NUCLEAR ENGINEERING
TEXAS A&M UNIVERSITY

# Experimental Characterization of Pu Separation by PUREX Process on a Low-Burnup, Pseudo-Fast-Neutron Irradiated $DUO_2$ for Product Decontamination Factors and Nuclear Forensics

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Dr. Craig Marianno
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AIEN 304

TEES | TEXAS A&M ENGINEERING EXPERIMENT STATION

NUCLEAR SECURITY SCIENCE & POLICY INSTITUTE

# Outline

# Motivation

- Current Events
  - Joint Comprehensive Plan of Action
  - Non-safeguarded reactors
  - Islamic State of Iraq and Syria
- Past Events
  - Septemer 11, 2001
- Limited scope of IAEA safeguards
- "the awful arithmetic of the atomic bomb" [1]
- Need for improved forensic capabilities[2, 3, 4]

# Definitions

- Special Nuclear Material (SNM)
  - Plutonium, $^{233}U$, or $^{235}U$
- Nuclear Forensics
  - The investiative activity that surrounds the search for attributes of undetermined radioactive specimens for the purpose of attribution.
- SNM origin attributes/indicators
  - Indicators or clues for SNM origin attribution. Examples include burnup, fluence rate, initial fuel enrichment, fuel age, and fast-to-thermal irradiation ratios
- Decontamination Factors (DF)
  - A measure of the effectiveness with which a product is decontaminated from a contaminant

$$DF_j = \frac{\frac{c_j}{c_{Pu}}\Big|_{\text{initial}}}{\frac{c_j}{c_{Pu}}\Big|_{\text{final}}}$$

# National Context

"The United States has developed a nuclear forensics capability that has been demonstrated in real-world incidents of **interdicted materials** and in exercises of actions required after a nuclear detonation. The committee, however, has concerns about the program and finds that without strong leadership, careful planning, and additional funds, these capabilities will decline" [2]

Major areas of concern include:

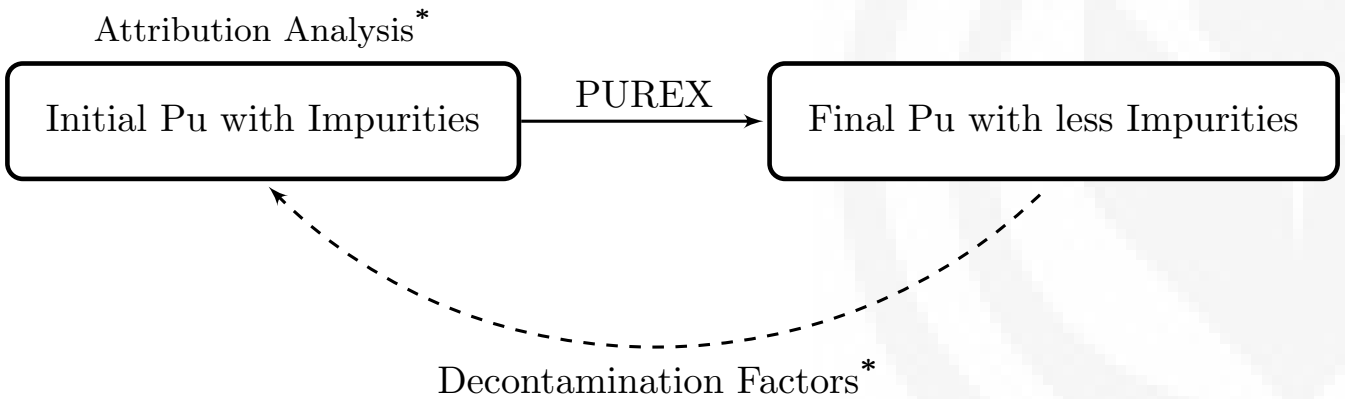
- Organization
- Sustainability
- **Workforce and Infrastructure**
- **Procedures and Tools**

# Forensic Context

- Nature of inverse problems
- Plutonium purification necessary for weapons production

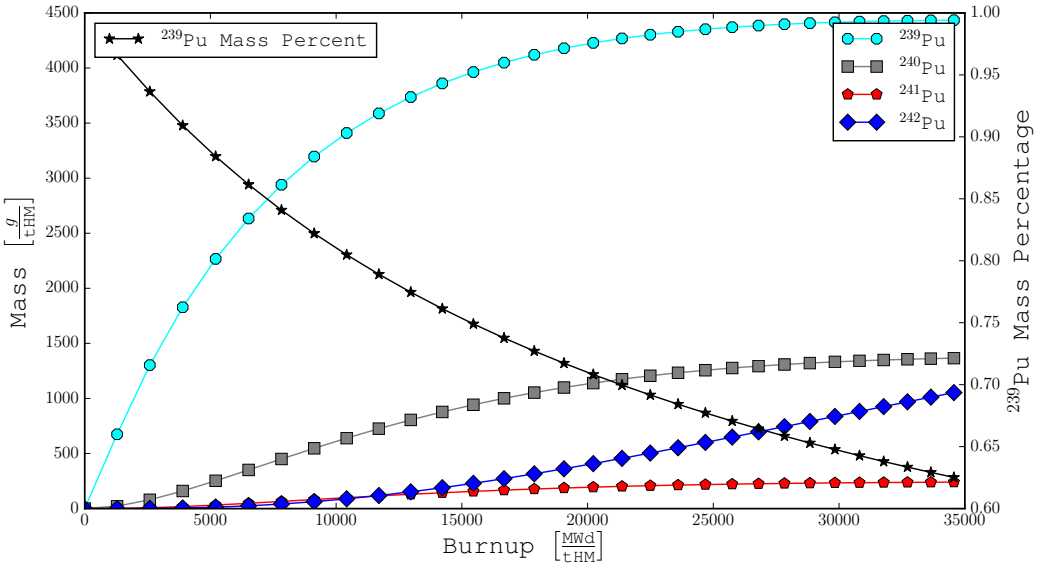
$$^{238}U + n \rightarrow ^{239}U \xrightarrow[T_{1/2}=23\text{ min}]{\beta^-} ^{239}Np \xrightarrow[T_{1/2}=2\text{ d}]{\beta^-} ^{239}Pu$$

- Attribution for unpurified Pu has been previously studied [5, 6, 7]



# Nuclear Context

- Weapons-grade Pu can be extracted from reactor discharged fuel with a burnup of about 1 (GWD/tU)
- Pu isotopes produced in irradiated fuel can vary
- Two examples of reactors which can intentionally discharge low burned fuel for extracting weapon-grade Pu are:
  - Fast Breeder Reactor, CANDU Reactor



# Chemical Context

- Plutonium Uranium Redox EXtraction (PUREX)
  - Liquid-liquid solvent extraction
  - Many stages:
- Distribution Coefficient (D): The ratio between the organic and aqueous phases (aka: D-values)

