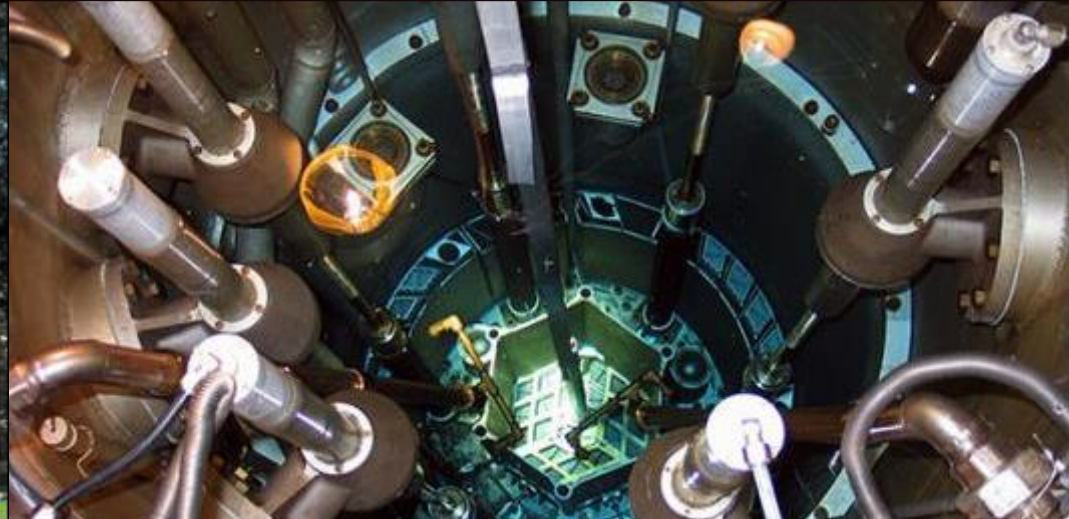


MIT NUCLEAR REACTOR LABORATORY

AN MIT INTERDEPARTMENTAL CENTER

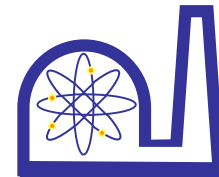


Experience with Tritium Evolution During Irradiation of MSRE Flibe in the MITR

David Carpenter

Group Leader, Reactor Experiments

10/27/15



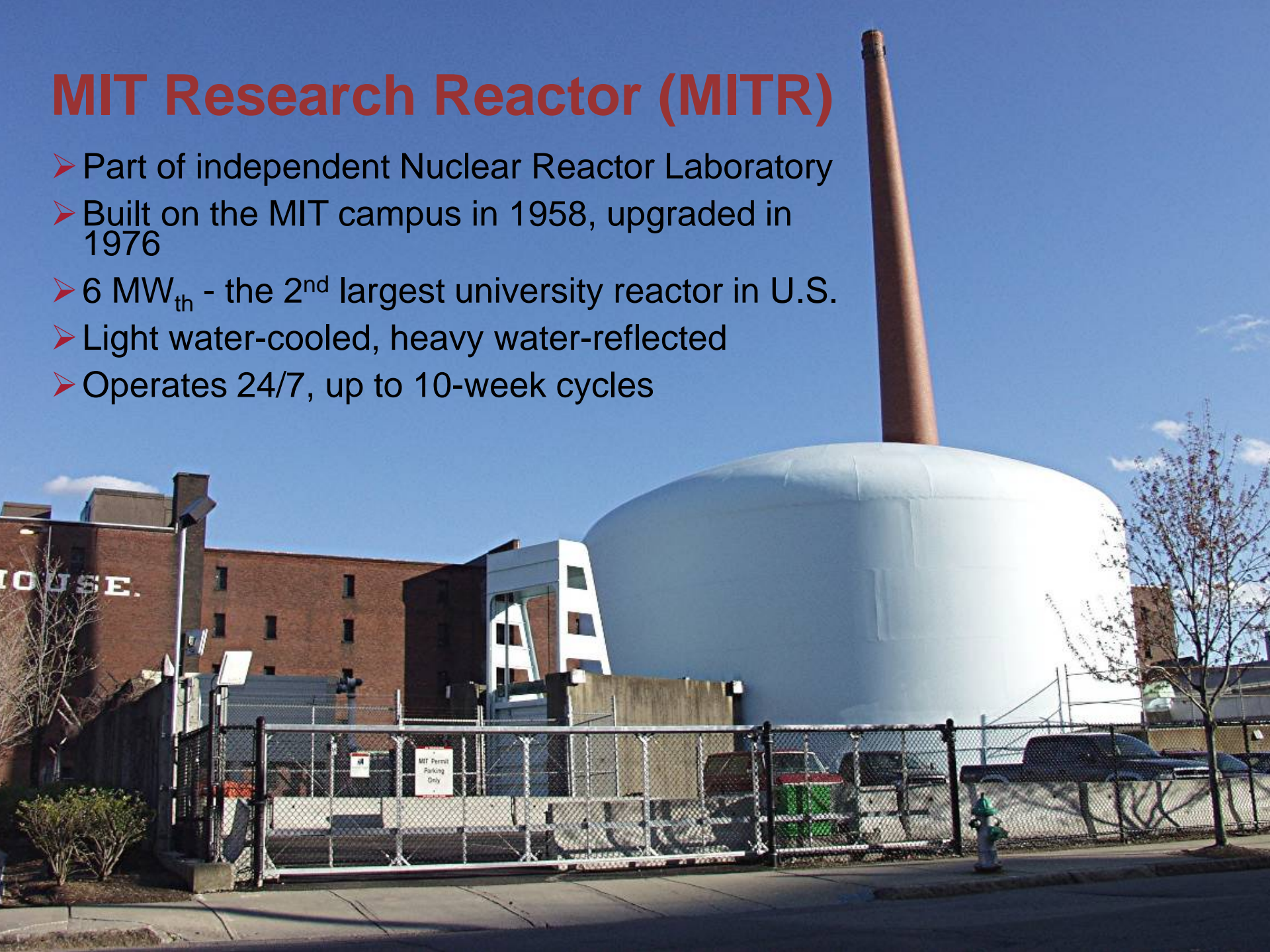
Outline

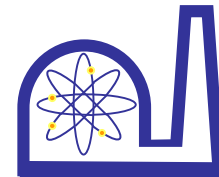
- MITR Introduction
- Tritium Experiments in the MITR
 - Goals
 - Limitations
- Tritium and Other Gas Release Measurements
 - FS-1
 - FS-2
- PIE Progress

- Please interrupt

MIT Research Reactor (MITR)

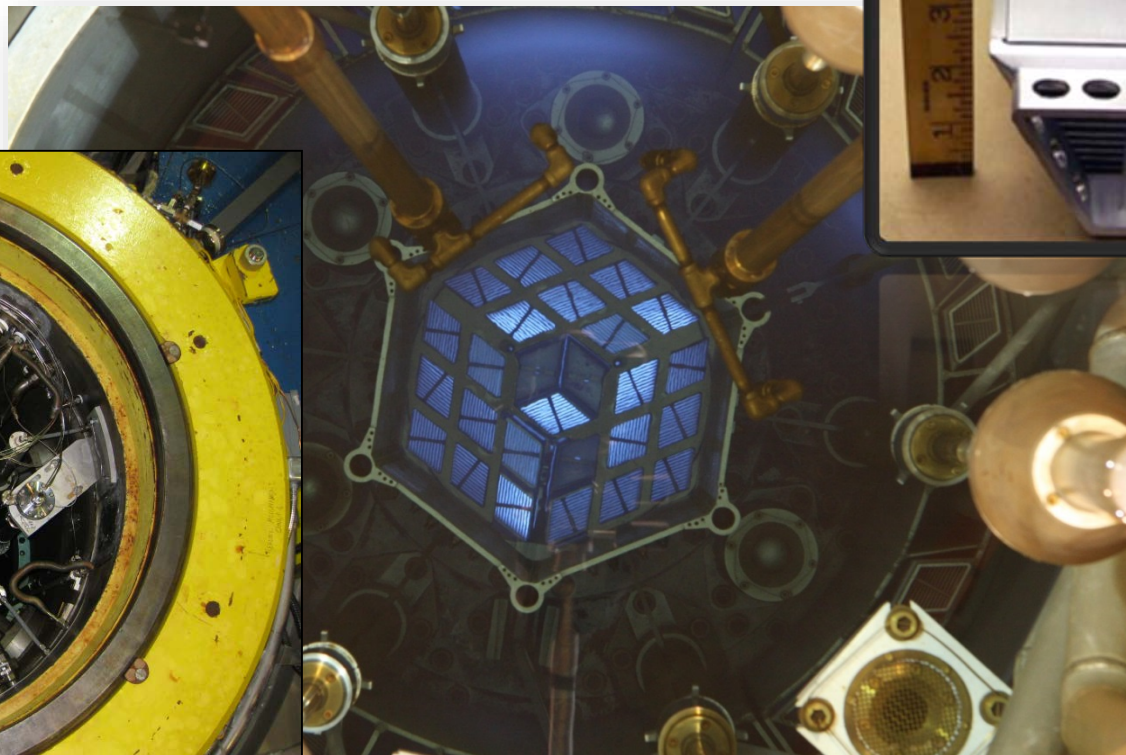
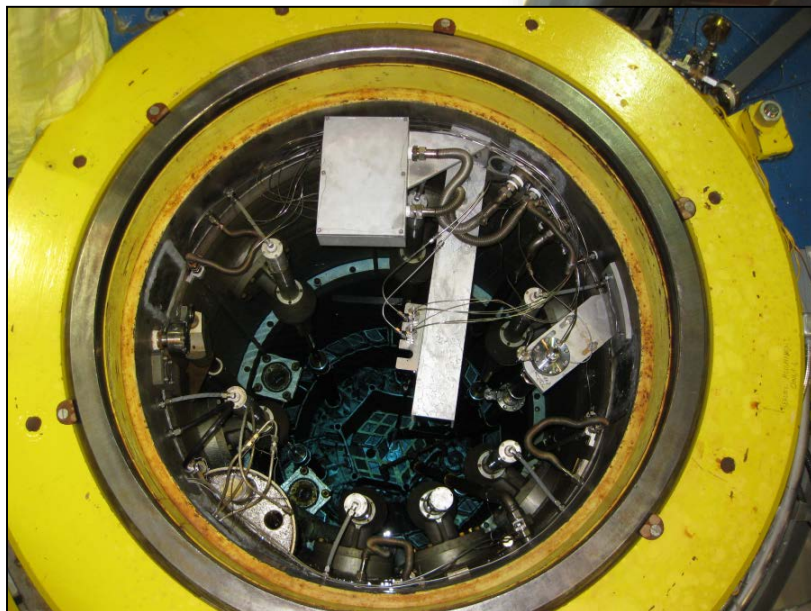
- Part of independent Nuclear Reactor Laboratory
- Built on the MIT campus in 1958, upgraded in 1976
- 6 MW_{th} - the 2nd largest university reactor in U.S.
- Light water-cooled, heavy water-reflected
- Operates 24/7, up to 10-week cycles

