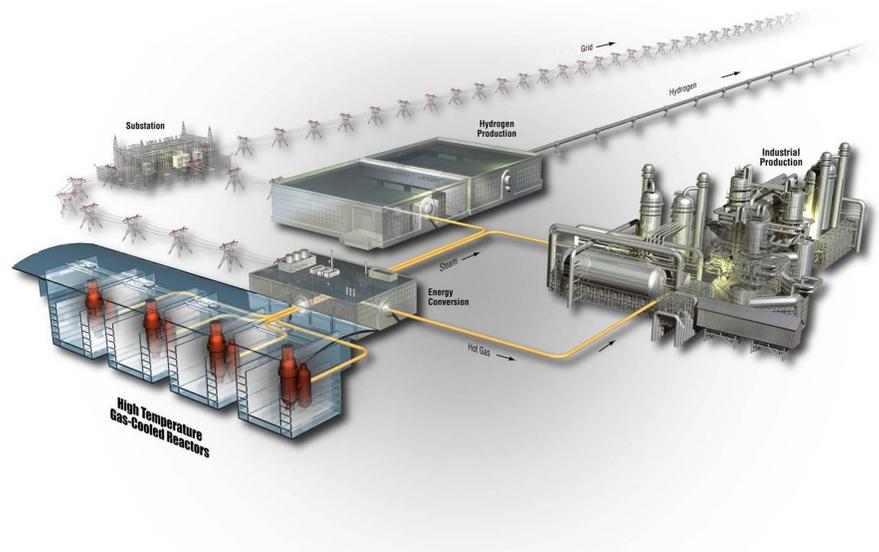


## Plan

Project No. 29412, 23841

# AGR-5/6/7 Post-Irradiation Examination Plan

The INL is a  
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**Idaho National Laboratory**

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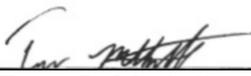
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## ACRONYMS

AGR	Advanced Gas Reactor Program
AL	Analytical Laboratory
AMIX	Air/Moisture Ingress Experiment
ART	Advanced Reactor Technologies
ASME	American Society of Mechanical Engineers
ATR	Advanced Test Reactor
AVR	Arbeitsgemeinschaft Versuchsreaktor
BWXT	BWX Technologies, Inc.
CCCTF	Core Conduction-Cooldown Test Facility
CIC	core-internal-changeout
CT	computed tomography
DAW	Dry Active Waste
DLBL	deconsolidation-leach-burn-leach
DTF	designed-to-fail
ECAR	engineering calculation and analysis report
EBSD	electron backscatter diffraction
EDMS	INL Electronic Document Management System
EDS	energy-dispersive X-ray spectroscopy
EELS	electron energy loss spectroscopy
EML	Electron Microscopy Laboratory
ENDF	Evaluated Nuclear Data File
EPMA	electron probe microanalyzer
FACS	Fuel Accident Condition Simulator
FGMS	Fission Gas Monitoring System
FIB	focused ion beam
FIMA	fissions per initial metal atom
FITT	Furnace for Irradiated TRISO Testing
FPMS	fission product monitoring system
HFEF	Hot Fuel Examination Facility
HOG	HFEF Out-of-cell Gamma
HTGR	high-temperature gas-cooled reactor
ICP-MS	inductively-coupled plasma mass-spectrometry
IFEL	Irradiated Fuels Examination Laboratory
IMCL	Irradiated Materials Characterization Laboratory
IMGA	Irradiate Microsphere Gamma Analyzer
INL	Idaho National Laboratory
IPyC	Inner-pyrolytic carbon layer
MFC	Materials and Fuels Complex
NDMAS	Nuclear Data Management and Analysis System
NEFT	Northeast Flux Trap
NRAD	Neutron Radiography Reactor

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NQA	Nuclear Quality Assurance
OD	outer diameter
OPyC	outer-pyrolytic carbon
ORNL	Oak Ridge National Laboratory
OPTAF	Optical anisotropy factor
PDF	Portable Document Format
PGS	Precision Gamma Scanner
PIE	post-irradiation examination
QAPP	quality assurance program plan
SEM	scanning electron microscopy
SPND	self-powered neutron detector
STEM	scanning-transmission electron microscopy
TA	time-averaged
TAVA	time-averaged, volume-averaged
TC	thermocouple
TEM	transmission electron microscopy
TEV	technical evaluation
TRIGA	Training Isotopes General Atomics
TRISO	tristructural isotropic
UCO	a heterogenous mixture of uranium carbide and uranium oxide
WDS	wavelength-dispersive spectroscopy

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**1. INTRODUCTION****1.1 Purpose**

The Advanced Gas Reactor (AGR) fuel development and qualification program was established to perform research and development on tristructural isotropic (TRISO)-coated particle fuel for high-temperature gas-cooled reactors (HTGRs). This work continues as part of the Advanced Reactor Technologies (ART) program. The overarching goal of the ART AGR program is to provide a baseline fuel qualification dataset to support licensing, deployment, and operation of HTGRs in the United States. To achieve these goals, the program includes the elements of fuel fabrication, irradiation, post-irradiation examination (PIE), fuel performance modeling, and fission product transport (Idaho National Laboratory 2020). Several fuel irradiation experiments have already been performed at the Advanced Test Reactor (ATR) at Idaho National Laboratory (INL), and the fourth and final irradiation, designated AGR-5/6/7, began in February 2018 and ended in July 2020.

This plan describes the PIE activities to be carried out on AGR-5/6/7 fuel and the non-fuel components of the AGR-5/6/7 irradiation test train. PIE of AGR-5/6/7 fuel and test-train components will provide data on fuel performance over a range of irradiation conditions (e.g., burnup, neutron fluence, and temperature), test fuel under postulated accident conditions, and support development of fuel performance and fission product transport models. Examination of the non-fuel test-train components will give information on fuel performance and releases of radioactive fission products from the fuel compacts. In cases of abnormal, in-pile performance of the fuel or test-train (see Section 1.4.4), inspecting the components of the test train (e.g., capsules, thermocouples, gas lines, and brazed joints, etc.) may help to explain such anomalies. PIE of the fuel compacts is used for many purposes including, but not limited to: updating the irradiation thermal calculations, measuring burnup, determining retention/release of fission products from the compacts and the TRISO particles, determining TRISO layer failure rates, determining fuel performance at elevated accident temperatures, determining fuel performance under oxidizing atmospheres, determining the distribution of fission products within the compacts and fuel particles, and observing compact and particle morphology to explain mechanisms of TRISO-layer failure and/or fission product release/retention.