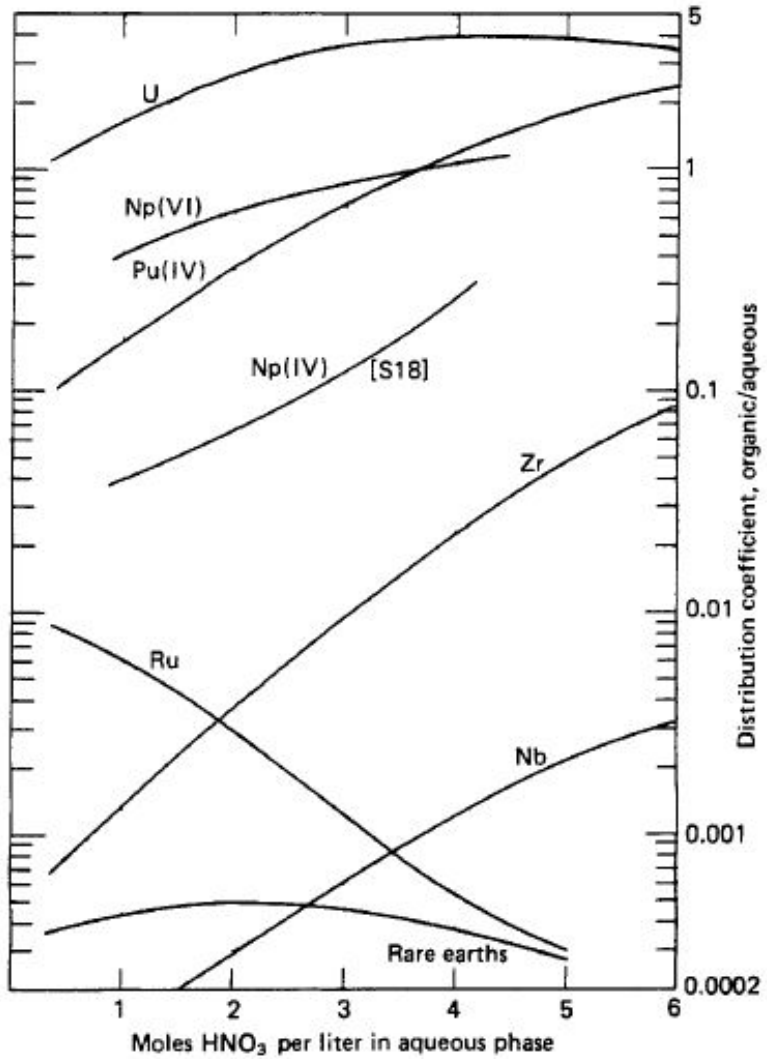
$$D = \frac{c_o}{c_{aq}}$$

  - Specific element to element
  - Vary widely[8]
  - The fraction of mass, $f_o$ deposited in the organic phase, assuming a volume ratio between the aqueous and organic phases, $V_R$, is:

$$f_o = (1 + D^{-1} V_R^{-1})^{-1}$$

# Chemical Context

- Lack of literature on decontamination factors and distribution coefficients for useful forensic elements (Cs, Sb, Eu, Rb, Sr, Nd, Pm, and Sm)
- With a known process and D-values, DF values for individual elements can be determined

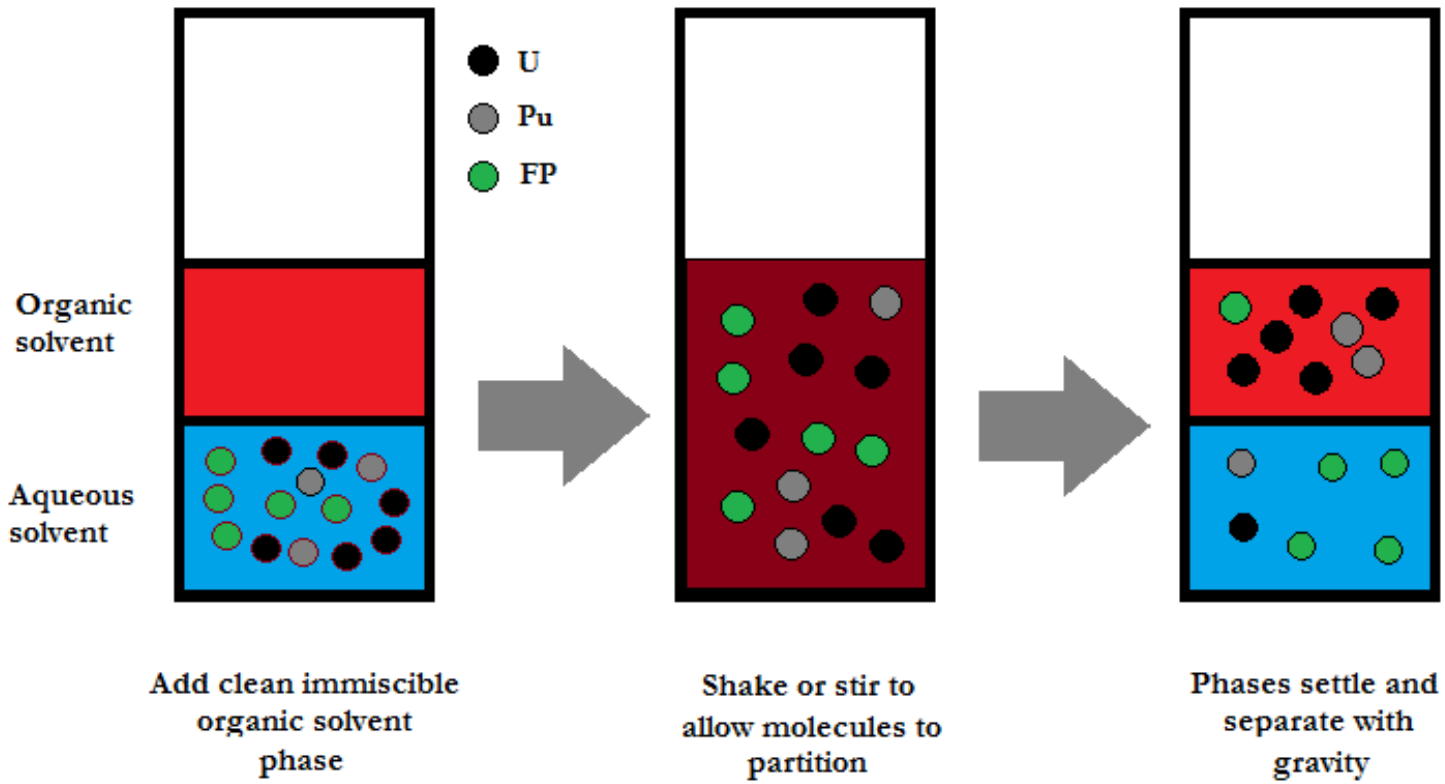



Adapted from Stoller[8]

# Extraction

$$UO_{2(aq)}^{2+} + 2NO_{3(aq)}^{-} + 2TBP_{(o)} \leftrightarrow UO_2(NO_3)_2 \cdot 2TBP_{(o)}$$ [9]

$$Pu_{(aq)}^{4+} + 4NO_{3(aq)}^{-} + 2TBP_{(o)} \leftrightarrow Pu(NO_3)_4 \cdot 2TBP_{(o)}$$



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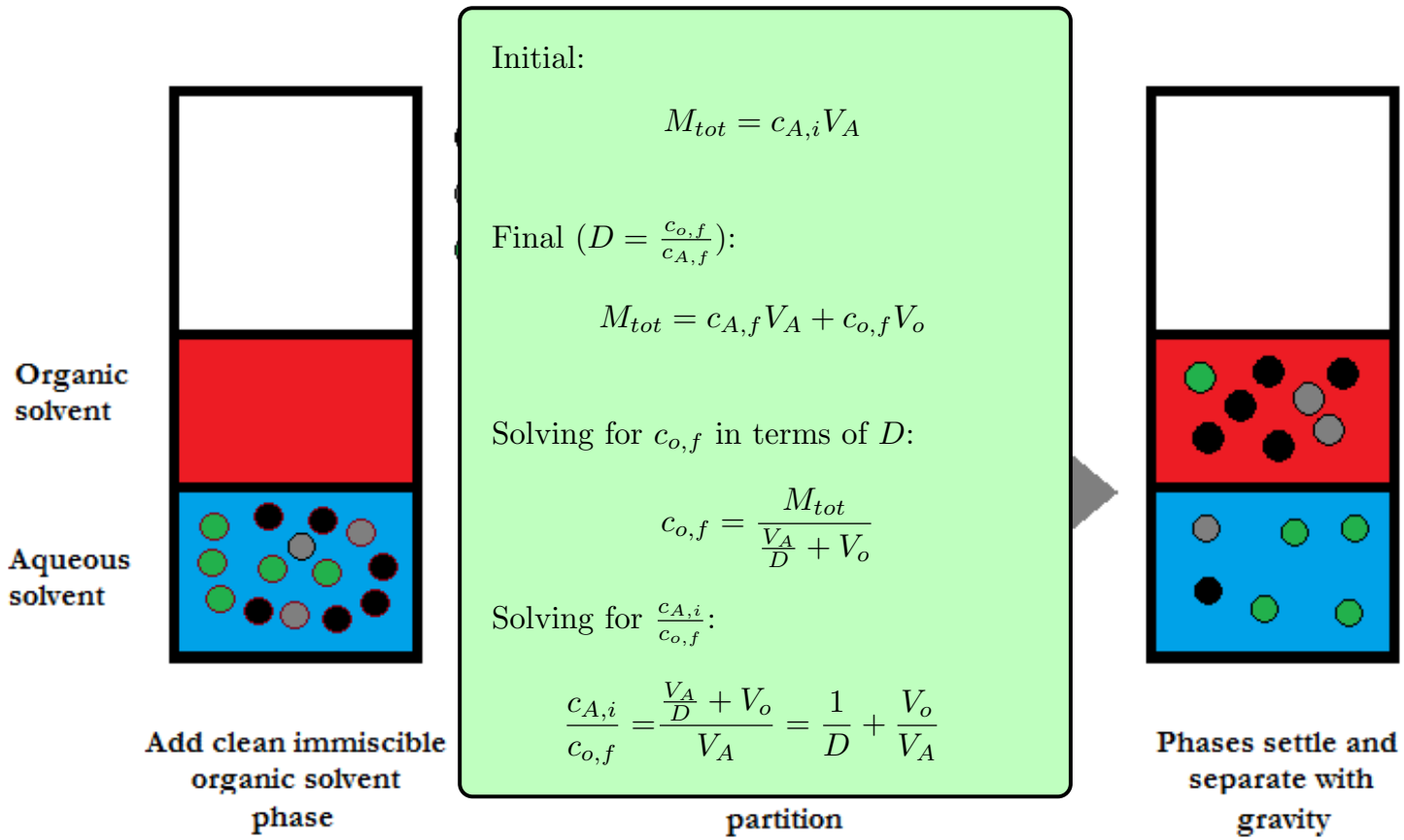

