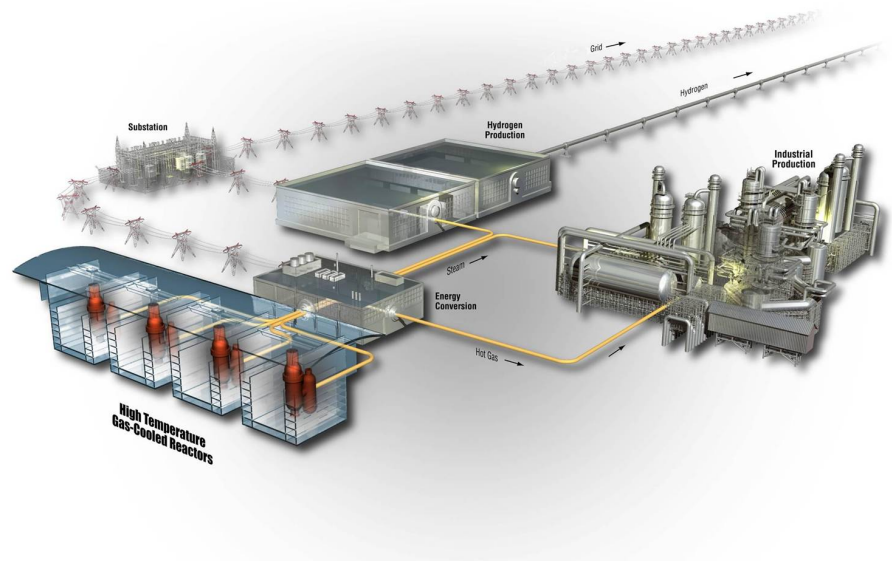


## Plan

Project No. 23843

# AGR-3/4 Phase 2 Post-Irradiation Examination Plan

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## Idaho National Laboratory

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INL ART TDO Program

Plan

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Manual: NGNP

**Author:**

\_\_\_\_\_  
Paul A. Demkowicz  
Advanced Gas Reactor TRISO Fuels Technical Lead

5/4/17

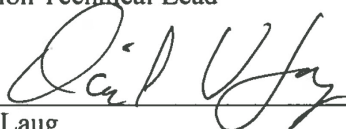
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**Approved by:**

\_\_\_\_\_  
John D. Stempien  
Advanced Gas Reactor TRISO Fuels Post-Irradiation  
Examination Technical Lead

5/04/2017

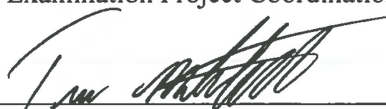
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\_\_\_\_\_  
David V. Laug  
Advance Gas Reactor TRISO Fuels Post-Irradiation  
Examination Project Coordination Lead

5/04/17

\_\_\_\_\_  
Date



\_\_\_\_\_  
Travis R. Mitchell  
INL Advanced Reactor Technologies Project Manager

5/4/2017

\_\_\_\_\_  
Date



\_\_\_\_\_  
Michelle T. Sharp  
INL Quality Engineer

5/4/17

\_\_\_\_\_  
Date



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## SUMMARY

The Advanced Gas Reactor (AGR)-3/4 irradiation experiment is the third in a series of test irradiations performed as part of the AGR Fuel Development and Qualification Program. AGR-3/4 was designed to investigate the migration of fission products in fuel kernels, compact matrix, and reactor graphite components. The experiment consists of fuel compacts containing driver fuel particles and special designed-to-fail (DTF) particles designed to release fission products during irradiation, which will migrate through the surrounding cylindrical rings of compact matrix and nuclear-grade graphite. Evaluation of fission product distribution in these rings following irradiation is a critical component of the experiment, providing data that will support refinement of fission product transport models.

Following conclusion of irradiation in the Advanced Test Reactor (ATR), the AGR-3/4 test train was removed from the reactor and shipped to the Hot Fuel Examination Facility (HFEF) at the Materials and Fuels Complex (MFC), which is located at Idaho National Laboratory (INL), for post-irradiation examination (PIE). The main objectives of PIE for this experiment are as follows:

- Determine distribution of fission products in matrix and graphite rings at the end of irradiation.
- Determine distribution of fission products in matrix and graphite rings at elevated temperatures following heating in pure helium and/or oxidizing atmospheres.
- Determine the fractional inventory of fission products remaining in fuel kernels and compact matrix at the end of irradiation.
- Determine the fractional inventory of condensable and gaseous fission product release from fuel kernels and compact matrix at elevated temperatures during heating in pure helium and/or oxidizing atmospheres.

The AGR-3/4 PIE has been separated into two phases. The majority of the first phase of PIE has been completed at the time of this writing and included test train shipment, receipt, and inspection; metrology of fuel compacts and capsule components; gamma scanning of the inner and outer rings; and gamma scanning of the fuel compacts. Planned Phase 2 PIE activities are described in this plan. The key activities are discussed in the following paragraphs.

The key components of the AGR-3/4 capsules will be analyzed to determine the inventory of fission products in/on these components in order to obtain a mass balance of fission product release from the compacts in each capsule. Destructive analysis of the inner and outer rings will be performed to quantify fission product distributions. These data will be used to compare with qualitative data obtained from gamma scanning of the rings and will be used to compare with fission product transport models.

Destructive examination of fuel compacts using a radial deconsolidation technique will be used to quantify the inventory of fission products in compacts outside the fuel particles (i.e., in the compact matrix). This will aid in determining the total release from DTF and driver fuel particles. Microanalysis of a limited number of compacts will also be performed to evaluate DTF kernel microstructures and driver particle morphology.

Post-irradiation heating tests are planned for fuel compacts, fuel bodies, and inner and outer rings. Heating tests of fuel compacts will provide information on fission product release from DTF kernels at elevated temperatures. Fuel body heating tests will provide information on fission product release from fuel kernels and transport through matrix and graphite at elevated temperatures. Heating tests of individual rings are considered to obtain additional data for refining fission product diffusion coefficients. These tests will be performed in both inert and oxidizing environments.

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Because of the complexity of the experiments and analysis of the data provided, much of the specific details of the proposed PIE are still provisional. It is expected that results from initial measurements will be used to inform decisions about remaining tests in order to best meet experiment objectives.

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AGR	Advanced Gas Reactor
ART	Advanced Reactor Technologies
ATR	Advanced Test Reactor
DFPA	destructive fission product analysis
DTF	designed to fail
FIMA	fissions per initial heavy metal atom
HT	heating test
ICP-MS	inductively coupled plasma mass spectrometry
INL	Idaho National Laboratory
MFC	Materials and Fuels Complex
ORNL	Oak Ridge National Laboratory
PGS	Precision Gamma Scanner
PIE	post irradiation examination
TAVA	time-average, volume-average
TRISO	tristructural isotropic
UCO	uranium oxide, uranium carbide (“uranium oxycarbide”)

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## 1. INTRODUCTION

### 1.1 Background

The Advanced Gas Reactor (AGR) Fuel Development and Qualification Program was established to perform the requisite research and development on tristructural isotropic (TRISO)-coated particle fuel to support deployment of a high-temperature gas-cooled reactor (HTGR). The work continues as part of the Advanced Reactor Technologies (ART) TRISO Fuel Program. The overarching goal of the program is to provide a baseline fuel qualification data set to support licensing and operation of a HTGR. To achieve these goals, the program includes the elements of fuel fabrication, irradiation, post-irradiation examination (PIE) and safety testing, fuel performance modeling, and fission product transport (INL 2016).

A series of fuel irradiation experiments is in being performed at the Advanced Test Reactor (ATR) at Idaho National Laboratory (INL). These experiments are intended to provide data on fuel performance under irradiation, support fuel fabrication process development, qualify fuel for operating and accident conditions, provide irradiated fuel for accident testing, and support development of fuel performance and fission product transport models.

The first two fuel irradiation experiments (AGR-1 and AGR-2) had similar designs and the primary objective was to test coated particle fuel performance. Both irradiations are complete. The major components of the AGR-1 PIE are complete (Demkowicz et al. 2015a), while the AGR-2 PIE began in July 2014 and is still in progress. The AGR-3/4 irradiation experiment was designed to investigate the migration of fission products in a fuel compact matrix and reactor graphite components. The experiment consists of fuel compacts containing designed-to-fail (DTF) particles that are designed to release fission products during irradiation, which will migrate through the surrounding cylindrical rings of compact matrix and nuclear-grade graphite. Evaluation of the fission product distribution in these rings following irradiation is a critical component of the experiment, providing data that will support refinement of fission product transport models.

### 1.2 AGR-3/4 Irradiation Experiment

#### 1.2.1 AGR-3/4 Fuel

The AGR-3/4 TRISO-coated particles contain low-enriched UCO (a uranium carbide, uranium oxide mixture) fuel kernels that are approximately 350  $\mu\text{m}$  in diameter and are manufactured at BWX Technologies Nuclear Operations Group (Lynchburg, Virginia). The U-235 enrichment was 19.7%. Driver fuel particles were fabricated at Oak Ridge National Laboratory (ORNL) by applying TRISO coatings to the kernels, with the following average thickness for each layer:

- Buffer: 109.7  $\mu\text{m}$
- Inner pyrolytic carbon (IPyC): 40.4  $\mu\text{m}$
- Silicon carbide (SiC): 33.5  $\mu\text{m}$
- Outer pyrolytic carbon (OPyC): 41.3  $\mu\text{m}$ .

The DTF particles were fabricated at ORNL by applying a single 20- $\mu\text{m}$ -thick pyrolytic carbon coating to the kernels. This layer was intentionally fabricated with a high anisotropy so it would be likely to fail during irradiation (Collin 2011).

The AGR-3/4 fuel compacts were fabricated at ORNL. The compacts are nominally 12.3 mm in diameter and 12.5-mm long (in contrast to the AGR-1 and AGR-2 compacts, which were approximately

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25-mm long). Figure 1 shows an image of an AGR-3/4 compact and an x-radiograph of a thin longitudinal compact section. Each compact contains approximately 1,872 driver fuel particles and precisely 20 DTF particles. The DTF particles were aligned in each compact roughly along the compact axial centerline (Figure 1). Note that DTF particles tended to slump toward the bottom of the compact during fabrication, leaving roughly the top 1/4 of the compact without DTF particles.

A summary of AGR-3/4 fuel properties is provided in the AGR-3/4 Experiment Irradiation Test Plan (Collin 2011). Detailed characterization data of fuel particles and compacts have been provided by ORNL (Hunn and Lowden 2007; Hunn, Trammel, and Montgomery 2011; Kercher et al. 2011).

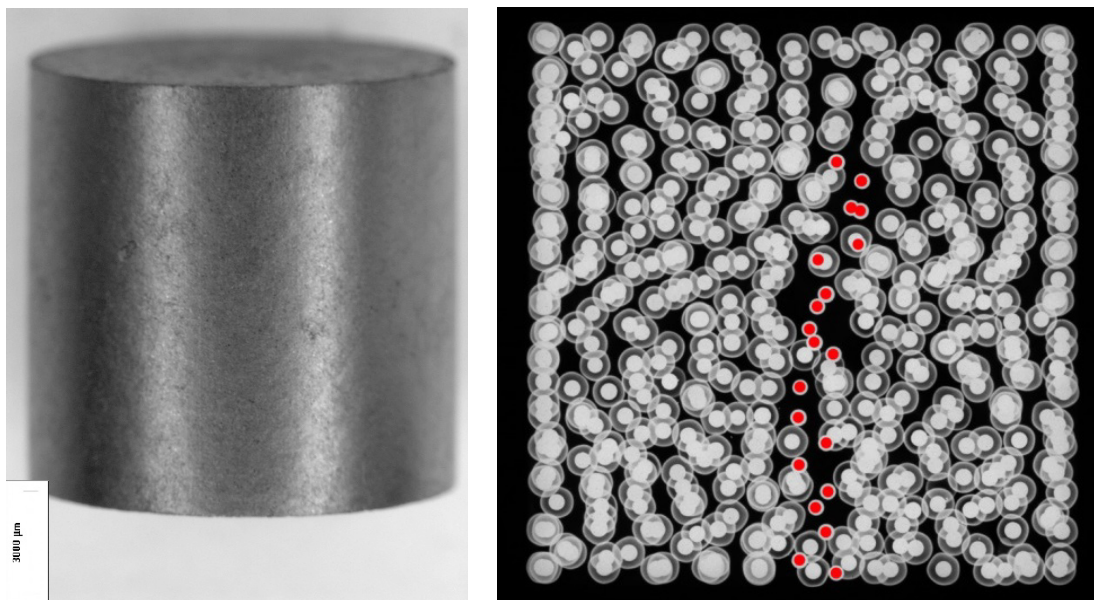


Figure 1. Image of an AGR-3/4 fuel compact (left) and x-ray side view image (right) (Hunn, Trammell, and Montgomery 2011). DTF particles are highlighted with red dots in the x-ray image.

