**Ph.D. Dissertation Proposal: Paul Mendoza**

**Dissertation Title:** Experimental Characterization of Pu Separation by PUREX Process on a Low-Burnup, Pseudo-Fast-Neutron Irradiated DUO2 for Product Decontamination Factors and Nuclear Forensics

1. **Introduction**

United States (U.S.) forensic capabilities have been demonstrated in scenarios of weapons testing, post-detonation exercises, and interdiction. Although it is hoped that these capabilities are never needed, their existence is important for national security. According to a report from the committee on nuclear forensics released in 2010, “the timeline for post-detonation analysis and evaluation is longer than desired”, and further, in context of our substantial forensic capabilities, this report notes that these capabilities are “fragile, under resourced, and, in some respects, deteriorating”1 The proposed approach for meeting the committee’s concerns would need to be addressed on a national scale. the Islamic State of Iraq and Syria, nuclear proliferation concerns are receiving utmost importance and attention. A nonproliferation focal point is the control and accounting of weapons usable special nuclear material (SNM), which has been the responsibility of the International Atomic Energy Agency (IAEA) with the goal of promoting the safe and peaceful use of atomic energy and nuclear technologies2. The IAEA’s efforts to meet this goal, backed by a U.S. “atoms for peace”3 program and the treaty on the non-proliferation of nuclear weapons (NPT)4, have hindered the development of weapons to a large extent5. However, efforts beyond nuclear material safeguards are necessary due to “the awful arithmetic of the atomic bomb”6. An effort outside of nuclear material safeguards for pre-detonation SNM, is to develop the nuclear forensics capability with a source attribution objective in the event of SNM interdiction.

SNM origin attribution, in this context, applies to deducing history of nuclear material, including its production process. The importance and time sensitivity of which varies widely in different circumstances. This problem is difficult to solve though, because of its inverse nature. For example, if purified plutonium were interdicted at a State border, information from a wide variety of sources will be used to arrive at a likely hypothesis for where the material came from. This hypothesis is an informed, studied, and tested conclusion, but is only one possible explanation for the history of the material.

The point is not to say the exercise is meaningless, but difficult and multi-layered. Multi-layered because there are many different sources of information and a large number of possible hypotheses. This study proposes a nuclear forensics characterization procedure for separated plutonium material, aiding an informed hypothesis for source attribution.

Previously, computational studies have indicated that analysis of contaminants in Plutonium Uranium Recovery by Extraction (PUREX) processed plutonium could give indications of material origins7. Unlike enrichment of 235U, 239Pu “enrichment” involves neutron irradiation, which introduces radioactive fission products and hence, purification by some means. Purification by PUREX is most common for low-enriched uranium fuel8 - important because 238U converts to 239Pu upon neutron capture and subsequent β- decay. Material attribution for separated plutonium is complicated because the separation process has a substantial amount of variability stemming from the changes in elemental decontamination factors (DFs, a measure of purification) that could be obtained in a PUREX process.

The measured effectiveness of a PUREX cycle is described by the DF, and measures the effectiveness with which the concentration,, of a contaminant, , is removed from a product. The product of interest in this work is plutonium, and the DF is defined by Equation 1.

Equation Decontamination Factor

|  |  |  |
| --- | --- | --- |
|  |  | (1) |

The problem of attribution for unpurified material has been previously studied7, 9, 10; ideally, if elemental DFs for interdicted plutonium were determined, then these previous methodologies could be used to narrow material origins by applying these correction factors, assuming DFs for different isotopes of the same element are equal. This is visually represented in Figure 1, where the initial plutonium impurity levels are estimated through DFs with attribution analysis ensuing. The asterisks in the figure indicate the main areas of study for this project.

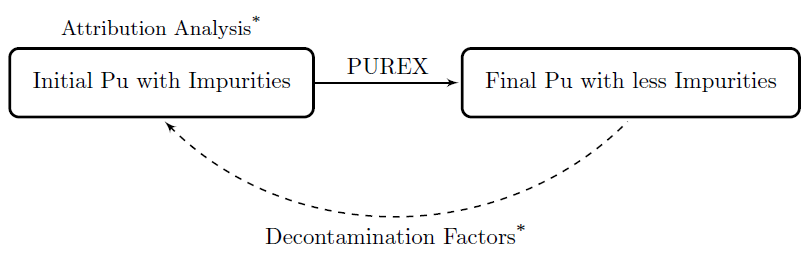


Figure Proposed research areas for irradiated fuel.

