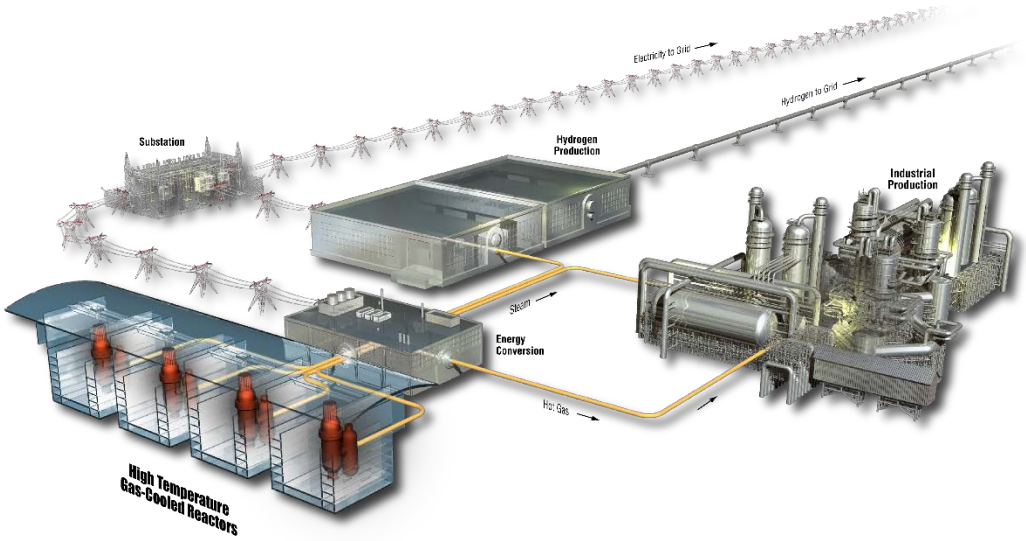
AGR-2 PIE Progress

John Stempien

AGR TRISO Fuels Program Review
Idaho Falls, ID
July 18-19, 2017

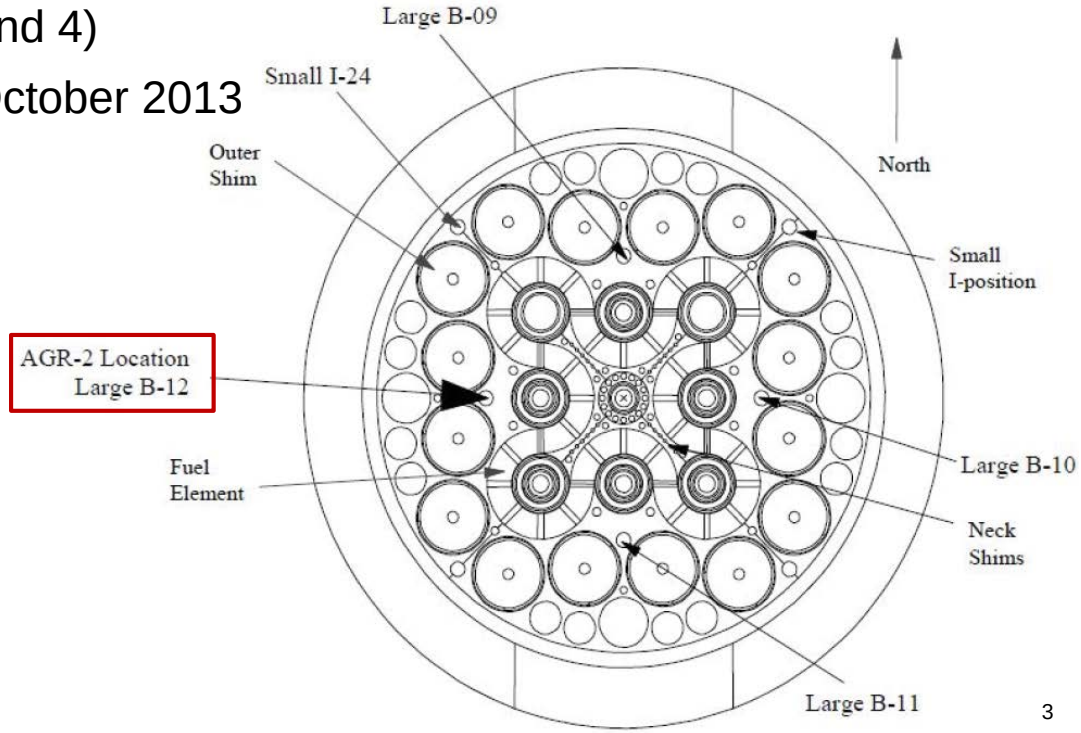


Outline

- AGR-2 irradiation experiment background
  - Fuel samples
  - Irradiation conditions
- Status of analyses on irradiation capsule components
- Fission product mass balance
- Fuel compact non-destructive/destructive analyses and safety testing
- Summary and future work

