Fission Product Decontamination Factors for Plutonium Separated by PUREX from Low-Burnup, Fast-Neutron Irradiated Depleted UO₂

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Abstract

Experimental investigations to determine fission product separation from actinides (U and Pu) while employing the Plutonium Uranium Recovery by Extraction (PUREX) process to purify plutonium produced in a fast neutron irradiated depleted uranium dioxide (DUO₂) target were conducted. The sample was a DUO₂ pellet (0.256 wt% ²³⁵U) irradiated to a low-burnup (4.43 ± 0.31 GWd/tHM) that was PUREX processed 538 days after neutron irradiation. Decontamination factors (DF) for the elements U, Mo, Ru, Ce, Sm, Sr, Pm, Eu, Nd, Pd, and Cd were measured with mass spectroscopy in two experiments using 30 vol.% tri-n-butyl phosphate (TBP) in a kerosene diluent. The first experiment characterized Pu DFs for a single stage extraction and back-extraction, while the second experiment had multiple stages with the goal of achieving greater Pu recovery. The benchtop scale PUREX process had overall Pu recoveries of ($83.4 \pm 9.5\%$) and ($99.7 \pm 4.2\%$) for the first and second experiments, respectively.

Keywords: PUREX; Decontamination Factor; Irradiated Depleted Uranium

1. Introduction

Background. In a recent publication, our group suggested that investigation of trace contaminants in Pu recovered using the Plutonium Uranium Recovery by

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Extraction (PUREX) process could give indications of material origins (Chirayath
5 2015). This computational study analyzed Pu and trace level contaminants of
decontaminated fuel irradiated in fast breeder and heavy water thermal reactors
due to their relative ease of producing weapons-grade Pu. A conclusion of the
paper was that a distinction can be made between decontaminated Pu from a fast
and thermal system based on fission product (FP) to Pu concentration ratios. In
10 practice, however, if a sample of Pu were intercepted through conventional
investigative techniques, it would likely have already been processed via PUREX.
Therefore, data on decontamination factors (DFs, a measure of contaminant
purification), are needed for each element individually. Due to a lack of literature
on DFs and distribution coefficients (DCs, the ratio of concentrations between
15 organic and aqueous phases) for the elements noted in our previous paper (Cs, Sb,
Eu, Rb, Sr, Nd, Pm, and Sm), the study for this paper was conducted.

Descriptions of various PUREX processes are provided in many sources with
explanations of chemistry including flow sheets and decontamination factors
(Lanham 1950, Arker 1954, Darby 1954, Irish 1957, Stoller 1961, Benedict 1982).
20 These sources generally report overall beta or gamma DFs of up to 10^8 with Pu
recoveries of 99.7% for industrial scale reprocessing facilities. While DCs for the
various process steps of PUREX have been previously reported, details about
elemental DFs for PUREX cycles have been largely limited to the major activity
contributors, such as ^{106}Ru and ^{95}Zr (Stoller 1961). A compilation of distribution
25 data for PUREX extraction processes provides data for U, Th, and Pu in a variety
of concentrations (Siddall 1957). DCs for Zr, rare earth metals, Pu, and Th are also

available (Alcock 1957, Best 1957, Hesford 1957, Scargill 1957, Alcock 1958, Best 1959, Hesford 1959). Additionally, Ga has been studied for separation (Collins 2000) because it is a common contaminant in weapons-grade Pu.

30 Although a DC, coupled with process information, can be used to calculate a reasonable estimate of DF (Colburn 1939, Sherwood 1952, Long 1967, Perry 2008), variability of DCs under different system conditions give rise to uncertainty in calculated results. For example, DCs between tri-n-butyl phosphate (TBP) and nitric acid (HNO_3) have been reported for U, Pu, Zr, Nb, Ru, and the rare earth
35 elements, but vary with HNO_3 concentration and U saturation in TBP by up to several orders of magnitude (Sherwood 1952, Stoller 1961). These sources also derive mathematical correlations between DC and DF, but experimental PUREX DFs for a large number of individual elements were not provided.