**1.2 Objectives of PIE**

Extensive PIE has been performed on uranium oxycarbide (UCO) TRISO fuel from AGR-1 (Demkowicz et al. 2018), AGR-2 (Hunn et al. 2018), and AGR-3/4 (Stempien et al. 2018a). General expectations for the behavior and performance of this fuel and the relevant phenomena related to UCO fuel performance were established via those efforts. The AGR-5/6/7 irradiation is the final qualification test of fuel made entirely at the engineering scale. The intent of AGR-5/6/7 PIE is to collect data on fuel performance, compare those data to results obtained in the earlier experiments (e.g., AGR-1 and AGR-2), and determine whether any new phenomena arise to challenge the existing understanding of UCO TRISO fuel irradiation performance. In so doing, the database supporting UCO TRISO fuel qualification will be expanded. There is strong motivation to complete AGR-5/6/7 PIE in a timely fashion. Accordingly, some simplifications to the PIE processes/activities used in earlier AGR experiments may be made when appropriate.

The following are major objectives of the AGR-5/6/7 PIE campaign:

1. Evaluate and characterize unexpected Capsule 1 behavior (see discussions in Sections 1.4.4 and 2.4.2.2).

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2. Determine if the performance and behavior of the fuel under normal irradiation conditions was acceptable (primarily Capsules 2, 4, and 5) and synthesize these results with PIE results obtained from the earlier AGR irradiations.
3. Evaluate performance and characterize behavior of fuel under high-irradiation temperatures (Capsule 3).
4. Conduct post-irradiation high-temperature testing in helium to verify acceptable fuel performance under conduction cool-down accidents.
5. Perform oxidation testing to characterize fuel behavior during exposure to air or moisture at nominal and accident temperatures.

These objectives are related to quantifying fission product release from the fuel under normal irradiation conditions, high-temperature irradiation conditions (sometimes referred to as margin testing), and accident conditions. One focus of as-irradiated PIE and accident testing is on quantifying the frequency of certain types of TRISO coating failures (e.g., SiC layer failure or complete TRISO failures) and the mechanism(s) that may have led to these failures. Destructive exams aim to observe changes to the fuel morphology (e.g., layer debonding, cracking, irradiation-induced dimensional changes, chemical reaction, etc.) that impact coating integrity and/or fission product retention. Certain destructive exams aim to quantify the fission product release associated with both intact and degraded TRISO coatings. Another focus of as-irradiated PIE is to determine the quantity of fission products that may have been released from intact TRISO particles that is still retained within the fuel compact matrix.

Aside from oxidation testing, the methods employed in AGR-5/6/7 PIE will be the same as those used in previous AGR PIE campaigns. AGR-5/6/7 PIE will be a collaborative effort between INL and Oak Ridge National Laboratory (ORNL). Irradiation test-train disassembly, non-destructive analyses, and capsule components analysis will take place at INL. Fuel compact destructive exams and post-irradiation heating tests will take place both at INL and at ORNL. The specific fuel compacts to be sent to ORNL will be determined based on the final irradiation conditions of those compacts and the results from initial PIE at INL.

### 1.3 Background

The first two AGR fuel irradiation experiments (AGR-1 and AGR-2) had similar test-train designs for the irradiations in ATR, and one objective was to test UCO TRISO-coated particle fuel performance over a range of irradiation temperatures and burnups. UCO fuel kernels are a heterogeneous mixture of uranium carbide and uranium oxide. AGR fuel fabrication was progressively scaled-up from the laboratory scale to the engineering scale (see Table 1). All engineering-scale kernels, including AGR-1 and AGR-2 kernels, were produced at BWX Technologies, Inc. (BWXT). AGR-1 TRISO coatings were applied to the kernels on a laboratory scale at ORNL. Some deliberate variations in coating process conditions were used in AGR-1 to produce a “baseline” coating along with three other variants. AGR-2 TRISO coatings were produced in a 6-inch-diameter, engineering-scale coater at BWXT using conditions derived from the AGR-1 Variant 3 fuel. For both AGR-1 and AGR-2 the TRISO fuel particles were overcoated and compacted into cylindrical fuel compacts at ORNL.

AGR-1 was irradiated in the B-10 position in ATR from December 2006 to November 2009 (Collin 2015a). In addition to AGR UCO fuel (in Capsules 2, 5, and 6), the AGR-2 experiment also had AGR-produced UO<sub>2</sub> fuel (in Capsule 3) to compare the performance of UCO versus UO<sub>2</sub> fuel and to compare to UO<sub>2</sub> fuel performance observed historically in the German TRISO fuel program. AGR-2 Capsules 1 and 4 contained fuel where the UO<sub>2</sub> TRISO particles were produced in France and South Africa, respectively. The French fuel was also compacted in France, but South African particles were compacted at ORNL. AGR-2 was irradiated in the B-12 position of ATR from June 2010 to October 2013 (Collin 2018a).

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AGR-1 PIE is complete (Demkowicz et al. 2015, Demkowicz et al. 2018). AGR-2 PIE began in July 2014 and encompasses as-irradiated analyses, reirradiations, heating tests, microscopy, and other activities.

The third irradiation experiment, AGR-3/4, was designed to investigate the migration of fission products in fuel compact graphitic matrix and reactor graphite components. The experiment consisted of fuel compacts containing TRISO-coated UCO driver fuel particles similar to AGR-1 baseline fuel (Collin 2015b; Hunn and Lowden 2007; Hunn et al. 2014) and designed-to-fail (DTF) particles that were intended to provide a well-defined source of fission product releases during irradiation to migrate through the surrounding cylindrical rings of graphitic matrix and nuclear-grade graphite. PIE activities are in progress to measure the fission product distributions in these rings and fuel compacts. These data will support refinement of fission product transport models and HTGR source-term analyses (Demkowicz 2017). Reirradiation/heating tests of the AGR-3/4 fuel compacts to determine the release of short-lived fission products (e.g., I-131) from exposed kernels are also in progress.

Table 1. AGR fuel fabrication scales. Contents based on (Petti et al. 2017).