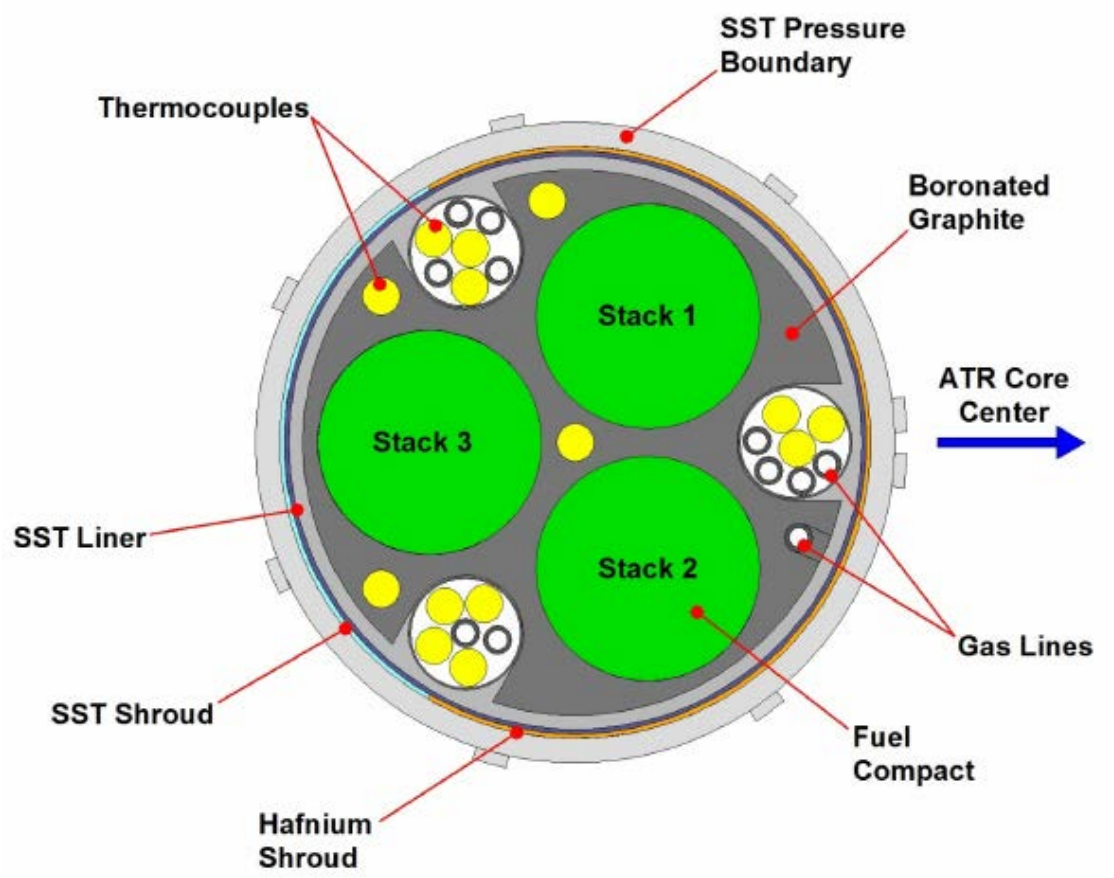
AGR-2 Irradiation

- TRISO fuel particles produced in engineering scale coater (6 in. diameter)
- Total of 6 capsules
- 4 AGR Program Capsules, total of 48 AGR-2 Compacts
  - 36 compacts with AGR-2 UCO fuel kernels (~3176 particles/compact)
  - 12 compacts with UO_2 fuel kernels (~1500 particles/compact) for comparison to UCO and to historic UO_2 data
- 2 CRADA capsules (Capsules 1 and 4)
- Irradiation in ATR: June 2010 to October 2013