 In the current work, 12.9 mg of depleted uranium dioxide (DUO_2) was
40 irradiated in a pseudo-fast neutron spectrum at the High Flux Isotope Reactor (HFIR) at Oak Ridge National Laboratory [described in detail in (Swinney 2014)]. The DUO_2 pellet, containing FPs and Pu, was dissolved in HNO_3 and subjected to two different PUREX experiments for DF characterization and Pu product recovery. Aqueous solutions were analyzed at each step with Inductively Coupled
45 Plasma-Mass Spectrometry (ICP-MS). The experimental work used benchtop scale methods to isolate a large fraction of Pu, measure DFs for FPs, and measure overall gamma DFs as part of a larger project to develop forensic radioanalytical capabilities at Texas A&M University, and first results are reported here. In order to properly utilize the information presented for nuclear forensics, additional

50 experiments are being conducted which will include different solution
concentrations and levels of heavy metal saturation, as well as providing a more
realistic simulation of an actual reprocessing plant. That additional information will
provide more avenues to determine purification process steps, and will be the
subject of a future publication.

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Terminology. A DC is defined as the concentration ratio between the organic (org)
and aqueous (aq) phases as shown in Equation 1, and describes the equilibrium
distribution of a species in the system during PUREX separation processes
(Benedict 1982):

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$$DC = \frac{c_{org}}{c_{aq}} \quad (1)$$

where c is the concentration of the specific species in the indicated phase. DCs are
specific to an element and vary widely with the concentration and temperature of
the solvents. They are also affected by saturation of U and Pu in the system and, in
some cases, time since solution preparation (Stoller 1961, Simpson 2010). For

65 PUREX, the fraction of mass, f_{org} , deposited in the organic (TBP) phase for a
single element, assuming a volume ratio between the aqueous and organic phases,
 V_R , is given by Equation 2.

$$f_{org} = (1 + DC^{-1}V_R^{-1})^{-1} \quad (2)$$

This is termed “extraction”, where a desired solute is extracted out of an aqueous solution through contact with an immiscible liquid phase. Ideally, a large fraction of desired solutes are extracted into the organic phase, while a small fraction of undesired solutes are extracted. Conversely, “back-extraction” is the process by which a desired solute is extracted out of an organic solution through contact with an immiscible aqueous solution. Both extraction and back-extraction will also be termed “stage”, where mass transfer occurs between two sufficiently mixed immiscible liquid phases. “Scrubbing” is the transfer of a large fraction of impurities to a second phase while having a minimal transfer of desired constituent.

After several cycles of Pu extraction/scrubbing/back-extraction are complete, the measured effectiveness of a PUREX cycle is described by the DF (which is ideally determined by DCs) and measures the effectiveness with which a contaminant, j , is removed from a product. The product of interest in this work is Pu, and the DF is defined by Equation 3.

$$DF_j = \frac{\frac{c_j}{c_{Pu}}|_{initial}}{\frac{c_j}{c_{Pu}}|_{final}} \quad (3)$$

85 Initial and final refer to the values before and after purification, respectively.
DFs are also characteristic of different process cycles, and may have larger values
($> 10^7$) for industrial scale PUREX compared to the benchtop scale version
presented here because of an additional scrubbing step as well as a larger number
of stages (Stoller 1961, Benedict 1982). Industrial processes often report either an
90 overall DF value, or a DF value for a single isotope.

2. Experiment

A commercially acquired pellet containing 12.9 ± 0.1 mg of DUO_2 was
irradiated over the course of three months with two shutdown periods in the
HFIR. The final burnup was 4.43 ± 0.31 GWd/tHM (Swinney 2015). The
95 irradiation produced 0.196 ± 0.013 mg of total Pu as measured by ICP-MS, with
the assumption that, due to the low burnup, all of the 241 mass was ^{241}Pu . After
the short-lived radioisotopes had opportunity to decay, the irradiated pellet was
shipped to Texas A&M University.