### 1.2.2 AGR-3/4 Test Train

The AGR-3/4 test train consisted of 12 capsules, each with independent gas supply, fission product monitoring, and temperature monitoring. A single stack of four compacts was placed at the center of each capsule, with concentric rings of fuel matrix materials and/or graphite surrounding the compacts. Radial and longitudinal cross-section schematics of a standard capsule are shown in Figure 2 and highlight the basic components. The capsules are numbered 1 through 12, with Capsule 1 at the bottom of the test train and Capsule 12 at the top.

Each fuel compact is assigned an identifier in the format  $X$ - $Y$ , indicating the Capsule ( $X$ ) and the Level ( $Y$ ) of the original location in the test train. The levels in a capsule are numbered from 1 to 4, with Level 1 at the bottom and Level 4 at the top of the capsule. For example, Compact 12-1 represents the compact in Capsule 12 at Level 1 (i.e., the bottom compact in the capsule).

There are two basic types of AGR-3/4 capsules: standard capsules and fuel body capsules. The fuel body capsules are slightly longer to incorporate an integrated graphite screw-top lid as part of the outer ring (Figure 3). These capsules are intended for post-irradiation heating tests to assess fission product transport at temperatures in excess of those achieved during irradiation.

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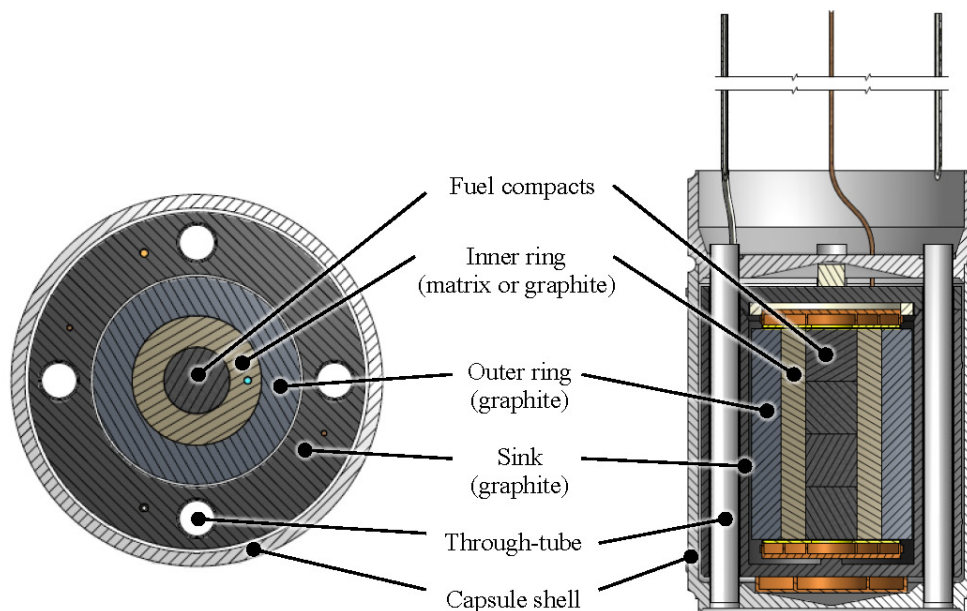


Figure 2. Axial (left) and longitudinal (right) cross-sectional diagrams of a standard AGR-3/4 capsule.

Some details about capsule design and materials of construction differ among the twelve AGR-3/4 capsules. While most of the capsules use an inner ring fabricated of fuel compact matrix material, selected capsules use graphite (either PCEA or IG-110) for the inner ring. The matrix inner rings were fabricated at ORNL and were made of a similar graphite/resin blend that was used to fabricate the AGR-5/6/7 fuel compacts. The outer rings were fabricated of either PCEA or IG-110 nuclear-grade graphite. All rings were machined to a length of approximately 51 mm at INL. Because of unique thermal control gaps used in each capsule, the thickness of the rings varies from capsule to capsule. The type of capsule, the material used for the inner and outer rings, and the inner and outer ring thicknesses are listed in Table 1.

Other details of capsule construction varied from capsule to capsule, including (a) the material used for the thermal insulators (either zirconium or zirconia), (b) the size of the gas gaps controlled by the inner and out diameters of the various rings, and (c) the number and azimuthal orientation of the centering nubs used to provide offset and center the concentric rings. These details can be found on the individual capsule drawings. INL drawing IDs for each capsule are given in Table 1

A variable gas mixture of helium and neon was continuously fed to each of the capsules during irradiation to provide temperature control and to sweep fission gases to the fission product monitoring system. In addition, gas impurities were injected into the sweep gas mixture in Capsule 11 to assess the effect of impurities that may be found in the primary HTGR coolant circuit. Details of the gas impurity flow history in Capsule 11 can be found in ECAR-2457 (Scates 2015).

Each capsule contained three separate neutron monitors intended to measure the thermal and fast neutron fluences. The monitors consisted of small wires encapsulated inside a small (i.e., about 1.25-mm outer diameter) vanadium tube. The wires used were (1) V + 0.1% Co, (2) iron, and (3) niobium. These fluence wire packages ranged from about 5 to 9 mm in length and were placed inside the graphite sink in each capsule (specific locations can be found on the capsule drawings).

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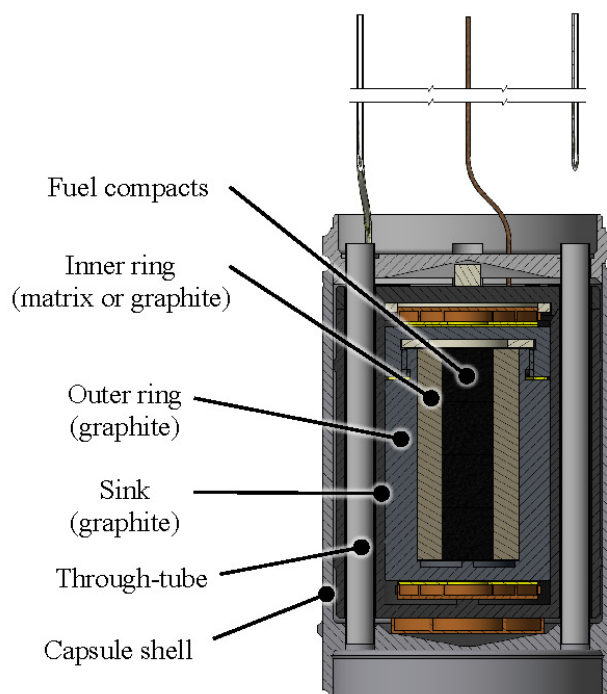


Figure 3. Longitudinal cross-section view of an AGR-3/4 fuel body capsule.

Table 1. List of AGR-3/4 capsules, noting capsule type, material for the inner and outer ring, and INL drawing number.

Capsule	Type	Material		Thickness (mm)		Capsule Drawing ID
		Inner Ring	Outer Ring	Inner Ring <sup>a</sup>	Outer Ring	
12	Standard	Matrix	PCEA	5.96	5.22	DWG-602712
11	Fuel body	Matrix	PCEA	5.02	4.50	DWG-602711
10	Standard	PCEA	PCEA	5.96	6.73	DWG-602710
9	Fuel body	Matrix	IG-110	5.96	7.49	DWG-602709
8	Standard	IG-110	IG-110	5.96	7.23	DWG-602708
7	Standard	Matrix	PCEA	5.96	6.72	DWG-602707
6	Fuel body	Matrix	PCEA	5.96	7.56	DWG-602706
5	Standard	Matrix	PCEA	5.96	7.56	DWG-602705
4	Fuel body	Matrix	PCEA	5.96	7.49	DWG-602704
3	Standard	PCEA	PCEA	5.96	4.50	DWG-602703
2	Fuel body	Matrix	PCEA	5.96	6.22	DWG-602702
1	Standard	Matrix	PCEA	5.65	4.51	DWG-602701

a. Drawing number for inner rings: DWG-602716.

Melt wire packages were also placed inside each capsule to provide verification of specific temperatures thresholds achieved in each capsule. Several different types of melt wires were used, with each wire contained inside a small (i.e., 1.25-mm outer diameter) vanadium tube, ranging from approximately 8 to 11 mm in length. Each capsule contains between one and three melt wire packages,

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selected based on the expected capsule temperatures and melting points of the wires. Melt wire packages were embedded in the inner rings (specific locations can be found on the capsule drawings). Details about specific melt wire compositions used in each capsule can be found in the AGR-3/4 Experiment Irradiation Test Plan (Collin 2011).

The capsules also contained Type N thermocouples (TC) with Inconel 600 sheath material. Two TCs were located in the sink ring in each capsule. In addition, a single TC is located in the inner ring in three capsules (i.e., Capsules 5, 10, and 12).

### 1.2.3 AGR-3/4 Irradiation

The AGR-3/4 test train was irradiated in the northeast flux trap of the Advanced Test Reactor (ATR) at INL from December 2011 to April 2014. Compact-average burnup ranged from 4.9 to 15.3% fissions per initial heavy metal atom (FIMA) and compact-average fast fluence ranged from  $1.19 \times 10^{21}$  to  $5.32 \times 10^{25}$  n/m<sup>2</sup>, with the peak burnup and fluence achieved in Capsule 6 (Sterbentz 2015). Capsule average burnup and fast fluence values, as well as temperature data for each capsule, are given in Table 2. Compact time-average, volume-average (TAVA), time-average peak, and time-average minimum temperatures for each compact are shown in Figure 4.

Release of short-lived fission gases from each capsule was monitored for the irradiation duration. The release-to-birth ratios derived from these data were used to estimate the number of failed DTF particles as irradiation progressed. Estimates for the number of failed DTF particles in each AGR-3/4 capsule at the end of life are given in Table 2 (Scates 2015). Note there is considerable uncertainty apparent in the estimates relative to the 80 DTF particles loaded into each capsule, because the estimates exceed 80 particles in several capsules. While the possibility of some driver particles failing cannot be completely excluded, this is deemed unlikely based on AGR-1 irradiation experience (i.e., zero in-pile failures among nearly 300,000 particles irradiated to a peak compact-average burnup of 19.6% FIMA).

Table 2. Irradiation data for each AGR-3/4 capsule.

