction properties of TBP and KSM-17 from nitric acid

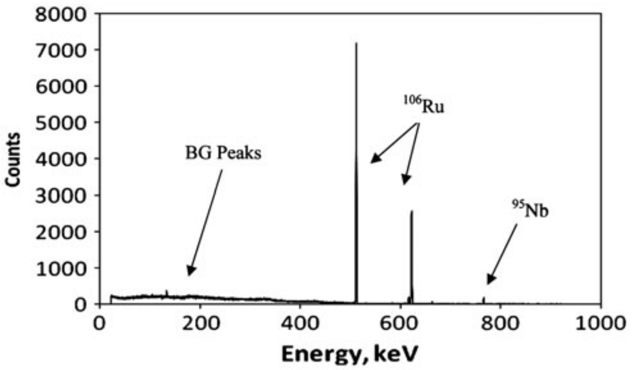

Fig. 1. Gamma spectra of an effluent sample from ion-exchange column.

medium and sorption properties of silica gel are adopted for the separation and purification of ^{106}Ru.

Tetravalent plutonium forms hexa nitrato complex [21] when present in nitric acid concentration of ~7.0 M. Since TBP forms extractable complexes with $UO_2(NO_3)_2$, $Pu(NO_3)_4$, and nitric acid, it was used for extracting U, Pu, and nitric acid from the effluent streams of ion exchange. Various aqueous to organic phase ratios were studied, and it was found that a phase ratio of 2:1 was most suitable to deplete U, Pu, and acidity without Ru loss in the feed. The results from solvent extraction studies using 30% TBP in *n*-dodecane in batch mode at an organic to aqueous phase ratio of 2:1 are given in Table 2.

It can be seen that >86% Pu and >98% uranium is removed from the feed solution in two contacts. Under these conditions, the acidity of the aqueous phase is lowered from 7.1 to 4.0 M. Extraction of Nb is about 80%, whereas that of Ru is <2% showing its preference in the aqueous phase. These studies clearly indicate that without affecting the Ru activity in the feed the concentration of uranium, plutonium, and niobium can be reduced. Introducing this solvent extraction step for the removal of small concentration of uranium and plutonium is amenable to adopt it within the process cycles as simple extraction and back extraction steps used in the process can be employed. The uranium, plutonium, and nitric acid bearing aqueous streams can be recycled in the process. Since Ru separation is intended for medical applications its purity is most important especially with respect to actinides such as Pu and U. Extraction chromatographic technique is relatively newer and better purification technique, and therefore it was thought for further purification. TBP-based extraction chromatography was not employed as it required longer column compared with KSM-17 column due to its relatively slower kinetics.

Table 2
Extraction behavior of U, Pu, Nb, Ru, and HNO_3 using 30% TBP

Analyte	Raffiante-1	Raffiante-2
HNO_3, M	5.50	4.02
Pu, mg/L	0.60	0.20
Uranium, mg/L	84	<10
^{106}Ru, mCi/L	101.10	98.88
^{95}Nb, mCi/L	0.16	0.06

Experimental parameters. Feed: Ion-exchange effluent solution. Organic to aqueous phase ratio: 2:1, (4 mL: 2 mL), contact time: 10 min.

KSM-17 is known for poor extraction of Ru and better extraction behavior of uranium and plutonium from nitric acid medium [22], it was selected for complete removal of uranium and plutonium from the raffinate phase. Extraction behavior of U, Pu, Ru, and Nb as checked in batch mode using 20% KSM-17 in *n*-dodecane at different concentration of nitric acid are shown in Table 3. These results show that the extraction of uranium and plutonium is above 95% in all the acidities in a single-batch contact. Nb extraction is in the range of 55–67% whereas the Ru extraction is very less (<2% in all the cases). These results show that the removal of uranium and plutonium from Ru containing feed solution can be achieved using KSM-17 based extraction retaining entire Ru in the aqueous phase. These results show similar trends as those observed earlier from our laboratory when studied individually from nitric acid medium [22].

Dealing with lower concentrations, extraction chromatography is more efficient separation technique in metal purification than liquid–liquid extraction technique. Therefore, KSM-17 based extraction chromatography was selected for Ru purification. Based on earlier experience [19,22] an extraction chromatographic material, KSM-XAD, was used for the removal of uranium and plutonium. Extraction behavior of all the relevant radionuclide as studied from feed solutions adjusted to different acidities in the range 2.0–7.1 M HNO_3 are presented in Table 4.