The kerosene and TBP ($>99\%$) used for these experiments were acquired
100 from Alfa Aesar (Haverhill, Massachusetts, USA), NaNO_2 was acquired from
Aldrich Chemistry (St. Louis, Missouri, USA), 69% nitric acid was acquired from
Mallinckrodt Chemicals (St. Louis, Missouri, USA), and iron sulfamate (40.26%)
was acquired from Strem Chemicals (Newburyport, Massachusetts, USA).
Chemicals were used as received without further purification. Degradation of TBP
105 due to radiolysis was not a significant concern because the activities were relatively
low, most of the FPs stayed in the aqueous phase, and the organic phase was only
in contact with the aqueous for a few minutes at a time.

The pellet, both prior to dissolution and after, was counted with a Canberra HPGe detector model number CC4018 which was connected to a Canberra Lynx multichannel analyzer (Zakrzewski 2013, Canberra 2013a). Canberra's software package GENIE-2000 version 3.2.1 (Canberra 2013b) was used to collect spectra while the samples were inside a lead tomb. The same detector was used to count the various process solutions. Inductively coupled plasma mass spectrometry (ICP-MS) was also conducted for aqueous samples using a PerkinElmer NexION 300X quadrupole ICP-MS (PerkinElmer 2009).

Samples of the pellet were prepared as described below. The sample was added to a round-bottom flask and 5.0 ml of 8 M HNO₃ was added to the flask, which was heated to 50 °C with constant 100 rpm stirring for 2 h. This solution will be referred to as the "dissolution solution". The flask was connected to a cold trap with the help of Schlenk line. The FP gases such as H₂, CO₂, Kr, Br₂, I₂ and N₂O were captured in the cold trap using molecular sieves that were chilled by liquid nitrogen. In order to reduce the amount of activity per sample, 500 µl from the dissolution solution was diluted to 5.0 ml and the concentration was changed to 4 M HNO₃. From this solution, referred to as the "stock solution", 0.5 ml aliquots, containing ~1% of the pellet, were used in benchtop scale PUREX experiments described in the two subsections below. The total activity concentration of the stock solution was approximately 80 µCi/ml.

The "working" solution was prepared by transferring a 500 µl aliquot of stock solution and 0.5 mg of NaNO₂ to a 15 ml centrifuge tube. The tube was subsequently stirred and covered to retain the resulting NO₂ gas. The solution was

left overnight to ensure complete oxidation of Pu(III) to Pu(IV). Pu exists in several oxidation states at room temperature, and interconversion can be rapid. As a result, it is possible for Pu(IV) to convert to other oxidation states after using NaNO_2 . Regardless, the transfer of Pu(IV) to the organic phase reduces the concentration of Pu(IV) in the aqueous phase. In order to restore the equilibrium, other oxidation states will convert to Pu(IV), which can then be further extracted. The end result is preferential formation of Pu(IV) in the aqueous phase. The high extraction yields discussed below suggest that production of Pu(IV) was dominant as expected. During extraction and back-extraction, both experiments had the aqueous and organic phases mixed on a vortex mixer for 15 min at 1500 rpm, after which the two phases were allowed to settle and separate. The phases were then physically separated into two different vials via pipetting.

Each extraction and back-extraction mixed organic and aqueous solutions with unequal volumes. The solution being added always contained an extra 200 μl to reduce the chance of accidentally pipetting both phases. For example, if TBP were being added to the working solution described above, 700 μl would be added initially and 500 μl removed. This excess volume will be referred to as hold-up volume in similar fashion to the differential extraction experiment described by Long (Long 1967). All results presented below have been corrected to those that would be expected for equal volumes between aqueous and organic solutions during both extraction and back-extraction. Results are also corrected to zero hold-up volume.