<b>Experiment</b>	<b>Kernels</b>	<b>TRISO Coatings</b>	<b>Compacts</b>
AGR-1	Engineering	Laboratory	Laboratory
AGR-2	Engineering	Engineering	Laboratory
AGR-3/4	Engineering	Laboratory	Laboratory
AGR-5/6/7	Engineering	Engineering	Engineering

## 1.4 AGR-5/6/7 Irradiation Experiment

AGR-5/6/7, the fourth and final irradiation experiment in the AGR campaign, is intended to serve as the fuel qualification irradiation (Idaho National Laboratory 2020, Collin 2018b). Data collected from earlier irradiations (i.e., AGR-1, AGR-2, and AGR-3/4) may be used to supplement data collected from AGR-5/6/7. The irradiation began in the Northeast Flux Trap (NEFT) position of ATR in February 2018. In order to remove the experiment from ATR and ship it to the Hot Fuel Examination Facility (HFEF) at the Materials and Fuels Complex (MFC) before ATR core-internal-changeout commences, the decision was made to end the irradiation on July 22, 2020 at the end of ATR Cycle 168A. Ending the irradiation early means the fuel accrued less burnup than was originally planned. In all capsules, the non-fuel test-train components will be inspected for signs of degradation or anomalous behavior and analyzed for fission products and select fuel compacts will be subject to a host of PIE. Special attention will be given to Capsule 1, which in-pile data show experienced unexpected TRISO particle failures and sweep gas flow obstructions (see discussions in Sections 1.4.4 and 2.4.2.2).

### 1.4.1 AGR-5/6/7 Fuel

All elements of the AGR-5/6/7 fuel (i.e., UCO kernels, TRISO coatings, and fuel compacts) were produced at engineering scale at BWXT to the specifications in (Marshall 2017). Several production kernel batches were combined into a single composite: Lot J52R-16-69317. The kernels from this lot were then TRISO-coated, and several of these coating batches were combined into a single TRISO-coated composite Lot J52R-16-98005. Table 2 summarizes selected properties from this composite particle lot.

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Table 2. Selected properties for AGR-5/6/7 kernel Lot J52R-16-69317 and TRISO-coated particle Lot J52R-16-98005. Except where noted, all values are from (Collin 2018b).

Property	Mean $\pm$ population standard deviation
Kernel diameter ( $\mu\text{m}$ )	425.78 $\pm$ 10.42
U-235 enrichment (wt%)	15.477 $\pm$ 0.013
Kernel density ( $\text{g}/\text{cm}^3$ )	11.048 $\pm$ 0.044
Buffer thickness ( $\mu\text{m}$ )	100.37 $\pm$ 5.55
IPyC thickness ( $\mu\text{m}$ )	39.24 $\pm$ 1.26
SiC thickness ( $\mu\text{m}$ )	36.15 $\pm$ 0.65
OPyC thickness ( $\mu\text{m}$ )	35.03 $\pm$ 1.99
Buffer density ( $\text{g}/\text{cm}^3$ )	1.031 $\pm$ 0.022
IPyC density ( $\text{g}/\text{cm}^3$ )	1.897 $\pm$ 0.010
SiC density ( $\text{g}/\text{cm}^3$ )	3.195 $\pm$ 0.002
OPyC density ( $\text{g}/\text{cm}^3$ )	1.897 $\pm$ 0.004
IPyC anisotropy (OPTAF) <sup>(a,b)</sup>	1.031 $\pm$ 0.002
OPyC anisotropy (OPTAF) <sup>(a,b)</sup>	1.021 $\pm$ 0.001
IPyC anisotropy post-compacting anneal (OPTAF) <sup>(a,b)</sup>	1.0388 $\pm$ 0.0085
OPyC anisotropy post-compacting anneal (OPTAF) <sup>(a,b)</sup>	1.0296 $\pm$ 0.0078
Particle diameter ( $\mu\text{m}$ ) <sup>(c)</sup>	866.7 $\pm$ 12.2
Particle mass (mg) <sup>(d)</sup>	1.014 $\pm$ 0.006
<p>a. OPTAF is calculated from the measured diattenuation, N, via the following equation: <math>\text{OPTAF} = \frac{1+N}{1-N}</math></p> <p>b. Measured diattenuation, N, and standard deviation of OPTAF for combined data from two samples from particle composite J52R-16-98005 from (Helmreich et al. 2017).</p> <p>c. The value here is from combining particle diameter measurements from multiple batches to give an average diameter for the particle lot (Marshall 2020a).</p> <p>d. From combined data from two samples from particle composite J52R-16-98005 (Helmreich et al. 2017).</p> <p>IPyC: Inner-pyrolytic carbon  OPyC: Outer-pyrolytic carbon  OPTAF: optical anisotropy factor</p>	

TRISO particles were overcoated in a resinated graphite and formed into right cylindrical compacts at BWXT. Compacts were produced in four different batches. Two of the batches had nominal particle packing fractions of 40%, and two had nominal particle packing fractions of 25%. Table 3 provides information on each of the four compact batches. Post-compacting analyses were performed to determine properties such as compact dimensions, compact graphitic matrix density, the fraction of TRISO-coating defects, and the fraction of uranium contamination in the compacts. Table 4 summarizes selected properties for the AGR-5/6/7 fuel compacts loaded into each of the five AGR-5/6/7 irradiation capsules. Additional details and references for these values are available in (Collin 2018b) and the other sources cited in Table 2 through Table 4.

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Table 3. AGR-5/6/7 compact batches, packing fractions, and irradiation capsule loadings. Table contents from (Collin 2018b).

Batch	Nominal Packing Fraction	Compact Serial Number (S/N) Range	Capsule	Number of Compacts
J52R-16-14154A	40%	0001 to 0108	1	55
J52R-16-14155A	40%	0109 to 0216	1 5	35 24
J52R-16-14156A	25%	0217 to 0324	2 3	32 24
J52R-16-14157A	25%	0325 to 0432	4	24

Table 4. Selected properties for AGR-5/6/7 fuel compacts. Values are from (Collin 2018b) unless noted otherwise. Additional as-fabricated fuel characterization data are available in the AGR-5/6/7 Fuel Fabrication Report (Marshall 2019b).