Above results clearly indicate that in a single-batch contact more than 98% removal of plutonium and uranium can be achieved without affecting Ru activity in the feed. Removal of ^{95}Nb is also found to be above 90% in all the cases.

Since radionuclidic purity is one of the most important criteria for any radionuclide to be considered for nuclear medicine, the final polishing of Ru product was thought to be carried out using silica gel which is commonly used for the sorption of Zr/Nb

Table 3
Extraction of ^{106}Ru, ^{95}Nb, and Pu using KSM-17 in *n*-dodecane

[HNO_3] (M)	Radionuclide	Activity		Extraction (%)
		Feed	Raffinate	
0.52	^{106}Ru, mCi/L	8.80	8.68	1.36
	^{95}Nb, mCi/L	0.03	0.01	67.00
	Pu, mg/L	0.12	0.005	95.83
	U, mg/L	67.73	<1	>98.52
2.05	^{106}Ru, mCi/L	33.98	33.50	1.41
	^{95}Nb, mCi/L	0.09	0.04	55.55
	Pu, mg/L	0.47	0.01	97.87
	U, mg/L	216	2.09	99.03
4.06	^{106}Ru, mCi/L	62.87	61.80	1.70
	^{95}Nb, mCi/L	0.16	0.07	56.25
	Pu, mg/L	0.86	0.02	97.67
	U, mg/L	484	4.50	99.07
7.10	^{106}Ru, mCi/L	101.10	99.20	1.88
	^{95}Nb, mCi/L	0.29	0.12	58.62
	Pu, mg/L	1.52	0.03	98.03
	U, mg/L	850	8.8	98.96

Experimental conditions. Aqueous feed: Ion-exchange effluent sample diluted to different concentration of HNO_3.
Organic: 20% KSM-17 in *n*-dodecane, organic to aqueous phase ratio: 1:1, (2 mL each),
contact time: 10 min.

Table 4
Sorption behavior of ^{106}Ru, ^{95}Nb, Pu, and U using KSM-XAD

[HNO_3]	Radionuclide	Feed (mCi/L)	Supernatant (mCi/L)	Retention in effluent phase (%)
2.05	^{106}Ru, mCi/L	33.98	33.05	97.26
	^{95}Nb, mCi/L	0.09	0.007	7.78
	Pu, mg/L	0.47	0.009	1.91
	U, mg/L	216	3.48	1.61
3.04	^{106}Ru, mCi/L	46.62	45.98	98.63
	^{95}Nb, mCi/L	0.12	0.008	6.67
	Pu, mg/L	0.64	0.011	1.72
	U, mg/L	358	5.46	1.53
4.06	^{106}Ru, mCi/L	62.87	62.47	99.36
	^{95}Nb, mCi/L	0.16	0.01	6.25
	Pu, mg/L	0.86	0.016	1.86
	U, mg/L	484	7.81	1.61
7.10	^{106}Ru, mCi/L	101.10	99.12	98.04
	^{95}Nb, mCi/L	0.29	0.02	6.70
	Pu, mg/L	1.52	0.01	0.66
	U, mg/L	850	10.70	1.26

Experimental conditions. Aqueous feed: Ion-exchange effluent sample diluted to different concentration of HNO_3.
Weight of sorbent, KSM-XAD: ~50 mg, contact time: 10 min.

Table 5
Sorption behavior of ^{106}Ru and ^{95}Nb using Silica gel

[HNO_3]	Radionuclide	Feed (mCi/L)	Effluent (mCi/L)	Sorption (%)
0.10	^{106}Ru	2.38	2.35	1.26
	^{95}Nb	0.007	<0.001	100
0.50	^{106}Ru	5.40	5.36	0.74
	^{95}Nb	0.001	<0.001	100
1.00	^{106}Ru	9.58	9.57	0.10
	^{95}Nb	0.001	<0.001	100

Experimental conditions. Aqueous feed: Ion-exchange effluent sample diluted to different conc. of HNO_3.
Weight of silica gel: ~50 mg, contact time: 10 min.