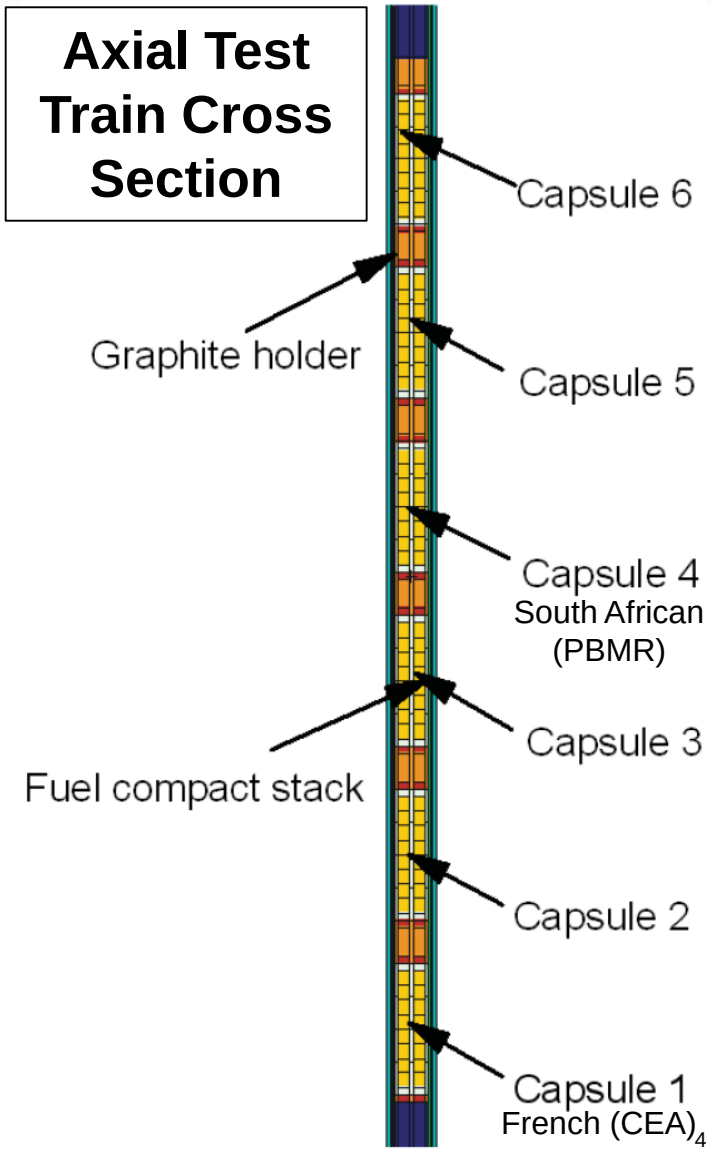


AGR-2 Irradiation Test Train

Radial Capsule Cross Section

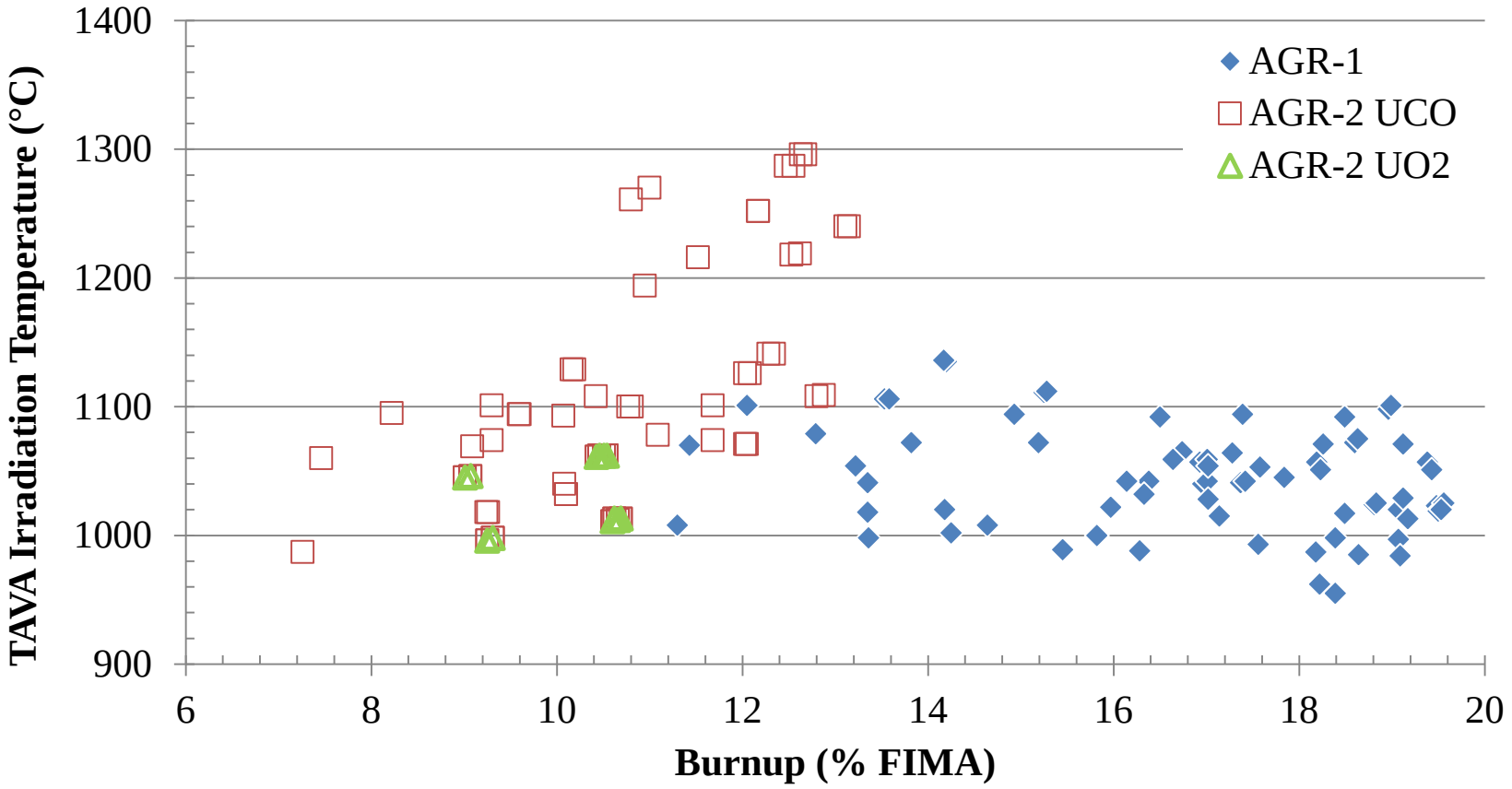


Axial Test Train Cross Section



AGR-2 Irradiation Temperature and Burnup

- Time-averaged, volume-averaged (TAVA) irradiation temperatures: 987 to 1296°C
- Burnup range: 7.3 to 13.2% fissions per initial metal atom (FIMA)
- AGR-2 UCO fuel lower burnup, but higher temperature than AGR-1



AGR-2 Components and Associated PIE

- Each AGR-2 irradiation capsule has the following components and analyses:

- Complete
- In-progress
- Planned

1 Graphite Compact Holder
- Precision gamma scanner (PGS)
- Burn-leach of holder
  - Gamma
  - ICP-MS
  - Sr-90

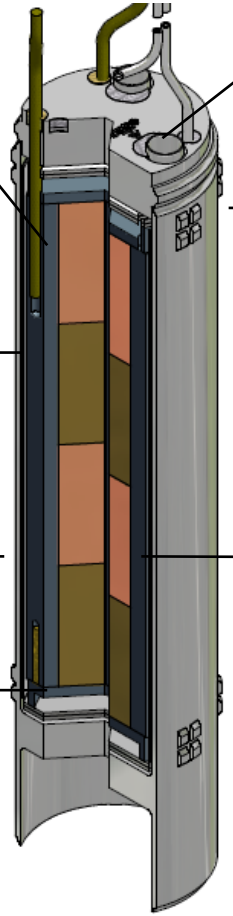
3 Through-tubes
- Leach
  - Gamma
  - ICP-MS
  - Sr-90

1 Shell:
- Leach
  - Gamma
  - ICP-MS
  - Sr-90

3 Flux Wires
- Gamma counting
- Nb wire dissolution and gamma count

1 Melt Wire
- Open package
- Observe if wire melted

2 Graphite Spacers (top and bottom):
- Gamma



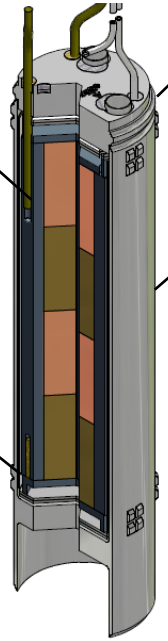
12 Fuel Compacts:
- Dimensional measurements
- Whole-compact PGS
- Burnup
- Optical/electron microscopy
- High-temperature safety testing
- Particle/**Compact** cross sectional analysis
- Re-irradiation and safety testing
- Deconsolidation-leach-burn-leach (DLBL)
- Particle gamma counting

Fission Product Mass Balance Outside of Fuel

- Capsule components from all capsules are analyzed for fission product inventory

1 Graphite Compact Holder
- **Precision gamma scanner (PGS)**
- Burn-leach of holder
  - Gamma
  - ICP-MS
  - Sr-90

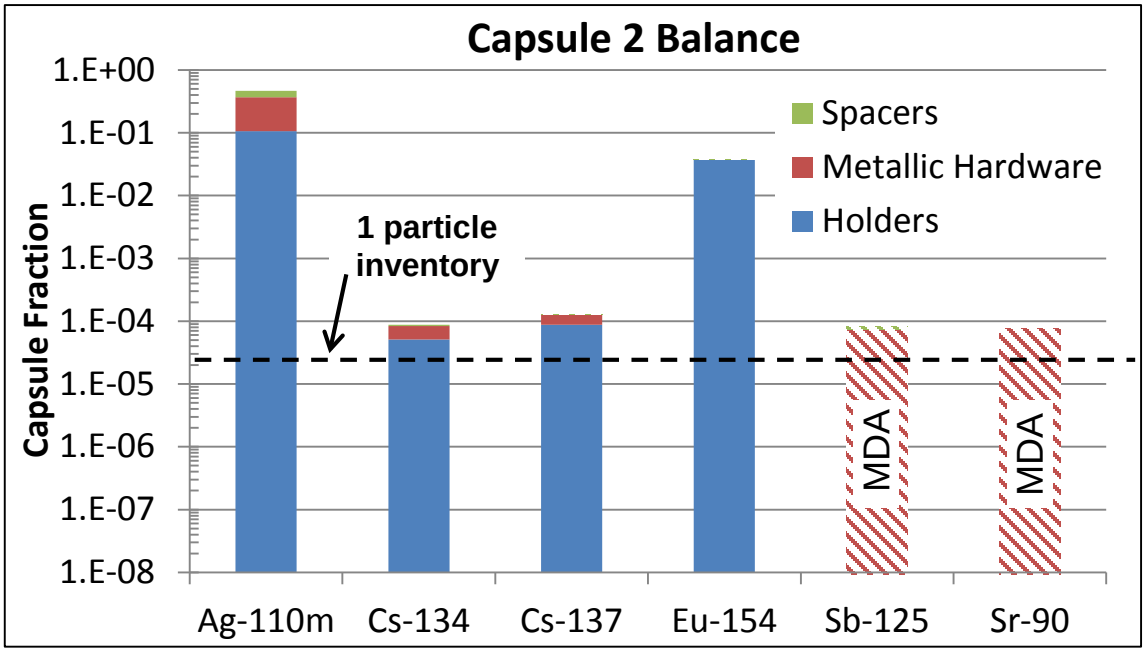
2 Graphite Spacers (top and bottom):
- **Gamma**