Property	Actual Mean Value ± Population Standard Deviation
Compact mass (g)	
Capsule 1	6.676 ± 0.065
Capsule 2	6.182 ± 0.026
Capsule 3	6.187 ± 0.021
Capsule 4	6.100 ± 0.034
Capsule 5	6.603 ± 0.021
Mean uranium loading (g U/compact)	
Capsule 1	1.362 ± 0.014
Capsule 2	0.898 ± 0.004
Capsule 3	0.898 ± 0.003
Capsule 4	0.871 ± 0.005
Capsule 5	1.346 ± 0.004
Diameter <sup>(a)</sup> (mm)	
Capsule 1	12.293 ± 0.007
Capsule 2	12.241 ± 0.007
Capsule 3	12.245 ± 0.006
Capsule 4	12.248 ± 0.006
Capsule 5	12.296 ± 0.006
Length <sup>(a)</sup> (mm)	
Capsule 1	24.947 ± 0.219
Capsule 2	24.991 ± 0.098
Capsule 3	25.000 ± 0.078
Capsule 4	24.770 ± 0.119
Capsule 5	24.675 ± 0.059
Estimated mean number of particles per compact <sup>(b)</sup>	
Capsule 1	3434
Capsule 2	2264
Capsule 3	2265
Capsule 4	2197
Capsule 5	3393
Particle volume packing fraction <sup>(b)</sup> (%)	
Capsule 1	38.4
Capsule 2	25.5

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Property	Actual Mean Value ± Population Standard Deviation
Capsule 3	25.5
Capsule 4	24.9
Capsule 5	38.4
Effective overall compact density <sup>(b)</sup> (g/cm <sup>3</sup> )	
Capsule 1	2.26
Capsule 2	2.10
Capsule 3	2.10
Capsule 4	2.09
Capsule 5	2.25
Compact matrix density <sup>(b)</sup> (g/cm <sup>3</sup> )	
Capsule 1	1.748 ± 0.007
Capsule 2	1.772 ± 0.005
Capsule 3	1.771 ± 0.005
Capsule 4	1.766 ± 0.006
Capsule 5	1.747 ± 0.007
Dispersed uranium fraction <sup>(c, d, e)</sup> (g leached U/g U in compact)	
Nominal 25% packing fraction	≤ 5.59 × 10 <sup>-6</sup> <sup>(f)</sup>
Nominal 40% packing fraction	≤ 5.68 × 10 <sup>-6</sup> <sup>(f)</sup>
Exposed kernel fraction <sup>(c, e)</sup> (kernel equivalent/particle count)	
Nominal 25% packing fraction	≤ 3.45 × 10 <sup>-5</sup>
Nominal 40% packing fraction	≤ 8.30 × 10 <sup>-5</sup> <sup>(f)</sup>
Defective SiC coating fraction <sup>(c, e)</sup>	
Nominal 25% packing fraction	≤ 5.64 × 10 <sup>-5</sup> <sup>(f)</sup>
Nominal 40% packing fraction	≤ 7.43 × 10 <sup>-5</sup>
Defective IPyC coating fraction <sup>(c)</sup>	≤ 7.6 × 10 <sup>-5</sup>
Defective OPyC coating fraction <sup>(c)</sup>	≤ 8.6 × 10 <sup>-5</sup>
<p>a. Allowable range corresponding to upper and lower critical limits specified with no compacts exceeding the limits, which requires 100% inspection of all compacts.</p> <p>b. Calculated value derived from other characterized properties.</p> <p>c. Upper limit at 95% confidence.</p> <p>d. The value given is the total dispersed uranium fraction in a compact. Some of the dispersed uranium appeared to be uniformly dispersed in the compacts, and some did not. The uniformly dispersed component was ≤5.7E-6 for 25% packing fraction compacts and ≤5.9E-6 for 40% packing fraction compacts (Hunn et al. 2019c). It was concluded that the majority of the dispersed uranium was in the TRISO particle composite and not the result of TRISO particle overcoating or compacting (Hunn et al. 2019c).</p> <p>e. Values from (Marshall 2020b).</p> <p>f. The 95% confidence fraction exceeds the specification; despite the non-conformance, it was decided to accept the fuel as is (Marshall 2020b).</p>	

#### 1.4.2 AGR-5/6/7 Irradiation Test Train

AGR-5/6/7 is the combination of what was originally conceived as three separate irradiations into a single irradiation. The AGR-5/6/7 irradiation test train consists of five independently instrumented and controlled capsules. Table 5 lists selected engineering drawings of major components of the AGR-5/6/7 irradiation test train. Multiple different capsule designs were used within the AGR-5/6/7 test train. Figure 1 shows a schematic of an axial cross section of the assembled test train that was loaded into NEFT at ATR, and Figure 2 shows schematic radial cross sections of each of the five different capsules. The stainless-steel capsule shells acted as the experiment pressure boundary, separating the experiment from the ATR coolant water that flowed around the outside of the test train. The stainless-steel shells for the capsules were welded together, one on top of the other, and numbered from bottom to top. Figure 2 shows that Capsule 1 has 10 stacks of fuel compacts, and Figure 1 shows that each of these 10 stacks is

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comprised of nine fuel compacts. Capsules 2, 4, and 5 each have four stacks of compacts; however, in Capsule 2, each stack has eight compacts (compared to six compacts per stack in Capsules 4 and 5). Capsule 3 has three compact stacks containing eight compacts each. Capsules 1, 2, 4, and 5 are considered to be the fuel qualification test, AGR-5/6, and Capsule 3 is considered to be the high-temperature margin test, AGR-7.

Each capsule had an independent helium and neon gas supply to control the temperature within the capsule by varying the gas composition. The gas supply was also used to sweep gaseous fission products into the fission product monitoring system (FPMS) for measurement. A number of thermocouples (TCs) were installed in each capsule for temperature monitoring. Capsule 1 does not have any through-tubes, but Capsules 2 through 5 each have four through-tubes to accommodate gas lines and thermocouple leads from capsules below them in the test train. Capsule 3 has two graphite holders: an inner holder for the fuel compacts and an outer holder. The other capsules each have only a single graphite holder with a hollow center designed to reduce graphite mass and graphite volumetric heating (Collin 2018b). All holders were made from IG-430 graphite. Figure 2 shows a series of “nubs” on the outside of each capsule shell. These nubs helped to center the test train in NEFT. A set of double nubs (shown in Figure 2 at the 7 o’clock position on each shell) points to the southeast direction and can be used to denote the orientation of the capsules when they were in ATR. Each fuel compact stack is numbered according to Figure 2, and noting the positioning of the compact stacks in relation to the nubs can be used to help determine which stack is which.