This work will start with the assumption that the specifics of the PUREX process used for plutonium separation are known, so that DFs are applied appropriately. In real world applications, this information would not be readily available, and this same analysis would have to ensue with best estimate PUREX processes and experimentally measured distribution coefficients (DCs, the ratio of concentrations between organic and aqueous phases).

1. **Objectives**

The objective of this research is to determine DFs and DCs for a benchtop scale PUREX process for important attribution elements noted in previous research (Cs, Eu, Rb, Sr, Nd, Pm, Sm, U, and Pu)7. Both DCs and DFs will be determined for this process so that the two parameters will be experimentally connected to the specific PUREX process utilized. This establishment is important because the nuances for a particular PUREX process vary widely and result in different DFs from process to process11. Mathematically, DC values, coupled with process information, can be used to calculate a reasonable estimate of a DF12-15, leading to original concentrations of impurities in purified plutonium, which in turn allows for use of more traditional forensic analysis.

The objective will be pursued by performing mass, gamma, and alpha spectroscopies on solutions/samples obtained from various PUREX process steps and analyzing the results. The PUREX process followed for this study will have four extraction and three back extraction steps for obtaining purified plutonium. These steps are chosen because they are the main steps for the separation of heavy metal from fission products, and uranium from plutonium13. Each step in the process will be analyzed for DC values, and the overall process will be analyzed so that the DFs can be determined through initial and final solutions and through using individual DCs concurrently.

Further, several forensic analyses will be pursued for the case of plutonium. The analyses performed will include fuel burnup which produced plutonium, neutron scalar flux (assuming constant), initial enrichment, fuel age, and energy dependence of the neutron scalar flux. These attributes are important for source attribution. These analyses will be first undertaken for unprocessed plutonium material. Then the experimentally and mathematically determined DFs for the PUREX process performed will be applied to determine the attributes of separated plutonium. The second step emulates a scenario where useful information is derived from experimentally processed plutonium using PUREX. These two determinations will then be compared to calculation results from the general purpose Monte Carlo N-Particle (MCNP) radiation transport code modeling the reactor fuel burnup operations which produced the plutonium in question.

The above points are summarized in the list below:

1. Complete four extraction three back-extraction PUREX process in triplicate
   1. Collect DCs for each step
   2. Determine DFs
      1. from DCs and
      2. the initial and final product
2. Determine attribution indicators of burnup, neutron scalar flux, initial enrichment, fuel age, and the energy dependence of the neutron scalar flux with data from:
   1. Unprocessed plutonium
   2. PUREX processed plutonium
   3. MCNP based reactor modeling and fuel burnup analysis
3. **Present Status of the Question**

There is a reasonably large body of literature on the work done in the area of using isotopic compositions in order to determine or verify the origin of plutonium and also the information on the process used to separate plutonium from reactor fuel. These works are varied in implementation and scope.

Isotopic compositions and ratios in spent reactor fuel have been used for burnup verification. One study utilized measurement of isotopic ratios of stable noble fission gases during reprocessing in order to verify burnup16. They used data analysis methods to determine specific fuel parameters and then compared them with the measured values of stable noble gases. Fuel burnup is important because low burned uranium fuel (<3000 MWD/tU) yields weapons-useable plutonium material. If a fuel assembly were burned to a lower level than reported, then further investigation is necessary to either alleviate or confirm suspicions of nefarious activities.

Others have analyzed later stages of processing used nuclear fuel. Given the spent fuel from a reactor that is used for a radiological dispersal device (RDD), a study sought to determine the burnup, enrichment, and age for the fuel 9. These combined data would then be subsequently used for reactor attribution purposes. This study is similar to our scenario in that it looks at FP in the matrix of the fuel itself and deduces reactor type and information about the history of the fuel, but this method does not consider any chemical processing.