Capsule	Burnup <sup>1</sup> (%FIMA)	Fast Fluence <sup>1</sup> ( $\times 10^{25}$ n/m <sup>2</sup> )	Time-Averaged Peak Temp (°C)			End of Life Number of Failed Particles
			Fuel	Inner Ring <sup>2</sup>	Outer Ring <sup>2</sup>	
12	5.4	1.50	888	802	748	40
11	9.1	2.87	1280	1166	975	69
10	11.8	3.94	1249	1055	986	47
9	13.7	4.65	1083	884	721	90
8	14.5	5.08	1257	1048	945	78
7	15.0	5.27	1418	1203	1045	52
6	15.2	5.31	1133	912	728	47
5	14.9	5.19	1102	858	706	54
4	14.2	4.85	1084	882	727	76
3	12.6	4.22	1242	1050	976	96
2	10.1	3.21	1113	977	875	91
1	6.1	1.76	978	889	785	41

1. Burnup and fast fluence are capsule-average values (Sterbentz 2015).

2. Ring temperatures are the volume average for a 1-in. tall axial section centered on the fuel stack (Hawkes 2016).

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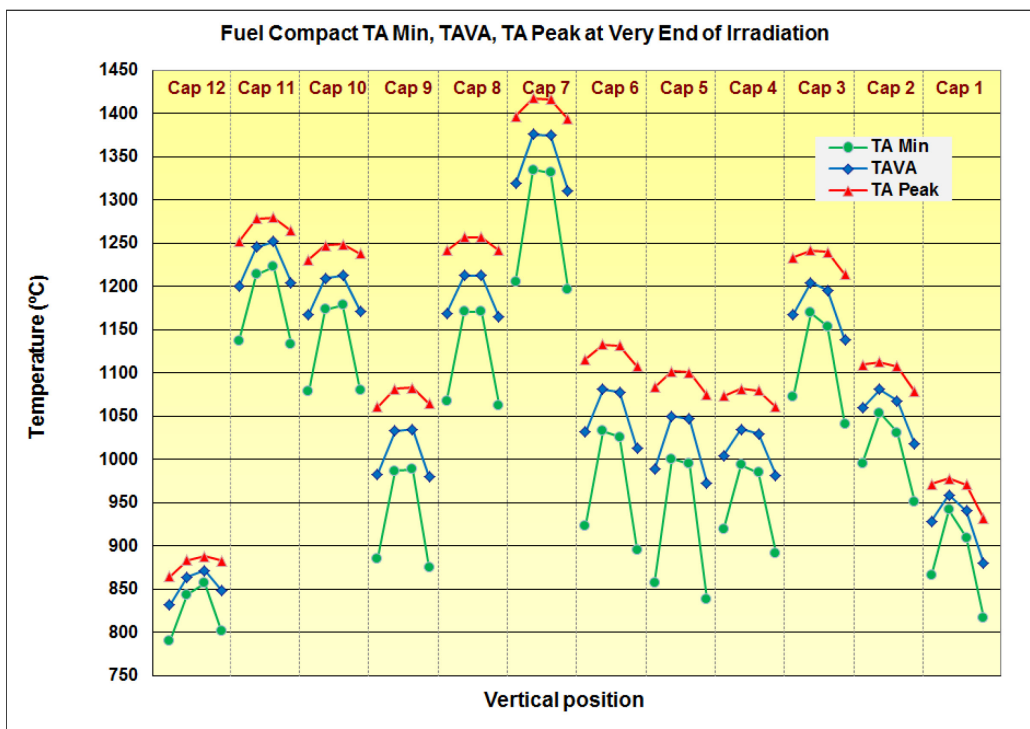


Figure 4. Time-average minimum, TAVA, and time-average peak temperatures for AGR-3/4 fuel compacts at the end of irradiation (Hawkes 2016).

### 1.3 AGR-3/4 Post-Irradiation Examination Objectives

The overarching objective of the AGR-3/4 experiment is to support development of fission product transport models through analysis of fission product migration in the kernels, matrix material, and graphite. In support of this, the primary objectives of the AGR-3/4 PIE are listed as follows:

- Determine distribution of fission products in matrix and graphite rings at the end of irradiation.
- Determine distribution of fission products in matrix and graphite rings at elevated temperatures following heating in pure helium and/or oxidizing atmospheres.
- Determine the fractional inventory of fission products remaining in fuel kernels and compact matrix at the end of irradiation.
- Determine the fractional inventory of condensable and gaseous fission product released from fuel kernels and compact matrix at elevated temperatures during heating in pure helium and/or oxidizing atmospheres.

AGR-3/4 PIE activities are intended to provide sufficient data to meet these objectives. Many of the activities planned for AGR-3/4 differ substantially from those used in AGR-1 and AGR-2 PIE.

## 2. PHASE 1 POST-IRRADIATION EXAMINATION ACTIVITIES

AGR-3/4 PIE has been divided into two phases. Phase 1 includes the following activities:

1. Test train shipment, receipt, and inspection
2. Test train and capsule disassembly



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3. Metrology of fuel compacts and capsule components
4. Gamma scanning of fuel compacts
5. Gamma scanning of inner and outer rings.

Items 1 through 4 were complete at the time of this writing (with the exception of fuel compacts and components in the fuel bodies, which will not be disassembled until after Phase 2 heating tests); Item 5 is still in progress. The basic flow of Phase 1 AGR-3/4 PIE activities is shown in Figure 5.

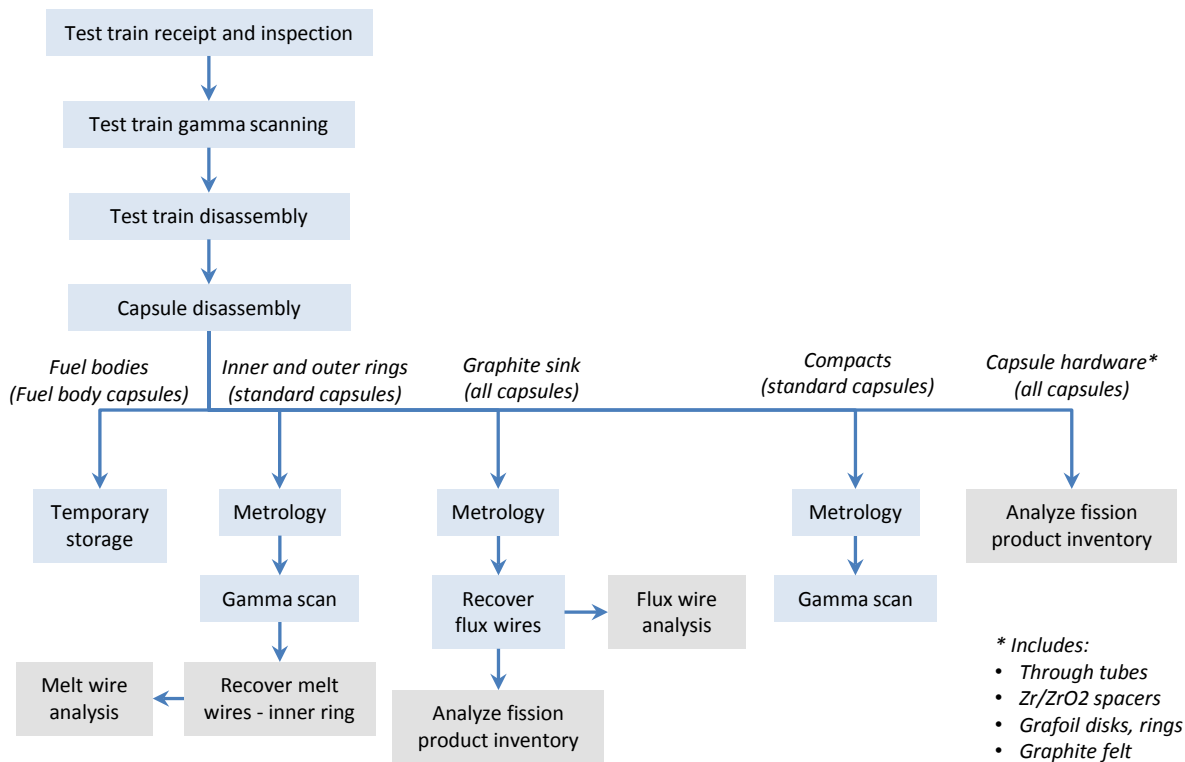


Figure 5. Flow chart of Phase 1 AGR-3/4 PIE activities. The chart indicates temporary storage of fuel bodies following removal from capsules until detailed PIE activities are specified. Capsule 4 is considered a “standard capsule” in the flow depicted here.

All Phase 1 PIE activities were performed under guidance in Demkowicz (2015). Following the irradiation and cool down period in the ATR canal, the AGR-3/4 test train was resized, sectioned in two pieces between Capsules 6 and 7, and transferred to the Materials and Fuels Compact (MFC) Hot Fuel Examination Facility (HFEF) in two separate shipments between February and April 2015. A visual examination of the test train was performed which did not reveal any significant damage. Each intact half of the test train was gamma scanned using the precision gamma scanner at HFEF. Gamma scans did not indicate any gross relocation of interior capsule components. All standard capsules and the fuel body Capsule 4 were disassembled to their constituent parts, which included the following:

1. Capsule body shell and base (1)
2. Capsule head (1)
3. Through tubes (4)
4. Graphite sink, lid, and screws (1)



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5. Inner ring
6. Outer ring (and lid, for fuel body Capsule 4)
7. Fuel compacts (4)
8. Miscellaneous capsule hardware (including zirconia or zirconium spacers, Grafoil® disks and rings, and graphite felt pieces)
9. Fluence wire packages.

All components were placed in storage containers upon disassembly. Fuel body Capsules 2, 6, 9, and 11 were disassembled in a similar manner, but the fuel bodies themselves (essentially comprising Items 5 through 7 in the above list) were left completely intact and placed in temporary storage. Because the irradiation conditions in Capsule 6 (particularly the fuel and ring temperatures) were similar to Capsule 4, it was decided to treat Capsule 4 as a standard capsule to provide as many irradiated rings as possible for destructive examination.

Following capsule disassembly, dimensions of the fuel compacts and certain components from the capsules were measured. Measurements included outer diameter and length of the fuel compacts, inner and outer diameter of the inner rings, inner and outer diameter of the outer rings, and inner and outer diameter of the graphite sinks. Note that for Capsules 2, 6, 9, and 11, the inner and outer diameter of the sinks and the outer diameter of the outer rings were measured, but the inner diameters of the outer rings, the inner and outer diameters of the inner rings, and the diameters of the compacts were not measured because the fuel bodies were left intact. The post-irradiation measurements were compared to the as-fabricated dimensions to determine the extent of dimensional change during irradiation. The measured dimensions were then fed back into the AGR-3/4 irradiation test as-run thermal analysis (Hawkes 2016).

Test train receipt, inspection, capsule disassembly, and dimensional measurements are all detailed by Stempien et al. (2016). Results of dimensional measurements can be summarized as follows:

- All measured compact diameters decreased during irradiation, with relative diameter change ranging from approximately  $-0.5$  to  $-2.0\%$ .
- Most measured compact lengths decreased, but several exhibited a net length increase in Capsules 4, 5, and 7. Relative length change in the measured compacts ranged from approximately  $+0.5$  to  $-0.9\%$ .
- All measured inner ring inner diameters exhibited net increase, with relative changes ranging from approximately  $0$  to  $+2.3\%$ . All measured inner ring outer diameters exhibited net decrease, with relative changes ranging from approximately  $-0.4$  to  $-2.8\%$ . The magnitude of the change in both cases tended to increase with increasing neutron fluence.
- Outer ring behavior was qualitatively similar to the inner ring behavior described above. Measured inner diameters exhibited net increase, with relative changes ranging from approximately  $0$  to  $+1.7\%$ . Measured outer diameters exhibited net decrease of approximately  $0$  to  $-1.6\%$ . As with the inner rings, the magnitude of change in inner and outer diameters increased with increasing neutron fluence.
- Nearly all inner and outer diameters of the graphite sinks decreased, with relative change ranging from approximately  $-1.2$  to  $-1.8\%$  (the exception was two sinks with inner diameter changes of  $+0.15$  and  $+0.7\%$ ).