Table 5. Selected engineering drawings of the AGR-5/6/7 test train and individual irradiation capsules.

Component	Drawing Number
AGR-5/6/7 Drawing Tree	604650
AGR-5/6/7 Test Train Assembly	604652
AGR-5/6/7 Capsules 1 thru 5 Assembly Weldment	604660
AGR-5/6/7 Capsule 1 Assembly and Details	604661
AGR-5/6/7 Capsule 2 Assembly and Details	604662
AGR-5/6/7 Capsule 3 Assembly and Details	604663
AGR-5/6/7 Capsule 4 Assembly and Details	604664
AGR-5/6/7 Capsule 5 Assembly and Details	604665

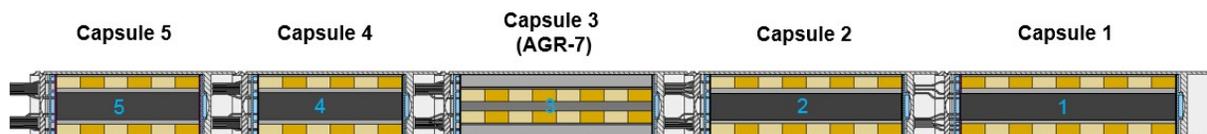


Figure 1. Axial cross-sectional schematic of the AGR-5/6/7 test train.

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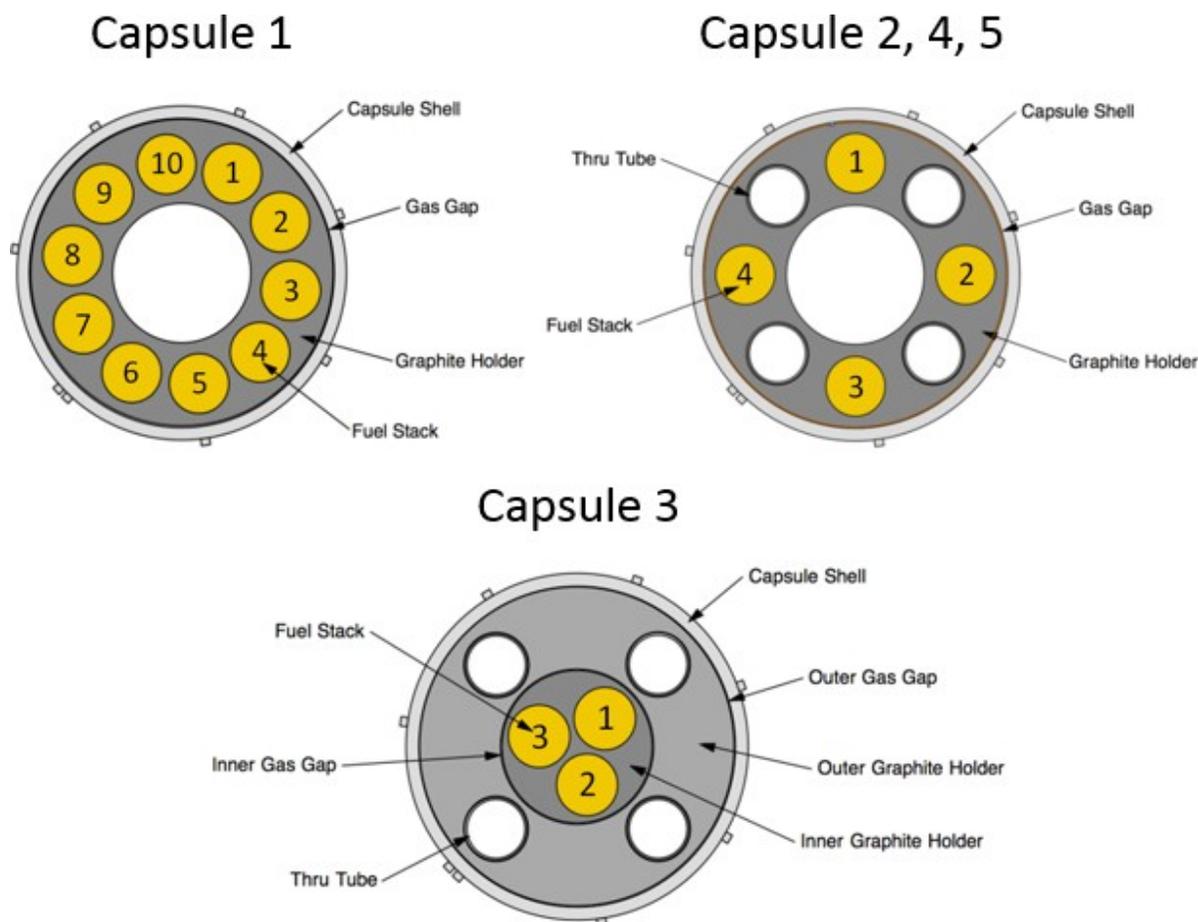


Figure 2. Radial cross sections of the AGR-5/6/7 capsules.

#### 1.4.2.1 Thermocouples

A discussion of TC type selection and placement is given in (Collin 2018b). The AGR-5/6/7 assembly weldment drawing and the drawings for the individual capsules (listed in Table 5) provide information on the TCs and their positions within the experiment. TCs penetrated the capsule heads and extended into small channels in the graphite holders. There were four different TC configurations in the AGR-5/6/7 test train. The major types of TC configurations were as follows:

- Type N (Ni/Cr/Si/Mg wire) with Inconel 600 (Ni/Cr/Fe/Mn alloy) sheath, MgO insulation, and sleeved with Nb (standard baseline)
- Type N with Cambridge low-drift pure Ni sheath, MgO insulation, and sleeved with Nb in AGR-5/6 capsules and with ZrO<sub>2</sub> in AGR-7 Capsule 3
- Type N with Inconel 600 sheath, Spinel (MgAl<sub>2</sub>O<sub>4</sub>) insulation, and sleeved with Nb
- High-temperature irradiation resistant (Mo/Nb wire) with Nb sheath, Al<sub>2</sub>O<sub>3</sub> insulation, and sleeved with Mo.

In addition to the TCs discussed above, some temperature-sensing devices considered nonessential “supplementary instrumentation” were also included (Collin 2018b). Capsules 2, 3, 4, and 5 had self-

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powered neutron detectors (SPNDs) in the through-tubes that also had TCs necessary for proper measurements via the SPNDs. Capsule 5 also had an ultrasonic temperature sensor and two fiber-optic temperature sensors in its graphite holders.