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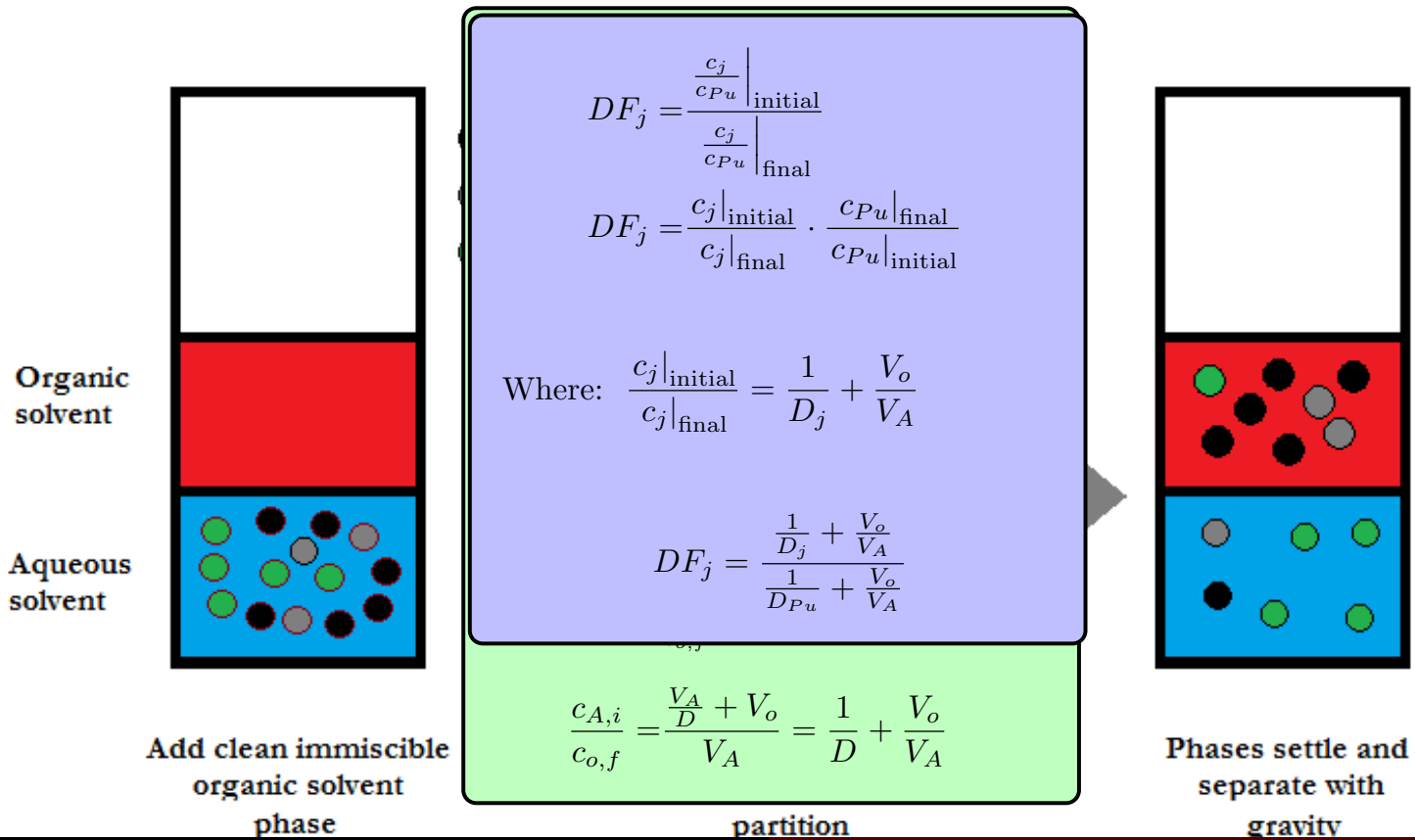
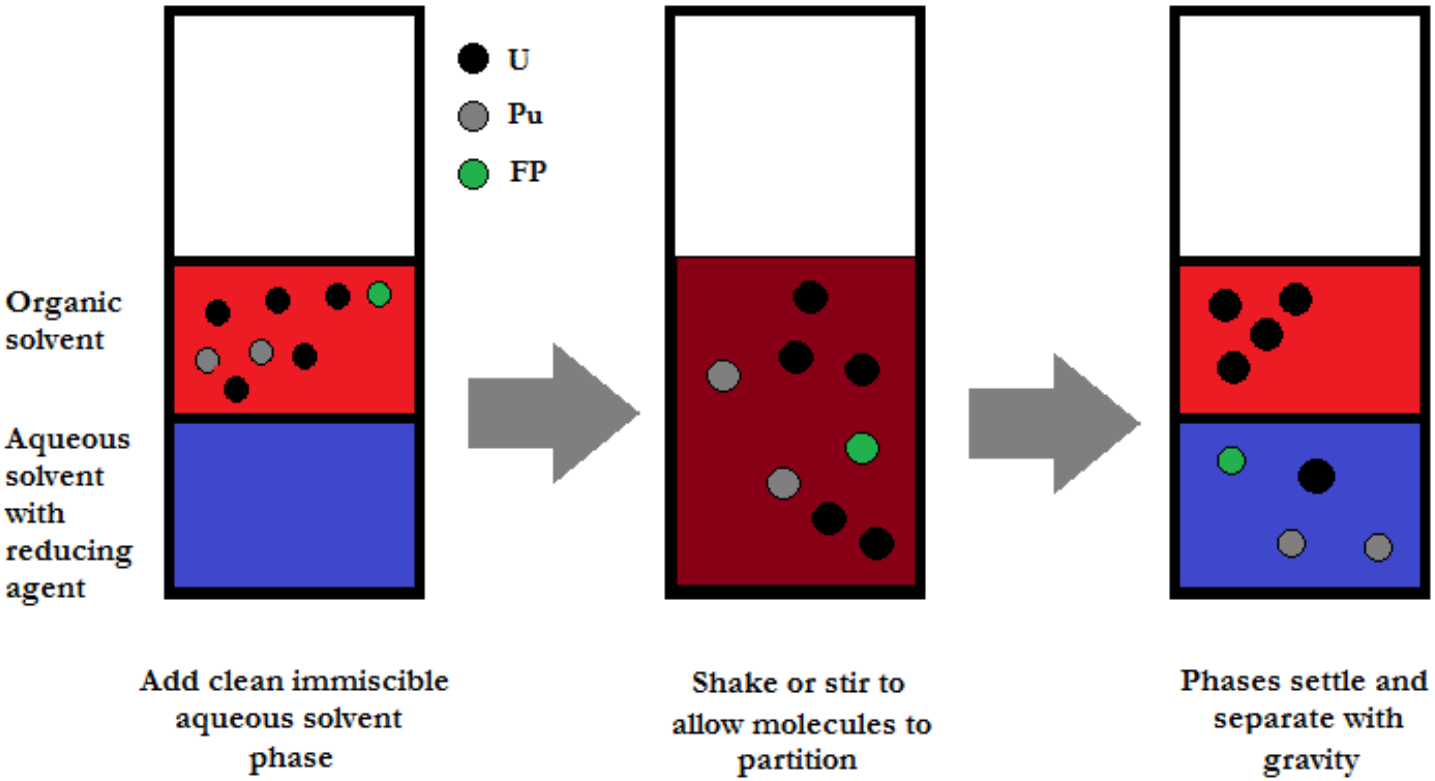