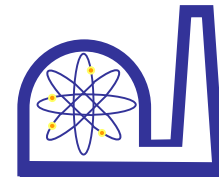




MITR Core

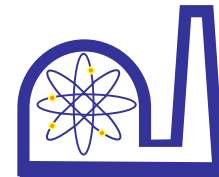
- 24 HEU rhomboidal elements with UAl_x plate-type fuel
- 50°C outlet, atmospheric pressure
- 3 dedicated in-core experimental positions





FHR IRP Irradiation Goals

- To test the interactions between the flibe and potential FHR fuel and structural materials
- To compare material test results with parallel test at University of Wisconsin (no irradiation)
- **To measure tritium production and partitioning among components**
- To evaluate the experimental components and methods for future FHR-related tests—the starting point that ultimately leads to larger experiments in HFIR and ATR



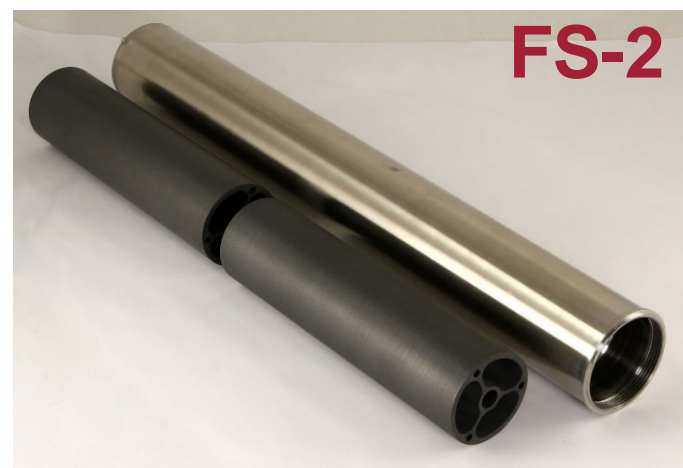
Initial Flibe Irradiations



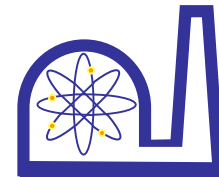
FS-1

- Study effect of different fluoride redox potentials on salt corrosion and tritium release
- Tritium and salt interaction with fuel compact graphite
- Cracking performance of TRISO particles
- 300 hours

- First in-core irradiation of MSRE flibe in 60 years
- Primary goal to identify potential safety and design issues for future experiments
- 1000 hours
- 700°C



FS-2



Irradiation Design Progress

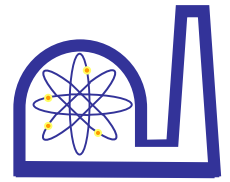
➤ Test conditions:

- 700°C
- Double encapsulation (Ni, Ti)
- He cover gas, He/Ne control gas
- 5.5MW reactor power

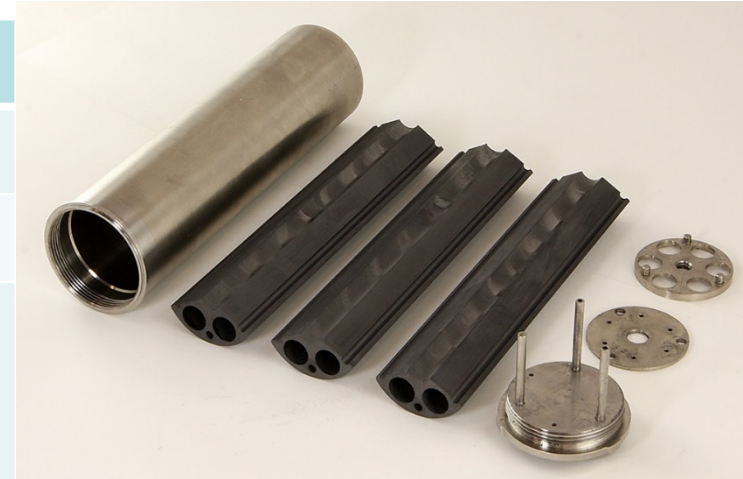
➤ FS-2 modifications:

- Independent irradiation facility
- 2.7x more salt volume
- Two fluoride potentials
- New C/C, graphite, and TRISO particles
- Improved gas flow and exhaust

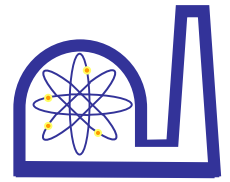
FS-1 Loading



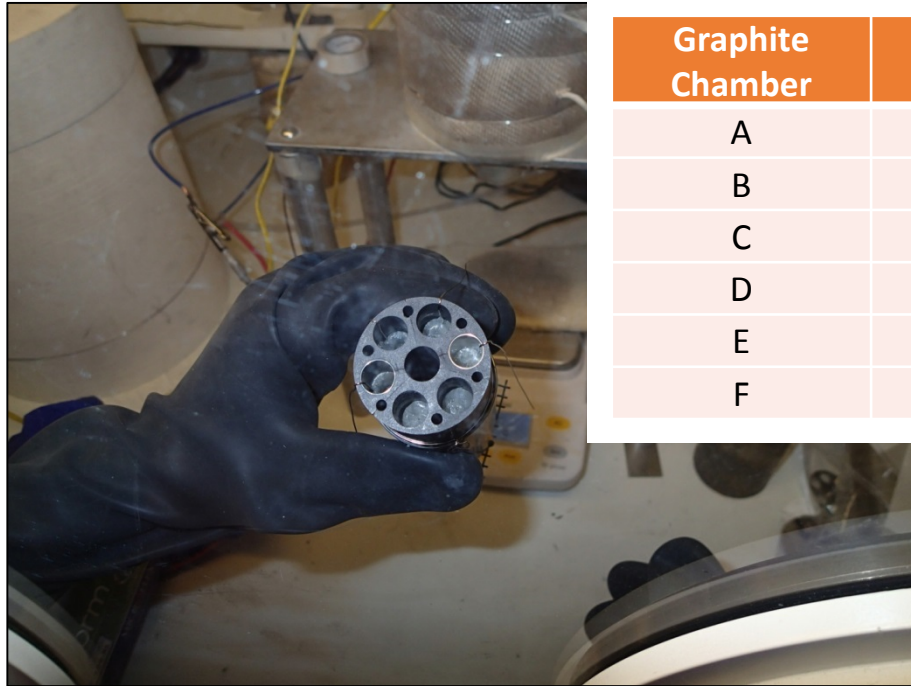
| Chamber | Container Material Combinations |
|---------|---|
| A | Two Hastelloy-N samples |
| B | Two 316 Stainless Steel samples |
| C | Three types of SiC samples: R&H CVD SiC SiC/SiC Tyranno-SA3 CVI SiC composites SiC/SiC Hi-Nicalon type-S CVI SiC composite |
| D | 300 surrogate TRISO particles |
| E | Two Hastelloy-N samples in nickel-lined hole |
| F | Two 316 Stainless Steel samples in 316SS-lined hole |



FS-1 Capsule Assembly



Graphite sample holder filled with samples and flibe



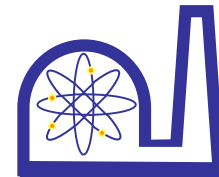
Flibe Loading

| Graphite Chamber | Flibe Mass (g) |
|------------------|----------------|
| A | 21.3 |
| B | 21.2 |
| C | 21.2 |
| D | 21.2 |
| E | 18.2 |
| F | 18.1 |