3 Through-tubes
- **Leach**
  - **Gamma**
  - **ICP-MS**
  - **Sr-90**

1 Shell:
- **Leach**
  - **Gamma**
  - **ICP-MS**
  - **Sr-90**

- **Complete**
- **In-progress**
- **Planned**



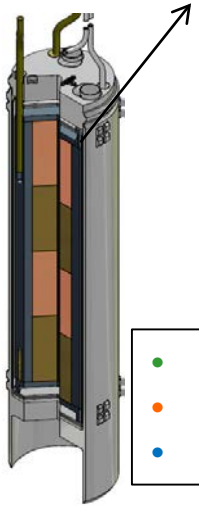
*MDA = minimum detectable activity. No activity was measured. This is the detection threshold.

PGS Helps Locate Potential Fuel Particle Defect

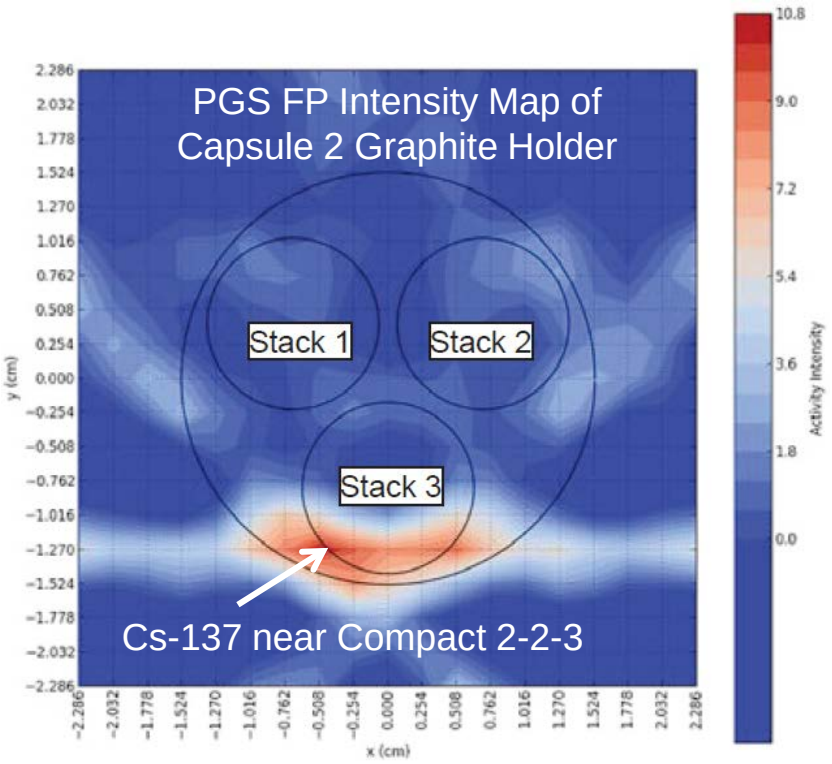
- PGS of graphite holder used to look for spots of elevated fission product activity, fuel compacts near hot spot can be targeted for additional PIE (e.g. Compact 2-2-3)
- Destructive PIE at ORNL found failed SiC layers in a few particles from Compact 2-2-3. Nickel contamination and migration from failed thermocouple near Compact 2-2-3 is believed to be the cause of these SiC failures. Not a fuel quality issue.

1 Graphite Compact Holder

- **Precision gamma scanner (PGS)**



- **Complete**
- **In-progress**
- **Planned**

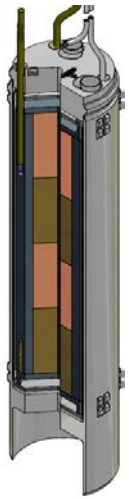


Neutron Fluence Wires and Melt Wires

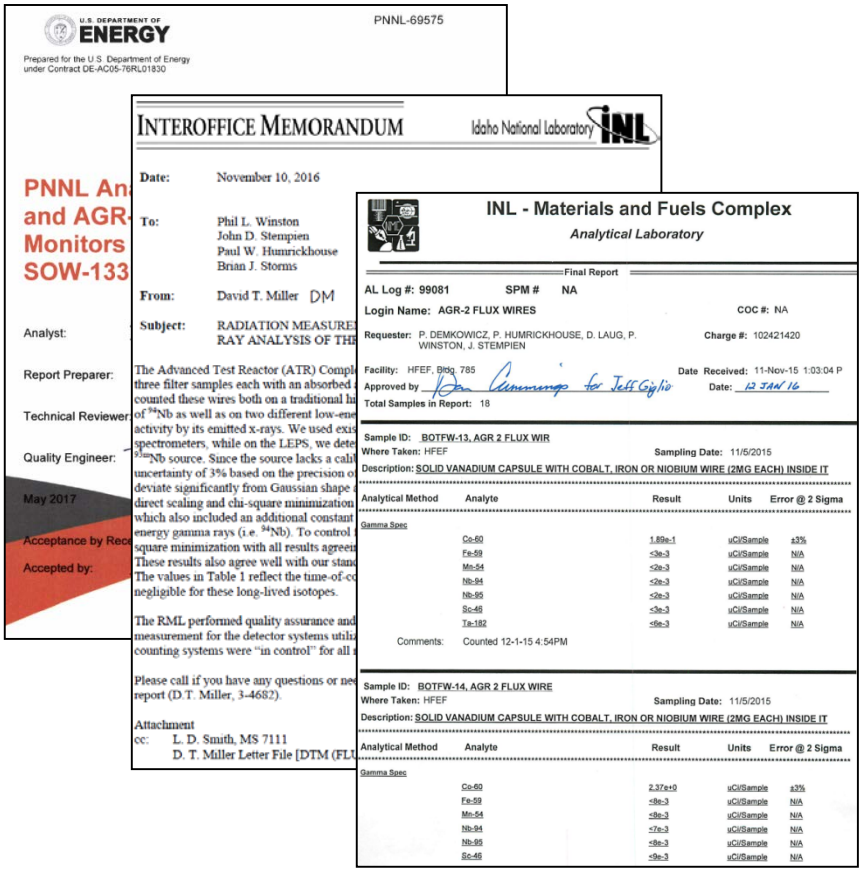
- Fluence wires counted at INL. Some sent to PNNL for comparison.
- Comparison of fluence derived from wires with predicted fluences in-progress
- Melt wires not successfully retrieved from AGR-2 holders; may attempt further inspections of holders for recovery