# Back-Extraction

$$Pu(NO_3)_4(TBP)_{2(o)} + Fe^{2+}_{(aq)} \leftrightarrow Pu^{3+}_{(aq)} + 4NO^{-}_{3(aq)} + 2TBP_{(o)}$$ [10]



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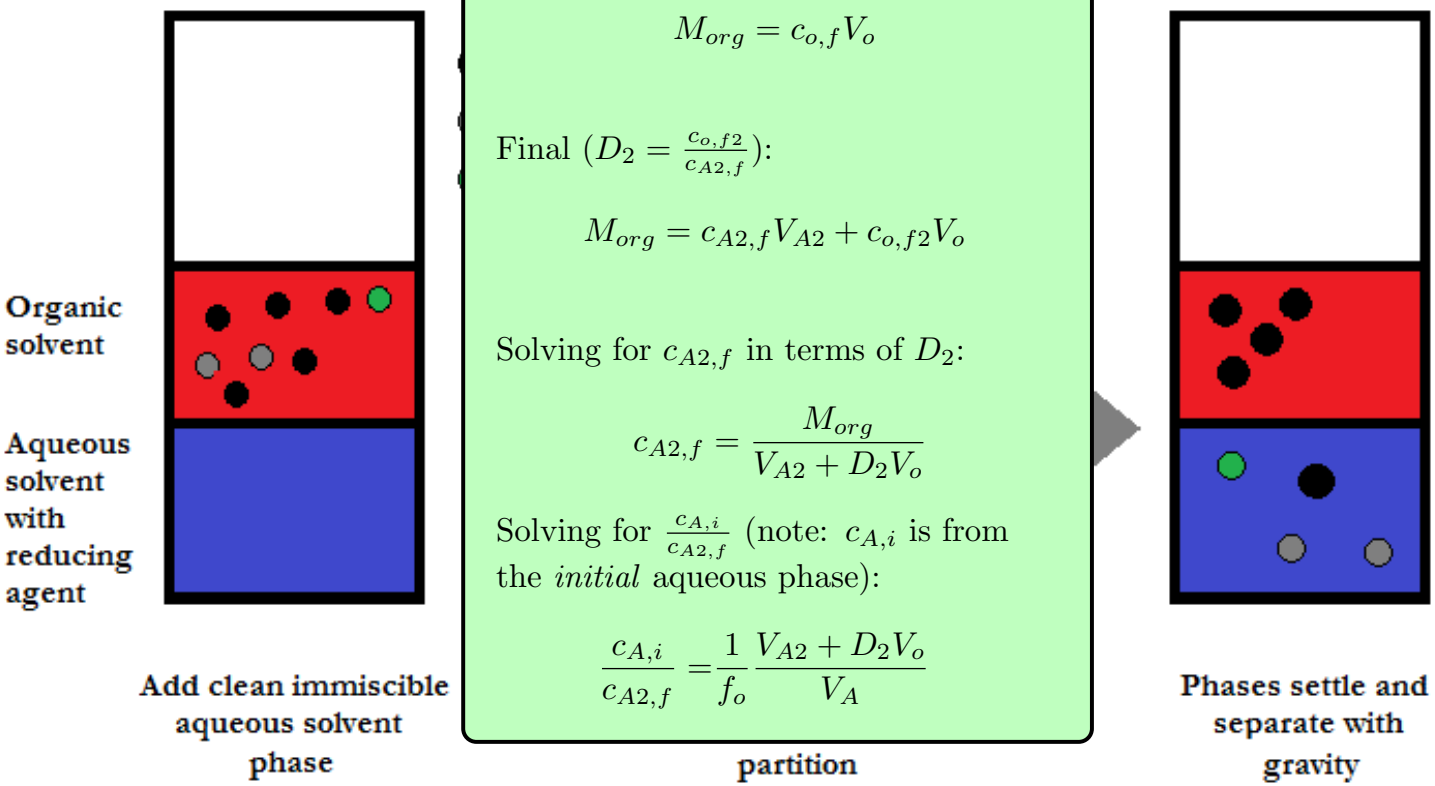

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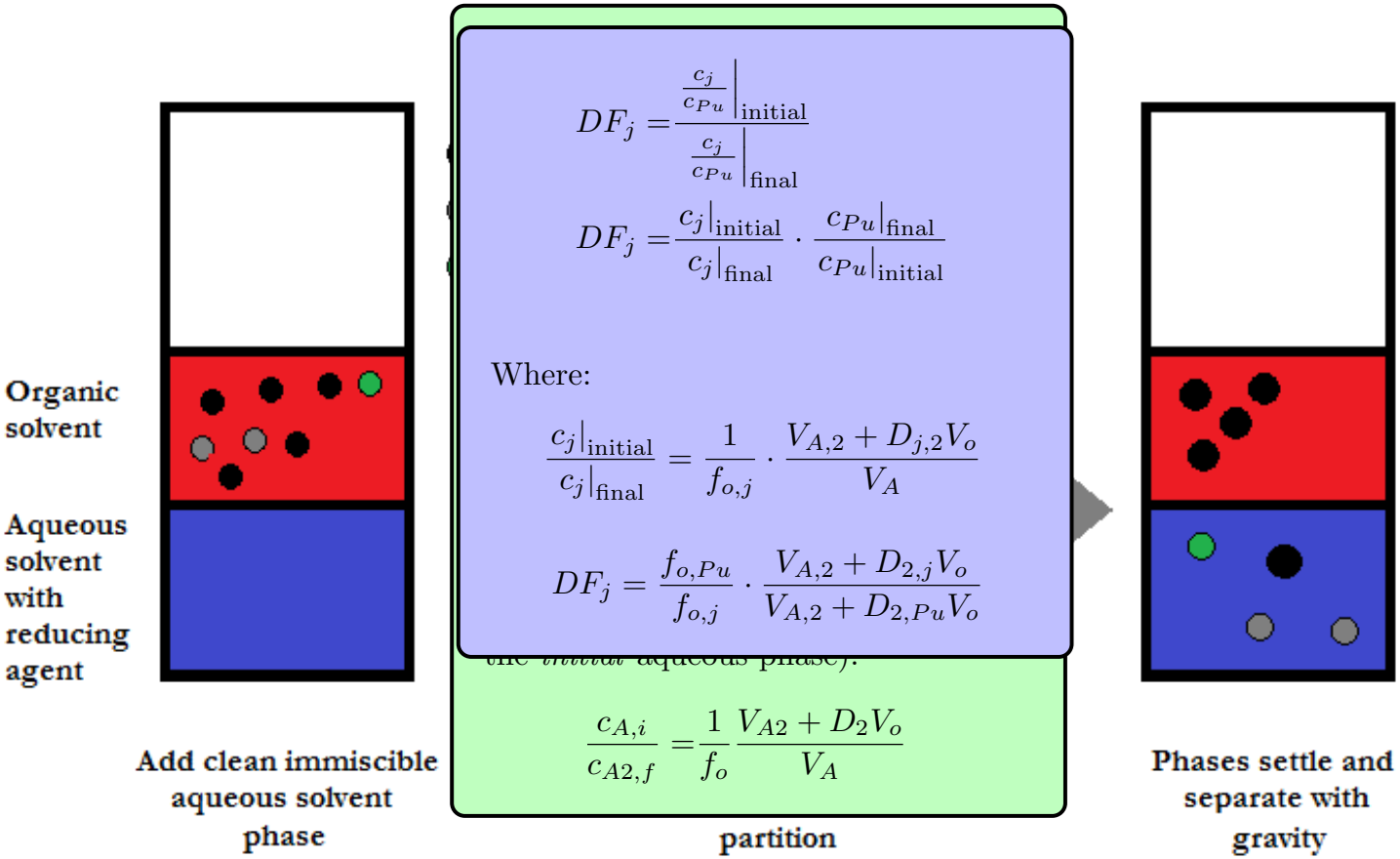