Capsule sealed with TCs and gas sampling lines

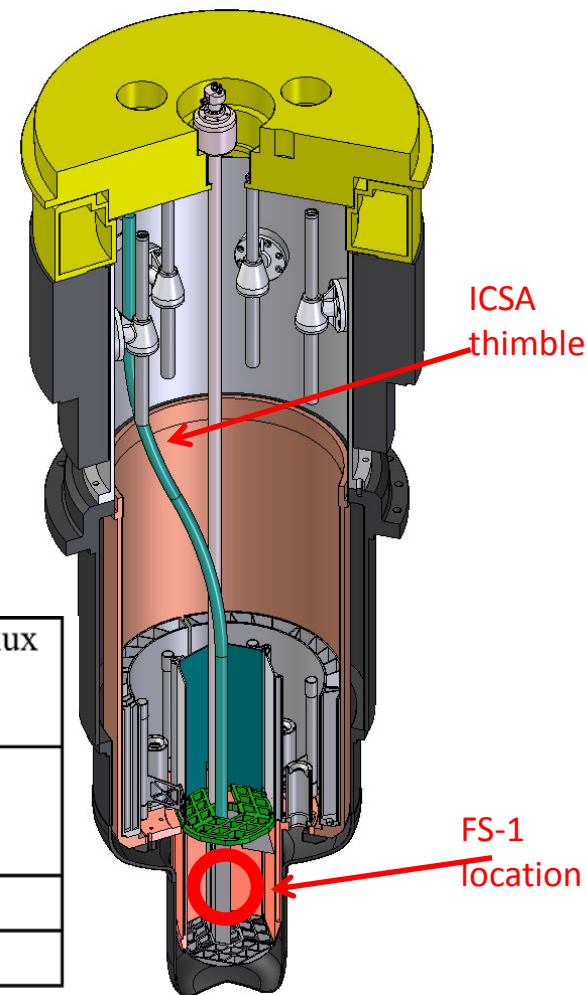


Samples in FS-1 are all identical to UW corrosion tests



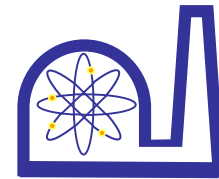
FS-1 Capsule Irradiation

- The FS-1 capsule runs inside the MITR In-Core Sample Assembly (ICSA) thimble
 - The ICSA is an instrumented inert gas irradiation facility in a central core position
- Separate gas flow in capsule and thimble
- Thermocouples in graphite for temperature measurement



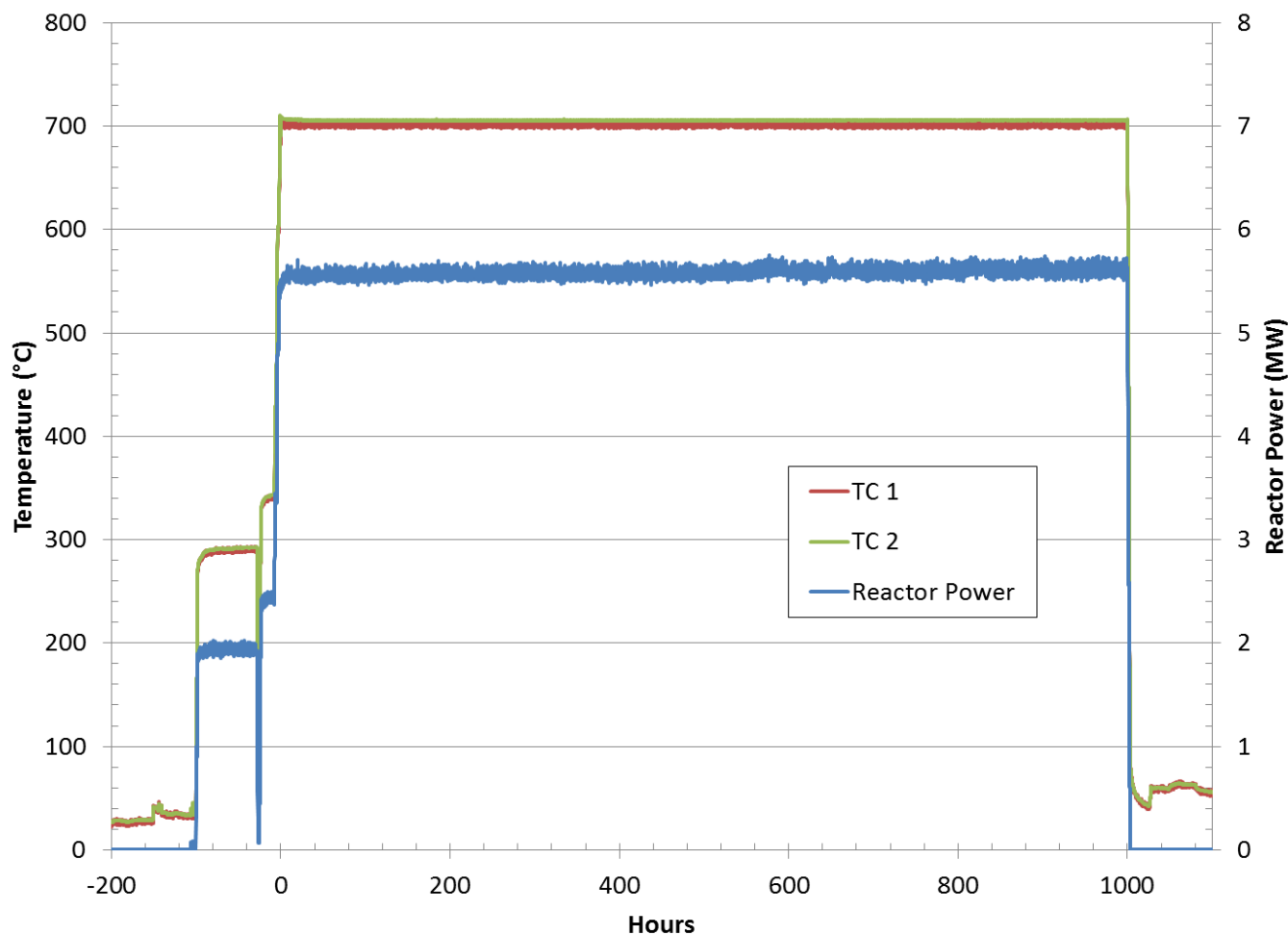
| | Fast flux (n/cm ² -sec) (E > 0.1 MeV) | Total neutron flux (n/cm ² -sec) | Fast flux/Total flux ratio, % |
|--|--|--|----------------------------------|
| FS-1 Flibe compartments (5.5 MW) | 1.06×10^{14} | 1.38×10^{14} | 76.8 |
| 2009 PB-AHTR* | 6.95×10^{13} | 4.04×10^{14} | 17.2 |
| 2011 AHTR* | 6.18×10^{13} | 3.64×10^{14} | 17.0 |

* FHR Materials, Fuels and Components White Paper, UCBTH-12-003, July 2013.

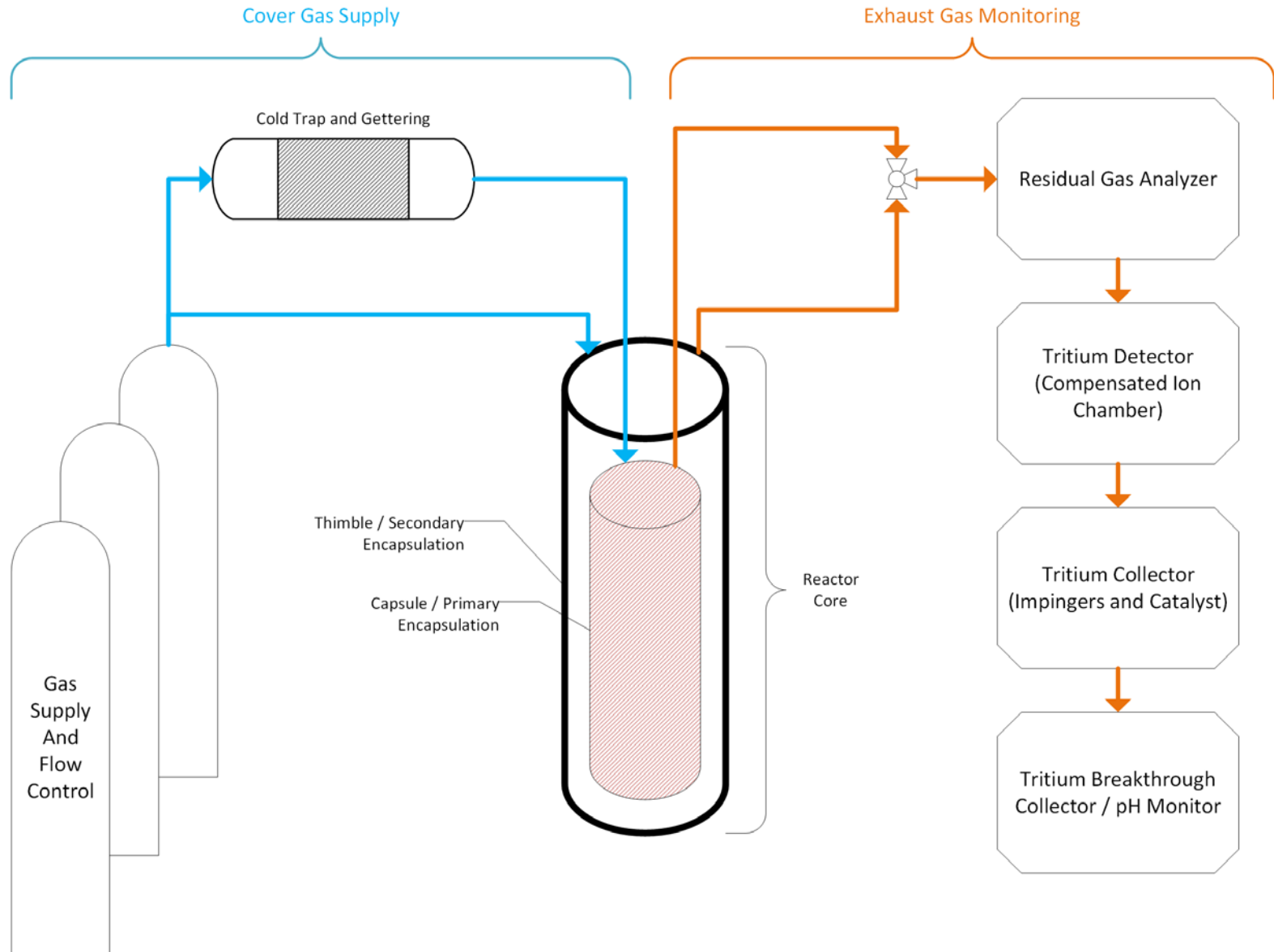
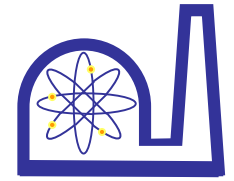