#### **1.4.2.2 Through-tubes and Gas Lines**

Through-tubes acted as conduits for TCs, gas lines, and nonessential supplementary instrumentation, running through the irradiation experiment test train and out of ATR to the FPMS and data collection systems. Capsule 1 did not have any through-tubes, but through-tubes ran the length of Capsules 2 through 5. The through-tubes penetrated the top and bottom heads of the capsules, were brazed at the top head, and slip-fitted at the bottom head. Neolube<sup>®</sup> lubrication was applied around the tubes where they passed through the bottom heads. In Capsules 2, 4, and 5, the through-tubes were made of stainless steel and wrapped in a 0.25-mm-thin Mo sleeve to prevent migration of transition metals in the TCs through the graphite holders. Capsule 3 was designed with Mo through-tubes only to allow additional space for instrumentation; however, the Mo through-tubes cracked during assembly and stainless-steel through-tubes were inserted inside the cracked Mo tubes (Collin 2018b).

Each capsule had a gas inlet and outlet. The gas inlet line penetrated the capsule head and ran in a small channel in the graphite holder to the bottom of the specific capsule to which it was supplying gas. In each capsule, the gas outlet line barely penetrated the capsule head to access the plenum at the top of the capsule.

#### **1.4.2.3 Miscellaneous Capsule Components**

Each capsule also has an assortment of smaller items used to support and position the compacts and graphite compact holders. This includes spacers, disks, insulators, gamma “heaters,” and the Capsule 1 spring. The spacers are made of zirconia or graphite. The disks are made of Grafoil<sup>®</sup>. The insulators are made of zirconia. In Capsule 1, the spring is made of Inconel, and the Capsule 3 heaters are made of tungsten. The Capsules 4 and 5 SPNDs are inside titanium tubes, which are inside the through-tubes.

### **1.4.3 AGR-5/6/7 Irradiation Conditions**

The AGR-5/6/7 irradiation test plan stated that the irradiation was scheduled for approximately 500 effective full power days in ATR (Collin 2018b). Significant ATR maintenance activities have been scheduled for March 2021, namely, core-internal-changeout (CIC). Access to and shipment of the AGR-5/6/7 irradiation test train will not be possible during CIC. Thus, the decision was made to end the AGR-5/6/7 irradiation on 7/22/2020 (earlier than scheduled) so that the test train has sufficient cooling time in the ATR fuel cooling channel and can be shipped to HFEF at MFC prior to the start of CIC. Due to ending the irradiation early, peak burnup and fluence will be lower than the targets listed in the AGR-5/6/7 Irradiation Test Specification (Maki 2015) and the intended conditions summarized in the test plan (Collin 2018b).

#### **1.4.4 Unusual Behavior in Capsule 1**

There have been gas flow issues (both restrictions and breaks in the gas lines, occurring intermittently) within Capsule 1 that have made online fission gas measurements difficult or impossible and made it difficult to control the neon/helium gas blend used for temperature control. Near the end of ATR Cycle 166A, in-pile fission gas measurements indicated significant numbers of TRISO coating failures in Capsule 1 (estimated to be on the order of thousands). Based on the performance of fuel in

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prior AGR irradiations, significant TRISO failures were not expected to occur in Capsule 1 at any point during the AGR-5/6/7 irradiation. The cause of these failures is unknown; however, a variety of causes have been postulated. The conditions of the fuel, graphite compact holder, gas lines, TCs and other capsule internals are also unknown.

The hypotheses that have been posited to explain the TRISO failures in Capsule 1 generally fall into two categories. The first category of hypotheses is that the fuel failed due to undetected fabrication defects or some to-be-determined irradiation-induced mechanisms. The second category of hypotheses is that the design or assembly of the test train itself, or unexpected operational issues during irradiation, damaged the fuel by an as-yet unknown mechanism(s). One hypothesis for how the test train could have damaged the fuel is that the graphite holder housing the compacts (see Figure 2) has undergone irradiation-induced shrinkage, reached its turn-around point, and then swelled, crushing particles on the outside of the compacts. This could make it difficult to remove the compacts from the holder during PIE. Another hypothesis is that a small leak in the Capsule 1 gas system allowed air to enter the capsule and oxidize the fuel. Other hypotheses include contamination of the fuel with metals from the braze material used on the capsule shell, contamination from failure of the thermocouple sheaths and subsequent transport of transition metals to the TRISO-coated fuel particles, or damage to the compacts during test train assembly. These are not the only potential pathways for the test train to damage the fuel.

Fuel performance-related factors that could damage the fuel in-pile include unexpected irradiation behavior of the new particle overcoating/compact matrix material or damage to the particles from the overcoating process that was not detected during fuel quality control inspections. Given all the uncertainties surrounding Capsule 1, observations of the capsule exterior and examinations of any and all components recovered from the capsule during PIE could be useful.

It is possible that the fuel compacts will be more difficult to retrieve from the Capsule 1 holder than from the other capsules. If the graphite holder and some or all of the compacts did come into hard contact, it may be impossible to push some or all of the compacts out of the holes. (Capsule disassembly is discussed in Section 2.4.2.) The approach and methods used to disassemble Capsule 1 and retrieve its contents shall be conducted with great care in order not to damage the fuel compacts or lose portions of the graphite holders.

## **2. AGR-5/6/7 POST-IRRADIATION EXAMINATION ACTIVITIES AND PRIORITIES**

### **2.1 PIE Activities Flow Diagram**

This PIE plan discusses all components of the AGR-5/6/7 irradiation test that will be subject to PIE. A simplified flow diagram of PIE activities is given in Figure 3. Some activities may be performed in parallel. Fuel destructive exams and heating tests are not depicted in Figure 3. Those activities begin after a compact has been through both analysis on the Precision Gamma Scanner (PGS) and metrology. This flow diagram assumes that all capsules can be readily disassembled and that no unforeseen challenges make disassembly and recovery of capsule contents (e.g., fuel compacts, holders, and hardware) more difficult. Any challenges in capsule disassembly will be dealt with on a case-by-case basis.