Gamma scanning of fuel compacts in Capsules 1, 3, 4, 5, 7, 8, 10, and 12 has been completed. Partial results are described by Harp, Demkowicz, and Stempien (2016b). The results were used to estimate burnup of the compacts and to quantify the inventory of several key fission products. In particular, the Ag-110m inventory was determined and compared with the predicted inventory from physics

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calculations. This provides an estimate of the fraction of Ag-110m retained in each compact during irradiation. The preliminary ratio of measured compact Ag-110m inventory to the predicted inventory<sup>a</sup> for the AGR-3/4 compacts is shown in Figure 6. Note for certain compacts there are relatively large uncertainty bounds on the values due to very low inventory generated in the compacts during irradiation (particularly for the relatively low-burnup compacts in Capsules 1 and 12). Also note that compacts from Capsules 2, 6, 9, and 11 have not yet been scanned, because these are in the unopened fuel bodies.

Gamma scanning of the inner and outer rings is still in progress at the time of this writing. Partial results are discussed by Harp, Demkowicz, and Stempien (2016b). Rings were scanned in the precision gamma scanner (PGS) in two axial passes that covered one half of the ring volume (Figure 7). The collimator slit was 2.22-cm (7/8-in.) wide and 0.254-cm (0.1-in.) high. The decay-corrected activity from all scans was totaled to obtain a total ring activity, which was compared with the predicted capsule inventory for each isotope. Table 3 provides the measured inventory of fission products in the inner and outer rings, expressed as a fraction of the total capsule inventory. Note that grey-shaded rows correspond to fuel body capsules that have not been disassembled at the time of this writing.

Following axial scanning, additional gamma scans were performed at selected axial levels if warranted by the axial scan data. These scans were performed to construct activity-intensity maps of the ring cross sections using the gamma emission computed tomography technique (Harp and Demkowicz 2014). Maps created from the data show the distribution of gamma-emitting isotopes in the ring cross sections.

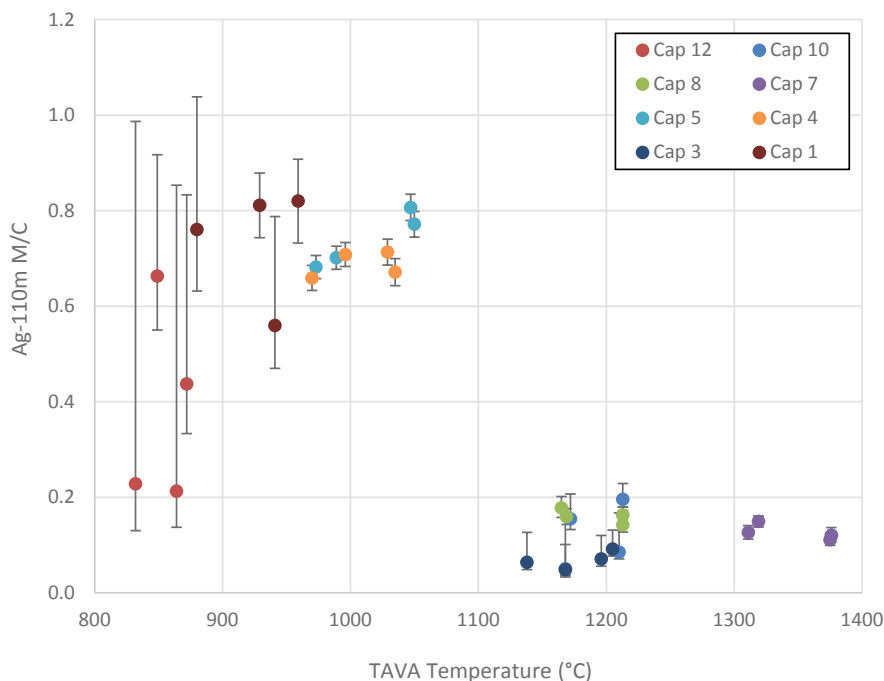


Figure 6. Preliminary data for the ratio of measured Ag-110m inventory to calculated inventory (M/C) for AGR-3/4 compacts plotted versus TAVA temperature.

<sup>a</sup> All measured fission product inventories determined during AGR-3/4 PIE will be decay-corrected to the end of the AGR-3/4 irradiation + 1 day (11:00 a.m. April 13, 2014, coordinated universal time) and compared with predicted values for each capsule at the same date and time, based on as-run physics calculations (Sterbentz 2015), in order to calculate the fraction of the capsule total that is represented.

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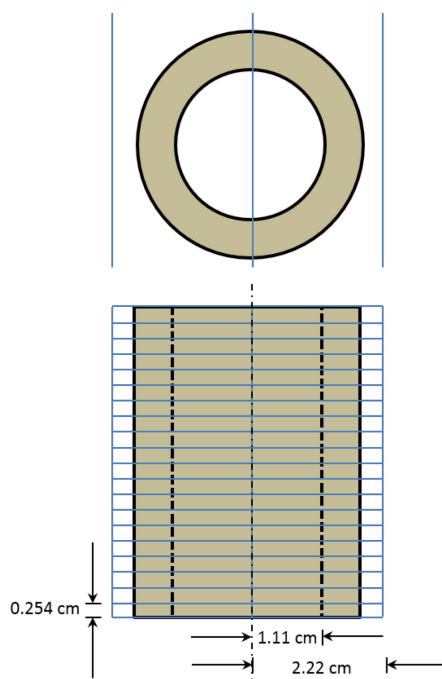


Figure 7. Diagram showing the geometry of the axial inner and outer ring gamma scanning. Outer ring geometry is shown, but the basic approach is the same for the inner rings.

Table 3. Fission product inventory measured in the AGR-3/4 inner and outer rings expressed as a fraction of the total predicted capsule inventory. Grey-shaded rows correspond to fuel bodies that have not been disassembled. “ND” indicates that no activity was detected in the ring.

Capsule	Inner Ring				Outer Ring			
	Ag-110m	Cs-134	Cs-137	Eu-154	Ag-110m	Cs-134	Cs-137	Eu-154
1	<5.1E-2	4.9E-4	7.3E-4	8.8E-4	<5.2E-2	1.9E-5	1.0E-4	ND
2	—	—	—	—	—	—	—	—
3	3.9E-2	5.9E-4	7.7E-4	4.4E-3	1.4E-1	5.0E-4	5.9E-4	ND
4 <sup>a</sup>	6.8E-2	8.6E-3	8.4E-3	ND	2.0E-3	7.9E-4	7.4E-4	ND
5	7.5E-4	7.7E-3	7.2E-3	ND	4.2E-4	7.2E-5	1.1E-4	ND
6	—	—	—	—	—	—	—	—
7	2.8E-4	6.2E-4	7.3E-4	3.0E-2	2.1E-2	9.7E-4	1.1E-3	ND
8	7.7E-2	7.1E-4	9.3E-4	8.0E-4	7.0E-1	3.4E-3	3.7E-3	ND
9	—	—	—	—	—	—	—	—
10	9.1E-2	8.4E-4	1.0E-3	4.5E-4	3.9E-1	1.2E-3	1.3E-3	9.8E-5
11	—	—	—	—	—	—	—	—
12	<1.8E-1	2.7E-4	6.4E-4	ND	<1.9E-1	ND	1.5E-5	ND

a. Note the outer ring in Capsule 4 differed from the outer ring in the standard capsules, because this was a fuel body capsule (Figure 3). The outer ring was in nearly direct contact with the top and bottom compacts during irradiation.

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### 3. PHASE 2 POST-IRRADIATION EXAMINATION ACTIVITIES

#### 3.1 Analysis of Fluence and Melt Wires

Fluence wire packages located in the graphite sink in each capsule were removed during disassembly as described in Stempien et al. (2016). The intact fluence wire packages will be gamma counted to measure the total inventory of key neutron activation products. The niobium wires will have to be opened, the niobium wires completely dissolved, and the solutions analyzed for low-energy emissions associated with the isomeric transition of Nb-93m. The measured isotopic inventories will be used to determine the neutron fluence at each location.

All melt wire packages were recovered from Capsules 1, 3, 4, 5, and 12. Melt wires from Capsules 7, 8, and 10 could not be pushed out of the inner rings without risking damage to the rings. Melt wires were not retrieved from any of the unopened fuel bodies (i.e., Capsules 2, 6, 9, and 11). The melt wires will be examined by manually cutting open and visually inspecting the packages in a glove box (similar to AGR-1).

#### 3.2 Analysis of Fission Products on Capsule Components

A total mass balance of fission products released from the fuel compacts is needed for each capsule. This will involve analysis of fission products on the following capsule components:

- Inner ring
- Outer ring
- Graphite sink
- Miscellaneous capsule components (zirconia or zirconium spacers, Grafoil® disks and rings, and graphite felt)
- Through tubes.

Analysis of each component is discussed in the following subsections. The basic work flow for analysis of each component is shown in Figure 8. Measured isotopic inventories in each capsule component will be compared with predicted values (Sterbentz 2015) for the entire capsule to determine the corresponding fraction of the total capsule inventory.

Based on the design of the AGR-3/4 capsules, the graphite sink is expected to absorb most of the fission products that escape from the outer ring, preventing them from traveling further to the outer steel capsule shell. Accordingly, early programmatic discussions on the AGR-3/4 PIE resulted in a decision to forego leaching and analysis of fission products on the metallic capsule components (i.e., capsule shells, top and bottom pieces) (Kendall 2012). Because the through tubes were located in the graphite sink a relatively small distance from the inner surface, these components will be leached and analyzed for fission products.

##### 3.2.1 Graphite Sinks

Each sink will be transferred from HFEF to the Analytical Laboratory and gamma-counted intact along with the sink lid. Each sink and lid will then undergo a burn-leach process to dissolve fission products, followed by solution analysis with gamma spectrometry and inductively coupled plasma-mass spectroscopy (ICP-MS). Aliquots of the leach solutions will also be subject to strontium separation and analyzed with gas-flow proportional counting for Sr-90.

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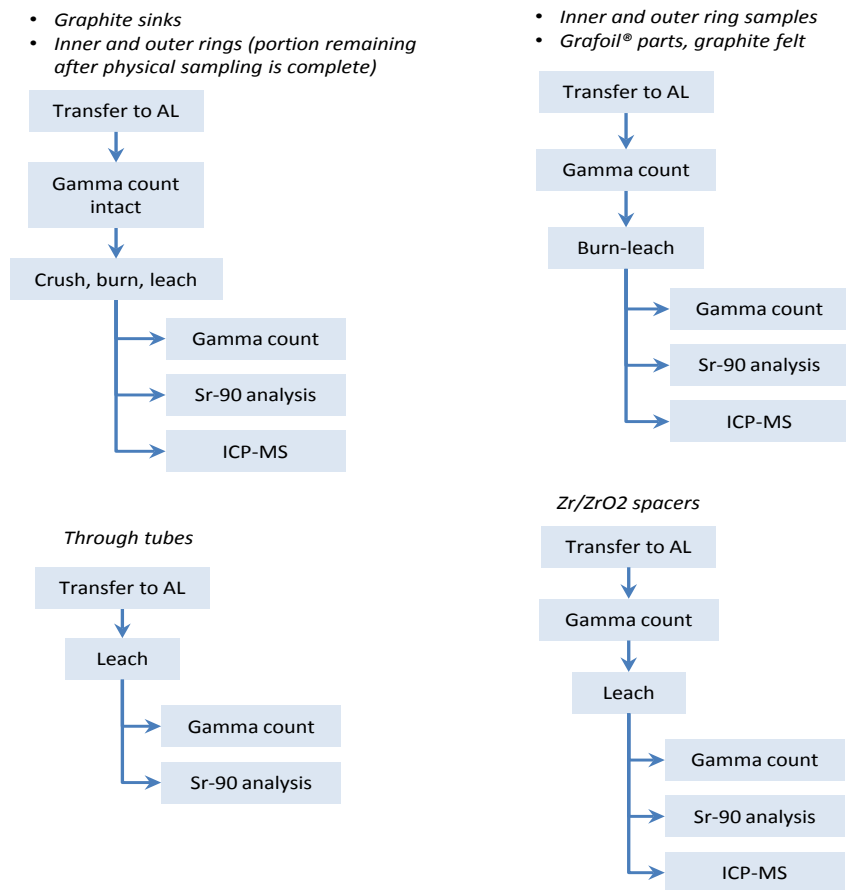


Figure 8. Flow charts for analysis of AGR-3/4 capsule components.