FS-1 Irradiation History

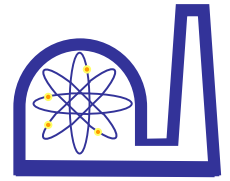
- Slow, stepped startup with fine control through melting
- Experimented operated at $700 \pm 3^\circ\text{C}$ for 1000 hours



ICSA Irradiation Facility



Tritium Measurements



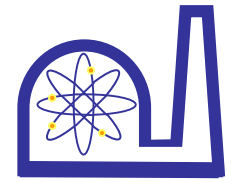
- First irradiations attempted combination of real-time and integrated measurements
 - Dual compensated ion chambers used to measure real-time tritium activity
 - Six-pass water bubbler system with catalytic furnace to capture tritium in water for later liquid scintillation counting (LSC)
- This system should allow separate collection of soluble (TF, HTO, T₂O) and non-soluble (HT, T₂) species
- Bubbling with LSC has been highly repeatable with good capture efficiency, and allows differentiation of H-3, C-14 activity



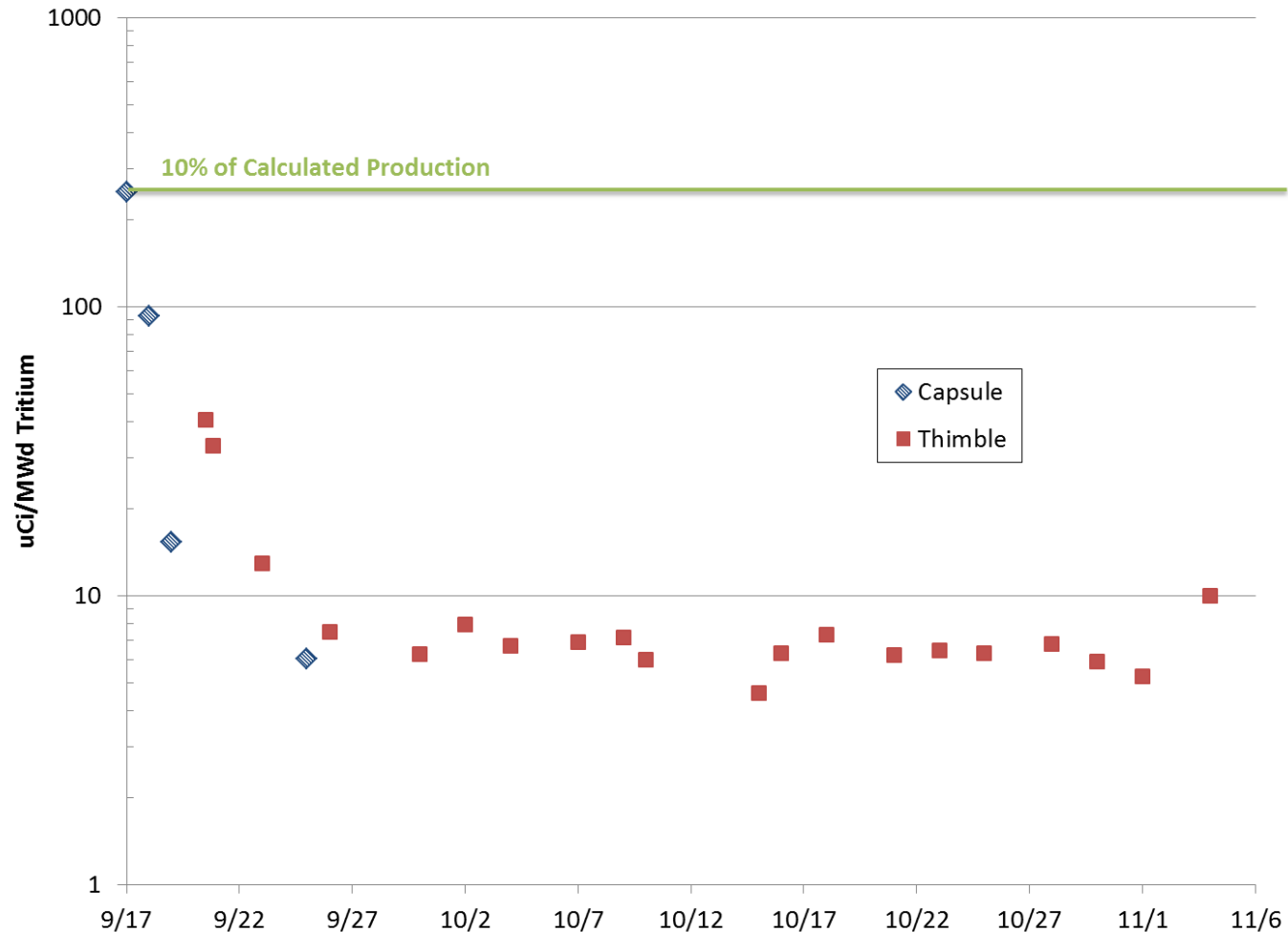
- Released activity below range of ion chamber sensitivity/background rejection



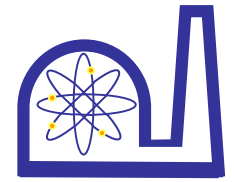
FS-1 Initial Findings – Tritium Activity



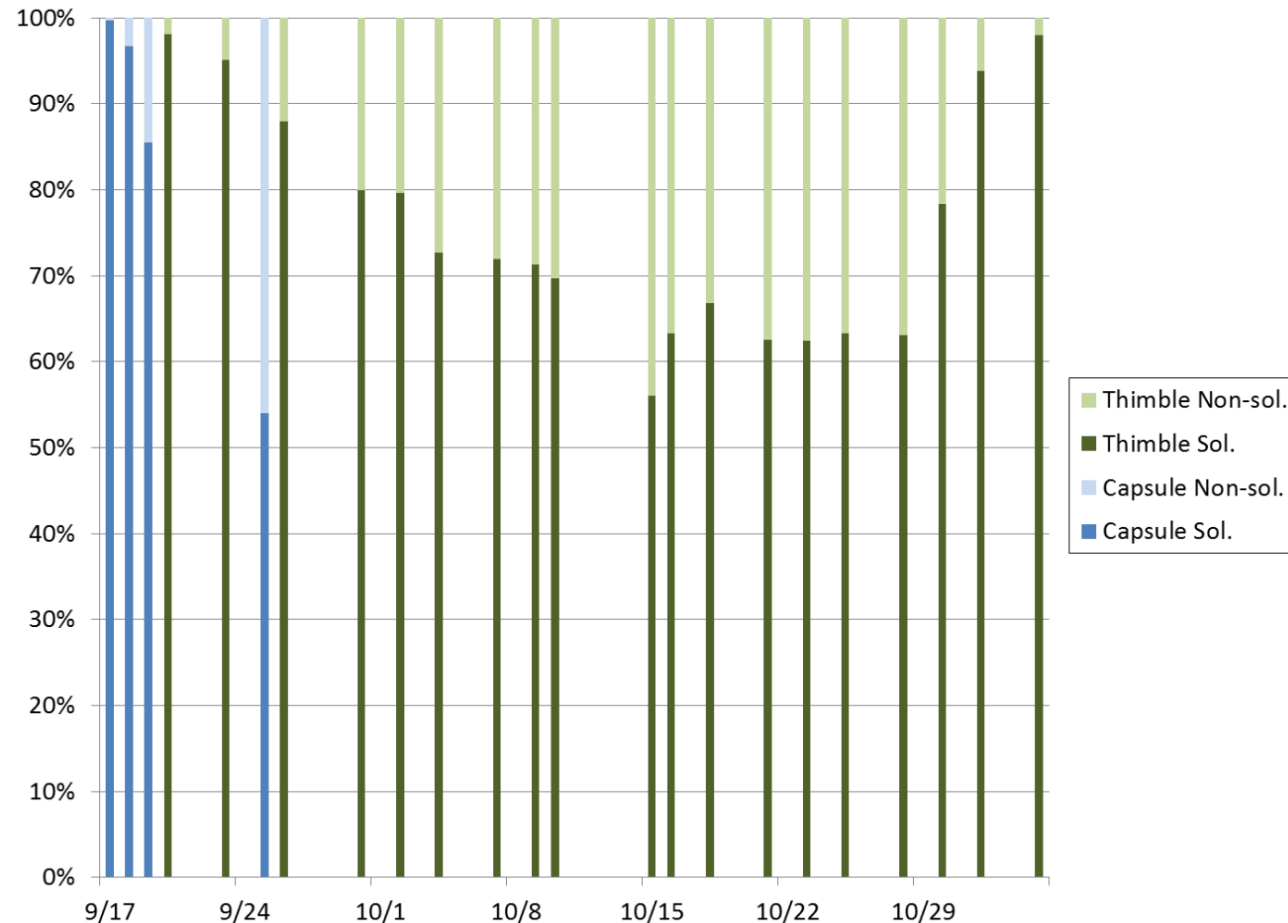
- Tritium counts integrated over 24-48 hour intervals
- Tritium collected during startup was ~10% of predicted production
- Subsequently, collected less than 1% of production
- Capsule and thimble tritium levels similar