$$DF_j = \frac{\left.\frac{c_j}{c_{Pu}}\right|_{\text{initial}}}{\left.\frac{c_j}{c_{Pu}}\right|_{\text{final}}}$$

$$DF_j = \frac{c_j|_{\text{initial}}}{c_j|_{\text{final}}} \cdot \frac{c_{Pu}|_{\text{final}}}{c_{Pu}|_{\text{initial}}}$$

Where:

$$\frac{c_j|_{\text{initial}}}{c_j|_{\text{final}}} = \frac{1}{f_{o,j}} \cdot \frac{V_{A,2} + D_{j,2}V_o}{V_A}$$

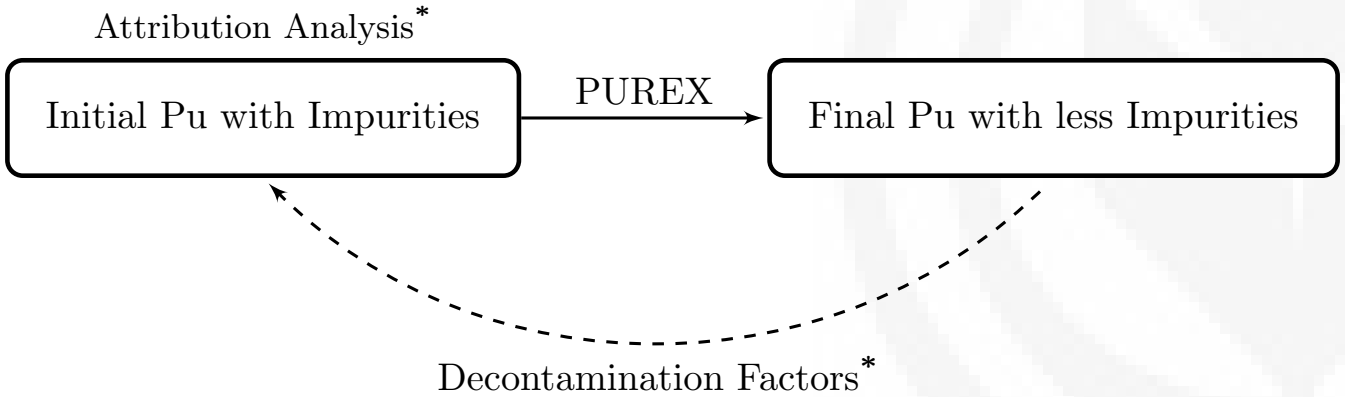
$$DF_j = \frac{f_{o,Pu}}{f_{o,j}} \cdot \frac{V_{A,2} + D_{2,j}V_o}{V_{A,2} + D_{2,Pu}V_o}$$

the initial aqueous phase):

$$\frac{c_{A,i}}{c_{A2,f}} = \frac{1}{f_o}\frac{V_{A2} + D_2V_o}{V_A}$$

# Decontamination Factors and their use

- After several cycles of Pu extraction/scrubbing/back-extraction are completed, the effectiveness of a PUREX cycle is described by the decontamination factor (DF):
- DFs are characteristic of different process cycles
- Larger values ($10^7$) for industrial scale PUREX (compared to benchtop)[8, 9]



# Objectives I

**Characterize a 4 extraction 3 back-extraction PUREX process**

- Collect D-values for each step
  - ✓ $^{144}Ce$,$^{155}Eu^{*}$,$^{154}Eu^{*}$,$^{125}Sb$, $^{106}Ru$, $^{134}Cs$, $^{137}Cs$ (Measured Gamma in triplicate)[5]
  - ✓ Convert Gamma Spectrum to D-values
  - ✓ $^{85}Rb^{*}$,$^{90}Sr^{*}$,$^{97,98,100}Mo$,$^{101,102,104}Ru$, $^{110}Pd$,$^{112}Cd$,$^{133}Cs^{*}$,$^{140,142}Ce$,$^{143}Nd^{*}$, $^{147}Pm^{*}$,$^{151}Sm^{*}$,$^{154}Eu^{*}$,$U^{*}$,$Pu^{*}$ (Mass Spec)
    - Convert all mass spec data to D-values
- Collect DF-values for the process
  - ✓ Prepare alpha samples for each step (triplicate)
    - Analyze alpha samples for each step
    - Convert alpha spec + gamma spec data to DF values
  - ✓ Convert Mass spec data to DF values (published)
- Mathematically connect D-values to DFs
  - Derive equations with uncertainty propagation
  - Analyze connection with uncertainty