Analysis of plutonium isotopes alone have been suggested to help identify reactor type at the same processing step as for the proposed project 10. The reactors considered were of the fast and thermal neutron spectrum type. This study concluded that while it is possible to identify with a high level of confidence differences in plutonium composition between fast and thermal reactors, it is difficult to make this same distinction in “dedicated production reactors fueled with natural uranium.” This should be contrasted with our study with depleted uranium in a fast neutron spectrum and specifically for low-burnup neutron irradiations.

PUREX co-processing, where uranium and plutonium are not separated during reprocessing, of spent LWR fuels has also been studied 11. Several decontamination factors and production yields for uranium and plutonium were determined. The specific goal of this paper was to demonstrate that uranium and plutonium could be co-extracted for reprocessing. This study was also limited in determining decontamination factors for a wide array of isotopes, which may not be as important for reprocessing as opposed to nuclear forensic analysis. This is because trace isotopes could give an indication of the origin of plutonium.

Descriptions of various PUREX processes are provided in many sources with explanation of chemistry including flow sheets and DFs8, 17-21. These sources generally report overall beta or gamma radiation DFs of up to 108 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 106Ru and 95Zr21. A compilation of distribution data for PUREX extraction processes provides data for U, Th, and Pu in a variety of concentrations22. DCs for Zr, rare earth metals, Pu, and Th are also available 23-28. Additionally, Ga has been studied for separation29 because it is a common contaminant in weapons-grade plutonium.

This work is unique in that elemental DFs will be determined and used on a PUREX processed plutonium with an attribution analysis ensuing.

1. **Procedure**

The objectives for this project were split into two distinct parts, chemical reprocessing, and forensic analysis. The procedure will also be broken up along these same lines. Although both parts require some element of experimental procedure, the first requires more and will be discussed in greater detail below under the heading of experimental, while the other will be more analytical in nature.

***Analytical Procedure****:* This experiment will utilize an irradiated 12.9 ± 0.1 mg of DUO2 irradiated to a lower burnup in a particular fast neutron environment. The specifics of this information is known to some, but not to the author, and working through the procedures for the forensic analysis will give some grounding for the history of the sample. Working from this “blind” perspective will help by removing bias towards particular results or conclusions.

The analytical attribution methodology will follow a series of calculations as depicted in Figure 2. Each individual calculation will be discussed briefly, but the flow chart depicts the order in which attributes will be determined. The following calculations will assume a point wise irradiation. Burnup is solved for first because it has no dependency on the neutron scalar flux or its energy dependence, whereas the rest of the calculations depend on one or both. In order to solve for the rest of the attributes, the calculations will be performed over a wide range of potential normalized energy-dependent neutron scalar fluxes and the neutron energy spectrum that produces the most consistent fuel age indicators will be deemed the “correct” answer.

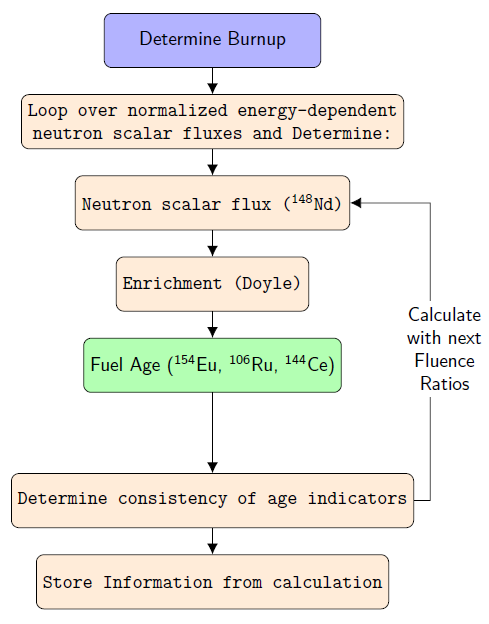


Figure Flow chart for analytical calculations.

Burnup is an operational parameter for the amount of energy produced from a mass of fissionable material, and is calculated with Equation 2, where refers to the mass of heavy metal in the irradiated fuel in metric tons. This is useful for reactor designers and operators, whose goal is to produce electricity efficiently. From an operational standpoint this term is relatively straightforward to determine. Knowing the amount of fuel put into a system, the amount of energy produced can be calculated with a specific heat calculation on the working fluid in the reactor system and the operation time. To precisely determine this parameter from a reactor physics point of view, the total number fissions from each fissionable isotope should be known along with respective recoverable energy. This information is more difficult to acquire because the concentrations of fissionable material in a fissioning system (nuclear reactor) is constantly changing. Burnup will thus be determined with a fission product that builds into the fuel matrix linearly as a function of burnup such as 137Cs or 148Nd30.