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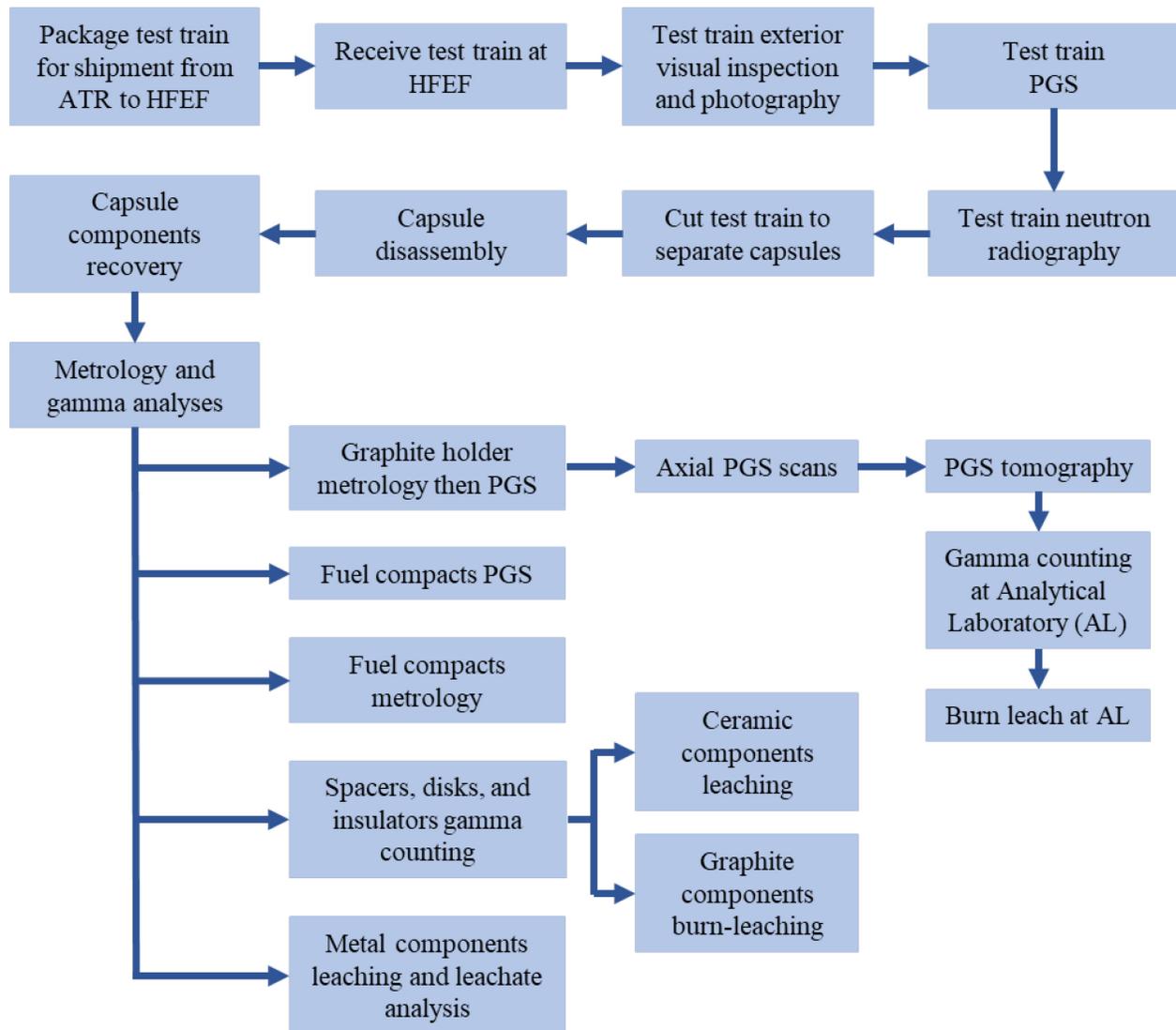


Figure 3. Flow diagram of major PIE activities beginning with shipping the test train from ATR to HFEF. Destructive fuel exams and heating tests are not shown here.

## 2.2 Preliminary Capsule Priorities

Preliminary priorities are given in Table 6, but it is possible that these priorities could change as data are acquired. The listed priorities will be performed in parallel to the extent possible. The focus of PIE is primarily on the fuel, but non-fuel capsule components will be inspected for signs of degradation or anomalous behavior and analyzed for fission products to quantify the fission product inventories released from the fuel compacts. Special attention will be given to all fuel and components in Capsule 1, which in-pile data show experienced unexpected TRISO particle failures and sweep gas flow obstructions (see Sections 1.4.4 and 2.4.2.2).

To quantify the SiC and TRISO failure rates in post-irradiation heating tests, a number of compacts must be tested. The required number of compacts may change based on the results obtained in early tests. Estimated numbers of compacts required to show certain safety-test failure rates are given in Section

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2.15. Given the lack of in-pile fission gas data from Capsule 1, destructive exams on a number of Capsule 1 compacts (see Section 2.10) could be used to estimate the number of TRISO failures in that capsule. That said, the primary goal for Capsule 1 is to determine the cause of significant failure in that capsule, not necessarily to quantify the number of particle failures which, based on limited in-pile fission gas data, could number in the thousands.

Table 6. Preliminary prioritization of the irradiation capsules.

Priority	Capsule	TAVA Temperature (°C)	Burnup (% FIMA)	Remarks
1	1	1100	11.5	Identify the cause(s) of in-pile TRISO failures. In-pile fission gas release indicates that significant fuel failure occurred in this capsule. Blockages of gas flows in this capsule were also observed. In addition to examining the fuel, all the capsule components shall be inspected to determine if the capsule design or capsule components could have caused fuel failure.
2	2	910	18	Determine if intact AGR-5/6 TRISO fuel behaves in-pile and in safety testing like previous fuel from AGR-1 and AGR-2. Determine if observed degradation pathways in AGR-5/6 are via the same mechanisms as observed in AGR-1 and AGR-2. Determine if the phenomena affecting performance in AGR-1 and AGR-2 are the same phenomena affecting AGR-5/6 performance. It is likely that Capsule 1 compacts cannot be used for fuel performance evaluations (e.g., post-irradiation heating tests); therefore, to widen the temperature range, the cooler Capsule 3 fuel compacts ( $\leq 1250^{\circ}\text{C}$ ) may also need to be used for these purposes.
	5	780	10.8	
	4	910	16.5	
3	All	N/A	N/A	Establish fuel compact and TRISO particle performance under oxidizing conditions in air and moisture.
4	3	1380	18	Analyze high-temperature margin test fuel to determine the effects of high-average irradiation temperatures on in-pile performance and accident performance.
FIMA: fissions per initial metal atom TAVA: time-averaged, volume-averaged				

### 2.3 Test Train Shipment to HFEF

After completion of the irradiation, the test train will be removed from ATR and stored in the water canal at ATR for approximately 2 months to allow radioactive decay of the short-lived isotopes. The test train may or may not be sectioned prior to loading into a shielded container. The most likely scenario is that the test train will be sectioned at ATR to give one section consisting of Capsules 1 and 2 and a second section consisting of Capsules 3, 4, and 5. Whether and how test train sectioning is performed shall be documented. After loading into the shielded container, the test train will be transported from ATR to HFEF by truck. The container will be mated to the hot cell, the test train will be removed, and then it will be transferred to a shielded window for external inspection.