# Objectives II

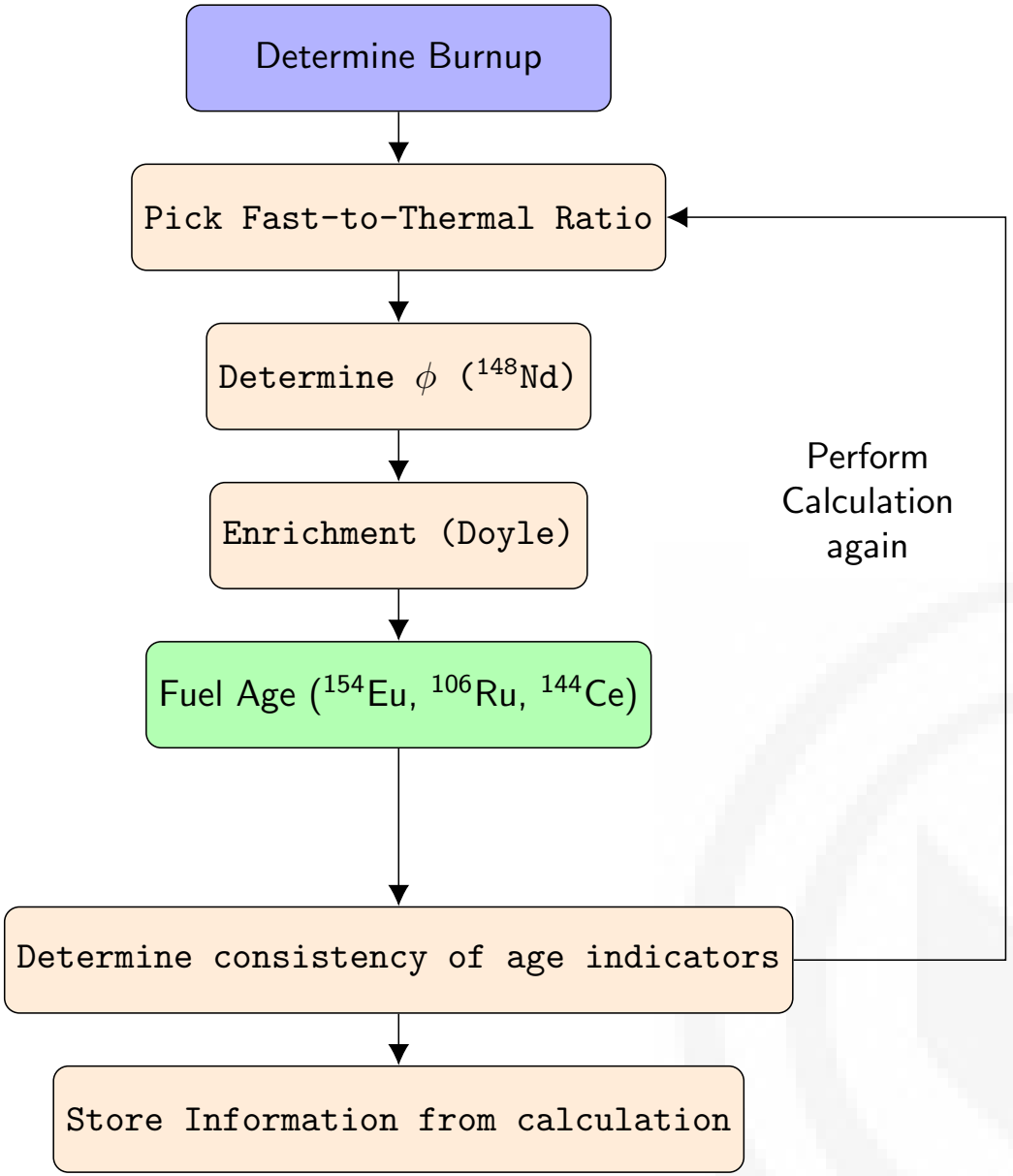
**Determine attribution indicators:**

- Mathematically derive equations for above indicators with respect to one of the isotopes determined above
  - ✓ Burnup
  - ✓ Fluence Rate
  - ✓ Initial Enrichment
  - ✓ Fuel Age
  - Fast-to-thermal ratios (requires iteration)
- Program a system to iteratively solve for these parameters given heavy metal concentration ratios
  - Make a program that can read ENDF files for x-sections
  - ✓ Create/Use a bateman solver with automated x-section modifications
  - ✓ Create program to calculate single group x-sections from ENDF data and an assumed fast-to-thermal ratio
  - Couple all programs together in a single program
- Use above information to determine indicators for three sets of data

# Present Status of the Question

- Stable noble fission gases as burnup verification[11]
- Determine burnup, enrichment, and fuel age from used fuel in a RDD[12]
- Analysis of purified plutonium isotopics for reactor type[13]
- PUREX co-processing DF values for U and Pu[14]
- PUREX D-values and DF values under numerous circumstances[9, 15, 16, 17, 18, 8]
  - DF values for $^{106}$Ru and $^{95}$Zr[8]
  - Compilation of D-values for U, Th, and Pu[19]
  - D-values for rare earths, Pu, Th[17, 20, 21, 22, 23, 24, 25, 26, 27, 28]
  - Ga D-values[28]

# Procedure


Determine Burnup
Pick Fast-to-Thermal Ratio
Determine $\phi$ ($^{148}$Nd)
Enrichment (Doyle)
Fuel Age ($^{154}$Eu, $^{106}$Ru, $^{144}$Ce)
Determine consistency of age indicators
Store Information from calculation
Perform Calculation again

# Analytical Procedure

- $12.9 \pm 0.1$ mg of $DUO_2$ irradiated at HFIR

$$\frac{dn_i}{dt} = -\lambda_i^{eff} n_i + \sum_{j=1}^{N} b_{j\rightarrow i}^{eff} n_j$$

$$\lambda_i^{eff} = \lambda_i + \phi \sum_{j=1}^{N} \sigma_{i\rightarrow j}$$

$$b_{j\rightarrow i}^{eff} = b_{j\rightarrow i}\lambda_j + \sigma_{j\rightarrow i}\phi + \gamma_{j\rightarrow i}\sigma_{j,f}\phi$$

$$\frac{d\vec{n}}{dt} = \mathbf{A}\vec{n}(t) \rightarrow \vec{n} = e^{\mathbf{A}t}\vec{n}_0$$

# Analytical Procedure - Burnup

$$BU = \frac{\text{Power[MW]} \cdot \text{days}}{m[HM]}$$
$$= \left[\frac{N^B}{N_0^{HM}}\right] \frac{N_A E_R}{\gamma_B} \cdot \frac{1}{M_0^{HM}}$$

# Analytical Procedure - Fluence Rate

$$\phi \approx \frac{\lambda_7}{\sigma_7 \left( \frac{\gamma_7}{\gamma_8^* - \gamma_8} - 1 \right)}$$

# Analytical Procedure - Initial Enrichment

$$\epsilon_0 = \frac{N^{U238}(T)}{N_0^U}\left[\frac{N^{U235}(T)}{N^{U238}(T)} + \frac{N^{U236}(T)}{N^{U238}(T)}\right] + \frac{M_0^U}{N_A E_R} BU(T) - G^{238} - G^{239} - G^{240} - G^{241}$$

# Analytical Procedure - Fuel Age

$$t_d = -\frac{1}{\lambda} ln \left( \frac{N_{\text{measured}}}{N_{EOI}} \right)$$

# Analytical Procedure - Fast-to-thermal ratio

- Iterative scheme
- Recalculate the above with new x-sections
- Cut off for thermal 0.5 ev, 100kev, 20MeV

# Experimental Procedure - Chemistry Procedure

- Grab part of procedure from lab notebook, or grab a picture

# Experimental Procedure - Mass Spectrometry

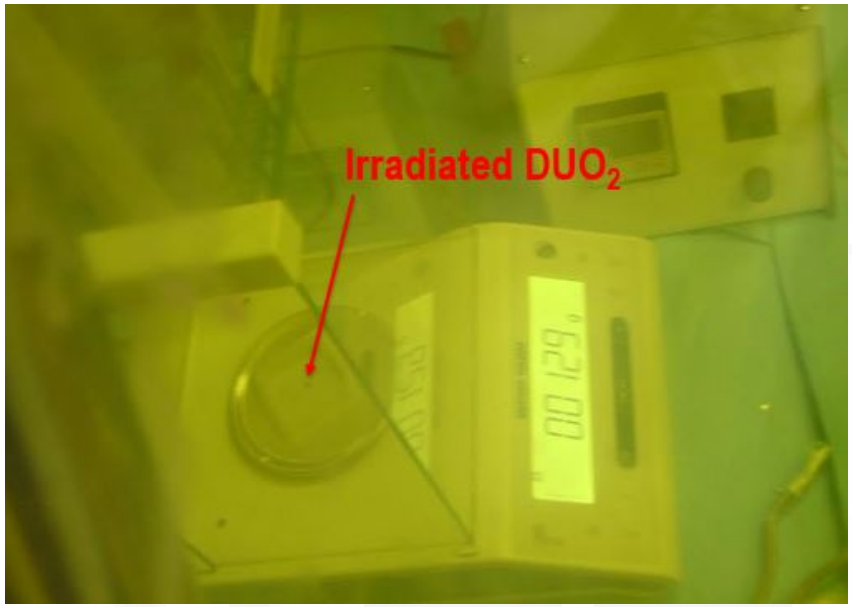
# Experimental Procedure - Gamma Spectrometry