Equation Burnup equation

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| --- | --- | --- |
|  |  | (2) |

The rest of the indicators are dependent on the energy-dependent neutron scalar flux the uranium sample was irradiated in. An issue with this dependency is the great complexity surrounding the shape of the energy-dependent neutron scalar flux. It varies with temperature, geometry, fuel composition, and density of the materials in the system. It also has a spatial dependency, and derives itself from the cross sections of the materials themselves. These complexities cannot be ignored or simplified for precise answers, and various transport codes like MCNP31 or WIMS32 can be used to resolve the scalar flux spectra, but in the case at hand, detailed descriptions of irradiated contexts are assumed to be unavailable for the sake of the forensic exercise, and the ensuing calculations will have a degree of error.

The energy-dependent neutron scalar flux will be split into an integrated magnitude, , and weighting factors for three different energy regions, as shown in Equation 3, where , , and are constant in their respective energy regions will be used to determine the percentage of flux in each energy region via integration because their integrated values will sum to unity. The percentage of neutron scalar flux is the scalar flux ratio for that particular energy region.

Equation Flux splitting

|  |  |  |
| --- | --- | --- |
|  |  | (3) |

The reason the energy-dependent neutron scalar flux is important for the following calculations is because the calculations use one group cross sections. In order to calculate a single group cross section to determine estimates on the attribution indicators, normalized energy-dependent neutron scalar fluxes will be assumed. These calculations also assume that the magnitude of the neutron scalar flux is assumed to be constant for an irradiation, when in reality it may not be. This assumption may be valid because lower burnups are more common for weapons grade plutonium. The indicators of neutron scalar flux magnitude, initial enrichment, and fuel age will be determined with these values. Then, another normalized energy-dependent neutron scalar flux will be assumed with the same calculation ensuing. This will be done for a wide range of energy-dependent scalar fluxes and the most internally consistent set of results will then be compared to results from MCNP and hopefully this methodology will provide a similar answer. This methodology should also provide an energy-dependent neutron scalar flux which would give an indication to what type of reactor the fuel was irradiated in.

The neutron scalar flux magnitude calculation will be completed with the typical burnup monitor 148Nd30, 33. These monitors are known to build into the fuel matrix linearly as a function of burnup, and typically a fission yield is used in order to calculate an actual value. In the case of 148Nd there is a potential for a sizeable amount of contribution from neutron capture in the 147 mass chain, and therefore, an effective yield value would be necessary for this calculation, especially in thermal systems34. If burnup for the system were already known, say with 137Cs, then the effective yield could be used to solve for flux from a balance equation of 148Nd. This is how this calculation will be performed.

Given that only a single isotope undergoes fission in a system, the estimate for the initial enrichment of a sample would increase linearly with burn-up. Most systems have fissile material transmuted into the system through capture reactions. Due to this, multiple isotopes fission, and more complicated analysis is required. Even so, a series of conservation equations may be solved iteratively with the assumption that burnup, heavy metal compositions, and single group cross sections are known. This methodology was presented by Doyle35, and will be used for this calculation.

The fuel age will be determined with a simple decay calculation, but requires information from all of the above analyses as well as a Bateman solver with readily changeable one group cross sections. These are needed to estimate the expected value of a radioactive species in a fissioning system at a particular burnup with one group cross sections. The radioactive species should have some level of independence for its yield for different fission isotopes and be readily found in the fuel matrix, such as 90Sr, 106Ru, 125Sb, 144Ce, 134Cs, 137Cs, or 154Eu.

***Experimental procedure:***The experimental part of this project will focus on developing a benchtop scale PUREX process, and analyzing this process. The PUREX process extracts cations of U and Pu from nitric acid by forming complex compounds with TBP and subsequently partitions uranium and plutonium by reducing plutonium (in this case with Fe[II]). First experiments will be conducted to verify that these two process steps (extraction and back-extraction) for PUREX are performing as expected for the heavy metals, and subsequent experiments will be conducted for the determination of DCs for the two steps as well as DFs for a four extraction/three back-extraction process.