### 3.2.2 Inner and Outer Rings

An estimate of the total activity of gamma-emitting radioisotopes in the inner and outer rings has been provided by the axial gamma scans (Table 3). Validation of these values (as well as quantification of non-gamma-emitting isotopes) will be obtained by performing quantitative radiochemical analysis of the entire rings from the standard capsules<sup>b</sup> following physical sampling. (See Section 3.3 for a discussion of ring physical sampling.) Quantitative radiochemical analysis will involve transferring to the MFC Analytical Laboratory portions of the rings remaining after physical sampling. The ring material will be gamma counted to determine inventory of gamma-emitting fission products. The material will then undergo a burn-leach process to dissolve fission products, followed by solution analysis with gamma spectrometry and ICP-MS. Aliquots of the leach solutions will also be subject to strontium separation and analyzed with gas-flow proportional counting for Sr-90. The measured inventory in the physical samples (i.e., Section 3.3) will be added to the inventory determined for the remainder of each ring to obtain a total inventory.

<sup>b</sup> Similar analysis on fuel bodies will be performed following heating tests and will be described in a subsequent revision to this plan.

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### 3.2.3 Miscellaneous Capsule Hardware

Extra components from each capsule (including zirconia and zirconium spacers, Grafoil® disks and rings, and graphite felt pieces) will be transferred to the Analytical Laboratory for analysis (see Figure 8). The components will be gamma counted first to quantify the inventory of gamma-emitting isotopes. Because chemical leaching or burn-leach analysis and subsequent analysis of the solutions are time-consuming and add considerable expense, the need for subsequent chemical processing will be evaluated based on the estimated inventory of parts from gamma scanning and other PIE data. If gamma data indicate insignificant fractions of gamma-emitting isotopes on the components relative to the amount present in the other major capsule components (e.g., inner and outer rings and graphite sink), it may be decided to forego further analysis in order to focus on other PIE priorities. If further analysis is required, this will be accomplished by leaching (i.e., zirconium and zirconia components) or oxidation followed by leaching (for graphite components) and subsequent solution analysis similar to that described above.

### 3.2.4 Through Tubes

The stainless steel through tubes that were originally embedded in the graphite sink in each capsule may collect fission products that have migrated through the graphite sink during irradiation. The through tubes from each capsule will be collected after capsule disassembly and sent to the Analytical Laboratory for leaching and solution analysis, which will include gamma spectrometry and strontium separation and analysis.

## 3.3 Physical Sampling and Analysis of the Inner and Outer Rings

Following nondestructive gamma scanning of the inner and outer rings from the standard capsules and Capsule 4, physical samples will be taken from the rings for subsequent fission product analysis. This activity will provide quantitative data on the inventory and distribution of fission products at specific locations in the rings to compare with gamma scanning data. This will be critical to assist in converting the gamma intensity maps to quantitative maps. In addition to analysis of gamma-emitting fission products, non-gamma-emitting fission products in the samples must be analyzed. Regardless of the technique used, the goal will be to provide data with the maximum radial resolution (i.e., maximum number of data points across the radial thickness of the rings) within the limits of the method. This will be determined in part by (a) the fission product concentration in the rings and (b) the limits of the physical sampling method used.

The primary method of sampling that is being developed for this purpose is milling thin circumferential sections from the rings. The basic approach is shown in the schematic diagram in Figure 9. It is proposed that the section width ( $w$ ) will range from 0.5 to 2.5 cm. The minimum radial thickness ( $t$ ) of each section removed will be approximately 0.2 to 0.3 mm, making it possible to generate at least 10 separate data points across the radial thickness. The radial thickness of each sample does not need to be uniform across the entire ring and can be varied during the process as needed. Samples will be extracted in consecutive cuts until the inner diameter of the ring is reached. The interior of each ring will be filled with two-part epoxy prior to milling in order to physically stabilize the ring and avoid loss of material near the inner diameter once the mill reaches this depth.

The axial location of sampling will be determined based on data from ring gamma scanning. In general it is expected that a sample will be taken from the axial center of each ring and an additional sample at one of the axial ends of the rings may also be taken if warranted based on fission product activity distributions indicated by gamma scanning.

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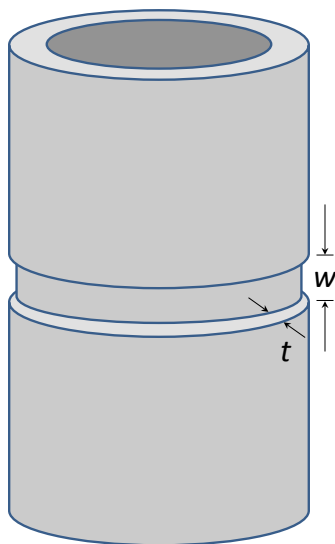


Figure 9. Diagram showing sampling approach for AGR-3/4 inner and outer rings.

The equipment being developed for ring sampling is shown in Figure 10 and Figure 11. As shown in Figure 10, rings will be held within a chuck. A motor (not pictured) will allow the ring to be rotated. A mill with two slides (horizontal and vertical) will enable cuts to be taken from the ring at the desired ring axial location to the desired radial depth. The graphite fines generated from milling the rings will be collected using a vacuum pump through a cyclone separator and deposited in individual vials (Figure 11). A hose with a funnel at one end (not pictured) will fit around the ring and carry the fines to the inlet to the cyclone separator (denoted “cyclone suction port” in Figure 11). The contents collected in each vial will be weighed and the vial gamma counted to determine the inventory of gamma-emitting fission products. The specimens will be oxidized in air and the residue leached in acid. The leachate will be analyzed with gamma spectrometry and ICP-MS. A strontium separation will also be performed and Sr-90 content analyzed. The measured inventory of fission products will be compared to the predicted inventories (Sterbentz 2015) in order to determine a total fraction in each specimen. Data will be used to determine a circumferentially averaged fraction of the total inventory at each radial thickness in the ring; therefore, fission product distribution will not provide any information on azimuthal uniformity that may exist in the rings.

The melt wire packages in each capsule are located in a hole drilled approximately in the middle of the inner ring thickness and approximately at the axial center of the ring (for more details see the drawings indicated in Table 1). In general, it is preferable to remove the melt wires prior to physical sampling activities. Section 3.1 discusses the results of melt wire removal for the capsules. In cases where melt wires could not be removed without risking damage to the rings, they must be left in place during sampling efforts. For circumferential milling, this means that it is likely the melt wire packages will be impinged by the mill during the sampling process and damaged, particularly when sampling at the axial center of the rings. Care will be taken during the process to prevent damage to the surrounding ring during this process if possible.

An alternate approach for obtaining physical samples of the rings is core-drilling specimens through the radius of the rings, followed by sampling of the core specimen in thin sections to determine the inventory at various radial displacements. Analysis of the core specimens will be similar to that described above for the specimens taken from the milling method. This method has the advantage of sampling a particular azimuthal location; therefore, azimuthal variation in fission product distribution could be explored. However, the volume of material at each radial displacement will be significantly less than with

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the circumferential milling technique discussed above and preliminary estimates based on predicted fission product inventories in the rings suggest that analysis of the small activity of fission products in these specimens may be challenging. It also will likely limit the number of specimens that can be taken along the radius of the ring (i.e., individual sections of the core sample), therefore, providing fewer radial thickness data points compared to the milling method.

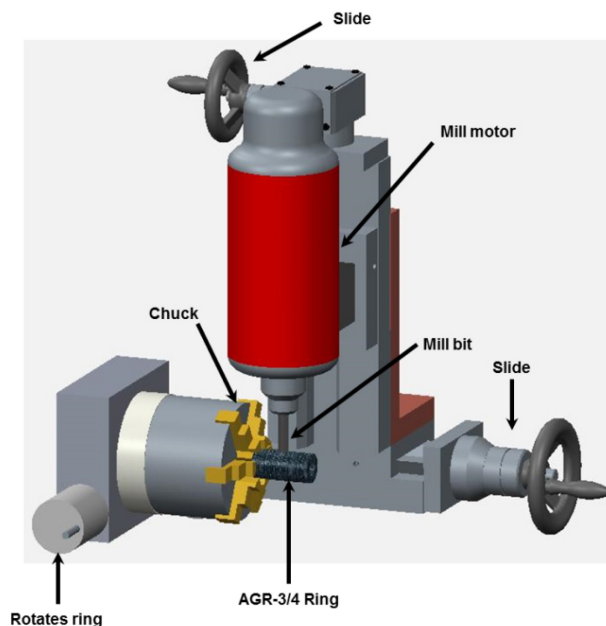


Figure 10. Equipment for ring milling.

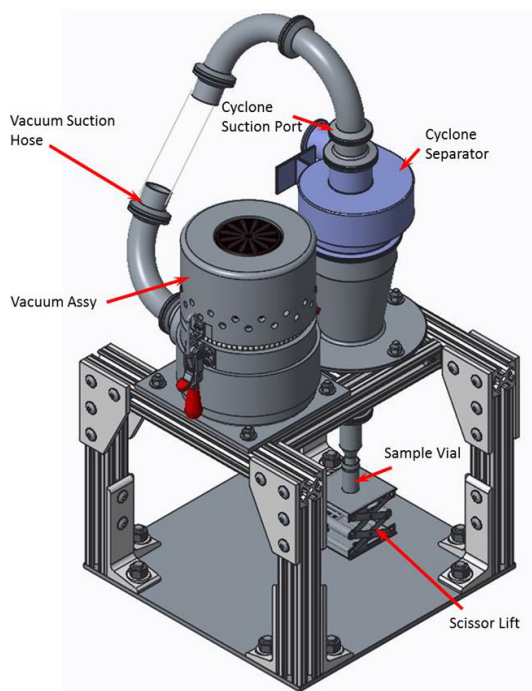


Figure 11. Collection of fines from ring milling.



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### 3.4 Compact Destructive Examination

#### 3.4.1 Compact Cross-Section Analysis

A limited amount of cross-section analysis on AGR-3/4 compacts is planned, primarily to inspect the state of the DTF particles. Interest in the morphology and microstructure of driver fuel particles is of secondary interest, because they are identical to particles irradiated in the AGR-1 experiment at higher total burnup and fluence. Several compacts will be sectioned longitudinally at a location displaced by approximately 1 to 2 mm from the centerline (see Figure 12). The larger portion of the compact will contain the majority of the DTF particles (stacked roughly along the compact centerline during fabrication). The exposed cross section in this piece will be mounted in epoxy and ground sequentially to expose as many of the DTF particles for inspection as practical. Mounting, grinding, and polishing of the mounts will be performed in a manner roughly similar to that performed for the AGR-1 (Ploger et al. 2012) and AGR-2 compacts (Rice, Stempien, and Demkowicz 2016). Examination of the polished cross-sections will focus on the condition of the DTF kernels and pyrocarbon coatings and document any interaction of the kernels with the surrounding matrix material. Microstructure and morphology of the driver particles will also be documented. Compacts best suited for this analysis would include both intact and failed DTF particles so comparisons can be made. This calls for selecting compacts primarily from capsules with estimated DTF failures at the lower end of the range observed for the AGR-3/4 experiment (Table 2).