3 Flux Wires
- Gamma counting
- Nb wire dissolution and gamma count

1 Melt Wire
- Open package
- Observe if wire melted



- Complete
- In-progress
- Planned


U.S. DEPARTMENT OF ENERGY
PNNL-69575
Prepared for the U.S. Department of Energy under Contract DE-AC05-76RL01830

INTEROFFICE MEMORANDUM
Idaho National Laboratory INL

Date: November 10, 2016
To: Phil L. Winston, John D. Stempien, Paul W. Humrickhouse, Brian J. Storms
From: David T. Miller DM
Subject: RADIATION MEASUREMENT RAY ANALYSIS OF THE

INL - Materials and Fuels Complex
Analytical Laboratory
Final Report
AL Log #: 99081 SPM # NA
Login Name: AGR-2 FLUX WIRES COC #: NA
Requester: P. DEMKOWICZ, P. HUMRICKHOUSE, D. LAUG, P. WINSTON, J. STEMPIEN Charge #: 102421420
Facility: HFEF, Bldg. 785 Date Received: 11-Nov-15 1:03:04 P
Approved by: for Jeff Gyllio Date: 12 JAN 16
Total Samples in Report: 18

Sample ID: BOTFW-13, AGR 2 FLUX WIR
Where Taken: HFEF Sampling Date: 11/5/2015
Description: SOLID VANADIUM CAPSULE WITH COBALT, IRON OR NIOBIUM WIRE (2MG EACH) INSIDE IT

Analytical Method	Analyte	Result	Units	Error @ 2 Sigma
Gamma Spec	Co-60	1.89e-1	uCi/Sample	±3%
	Fe-59	<3e-3	uCi/Sample	N/A
	Mn-54	<2e-3	uCi/Sample	N/A
	Nb-94	<2e-3	uCi/Sample	N/A
	Nb-95	<2e-3	uCi/Sample	N/A
	Sc-46	<3e-3	uCi/Sample	N/A
	Ta-182	<6e-3	uCi/Sample	N/A

Comments: Counted 12-1-15 4:54PM

Sample ID: BOTFW-14, AGR 2 FLUX WIRE
Where Taken: HFEF Sampling Date: 11/5/2015
Description: SOLID VANADIUM CAPSULE WITH COBALT, IRON OR NIOBIUM WIRE (2MG EACH) INSIDE IT

Analytical Method	Analyte	Result	Units	Error @ 2 Sigma
Gamma Spec	Co-60	2.37e+0	uCi/Sample	±3%
	Fe-59	<8e-3	uCi/Sample	N/A
	Mn-54	<8e-3	uCi/Sample	N/A
	Nb-94	<7e-3	uCi/Sample	N/A
	Nb-95	<8e-3	uCi/Sample	N/A
	Sc-46	<9e-3	uCi/Sample	N/A

Fuel Compact Analyses

- Majority of PIE focuses on fuel compacts
- Large test/exam matrix
  - Each capsule had different burnup/irradiation temperature ranges
  - One capsule had UO_2 fuel for comparing to AGR UCO fuel performance

12 Fuel Compacts per Capsule:

- **Dimensional measurements**
- **Whole-compact PGS**
- Burnup
- High-temperature safety testing
- Optical/electron microscopy
- Particle/**Compact** cross sectional analysis
- Re-irradiation
- Deconsolidation-leach-burn-leach (DLBL)
- Particle gamma counting