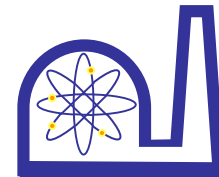


FS-1 Initial Findings - Solubility



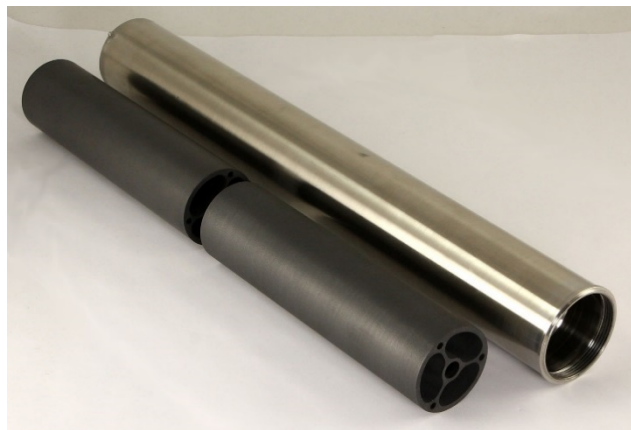
- Tritium collected in initial impingers expected to be only water-soluble form,
- Post-catalyzing furnace the previously non-soluble species will be captured
- Only non-soluble species (T_2) should be released through diffusion
- Additional tests are required to identify species to explain apparent contradiction





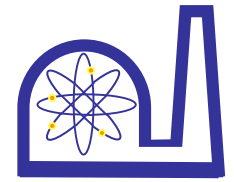
FS-2 Loading

- Added C/C and matrix graphite, removed Hastelloy
- Redox potentials straddle the historical MSRE potential (-665 to -707 kJ/mol F₂)



| Graphite Holder | Compartment | Material | Flibe F ₂ Potential (kJ/mol) |
|-----------------|-------------|----------------|---|
| Upper | A | Graphites | -632 |
| | B | SiC/SiC, TRISO | -632 |
| | C | SiC/SiC, TRISO | -711 |
| Lower | A | C/C | -632 |
| | B | SS316 | -632 |
| | C | SS316 | -711 |

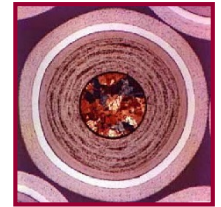
FS-2 Loading



ARB-B1-Z009



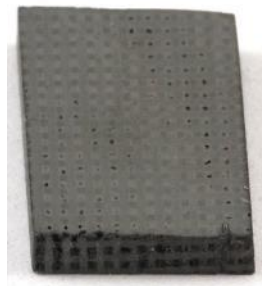
IG-110U



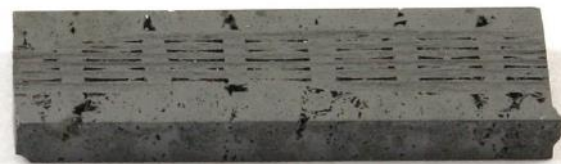
B&W TRISO
G73F-NF-93044



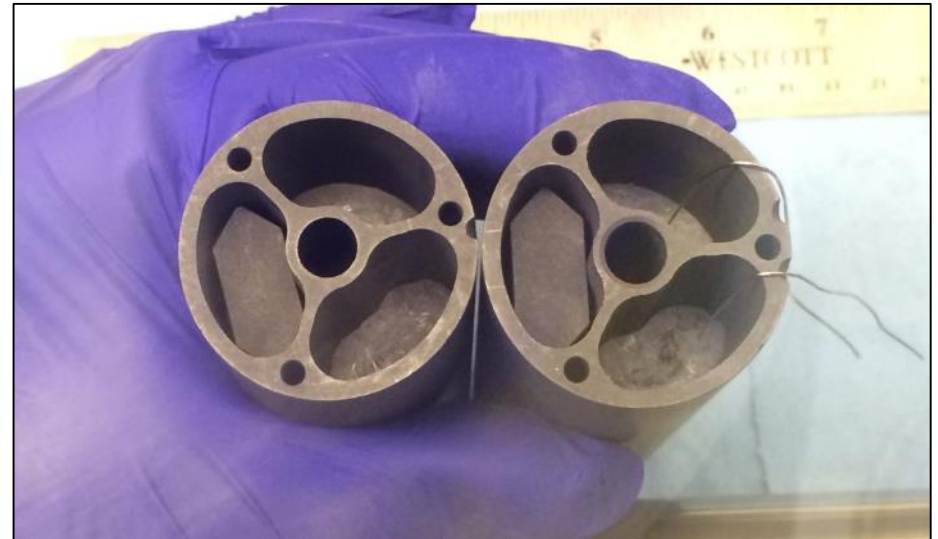
SS316



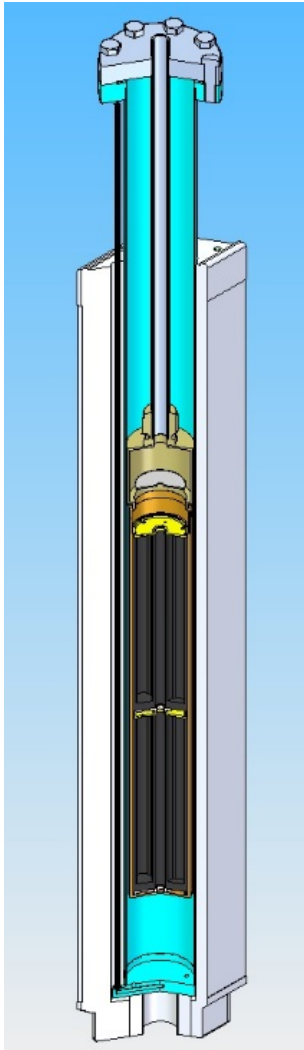
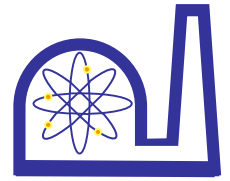
C/C



SiC/SiC



FS-2 In-Core Facility Design



Legend:

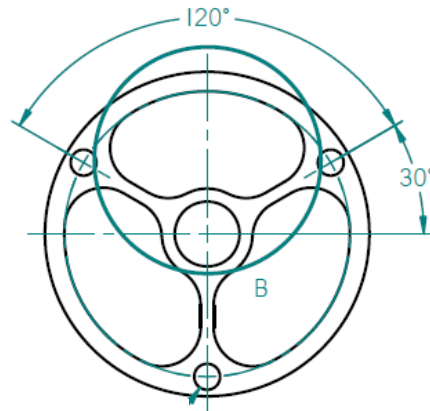
White – Aluminum dummy element

Blue – Titanium thimble

Orange – Nickel capsule

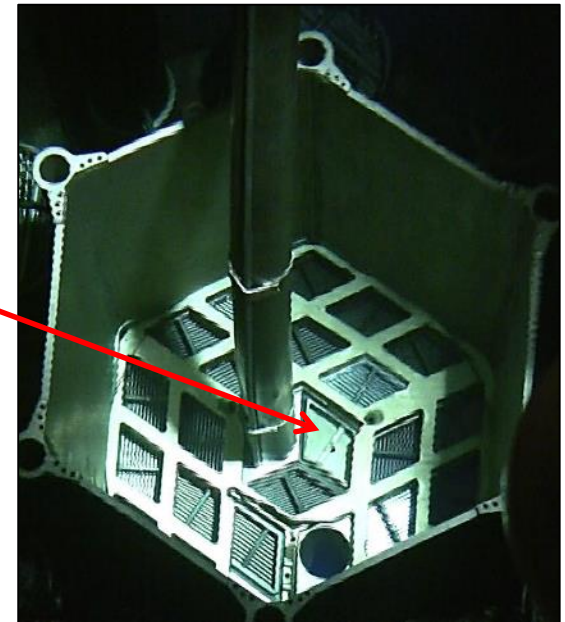
Yellow – Internal nickel parts

Gray – Graphite sample/flibe holder

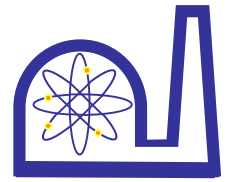


Graphite cross-section

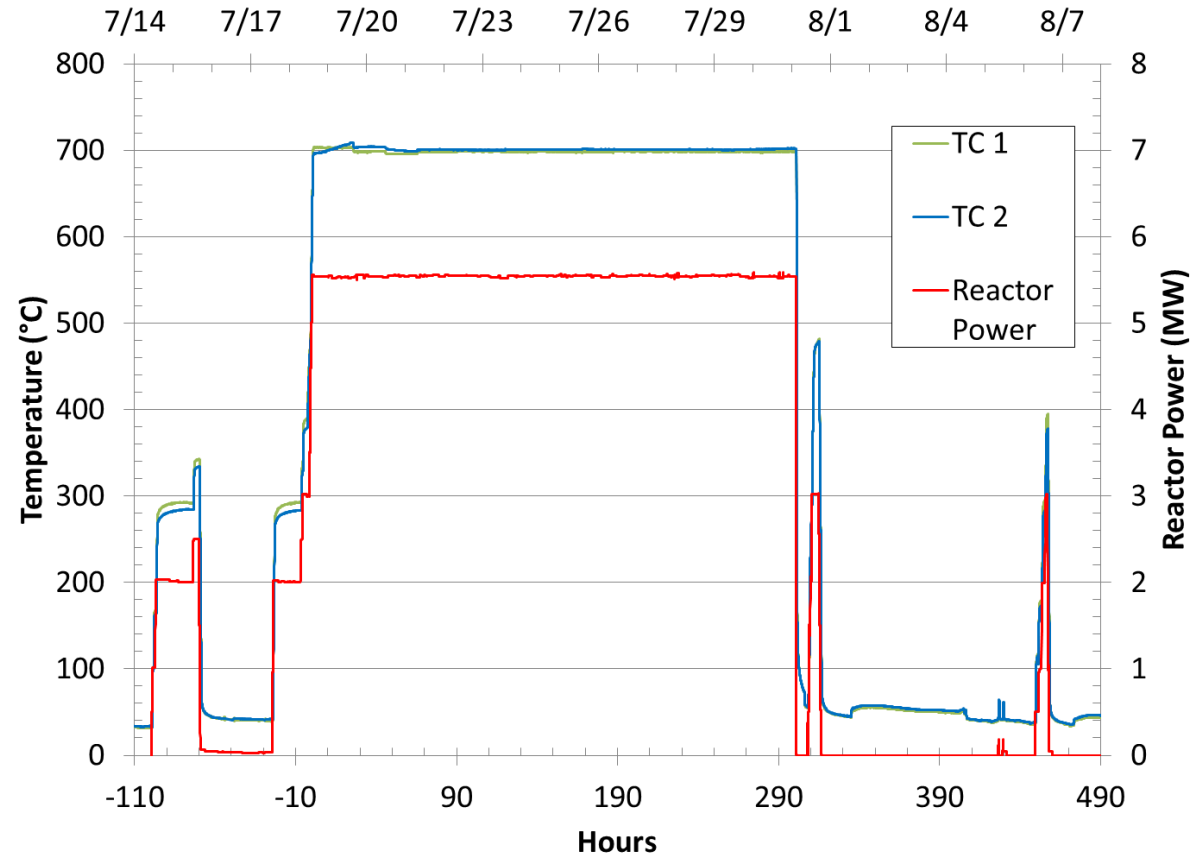
FS-2 Position
(A-1)



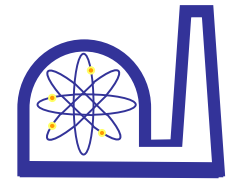
FS-2 Irradiation History



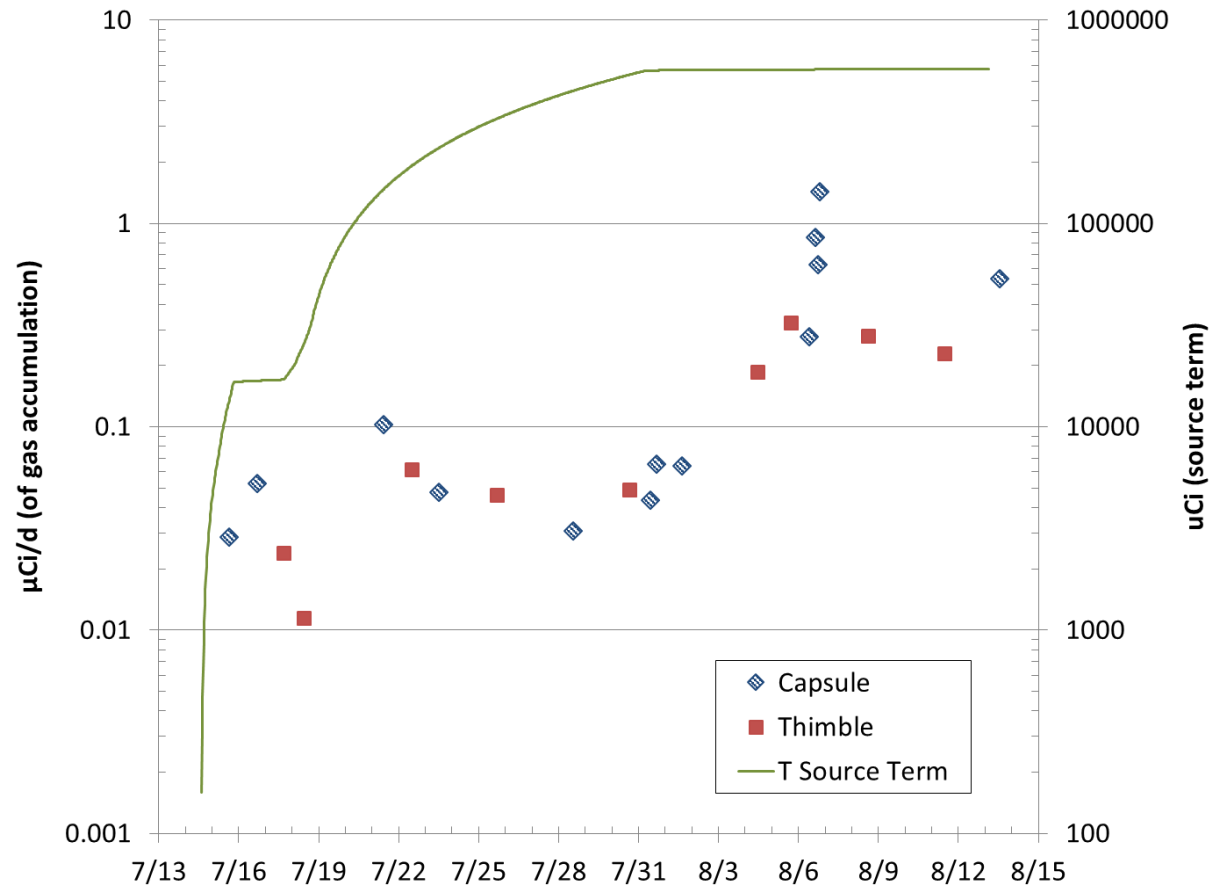
- First startup did not reach the flibe melting point
 - Reduction in power to 50kW for maintenance (~50°C)
- Eventual startup to 700°C was uneventful
- Unrelated reactor SCRAM at 300hrs, restart attempt
- After several days of troubleshooting, performed short test to 3MW, then removed experiment



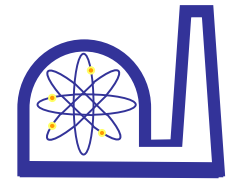
Tritium Release vs. Source Term



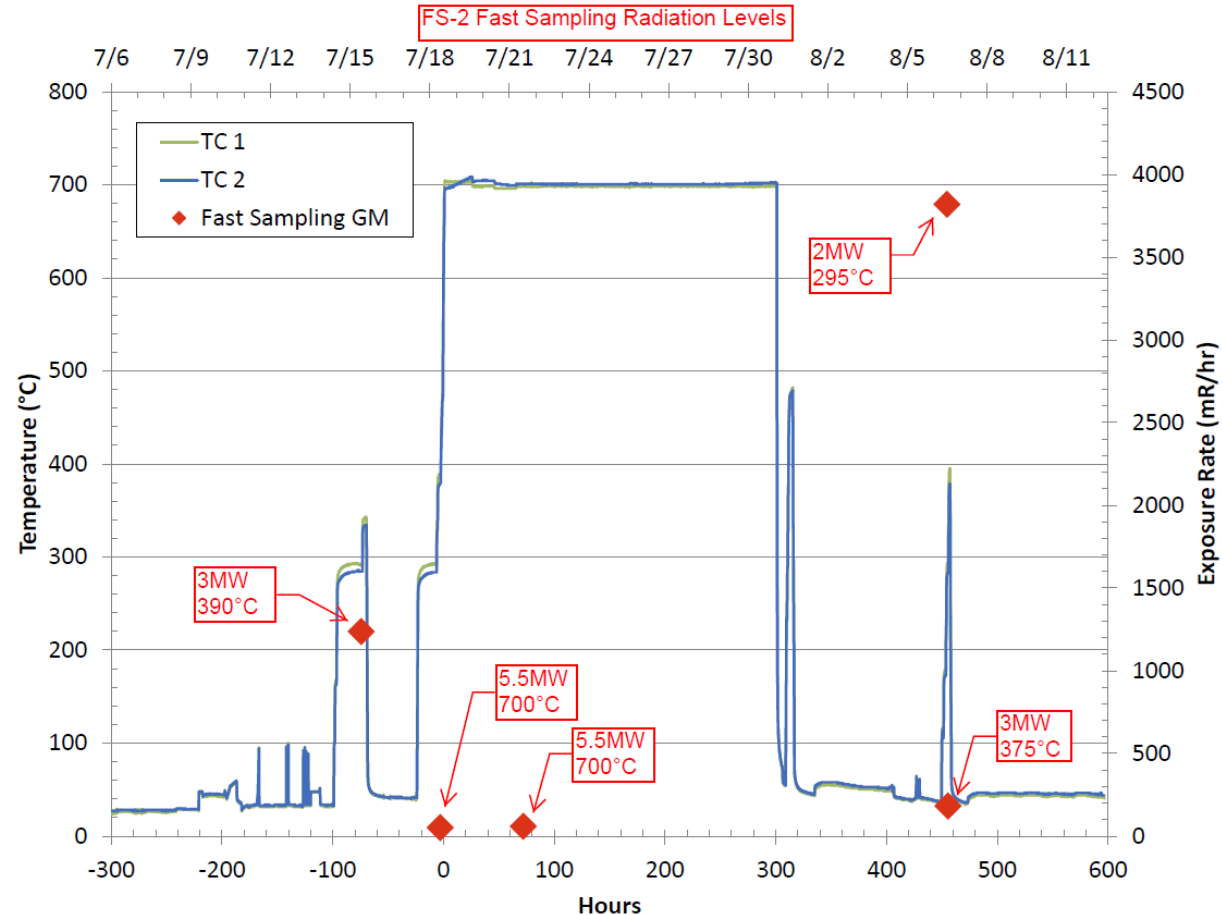
- Capsule and thimble gas tritium levels similar
 - Easy diffusion through capsule
- Majority of tritium is immediately soluble in water
 - Additional catalyzing had small effect
- Tritium release rate increases during temperature changes
- Tritium release during normal operation (5.5MW, 700°C) is same order of magnitude as FS-1 (<1% of generation)