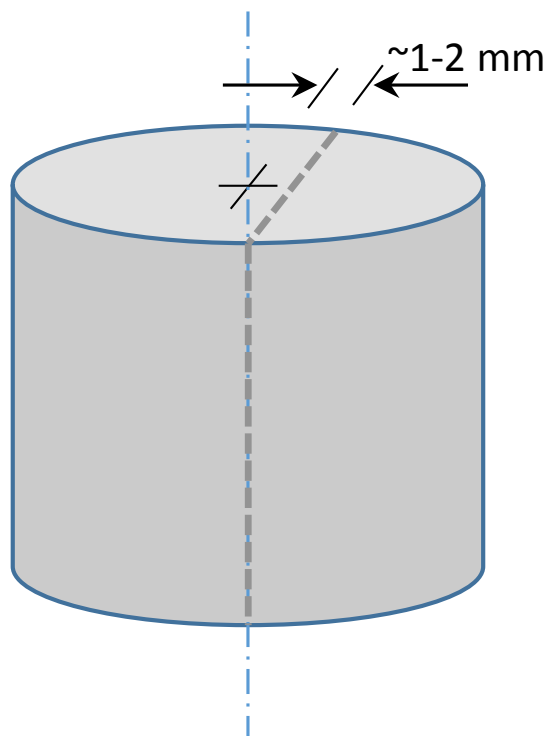


Figure 12. Diagram showing the approximate location of the longitudinal cut to be made on AGR-3/4 compacts prior to mounting, grinding, and polishing.

#### 3.4.2 Compact Radial Deconsolidation

A key data need for the AGR-3/4 experiment is the inventory of fission products retained in the matrix of compacts during irradiation or following post-irradiation heating tests (described in Section 3.5.1). Standard deconsolidation-leach-burn-leach analysis (as used on AGR-1 and AGR-2

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compacts; Demkowicz et al. 2015b), where the entire compact is deconsolidated axially in one step, is inadequate for this purpose because it would result in leaching of kernels from all failed DTF particles. Instead, deconsolidation of AGR-3/4 compacts will occur in several small radial steps and a single axial step (Figure 13). Figure 13 shows three radial steps; however, more radial steps could be considered. Radial steps will enable determination of the fission product inventory retained in the compact outside of the fuel silicon carbide layer within a particular radial segment of the compact without deconsolidating and leaching the DTF kernels. After completing the radial deconsolidation steps, a compact core that contains DTF particles will remain. This core will be axially deconsolidated using standard methods. Figure 14 shows equipment currently being developed to enable radial deconsolidation. The system will allow a compact to be suspended horizontally, with the bottom edge submerged in the acid electrolyte. Rotation of the compact while applying an electric current via a platinum screen (anode) and a platinum wire (cathode) in the acid solution will result in gradual deconsolidation of the compact matrix and liberation of particles in a relatively uniform manner around the compact circumference. (Note that in Figure 14 the platinum screen is not pictured, but its location is marked as “anode”. Likewise, the platinum wire serving as the cathode is not pictured, but it will be placed into the beaker holding the nitric acid solution.) The micrometer allows adjustment of compact depth in the acid solution.)

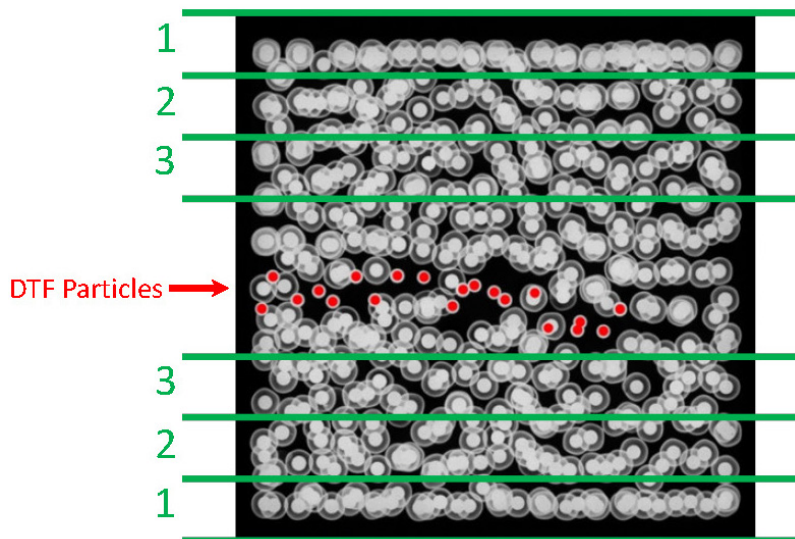


Figure 13. Representation of removing three radial segments from an AGR-3/4 compact by sequential electrolytic deconsolidation, exposing the core of DTF particles in the process. The number of radial segments and their thickness as depicted here are approximate.

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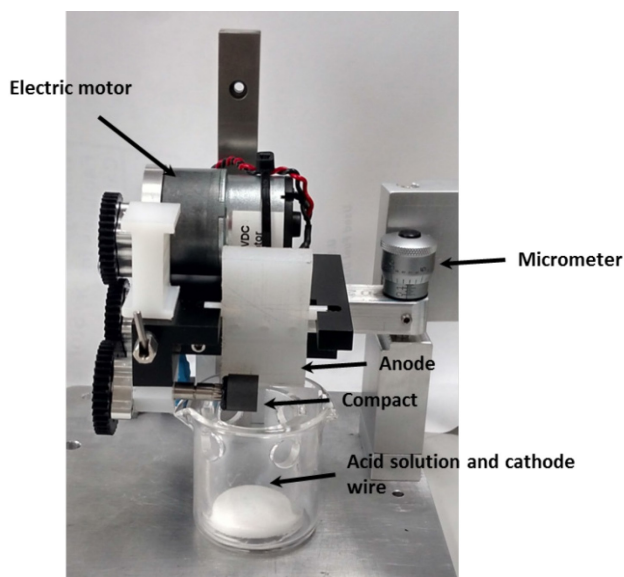


Figure 14. Apparatus for radial deconsolidation of AGR-3/4 compacts.

Figure 15 provides a flow chart of the basic process for radial deconsolidation, where the plan is to remove radial sections of the compact in approximately 1 to 2-mm increments. After each step, the process will be interrupted and the deconsolidation solution removed and replaced with fresh solution to continue the process. The material in each deconsolidation solution will be subjected to the standard leach-burn-leach process using a Soxhlet extraction apparatus (Demkowicz et al. 2015b). The radial deconsolidation will be terminated when the remaining compact core is approximately 6 mm in diameter; all DTF particles should be retained within this core. The remaining core will then be subject to a single-step axial deconsolidation. Video of the process will be recorded and the amount of material removed at each step of the radial deconsolidation will be estimated through analysis of the video and still images.

An attempt will be made to locate and separate any intact DTF particles from the driver particles and the matrix debris (note that best estimates of the number of failed DTF particles are significantly below the total of 80 particles in numerous capsules, implying the presence of intact DTF particles; see Table 2). The driver particles and matrix debris will be subjected to the standard leach-burn-leach analysis. All leach solutions will be analyzed with gamma spectroscopy, ICP-MS, and for Sr-90. The presence of significant quantities of uranium in the solutions could indicate that DTF kernels were present in the deconsolidated material and leached during processing. Any recovered intact DTF particles will be retained for gamma spectrometry analysis to quantify the inventory of fission products, providing information on fission product retention of these particles.

A subset of driver particles from each deconsolidation step will also be gamma counted, similar to the approach used for deconsolidated AGR-1 particles (Demkowicz et al. 2015b). Data will be used to determine the inventory of fission products in the particles taken from the different regions in the compact. The extent of fission product release will be estimated by comparing measured inventories to predicted particle inventories. This is likely to be significant for Ag-110m, which is often observed to have been released significantly from intact TRISO particles (Demkowicz et al. 2015a). In addition, appreciable europium release may be observable in particles from Capsule 7 compacts, based on the level of europium observed in the inner ring from that capsule, which indicated appreciable release from the compacts. In that capsule, it is estimated that approximately 3% of the total Eu-154 inventory was located in the inner ring (Table 3). In addition, the fuel compact TAVA temperatures for that capsule significantly

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exceeded those of compacts from AGR-2 Capsule 2, where significant Eu-154 inventory was also observed in the graphite holder (Harp, Demkowicz, and Stempien 2016a).

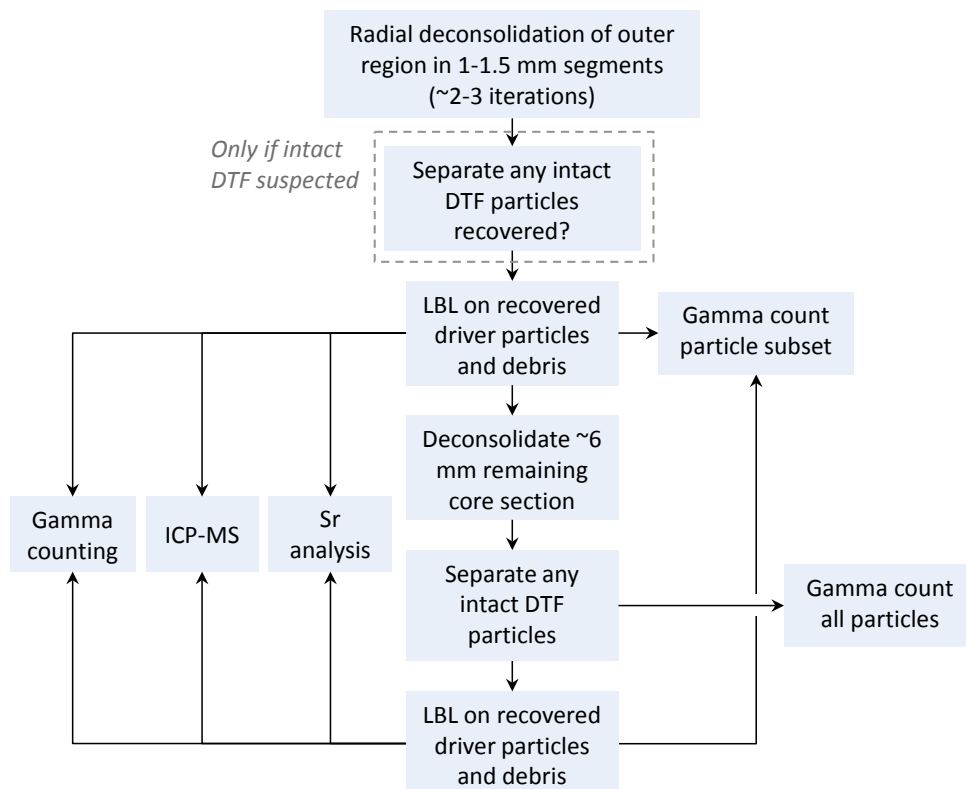


Figure 15. Flow chart of the fuel compact radial deconsolidation procedure.

### 3.4.3 Particle Analysis

No extensive microanalysis of individual driver particle cross-sections is planned unless warranted by other observations during PIE. Microanalysis of similar particles was performed during the AGR-1 PIE campaign (Demkowicz et al. 2015a). One area where a limited amount of driver fuel microanalysis may be of programmatic interest is fuel from Capsule 7, where fuel temperatures were higher than those of either AGR-1 or AGR-2. For example, the highest compact TAVA temperature in AGR-3/4 Capsule 7 was 1376°C, while the highest in AGR-2 Capsule 2 was 1296°C.

Note that optical micrographs of random driver fuel and DTF particles will also be collected as part of the compact cross-section analysis activity (Section 3.4.1).