Experiment 1. The purpose of the first experiment was to quantify product recovery and DF values for a single stage extraction and back-extraction of Pu. U(VI) and Pu(IV) were extracted and decontaminated by contacting the prepared working solution with a solution of 30 vol.% TBP with a kerosene diluent. After mixing and separation of the two phases, Pu(IV) was reduced to Pu(III) and back-extracted by contacting the physically separated TBP solution with a solution of 0.024 M ferrous [Fe(II)] sulfamate in 0.75 M HNO₃. The influence of Fe²⁺ concentration on FP DFs was not studied but is expected to be small because its concentration is already multiple orders of magnitude greater than the FPs, and any extraction enhancement would be expected to be observed even at low concentrations. Additionally, the extraction of FPs would depend on their ability to form neutral species, but this does not happen with Fe²⁺. All aqueous solutions were analyzed with ICP-MS.

Experiment 2. The purpose of the second experiment was to recover a large fraction of Pu. Utilizing the results from the first experiment, it was determined that contacting the prepared working solution four times with TBP would extract over 90% of the Pu. Therefore, this experiment had four extraction stages with the prepared working solution. The four TBP solutions were then collected into a single vial, and contacted three times with 0.024 M ferrous sulfamate in a 4 M HNO₃ solution. These three contacts were then collected into a single vial.

In order to ensure minimal U back-extraction, the HNO₃ concentration for this experiment was increased to 4 M because higher concentrations reduce the degree to which U is back-extracted (Stoller 1961). Three contacts of the ferrous

sulfamate solution ensured nearly complete back-extraction of Pu, while the higher nitric acid concentration minimized back-extraction of U. The same solutions as described in the first experiment were analyzed with ICP-MS.

The four-contact extraction, three-contact back-extraction process described above for the second experiment will be referred to as a cycle. The combined ferrous sulfamate solution containing back-extracted Pu underwent a second cycle to verify the repeatability of the process and for comparison with the first cycle. The ending solution of the first cycle Pu was “reset” with the addition of 0.5 mg of NaNO₂ to convert all Pu(III) to Pu(IV).

3. Results

The U and Pu recovery after back-extraction for the first and second experiments are shown in Table 1. The DCs for U and Pu were determined by analyzing the working solution both before and after extraction for experiment 1. These were determined to be 26.7 ± 2.9 and 11.6 ± 1.1 , for U and Pu respectively. Experiment 2 successfully recovered approximately 93% of the original Pu with less than 1% of the original U remaining.

Table 1 Recoveries of U and Pu for the different experiments.

	Pu Recovery	U Recovery
Experiment 1	$(83.4 \pm 9.5)\%$	$(11.2 \pm 1.3)\%$
Experiment 2 Cycle 1	$(99.7 \pm 4.2)\%$	$(6.8 \pm 0.3)\%$
Experiment 2 Cycle 2	$(93.0 \pm 4.6)\%$	$(6.6 \pm 0.3)\%$
Overall Experiment 2	$(92.7 \pm 6.0)\%$	$(0.45 \pm 0.03)\%$

In experiment 1, approximately 10% of the U in the TBP phase was back-extracted with a single contact of a solution of 0.024 M iron sulfamate in 0.75 M HNO₃. Experiment 2 had much smaller U back-extraction due to the higher molar

concentration of HNO_3 in the back-extraction solution (Benedict 1982). This can potentially led to lower Pu recovery because the stability of ferrous ion decreases as the concentration of nitric acid increases (Stoller 1961). The half-life of the ferrous ion in the back-extraction solution of experiment 2 was estimated to be approximately 4 d (Stoller 1961). The effect is shown in the differences in Pu recovery between cycle 1 and cycle 2 of experiment 2, where cycle 2 back-extraction utilized a Fe(II) solution that was 1 d older. This effect was expected to be small for the FPs because of their strong preference for the aqueous phase (low DCs).