The irradiated DUO2 pellet, containing fission products and Pu, will be dissolved in HNO3 for the experiments. The two main solutions used in this process are HNO3 and 30 vol.% tri-n-butyl phosphate (TBP) in a kerosene diluent. Quantification of material in these two respective solutions at various process steps will be done with a standard Canberra electrode coaxial High Purity Ge (HPGe) detector. The HPGe is a semiconductor detector which has the advantage of distinguishing gamma ray peaks at resolutions of 3-5 keV, which is important because the fuel matrix has several gamma emitters. The data collected will be in the form of count rates in a gamma spectrum, and calibration sources will be used to convert these values to mass. Due to the large number of gamma spectra that need to be collected and analyzed, a program will be written to automate analysis for a number of radionuclides.

Aqueous samples will additionally be analyzed for plutonium content through mass spectroscopy and alpha analysis. Due to cost constraints, mass spectrometry will only be applied to a single process, while alpha analysis will ensue for the others. A NexION 300X quadrupole ICP-MS will be utilized for gathering mass spectrometric data. This instrument works by drying, atomizing, and ionizing liquid samples. These samples are then passed through cone filters, curved in a quadrupole ion deflector, purified in a universal cell, refined in another quadrupole with a final-mass to-charge filtration and focused on a dual mode detector. The detector registers counts per second for particular isotopic masses, and calibration with seven different standards will be used to determine isotopic concentrations for a range of isotopes and elements.

Alpha analysis on will occur with a passivated implanted planar silicon (PIPS) detector produced under the auspices of Canberra. The spectrometer model 7401, multichannel analyzer, and NimBin model 2100, were also made by Canberra. Calibration will utilize a 4-peak Pu, Am, Gd, and Cm source from Eckert and Ziegler. Samples from aqueous solutions will first be diluted and then 10-20 μl samples will be dried on an aluminum surface.

Each extraction or back-extraction removes a certain percentage of dissolved material based on DCs. This is here defined as the ratio of grams per liter of solute in organic solution over grams per liter of solute in the aqueous solution, shown in Equation 4, describes the steady state location of any species in the system8. DCs are specific to an element and vary widely with the concentration and temperature of the solvents. DCs will be determined with the above analytical tools.

Equation Distribution Coefficient

|  |  |  |
| --- | --- | --- |
|  |  | (3) |

1. **Expected Results**

***Completed Analytical*:** Equations for burnup, flux magnitude, initial enrichment, and fuel age have been settled upon. A program was written to set up and solve an ORIGEN236 type system with options for tweaking cross section information, something that ORIGEN2 does not allow simply. The solver uses a best rational approximation, which reduces calculation time for each solution, which also presents an option for quantifying uncertainty in these calculations. Additionally programs have been set up for calculation of single group cross sections given a flux spectrum. These pieces of code still need to be linked together in a single package so that a fast-to-thermal ratio can be determined through brute force and final results for the forensic analysis presented. These results will include pre-processed and post-processed plutonium with the expectation that pre-processed plutonium will produce more accurate results. “Accuracy”, in this instance, will be determined by the closeness of these determined values to what MCNP predicts.

***Completed Experimental*:** The experimental procedure for separating uranium and plutonium from fission products (extraction) as well as Pu and U from each other (back-extraction) has been experimentally verified. Experiments with natural uranium analyzed with mass spectrometry were conducted to ensure that uranium does not proceed to the product solution. Initially a low nitric acid concentration in the back-extraction solution caused the uranium to proceed to the product, but increasing the concentration remedied this problem because the aqueous phase already had nitrate ions. Also a single extraction and back-extraction was conducted to ensure that plutonium separates from uranium in the final aqueous solution.

A process, containing four extractions and three back-extractions, has been completed and analyzed with mass spectrometry. The results, containing DCs and DFs for several elements, for this and the previous experiment were published37. This process was improved, and completed three full times, with several other partial experiments. All of the process steps have been analyzed with gamma spectroscopy and several still need alpha spectrums collected. The DF results for the second round of experiments are expected to be better in the sense that purer plutonium is being produced by this process.

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