## 3.5 Post-Irradiation Heating Tests

### 3.5.1 Compacts

Data on release of fission products from irradiated AGR-3/4 compacts during isothermal post-irradiation heating tests is needed, because it can potentially provide information on release from DTF particles at elevated temperatures. Compacts will be heated in the Fuel Accident Condition Simulator (FACS) furnace system to assess fission product release. Temperatures for the tests are expected to range from approximately 1200 to 1600°C. It is also desirable to explore the effect of irradiation conditions—most notably fuel burnup—on fission product release. A provisional test matrix is discussed in Section 3.6.3.

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The AGR-3/4 compacts are also considered attractive specimens for re-irradiation prior to heating in order to measure the release of short-lived I-131 at elevated temperatures. An option for this is to insert the compacts in the C-4 location in the neutron radiography (NRAD) reactor located in HFEF. The program is currently exploring the feasibility of irradiating compacts in the NRAD reactor. If this can be pursued, an update to this plan will be issued that will include details of the planned re-irradiations and heating tests.

In addition, heating of the AGR-3/4 compacts in oxidizing environments is of interest; therefore, the effects of air and moisture on fission product transport can be investigated. This will include the effects of oxidants on volatilization of fission products located both in the exposed kernels of DTF particles and in the compact matrix. The AGR Program is currently developing a capability for post-irradiation heating of fuel specimens in gas mixtures, including air and moisture (Stempien 2016). Additional details on heating of compacts in oxidizing atmospheres will be included in a revision of this PIE plan.

### 3.5.2 Fuel Bodies

The four intact fuel bodies (i.e., Capsules 2, 6, 9, and 11) will be heated to assess total release from the compacts and retention and release from the matrix and graphite rings. The detailed plan for these heating tests is currently being developed; it is likely the plan will be significantly influenced by initial results from PIE activities described in earlier sections. Data from heating the fuel bodies in inert (i.e., pure helium) and oxidizing (i.e., air and/or steam mixtures) atmospheres is of interest to the program. Heating tests in pure helium will be useful for assessing fission product release and diffusion in the components, while tests in an oxidizing atmosphere can additionally address the more complicated effects of oxidants on fission product mobility in the kernels, matrix, and graphite.

Analysis of as-irradiated rings in standard body capsules is expected to allow confirmation or modification of the existing transport model, as appropriate, in a way that refined predictions of fission product distributions in the fuel body rings can be performed. These predictions can be used to help guide subsequent analysis of these capsules and aid in interpretation of post-heating-test data, because no pre-heating test data regarding fission product distributions in the components will be available.

### 3.5.3 Individual Rings

Estimation of final fission product distributions and releases in AGR-3/4 involves modeling of many coupled physical phenomena, including temperature-dependent diffusion through each TRISO layer, compact, and cylindrical ring, coupled by temperature-dependent sorption isotherms at gas gaps between rings. This introduces a large number of parameters to the model and may prove infeasible to estimate all of these using only data obtained from destructive analysis. Many parameter sets may exist that reasonably describe these data, or it may prove difficult to determine a set that consistently describes all capsules. In the event either event appears to be the case, we propose using isolated inner or outer rings from selected capsules to perform separate effects experiments to measure fission product diffusion coefficients more directly. One proposed approach is to heat individual rings to a constant temperature in an inert atmosphere while monitoring fission product release as a function of time using the FACS furnace. Post-test analysis of the ring would provide information on the total inventory and distribution of fission products. Provided the initial distribution is known or can be reasonably estimated (i.e., this is a point that will be investigated further if such tests are to be undertaken), this can be compared directly to an analytical solution where diffusion coefficient is the only parameter; therefore, it is easily estimated.

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In addition, data on fission product release from rings heated in oxidizing atmospheres (containing air or moisture) are needed in order to determine the ability of oxidants to volatilize fission products previously deposited in graphite and matrix materials. While heating of fuel bodies in oxidizing atmospheres is also tentatively planned (Section 3.5.2), heating of rings is of value because it allows the behavior of fission products in the graphite and matrix to be studied specifically, without the confounding effect of fission products from the exposed DTF kernels. Selected rings may be heated in the air/moisture ingress testing furnace, which is currently being developed.

Details of ring heating tests will be developed once an initial comprehensive set of PIE data from several AGR-3/4 capsules is available (including nondestructive and destructive compact and ring analysis). These data will help to determine the practicality of deriving fission product transport parameters in the AGR-3/4 capsules based on the end-of-irradiation fission product distributions, inform decision about whether additional ring heating tests would be useful, and, if so, help determine appropriate test parameters. Determining the optimum temperature for heating tests will be particularly challenging given that the ideal temperature for studying fission product transport will be different for different elements. Test specimens and conditions will be included in a future revision to this PIE plan, as appropriate.

## **3.6 Phase 2 Post-Irradiation Examination Test Matrices**

### **3.6.1 Inner and Outer Ring Analysis**

A provisional matrix is provided in Table 4 and it outlines end use (either as-irradiated destructive analysis or heating tests) for each of the inner and outer rings in the standard capsules and Capsule 4. These provisional assignments were based on the following considerations and limitations:

- It will be optimal to have rings for destructive fission product analysis that span as much of the available temperature range as possible.
- When performing destructive analysis of fission product distribution in the rings, it will be ideal to examine both the inner and outer ring from a single capsule so that concentration changes across the gaps can be examined. This will provide information on sorption/desorption behavior.
- It will be ideal to include all types of ring materials (matrix, IG-110, and PCEA) for both studies. However, because there are limited available rings for each type of material (particularly in the case of IG-110), compromises will have to be made. A preference was given to the destructive fission product analysis measurements; therefore, Capsule 8 was assigned to this analysis because it represents the only two available IG-110 rings.
- Some rings have sufficiently low activity for certain isotopes that the inventory could not be determined using the gamma scanning technique (see Table 3). These rings would be less useful for heating tests, because the pre-test inventory could not be accurately determined prior to the test. Pre-test inventory would have to be determined based on total releases during the heating test and the post-test ring inventory determined using more sensitive methods than scanning with the PGS.

The priority for rings to be used for destructive fission product analysis is listed in Table 5. The nominal width (dimension  $w$  in Figure 9) of all specimens is 1 cm. Nominal thickness ( $t$ ) is 0.5 mm, but can be adjusted during sampling as needed. Sampling locations (i.e., top, center, or bottom) are selected based on results from axial gamma scanning, which indicate the general trends in distribution of fission products. Generally speaking, cesium is relatively uniformly distributed along the ring axes, while silver (if detectable) tends to be highly concentrated toward the axial ends of the rings. Samples taken at the top or bottom of the ring will be oriented so the region  $w$  mm from the end of the ring is removed during

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sampling. Samples taken from the center will have the 1-cm sample width centered approximately on the axial center of the ring.

Table 4. Provisional matrix of AGR-3/4 inner and outer rings and the preliminary end use for each ring, either destructive fission product analysis (DFPA) or post-irradiation heating tests (HT). The grey-shaded rows correspond to the intact fuel bodies that will be used for separate tests.

Capsule	Type	Material		TA Peak Temp (°C)		Use	
		IR	OR	IR	OR	IR	OR
12	Standard	Matrix	PCEA	802	748	HT	HT
11	Fuel body	Matrix	PCEA	1166	975	—	—
10	Standard	PCEA	PCEA	1055	986	HT	HT
9	Fuel body	Matrix	IG-110	884	721	—	—
8	Standard	IG-110	IG-110	1048	945	DFPA	DFPA
7	Standard	Matrix	PCEA	1203	1045	DFPA	DFPA
6	Fuel body	Matrix	PCEA	912	728	—	—
5	Standard	Matrix	PCEA	858	706	DFPA	DFPA
4	Fuel body	Matrix	PCEA	882	727	HT	HT
3	Standard	PCEA	PCEA	1050	976	DFPA	DFPA
2	Fuel body	Matrix	PCEA	977	875	—	—
1	Standard	Matrix	PCEA	889	785	HT	HT

The nominal plan is to begin by examining fission product distributions in rings from two AGR-3/4 capsules and use these data (along with data on fission product mass balance in the compacts and in the other capsule components) to construct a complete fission product distribution and mass balance data set for these capsules. These data will be compared with predictions from the existing transport model (Humrickhouse et al. 2016). Based on results from these comparisons, the path forward for remaining capsules (as outlined in Table 4 and Table 5) may be refined.

Table 5. Tentative priority for AGR-3/4 ring sampling and sample locations for each ring.

Ring ID	Sampling Locations <sup>1</sup>	Comments <sup>2</sup>
IR-03	Top Center	Ag-110m peaked at the top
OR-03	Top Center	Ag-110m peaked at both the top and bottom
IR-07	Top Center	Eu-154 skewed toward top; no Ag-110m detected in axial scans
OR-07	Top Center	Ag-110m peaked at top and bottom, but not uniform between left and right half
IR-05	Center	No fission product concentration at the ring ends
OR-05	Center	No fission product concentration at the ring ends
IR-08	Bottom Center	Ag-110m peaked at the top and bottom
OR-08	Bottom Center	Ag-110m peaked at the top and bottom

1. Nominal width ( $w$ ) and thickness ( $t$ ) for all samples is 1 cm and 0.5 mm, respectively, subject to change.

2. Comments are based on isotope distributions in the rings, obtained from axial gamma scanning data.

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### 3.6.2 Ring Heating Tests

A detailed plan for post-irradiation heating tests using isolated rings from the standard body capsules and Capsule 4 will be developed at a later time. It is expected that results from initial destructive exams on compacts and rings (as well as available data from nondestructive exams) will contribute to the decision on how best to use these rings. Additionally, results from fission product transport model refinement based on PIE data and subsequent predictions will aid in determining the optimal test conditions (i.e., duration and temperature) for ring heating tests.

### 3.6.3 Compact Heating Tests

Table 6 provides a provisional list of inert-atmosphere post-irradiation compact heating tests. The compacts in Table 6 are also indicated in Figure 16. Tests are planned to explore the effect of burnup and heating test temperature on fission product release. As such, compacts with relatively similar burnup and irradiation temperature are selected for determining the effect of test temperature. Tests at 1200, 1400, and 1600°C are proposed, with additional tests at 1300 and 1500°C considered as additional optional tests to be completed if time and budget allow. Three compacts (10-4, 1-2, and 8-2) are included to explore the effect of burnup on fission product release. A fourth compact (5-1) is considered as an optional test to explore the effect of irradiation temperature if time and budget allow. The tentative temperature for these tests is 1400°C, although this may be changed based on results of the initial tests spanning temperatures 1200 through 1600°C. Note that blue text in Table 6 indicates compacts that have a counterpart from the same capsule that will undergo as-irradiated deconsolidation analysis (Section 3.6.4). Following heating tests, some of the compacts may be examined using the radial deconsolidation technique described in Section 3.4.2.

If re-irradiation of compacts to produce I-131 is feasible and the capability is available (Section 3.5.1), then one or more of the compacts indicated in Table 6 will be re-irradiated prior to the planned heating tests so the short-lived iodine release also can be determined.

Additional heating tests in oxidizing atmospheres will also be pursued, using the in-cell furnace system currently under development. A detailed test matrix will be developed at a later time.

Table 6. Provisional list of compacts for post-irradiation heating tests along with test temperature.