# Experimental Procedure - Alpha Spectrometry

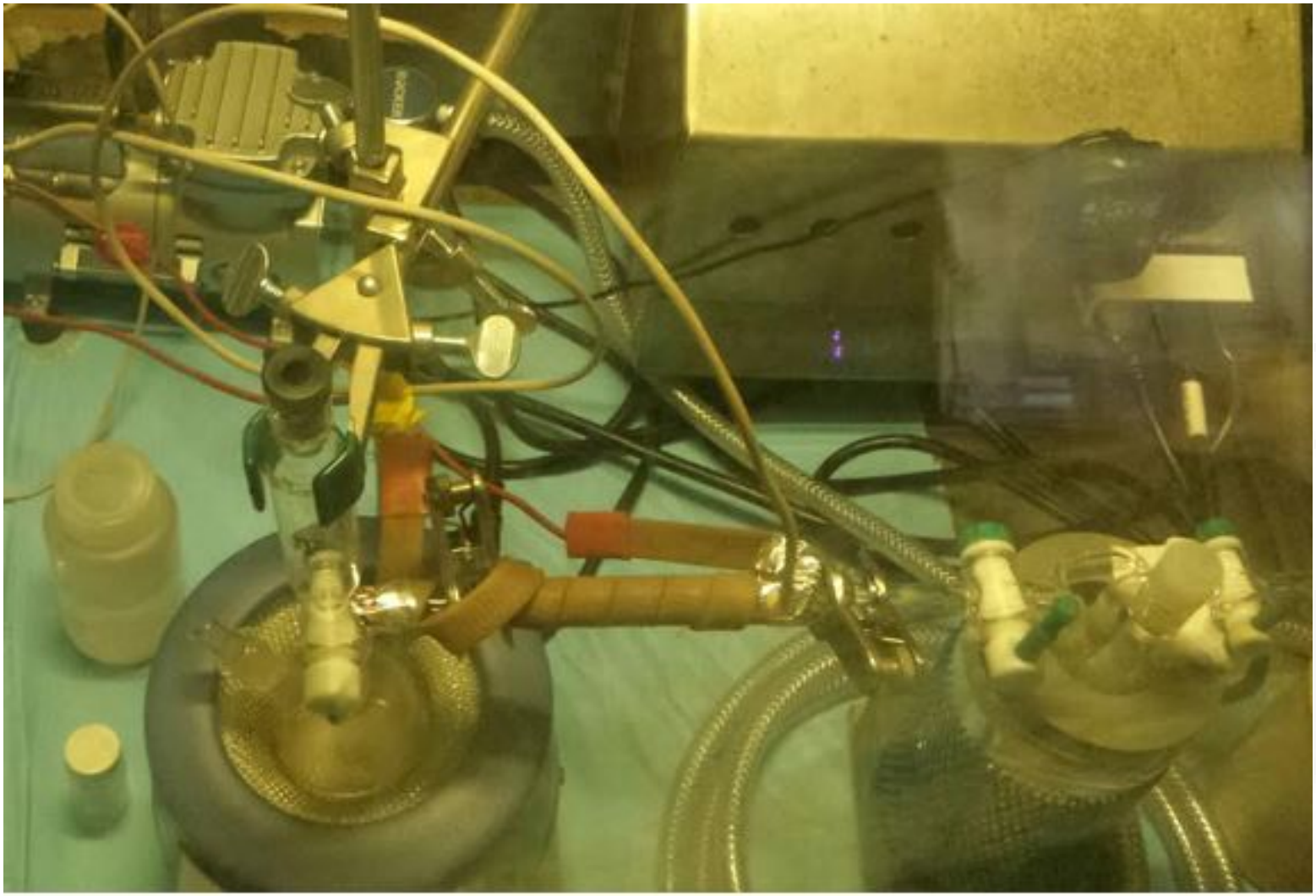
# Current and Expected Results

# Irradiation

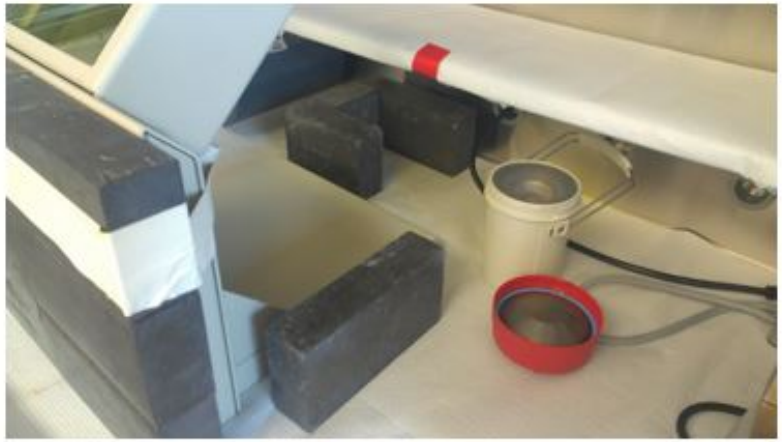
- $12.9 \pm 0.1$ mg of $DUO_2$ was irradiated
  - High Flux Isotope Reactor at Oak Ridge National Laboratory
- Burnup was $4.43 \pm 0.31$ GWd/tHM[29] from $^{137}Cs$
- $0.196 \pm$ mg of total Pu was produced as measured by ICP-MS

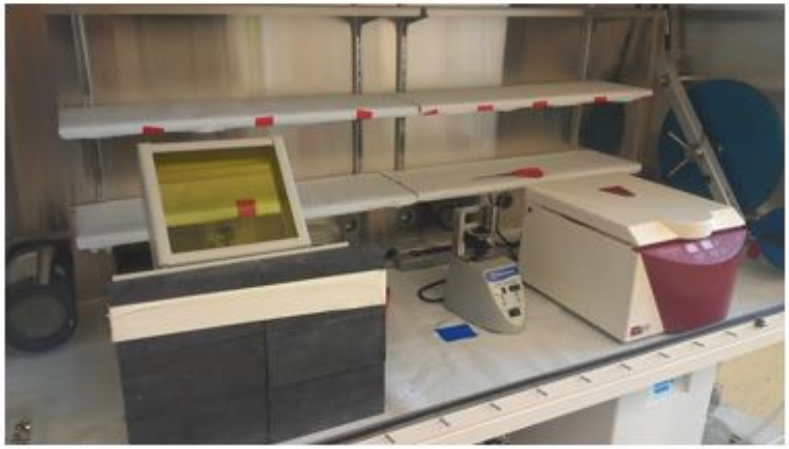


# Dissolution of the spent fuel pellet



# Glovebox





# Experiments

- Single stage extraction and back-extraction
  - Purpose: quantify product recovery, D-values and DF values for single stage extraction and back extraction
  - Conditions:

| Starting Solution | Extraction Solution | Back extraction solution |
|---|---|---|
| 4 M nitric acid | 30% vol.% TBP, 70 vol.% kerosene | 0.024 M ferrous sulfamate in 0.75 M nitric acid |

- Multi-contact extraction and back-extraction
  - Purpose: Quantify DF for a process with 4 extractions, 3 back extractions
  - Conditions:

| Starting Solution | Extraction Solution | Back extraction solution |
|---|---|---|
| 4 M nitric acid | 30% vol.% TBP, 70 vol.% kerosene | 0.024 M ferrous sulfamate in 4 M nitric acid |

# Mass spectrometry Results

## Recoveries of U and Pu

| | Pu Recovery | U Recovery |
|---|---|---|
| Single stage | (83.4±9.5)% | (11.2±1.3)% |
| Multi-contact Cycle 1 | (99.7±4.2)% | (6.8±0.3)% |
| Multi-contact Cycle 2 | (93.0±4.6)% | (6.6±0.3)% |
| Overall Experiment 2 | (92.7±6.0)% | (0.45±0.03)% |

# Mass Spectrometry Results

## Decontamination Factors

| Element (Z) | SS | Error | MC Cycle 1 | Error | Isotopes 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 |