The DF calculations utilized concentration ratios between contaminants that were normalized to the Pu concentration per Equation 3, with the initial solution being the working solution and the final solution being the back-extracted Pu. Both experiment 1 and experiment 2 first cycle DF values are shown in Table 2. FPs of interest were selected based on our previous publication (Chirayath 2015) with additions of Mo, Ru, Pd, Cd, and Ce. ^{237}Np and ^{241}Am were not considered due to their low forensic value in this case. ^{99}Tc was also not considered due to its low forensic value and difficulty of assaying the low-intensity photons produced by this nuclide. Cycle 2 of experiment 2 is not shown because the data were below background for these samples, which was generally less than 1 ppb. The low DF values reported were expected due to the reasons described above: the extraction and back-extraction were performed without intermittent scrubbing, and the number of stages was low.

Table 2 Decontamination factors for single and multiple-contact PUREX.

Element	Exp. 1	Error	Exp. 2 Cycle 1	Error	Isotopes
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(Z)					Used
Rb (37)	39.0	5.9	11.8	0.8	⁸⁵ Rb
Sr (38)	283	43	84.6	5.9	⁹⁰ Sr
Mo (42)	5.7	0.8	1.9	0.2	^{97,98,100} Mo
Ru (44)	59.2	6.4	16.6	2.5	^{101,102,104} Ru
Pd (46)	65	14	8.9	1.2	¹¹⁰ Pd
Cd (48)	74	17	22.1	2.5	¹¹² Cd
Cs (55)	177	28	52.9	3.9	¹³³ Cs
Ce (58)	43	16	11.5	4.9	^{140,142} Ce
Nd (60)	19.2	2.1	5.9	0.4	¹⁴³ Nd
Pm (61)	12.8	1.9	3.9	0.3	¹⁴⁷ Pm
Sm (62)	11.5	1.5	3.6	0.3	¹⁵¹ Sm
Eu (63)	10.0	1.4	3.6	0.3	¹⁵⁴ Eu
U (92)	7.4	1.2	14.7	0.9	²³⁸ U

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The common trend is that DF decreases from experiment 1 to experiment 2.

The major exception is U, which has a higher DF value. This is expected due to the change in HNO₃ concentration in the iron sulfamate solution, where higher concentrations of HNO₃ reduce the degree of U back-extraction, as mentioned

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above. The rest of the elements have lower DF values because of the four extraction and three back-extraction steps in cycle 1 of experiment 2 compared to one extraction and one back extraction in experiment 1. This is illustrated in Figure 1, where theoretical DFs as a function of extraction step and volume ratio for a product with a DC of 10 and a contaminant with a DC of 0.1 are shown. Higher

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numbers of contacts decrease DF because less and less product is extracted with each step, while the amount of contaminant extracted is approximately unchanged. Experiment 2 DF values decrease relative to experiment 1 DF values because of this.

235 Figure 1 DFs as a function of volume ratios for the first to fourth contact in TBP.

4. Conclusions

240 Two PUREX experiments were conducted. The first experiment determined DC values for Pu, U and several FPs. The second experiment utilized these values to recover over 92% of the Pu while leaving less than 1% of the U. Overall Pu and U recoveries for experiment 1 were $(83.4 \pm 9.5)\%$ and $(11.2 \pm 1.3)\%$, respectively, while the same values for experiment 2 cycle 1 were $(99.7 \pm 4.2)\%$ and $(6.8 \pm$
 245 $0.3)\%$, respectively. DF values were measured for 12 FP elements. DF values were lower than those typically found in industrial scale PUREX plants due to multiple

extraction and back-extraction steps without an intermittent scrubbing step. This work provides critical DF data that will be needed for potential nuclear forensic investigations of interdicted Pu. Such a sample might have already been purified, so accurate DF data will allow for estimation of the original amounts of forensic isotopes. Regardless, additional research is needed to study the effects of additional scrubbing and stripping steps on individual DFs.

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