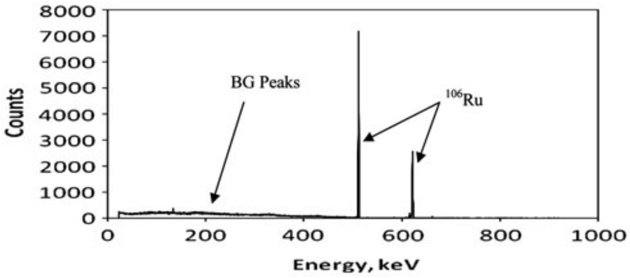

Fig. 2. Gamma spectrum of Purified ^{106}Ru product.

activity in PUREX process for uranium polishing [2]. Prior to its use for the removal of Nb, sorption behavior of Ru and Nb was studied in batch mode using silica gel from lower acidity ranges. Results from studies carried out in the acidity range of 0.10–1.00 M HNO_3 are shown in Table 5.

Quantitative removal of Nb is observed when feed solutions are contacted with small weight (~50 mg) of silica gel. Under these conditions, no Ru loss is observed. These studies clearly suggest that silica gel can be used for final Ru purification from the effluent stream of extraction chromatographic column KSM-XAD after lowering its acidity to ~0.50 M.

Based on the above results, Ru separation and purification experiment was carried out in three steps using 100 mL ion-exchange effluent collected from Plant. In first step, major uranium, plutonium, and niobium were depleted in solvent extraction mode using 30% TBP in *n*-dodecane without affecting activity of Ru in the feed. The raffinate from second TBP contact was used as feed for extraction chromatographic column containing 0.50 g KSM-XAD. Complete removal of U, Pu, and major removal of Nb was observed. The effluent after adjusting to ~0.50 M was passed through a column containing 0.50 g silica gel for Nb removal. The effluent from silica gel column had Ru activity of 92.00 mCi/L. This gives Ru recovery >90%. The gamma spectrum of pure Ru is shown in Fig. 2. The Ru product solution was subjected to radiometry for Pu alpha activity and was found to be absent. The minimum detection limit for gross alpha in our counting system was <0.2 µg/L. Even after 24 h counting, no counts above background were observed. Uranium in the product was subjected to ICP-OES and could not be detected. Minimum detection limit for uranium by the technique was 20 ppb.

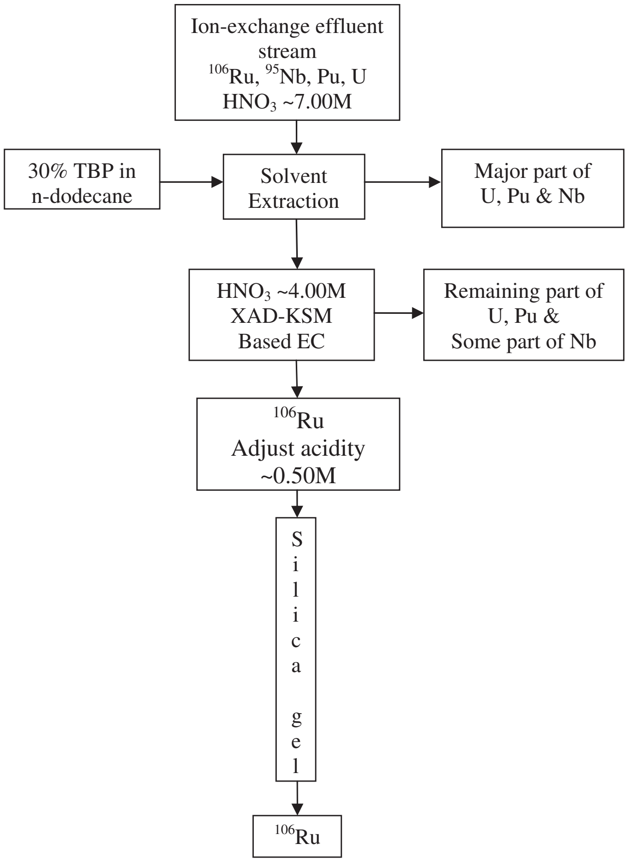

Fig. 3. Flow sheet for the separation and purification of ^{106}Ru.

No other radionuclides were found to be present in the final product. Based on the above studies, a flow scheme for separation and purification of Ru has been proposed (Fig. 3) which can be adopted for large scale purification of Ru.

4. Conclusions

These results clearly illustrate that the combinations of solvent extraction based on TBP, extraction chromatography based on KSM-17 and ion-exchange based on silica gel can be used for the separation and purification of ^{106}Ru from ion-exchange effluent streams of PUREX process. The recovered Ru activity can be used in nuclear medicine. The method is amenable to scale up.

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