- **Complete**
- **In-progress**
- **Planned**

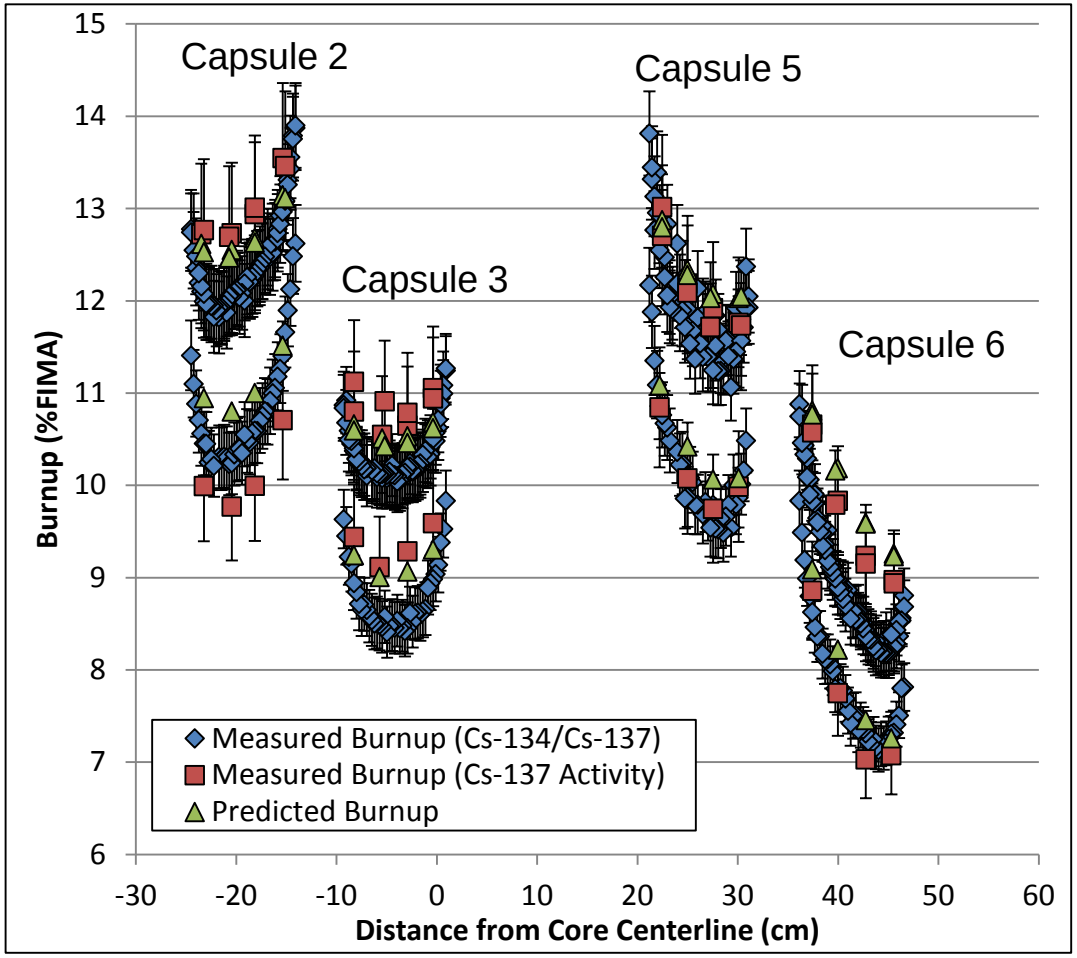
Fuel Compact PGS

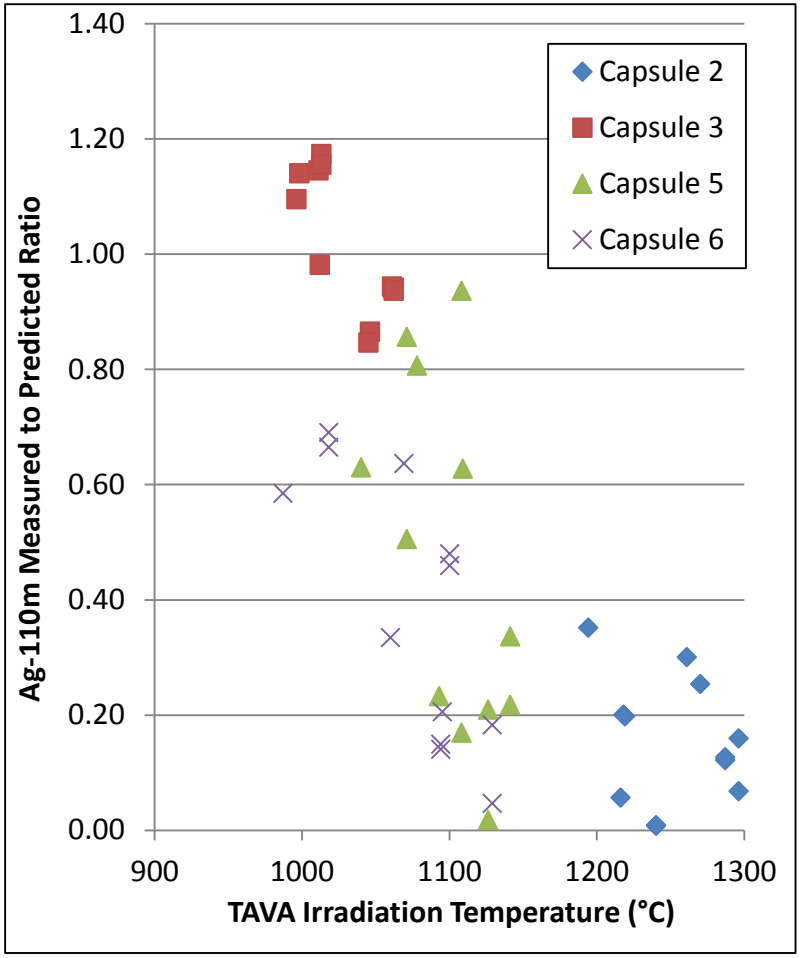
- Complete
- In-progress
- Planned

12 Fuel Compacts per Capsule:

- Whole-compact PGS
- Burnup by chemistry methods

- Estimate burnup
- Compare measured inventories to predicted (e.g. Ag-110m)





Fuel Compact Safety Testing, Destructive, and Microscopy

	Capsule 2 (UCO)												Capsule 3 (UO_2)												Capsule 5 (UCO)												Capsule 6 (UCO)											
	2-1-1	2-1-2	2-1-3	2-2-1	2-2-2	2-2-3	2-3-1	2-3-2	2-3-3	2-4-1	2-4-2	2-4-3	3-1-1	3-1-2	3-1-3	3-2-1	3-2-2	3-2-3	3-3-1	3-3-2	3-3-3	3-4-1	3-4-2	3-4-3	5-1-1	5-1-2	5-1-3	5-2-1	5-2-2	5-2-3	5-3-1	5-3-2	5-3-3	5-4-1	5-4-2	5-4-3	6-1-1	6-1-2	6-1-3	6-2-1	6-2-2	6-2-3	6-3-1	6-3-2	6-3-3	6-4-1	6-4-2	6-4-3
Shipment # to ORNL	7	4		2	2	1	3	3					5	5					1	1		5	1					7	3	2			4	2	3					7	7	5			4		4	
Safety Testing 1600°C																																															
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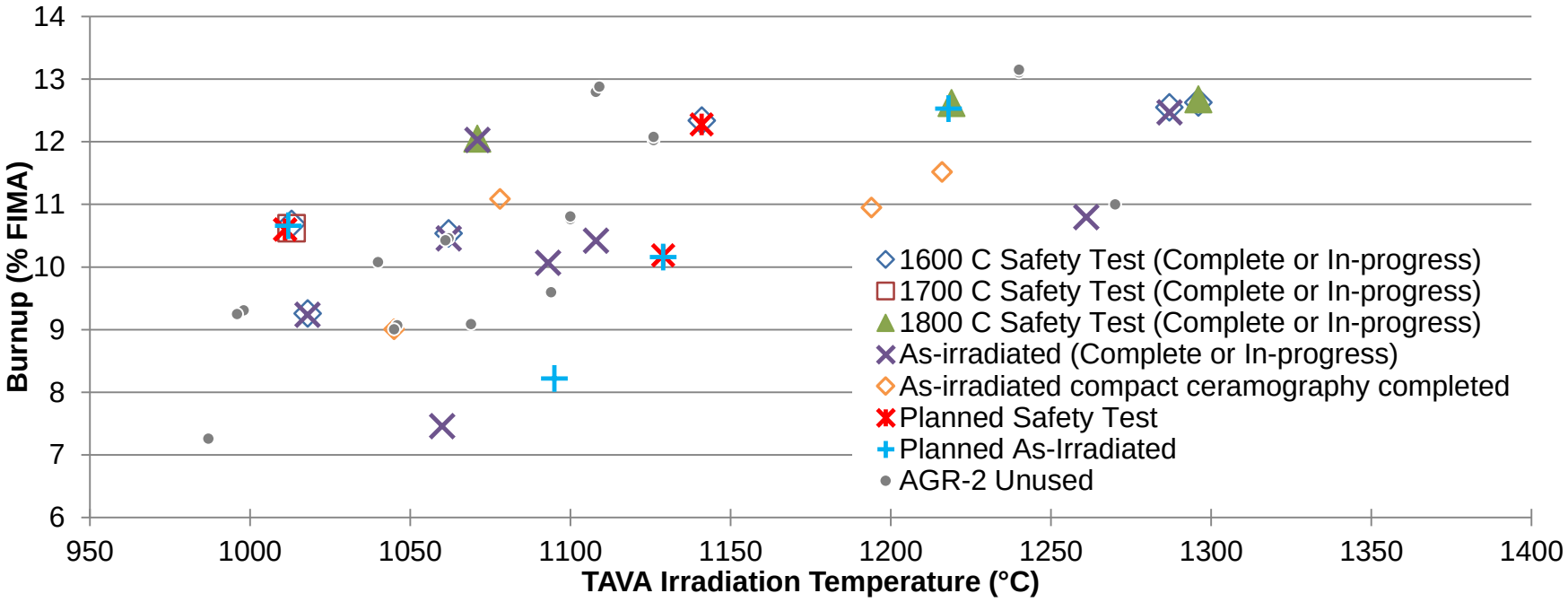
Safety Test (TBD)																																															
As-irradiated																																															
DLBL																																															
IMGA																																															
Basic micro analysis[a]																																															
Advanced microanalysis[b]																																															
Compact Ceramography																																															
Loose particle multi-level																																															
PIE Report																																															