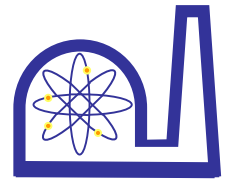
Short Half-Life Detection



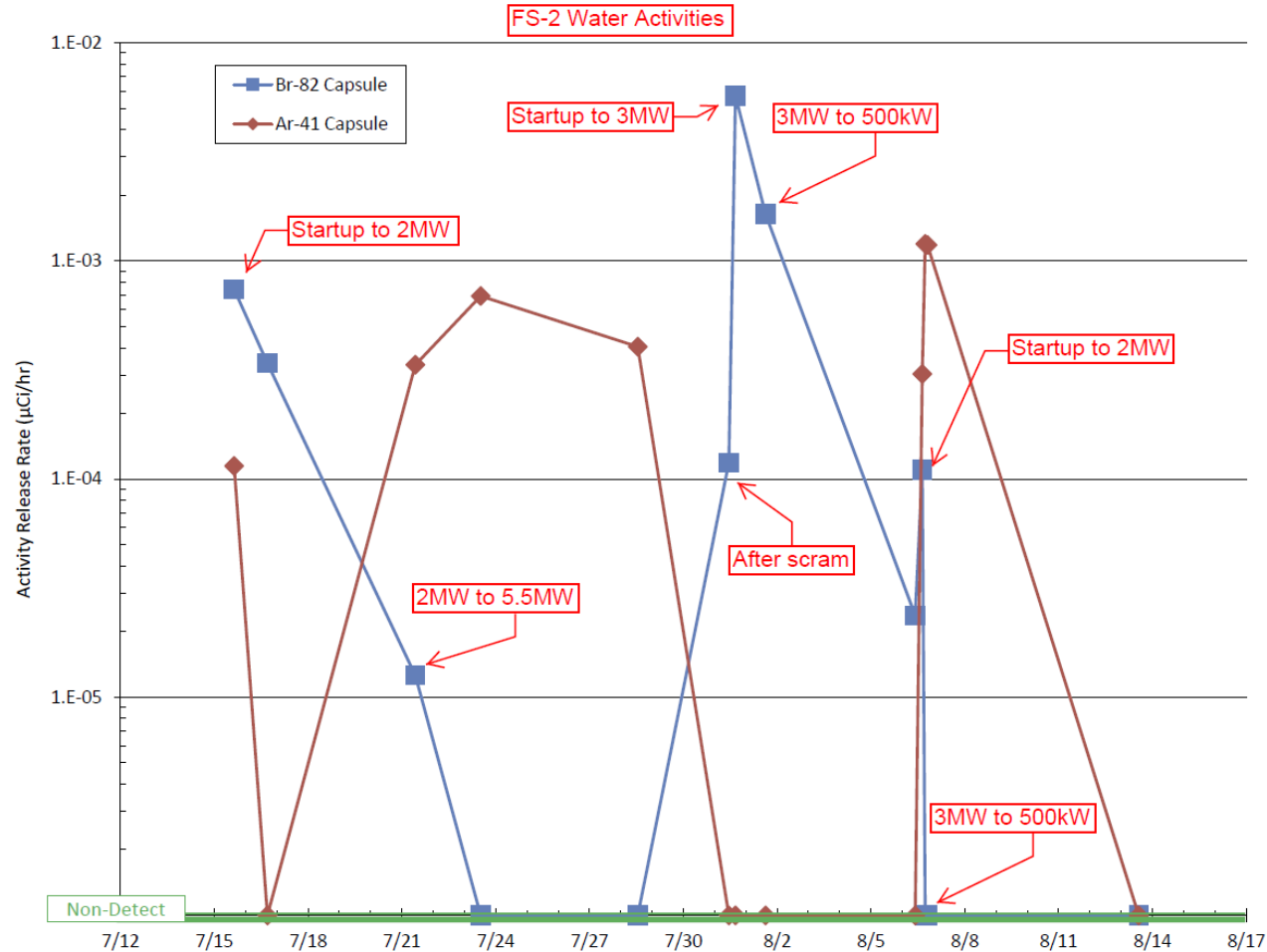
- In FS-1, problem of short-lived activation products in capsule exhaust
 - Added special gas system for FS-2
- In FS-2, highest N-16 and O-19 release when flibe was solid
 - Produced by F-19 neutron activation
- Almost no detectable release at full reactor power, 700°C



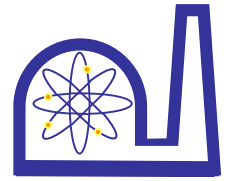
Bromine Release



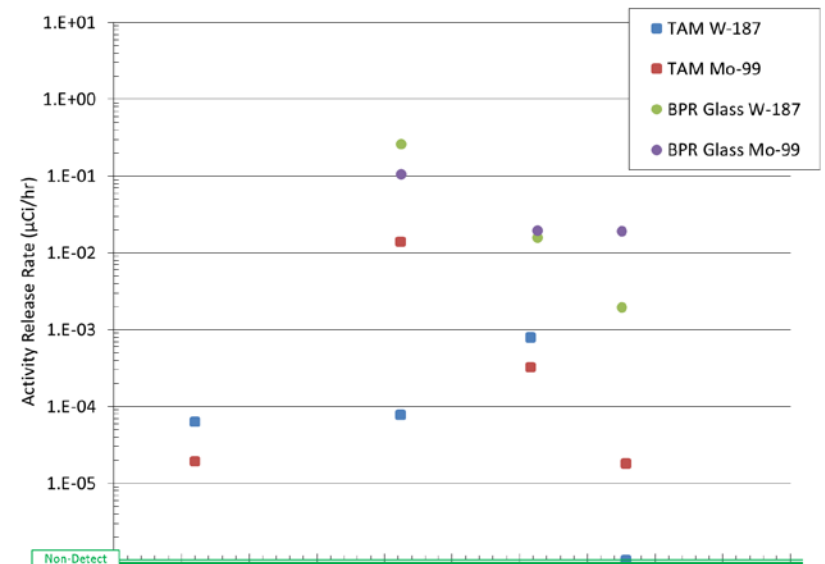
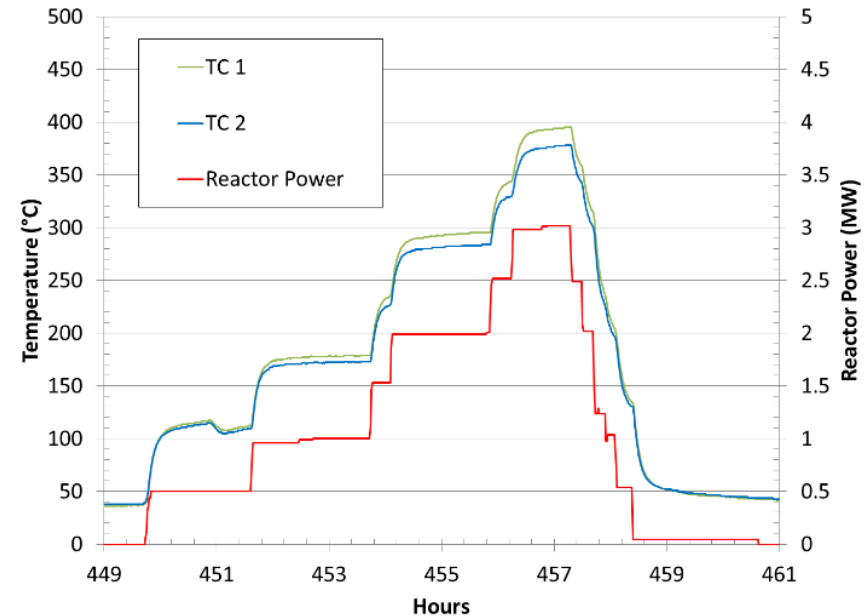
- Bromine was detected in tritium collection system
 - Only from capsule gas
 - Here contrasted with Ar-41, an expected He impurity
- Highest Br levels when at low power – flibe is solid
- Not detected at full power, 700°C