# Gamma Spectroscopy Results

# Initial vs Final solutions

Subsection 5

# Future Work

# Objectives I

**Characterize a 4 extraction 3 back-extraction PUREX process**

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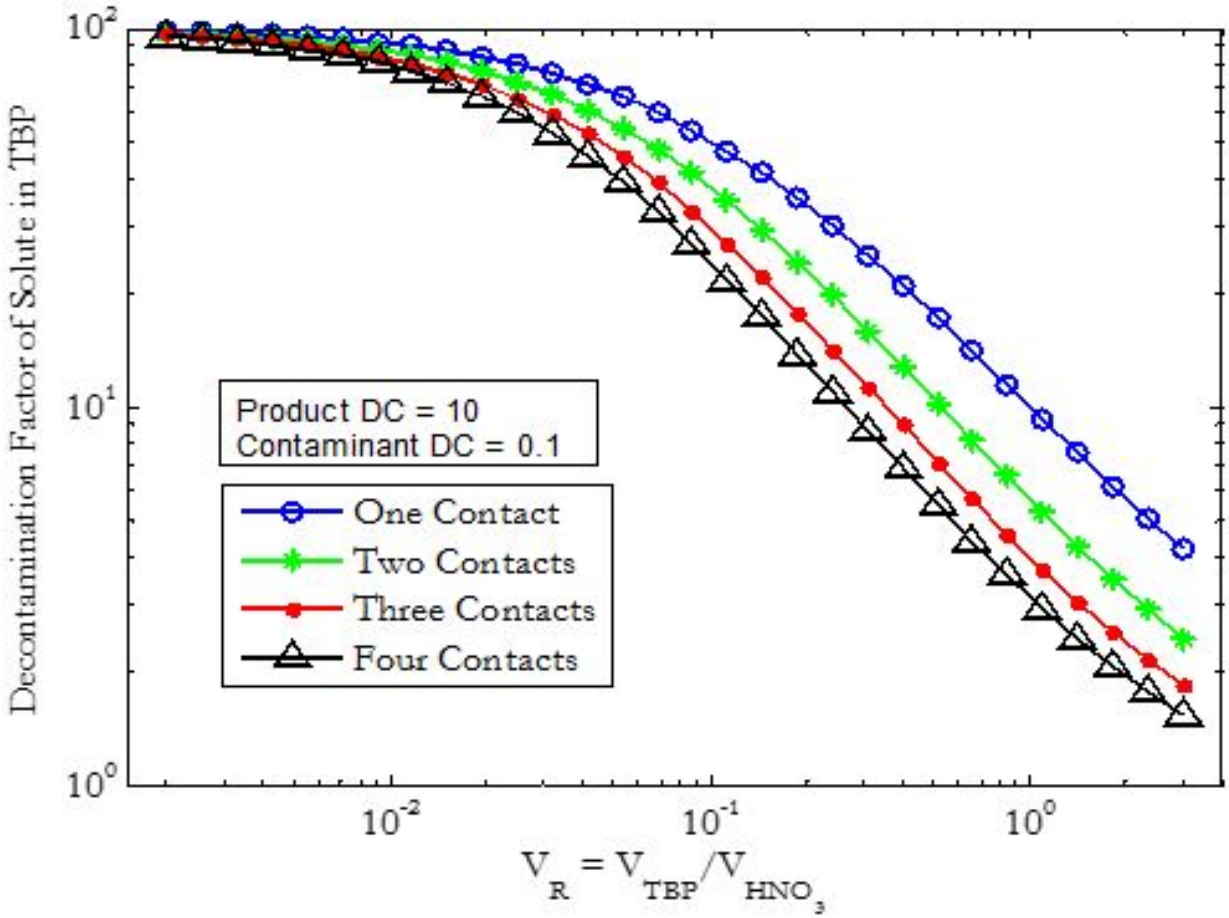
# Questions?

# Previous Experiment Results




Decontamination Factors for multi-contact extraction.

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[11] William S Charlton, Bryan L Fearey, Charles W Nakhleh, Theodore A Parish, Robert T Perry, Jane Poths, John R Quagliano, William D Stanbro, and William B Wilson. Operator declaration verification technique for spent fuel at reprocessing facilities. *Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms*, 168(1):98–108, 2000.

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[18] E.R. Irish and Reas W.H. The purex process-a solvent extraction reprocessing method for irradiated uranium. 1957.

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[21] K Alcock, FC Bedford, WH Hardwick, and HAC McKay. Tri-n-butyl phosphate as an extracting solvent for inorganic nitrates-i: Zirconium nitrate. *Journal of Inorganic and Nuclear Chemistry*, 4(2):100–105, 1957.

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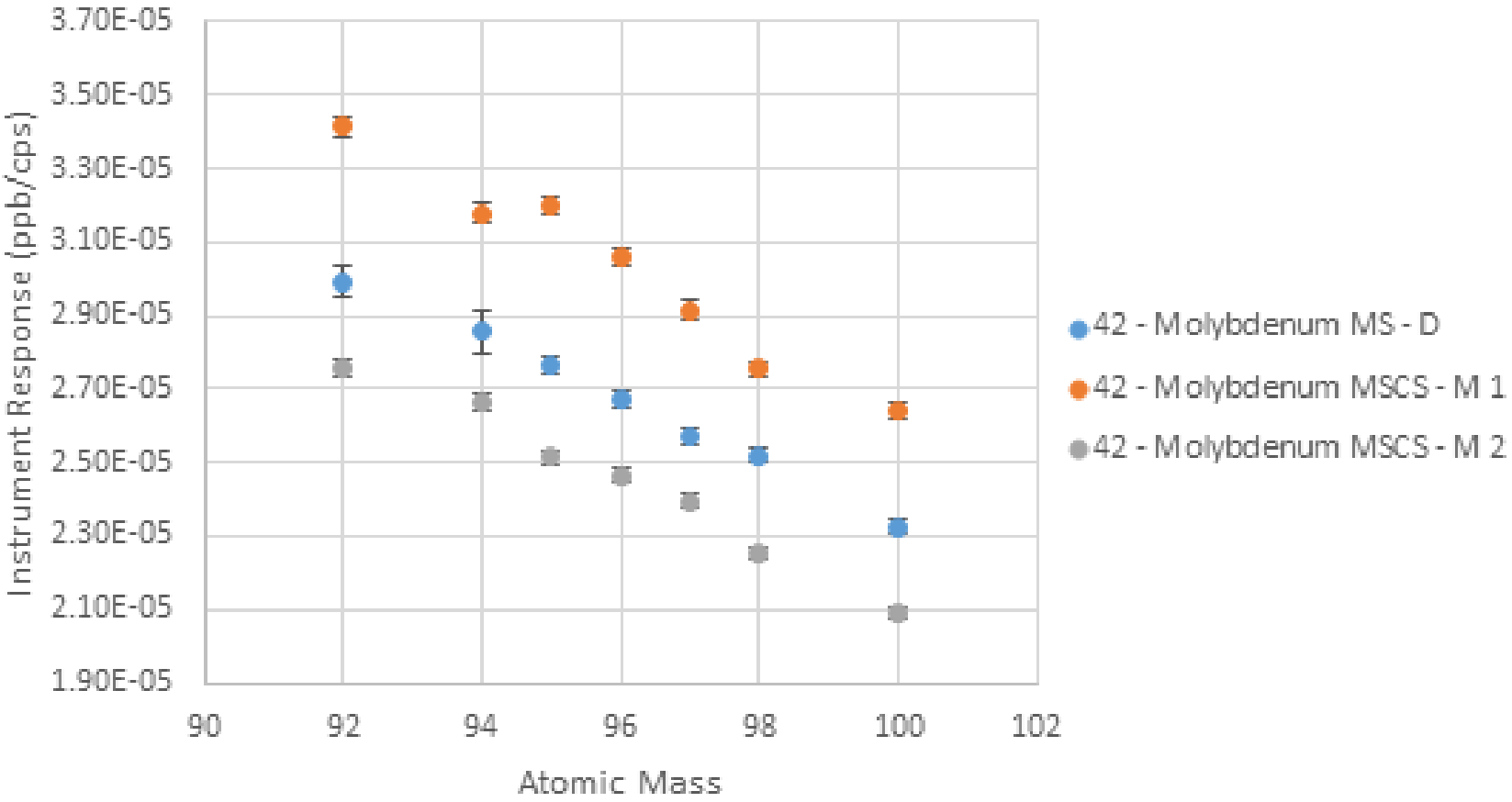
# Mass Spec


3.70E-05
3.50E-05
3.30E-05
3.10E-05
2.90E-05
2.70E-05
2.50E-05
2.30E-05
2.10E-05
1.90E-05
Instrument Response (ppb/cps)
90
92
94
96
98
100
102
Atomic Mass
42 - Molybdenum MS - D
42 - Molybdenum MSCS - M 1
42 - Molybdenum MSCS - M 2