DLBL: deconsolidation-leach-burn-leach
IMGA: irradiated microsphere gamma analyzer

[a] Optical microscopy and/or SEM/EDS
[b] Techniques include: TEM, STEM, HRTEM, EFTEM, EELS, APT, EBSD, EPMA, HRSTEM, HAADF, PED, etc. (See presentation from van Rooyen)

Compact Safety Testing, Destructive, and Microscopy Cover Range of Irradiation Conditions

- Completed 10 safety tests (3 in FY17 for **L2 Milestone** to "complete safety testing of 2 compacts")
- Completed 5 as-irradiated analyses (including 4 compact ceramography), 7 more in-progress
- The following presentations will elaborate on these activities in more detail:
  - "Re-irradiation Testing", Paul Demkowicz
  - "AGR-2 Fuel Compact Destructive Exams", John Hunn
  - "AGR-2 Safety Testing", Bob Morris
  - "AGR Advanced Microscopy", Isabella van Rooyen
  - "Long duration heating tests for silver release at intermediate temperatures," Tyler Gerczak

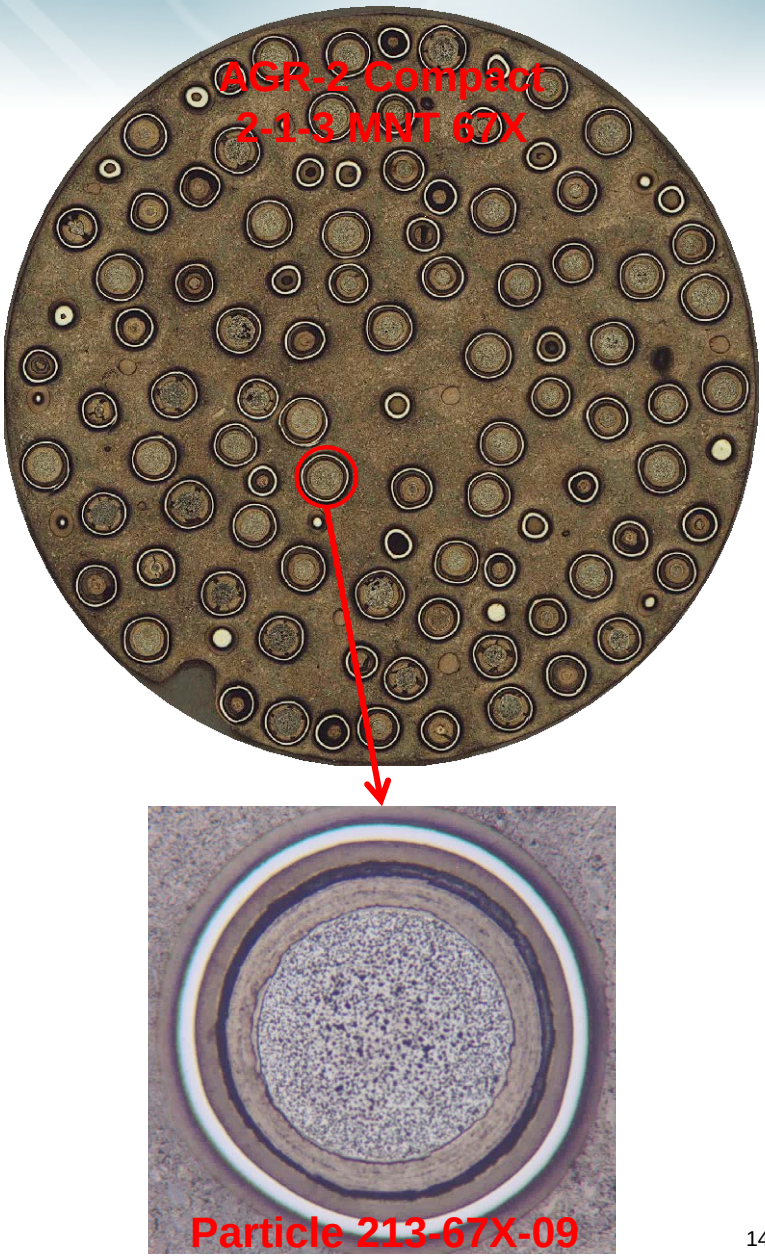


AGR-2 Compact Ceramography

- Examined 538 UCO particles
  - Compacts 2-1-3, 2-4-3, and 5-1-3
- Examined 102 UO_2 particles
  - Compact 3-2-3
- Compared to AGR-1:

	AGR-1	**AGR-2**
Partial IPyC Tears (%)	2.3	**3.5**
Through-layer IPyC Fracture (%)	3.5	**None**
Complete and Partial Buffer-IPyC Separation (%)	94	**100**
Buffer Fracture (%)	23	**20**