Capsule	Compact	Irradiation Temperature (°C) <sup>1</sup>	Burnup (% FIMA)	Test Temperature (°C)	Remarks
<i>Test temperature effect</i>					
10	2	1213	12.0	1200	
3	2	1196	12.5	1600	
10	4	1168	11.4	1400	
10	1	1172	12.1	1300	Optional test to improve temperature resolution
3	1	1138	12.2	1500	Optional test to improve temperature resolution
<i>Burnup effect<sup>2</sup></i>					
1	2	941	5.9	1400	
8	2	1213	14.6	1400	
5	1	973	14.7	1400	Optional to compare with Compact 1-2 at similar temperature

1. TAVA temperatures.

2. Note that another 1400°C test (Compact 10-4) is included in the *test temperature effect* list and will contribute to the burnup comparison. Blue text denotes compacts with a same-capsule counterpart that will undergo as-irradiated deconsolidation



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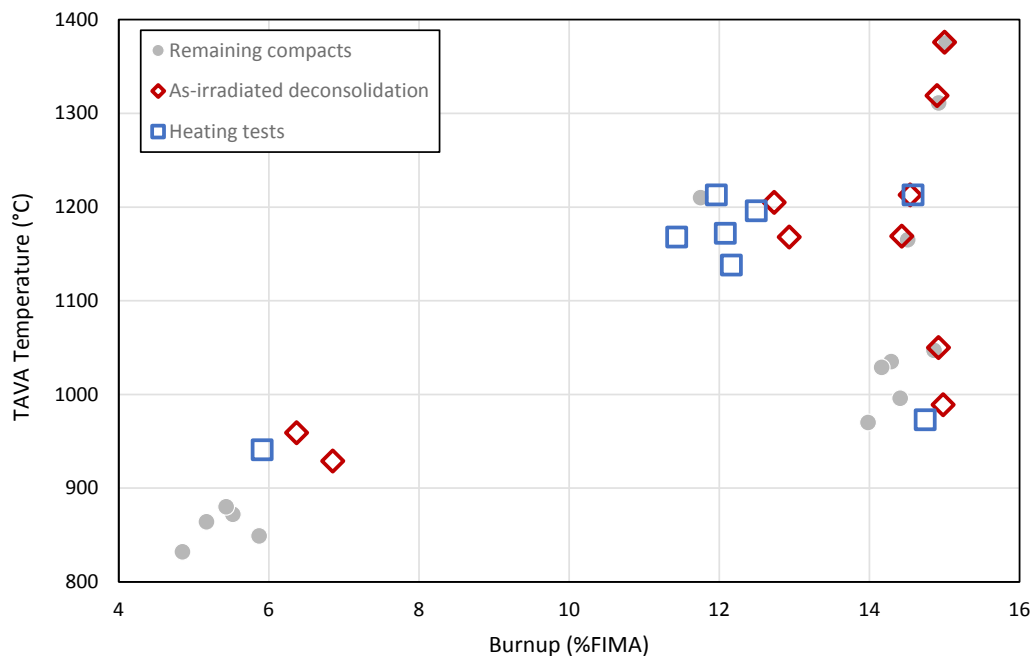


Figure 16. Burnup and TAVA temperature for compacts selected for radial deconsolidation and compact heating tests, as well as the remaining available compacts from Capsules 1, 3, 4, 5, 7, 8, 10, and 12.

### 3.6.4 Compact Radial Deconsolidation

Compacts for as-irradiated radial deconsolidation will be taken from the same capsules where rings were taken for destructive fission product analysis (Table 5). This will allow a complete mass balance of fission products to be determined in these capsules, with compact radial deconsolidation providing estimates for the inventory in the matrix and in the driver particles (of particular interest for silver). In each capsule, there is a range of temperatures across the four compacts (Figure 4). Note that the center compacts (i.e., compacts at Levels 2 and 3) can have TAVA temperatures as much as 50°C higher than the outer compacts (compacts at Levels 1 and 4) and that time-average minimum temperatures can be as much as about 150°C higher, resulting in a much greater range of temperatures within the two outer compacts. Because it is expected the inventory in the matrix and in the intact particles will be influenced by compact temperature, analysis of only one compact and extrapolation of the results to the other three (to determine total mass balance for the capsule) may bias the results high or low, depending on the specific compact chosen. Therefore, two compacts from each capsule will be chosen in order to better represent the range of temperatures experienced by the compacts. The upper two compacts in the selected capsules will be analyzed (i.e., compacts at Levels 3 and 4 in the capsule) in order to better represent the range of temperatures in the capsule; results from the remaining two compacts will be assumed to be similar for the purposes of calculating total capsule fission product mass balance. If it is observed during early analyses that differences in compact behavior between the Level 3 and 4 compacts are not significant, the decision may be made to only analyze the Level 3 compact in the remaining capsules to accelerate the schedule and minimize costs.

An additional consideration is to analyze compacts that span the range of irradiation temperatures and burnups. While compacts from Capsules 3, 5, 7, and 8 span a considerable portion of this range, compacts from Capsule 1 will also be deconsolidated to represent the lower-temperature, lower-burnup irradiation

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conditions. Figure 16 shows the burnup and temperature of the compacts selected for radial deconsolidation and compact heating tests (discussed above), as well as the remaining available compacts from Capsules 1, 3, 4, 5, 7, 8, 10, and 12.

In addition to the compacts selected for as-irradiated deconsolidation as discussed above, compacts may be deconsolidated following heating tests (both individual compact heating tests and fuel body heating tests). Table 7 provides a matrix of all compacts identified for radial deconsolidation in approximate order of priority. Given that radial deconsolidation equipment and procedures are being deployed for the first time for the AGR-3/4 PIE, one or more other compacts may be selected from the remaining available compacts as test cases to evaluate the efficacy of the method before beginning the test matrix given in Table 7.

Selected fuel body compacts may also be deconsolidated following fuel body heating tests. Specific compacts will be identified once a test matrix is established for the fuel body heating tests.

### 3.6.5 Fuel Body Heating Tests

A detailed test matrix for post-irradiation fuel body heating tests governing the use of fuel bodies and compacts in Capsules 2, 6, 9, and 11 will be developed at a later time.

Table 7. Provisional list of compacts identified for destructive radial deconsolidation and subsequent analysis.

Capsule	Compacts	Burnup (% FIMA)	TAVA Temp. (°C)	Comments
<i>As-irradiated compact analysis</i>				
3	3-4	12.9	1168	Rings used for DFPA
	3-3	12.7	1205	
7	7-4	14.9	1319	Rings used for DFPA
	7-3	15.0	1376	
5	5-4	15.0	989	Rings used for DFPA
	5-3	14.9	1050	
8	8-4	14.4	1169	Rings used for DFPA
	8-3	14.5	1213	
1	1-4	6.9	929	No ring DFPA, but provides compact data at low temperature and burnup
	1-3	6.4	959	
<i>Post-heating test compact analysis</i>				
10	10-4	11.4	1168	
	10-2	12.0	1213	
	10-1	12.1	1172	
3	3-2	12.5	1196	
	3-1	12.2	1138	
8	8-2	14.6	1213	
5	5-1	14.7	973	
1	1-2	5.9	941	
<i>Fuel body compacts following heating tests</i>				
(Compact IDs TBD)				

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#### **4. WASTE HANDLING**

PIE activities will generate small amounts of radioactive waste (estimated at less than 0.6 m<sup>3</sup> per year) that must be properly dispositioned. This waste will be generated by disassembly, post-irradiation heating, equipment maintenance activities, and Analytical Laboratory activities associated with AGR-3/4 examination and analysis. Typical waste will include short sections (i.e., less than 2 m) of 1/16 to 1/8-in. diameter sheathed thermocouples and gas lines, turnings from the tubing cutter, and Analytical Laboratory solids and solidified liquids. Additionally, after analysis activities of test train capsule components (i.e., capsule head and base, through tubes, outer shell, and zirconia/zirconium spacers) are completed, these components will be dispositioned as waste. Most waste will be classified as remote-handled low-level waste. Some of the waste, such as activated stainless steel, may be classified as greater-than-Class-C waste. Waste will be gathered, placed into appropriate disposal containers, and stored in the Radioactive Scrap and Waste Facility located at MFC until final disposal arrangements can be made.

#### **5. QUALITY ASSURANCE**

The ART Technology Development Office (TDO)'s Program Management Plan (Croson 2015), identifies that all ART TDO activities are conducted in accordance with requirements identified in the quality assurance program plan (INL 2015).

Work activities associated with this plan are conducted under a quality program implementing American Society of Mechanical Engineers NQA-1 2008/-1a-2009. Organizations or services subcontracted to support quality-affecting PIE work activities will be on the INL Qualified Suppliers List for the selected activities to be performed. Activities affecting quality include, but are not limited to, procurement, handling, shipping, storing, inspecting, testing, training, data collection, records, electronic data storage, software control for software used in data analysis, and generation of reports from collected data. ORNL will perform PIE support services in accordance with their project AGR-specific quality assurance program plan (QAP-ORNL-NR&D-01; Vance 2013).

#### **6. DATA MANAGEMENT**

INL is responsible for maintaining a record copy of all data associated with PIE activities. These data may come from INL, ORNL, Pacific Northwest National Laboratory, universities, or other partners in the PIE effort. INL will work with these institutions to define desired data formats. PIE and safety testing data will be kept as project records and will be transferred from their original source to either the Nuclear Data Management and Analysis System (NDMAS) or the INL Electronic Document Management System. Primarily, NDMAS will be the data storage forum for machine-readable data (e.g., database, spreadsheet, or tab delineated) and the Electronic Document Management System will be the storage forum for other types of information, including pictures, evaluation reports, pdf documents, technical evaluations, and engineering calculation and analysis reports. Because NDMAS will have provisions that allow access to data outside the INL computer firewall, data that would normally be stored on the Electronic Document Management System may be moved to NDMAS to allow access by users outside INL. The Very High-Temperature Program Data Management and Analysis Plan (Hull 2016) details how data will be stored, controlled, categorized, and qualified.

Nuclear data from the latest Evaluated Nuclear Data File (ENDF) database (currently ENDF/B-VII.1, Chadwick et al. 2011) will be used for decay-corrections of measured radioisotopic inventories (for comparison with predicted values) and for relevant gamma ray yields used in spectral processing.

#### **7. REPORTING**

Program staff will create reports pertaining to results from PIE of AGR-3/4 capsules to ensure pertinent data from PIE activities are available for various programmatic decisions as necessary. Because

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of the number of AGR-3/4 capsules and components involved and the time required for some of the analyses, it may be advantageous to issue some results in phases so data are available earlier in the PIE process. Reports that are completed or planned include the following:

- *Test train inspection, disassembly, and metrology report.* This report (INL/EXT-16-38005) provides information on test train receipt and inspection, test train gamma scanning, capsule disassembly and inspection, and dimensional measurement of fuel compacts and capsule components.
- *Topical reports.* Topical reports will be prepared to provide details on specific portions of AGR-3/4 PIE. These will include topical reports summarizing results of inner and outer ring and compact gamma scanning, inner and outer ring sampling, and analysis of fission product inventory on capsule components. These topical reports may be issued in phases, with only data from selected capsules included to accelerate dissemination of results and avoid excessive delays that would be required if data from all capsules were to be included. These will also include reports detailing results from destructive examination of specific compacts and reports detailing results from heating tests.
- *Final AGR-3/4 PIE data report.* This report will be prepared after completion of the AGR-3/4 PIE and when all data have been obtained from ongoing experiments and analyses. It will include data summaries taken from relevant topical reports and will present pertinent conclusions from the AGR-3/4 PIE and heating tests.

Regular input on PIE activities and experimental results will be provided as needed for the ART TRISO Fuel Program's monthly and quarterly reports and weekly highlights. ART-TRISO PIE staff will make selected PIE data available to program participants as it is generated and will participate in bi-weekly teleconferences, ART-TRISO Fuel Program meetings, and ART-TRISO annual technology review meetings to facilitate dissemination of experimental data as needed by the program and to discuss relevant issues. It is expected that very early results (covering examination of the first few AGR 3/4 capsules) will need to be distributed in this manner so the program can make decisions regarding Phase 2 AGR-3/4 PIE activities.

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