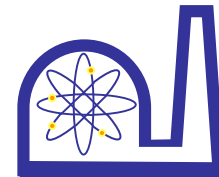


FS-2 Gaseous Release Test



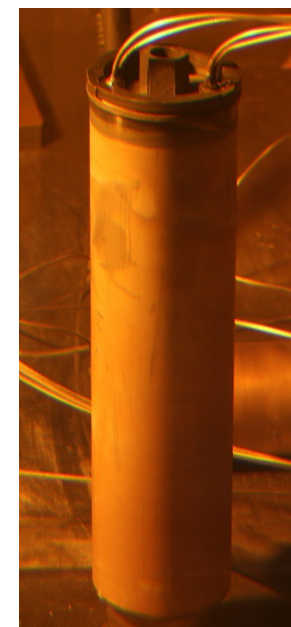
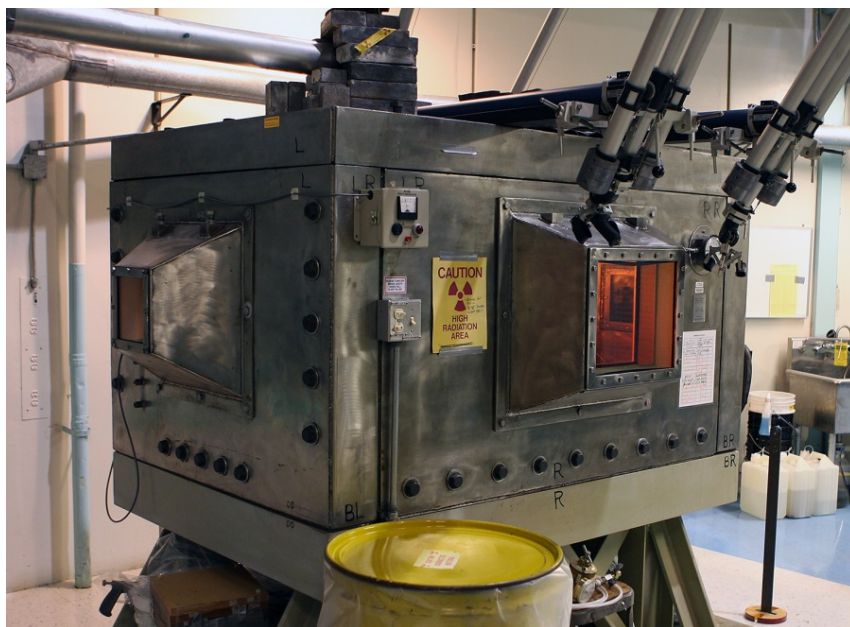
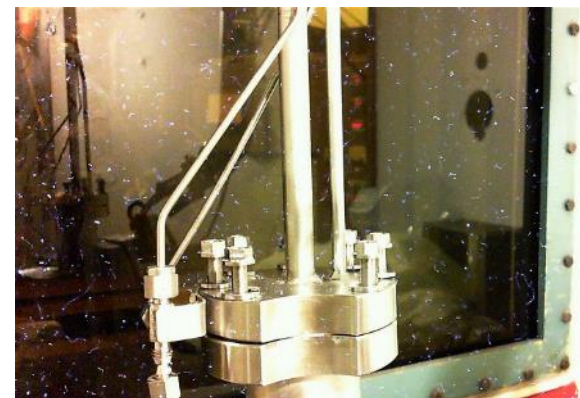
- Following the SCRAM, conducted a test to 3 MW to allow additional sampling
 - Added glass, paper, and charcoal filters prior to tritium capture bubblers
 - Activation products only seen from capsule
- Molybdenum release and capture on filters highest from 0-2MW (<math><200^{\circ}\text{C}</math>)
- W and Mo captured on glass and paper filters effectively, not on charcoal
- Br not captured on filters, minimal tritium capture on filters
- Experiment removed from reactor following the test

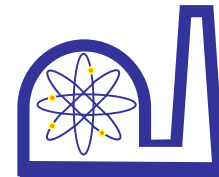




Post-Irradiation Examination

- FS-1 and FS-2 irradiation capsules extracted from in-core facility and transferred to Hot Box and Hot Cell, respectively
- Pressurized with helium to prevent moisture uptake
- Exterior condition of titanium pressure vessel and the nickel/Inconel capsule is excellent



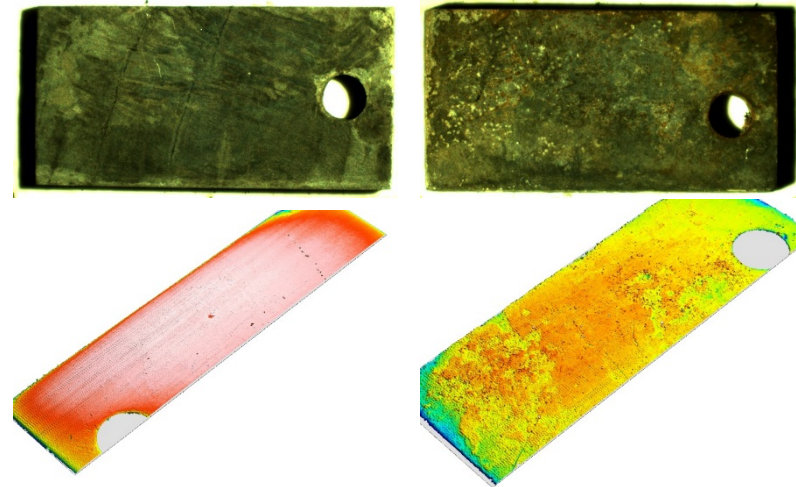
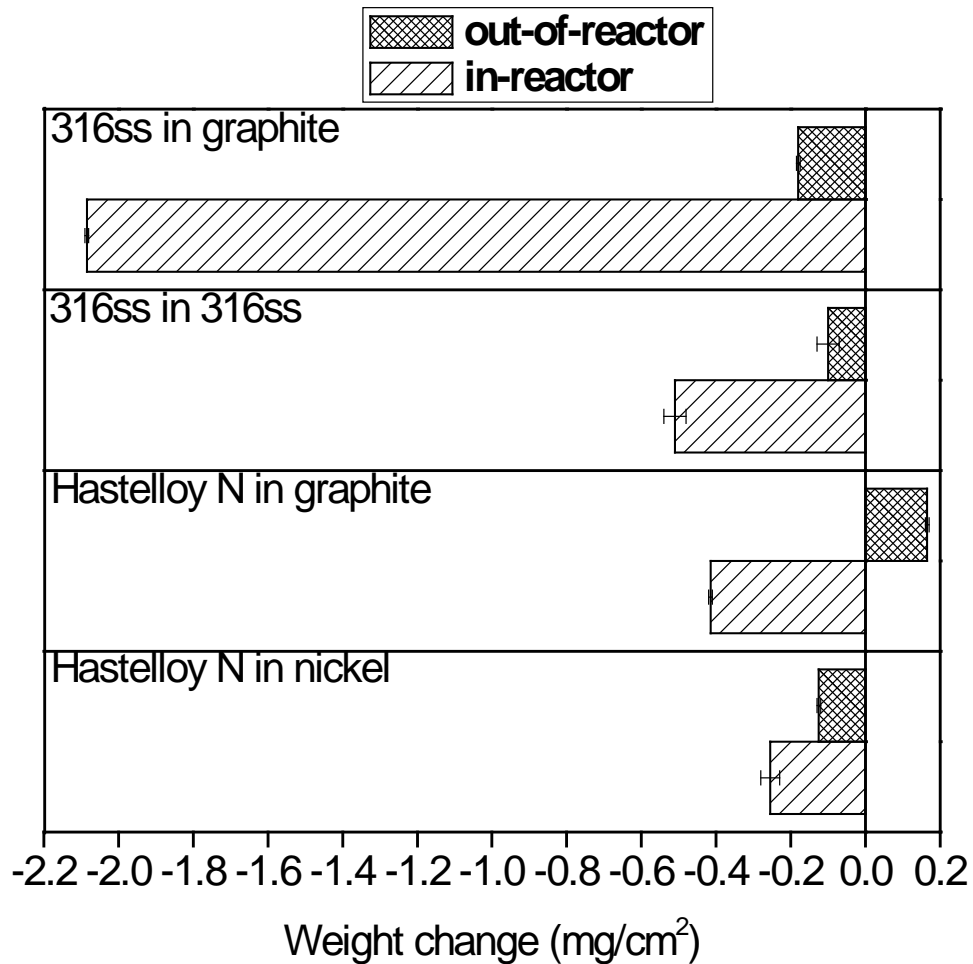
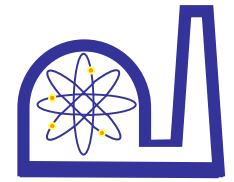


Specimen Extraction

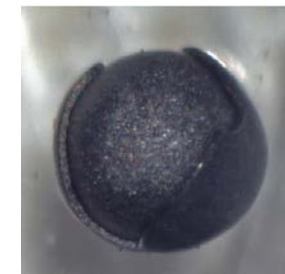
- All specimens have been removed from the FS-1 capsule and cleaned of flibe
 - Furnace operated in dry helium glove box to prevent HF generation and contain tritium
- Cleaning involved melting and collection of flibe $\sim 500^{\circ}\text{C}$, followed by soaking in water at ambient conditions until weight decrease arrested
- Did not see evolution of other activation products or substantial tritium release during salt melting



Initial PIE – Corrosion & Cracking



SS316 irradiated in lined (left) and unlined (right) chambers



Surrogate TRISO particle with OPyC damage