- Evidence that longer fluidization time between buffer layer deposition and IPyC layer deposition during fabrication promotes buffer-IPyC separation
- Easy and complete buffer-IPyC separation desirable
- Partial separation concentrates stresses at IPyC layer
- Also observed decreasing buffer fracture frequency as irradiation temperature increased above 1080°C to 1220°C



Summary

- PGS has been used to identify compacts that may have degraded SiC layers
  - 5 Compacts (2-2-3, 5-2-3, 5-3-3, 6-3-3, and 6-2-3)
  - 2-2-3: possibly 1 as-fabricated TRISO defect and 3 -5 SiC failures due to Ni contamination from a failed thermocouple
  - 5-2-3: possibly 2 or 3 particles with failed coatings
  - Others still in analysis
- Flux wires retrieved and counted. Comparison to to model predictions in-progress
- Completed Compact Analyses/Tests:
  - PGS of all compacts
  - 10 safety tests
  - Cross-section ceramography showing desirable enhanced buffer-IPyC separation
  - 10 as-irradiated compact analyses (including compact ceramography)

Future Work

- Fission product mass balance: ***remaining planned work*** is burn-leach of graphite holders for Sr-90
- Destructive compact analyses:
  - Safety testing: ***planning*** to complete **at least 3 more** safety tests before end of FY18
  - As-irradiated: ***planning*** to complete **at least 4 more** before end of FY18
- Particle analyses:
  - ***Planning*** to do cross sectional analysis of 100 particles from each of 4 compacts for kernel swelling and buffer shrinkage (beginning in FY17)
  - ***Continue*** advanced microscopy of kernel and layer samples from particles from 2 compacts
  - ***Begin*** long-term (~1000 hrs +) heating test of loose particles for long-term fission product release measurements

Questions and Discussion

John Stempien **Idaho National Laboratory**

john.stempien@inl.gov (208) 526-8410

AGR-2 Components and Associated PIE

- Each AGR-2 irradiation capsule has the following components and analyses:

1 Graphite Compact Holder
- Precision gamma scanning (PGS)
- Burn-leach of holder
  - Gamma
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3 Flux Wires
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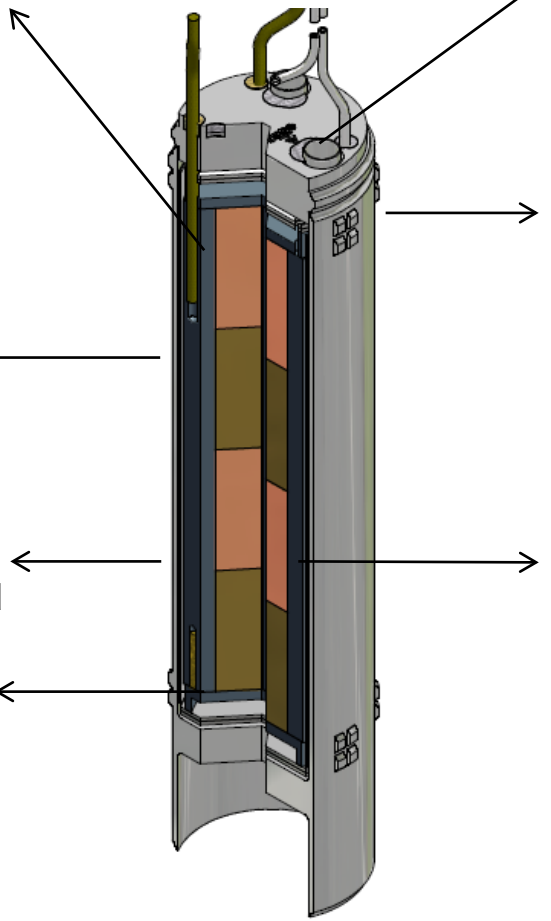

3 Through-tubes
- Leach
  - Gamma
  - ICP-MS
  - Sr-90

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- Re-irradiation
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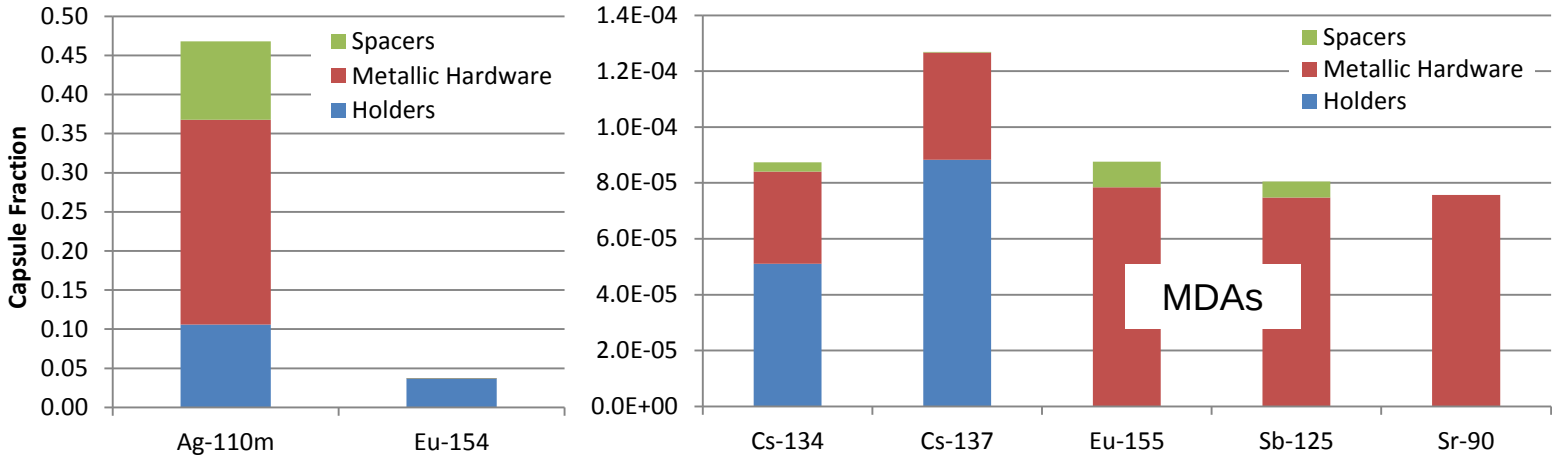
Example of Recent AGR-2 FP Analysis

- Capsule 2: UCO, TAVA Temp 1194°C



- Capsule 3: UO_2, TAVA Temp 1045°C