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### 2.3.1 Test Train Visual Inspection

After unloading from the shipping cask, the exterior of the train will be visually inspected to identify any significant abnormalities, damage, or degradation. Requirements for visual inspection (including high-resolution photography) of the test-train exterior are given in (Marshall 2019a). The entire test train will be inspected and photographed in segments at a macroscopic scale (using an approximately 5-inch field of view) with a high-resolution digital camera. Fine features of interest, such as the weld seams and brazed joints, can be visually inspected and photographed with a smaller field of view, if necessary. All important features will be examined, and all significant observations will be entered into an inspection log as permanent records to accompany digital photographs.

### 2.3.2 Test-Train Gamma Scanning

At a minimum, the intact Capsule 1 will be examined using the PGS at HFEF. Other capsules may be scanned as time allows. The goal is to verify that no gross relocation of fuel or test-train components occurred. This will be accomplished by locating the fuel within the test train by measuring the gamma rays emitted from the fuel and the gamma rays emitted from neutron activation of the structural components of the test train (e.g. Co-60 gamma rays from activation of stainless-steel). In prior AGR experiments, Cs-137 counts were used to locate the fuel compacts, and Co-60 counts were used to locate major structural components of the test train. It is desirable to scan at least portions of all the compact stacks within a given capsule. This may be accomplished in multiple axial scans. Requirements for PGS analysis of the test train are given in (Marshall 2019a).

### 2.3.3 Test Train Neutron Radiography

The Neutron Radiography (NRAD) reactor beneath the HFEF main hot cell shall be used to perform neutron radiography of the AGR-5/6/7 experiment. Radiography of the intact Capsule 1 is of particular interest. Depending on the availability of NRAD relative to the AGR schedule, the entire test train could be scanned (either as one piece or in segments), or Capsule 1 could be separated from the rest of the test train and scanned intact on its own. NRAD is a pool-type Training Isotopes General Atomics (TRIGA) reactor with multiple neutron beam ports. The radiographs will be useful for assessing the condition of the interior the capsule(s). In AGR-1, items such as the spacers, capsule heads, and compacts were visible (Demkowicz et al. 2011). Recent upgrades to the radiography equipment at NRAD may enable enhanced imaging.

## 2.4 Disassembly, Inspection, Sorting, and Dimensional Measurements

### 2.4.1 Test-Train Disassembly

The overall AGR-5/6/7 test-train length and capsule shell outer diameters are similar to those of AGR-3/4 (Stempien et al. 2016a), and equipment and processes similar to those used for AGR-3/4 will be used for AGR-5/6/7 test-train disassembly. The first step of the process is to cut the capsule-to-capsule segments, separating the capsules from each other one at a time. The test train will be disassembled in the opposite order that it was assembled, meaning that the capsules will be cut from the test-train from top to bottom in descending numerical order. This order of operations is necessary because the gas lines and TCs of each of the lower capsules are routed into the through-tubes of each of the above capsules in the test train. Thus, the top capsule must be cut first in order to pull each capsule free of the gas lines and TCs coming from the capsules below it.

Each capsule will be examined as it is separated from the test train. Along with outer capsule regions, exposed metallic capsule components (e.g., top head and floor/bottom, gas lines, and brazed joints) will be photographed and visually inspected to identify any degradation such as evidence of chemical reactions between components, cracking, discoloration, or failure of the brazed joints. This is especially

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important for the top of Capsule 1, where any and all observations may be useful in explaining the unusual gas flow behavior in that capsule. Gas lines retrieved from Capsule 1 shall be visually examined and photographed to the extent possible.

It is likely that the test train will be sectioned between the top of Capsule 2 and the bottom of Capsule 3 at ATR prior to shipping to HFEF. This will involve severing the gas lines, TCs, and other instrumentation lines between Capsules 2 and 3. All these instrumentation lines will be brittle after irradiation, but attempts shall be made to recover them. In working with the section of the test train comprised of Capsules 1 and 2, the Capsule 1 instrumentation and gas lines will be the only items inside the Capsule 2 through-tubes. In addition to attempting to retrieve instrumentation and gas lines from within Capsule 1, attempts shall be made to retrieve the Capsule 1 instrumentation and gas lines from within the Capsule 2 through-tubes.

Requirements for disassembling and inspecting the separated capsules are given in (Marshall 2019a). Requirements for the equipment used to accomplish the test-train disassembly and inspection are given in (Marshall 2020c).

## 2.4.2 Capsule Disassembly

### 2.4.2.1 All Capsules

All steps of the disassembly process shall be documented by digital photography and process engineer notes. After separating the capsules, but prior to cutting the head of each capsule, the TC leads and gas line leads above the head will be cut and retained. This is especially important for Capsule 1. Then the capsule head will be cut from each capsule so that the contents of the capsule can be accessed. The interior of the capsule, including the top of the compacts and graphite holder, shall be photographed and visually inspected after the head has been removed, and before attempting to remove the holder or any compacts.

Requirements for inspection, recovery, sorting, and storage of all capsule components, graphite holders, and fuel compacts are given in (Marshall 2019a) and are briefly discussed in the remainder of this section. In each capsule, the fuel compacts, graphite holders, through-tubes, TCs (including sheaths/sleeves), and gas lines shall be recovered, inspected, and retained for gamma spectrometric and radiochemical analyses discussed in Sections 2.5 through 2.7. Table 7 lists the other specific components found in each capsule: springs, disks, spacers, insulators, heaters, and SPND spacer tubes (which were located inside the through-tubes). The identity of each component shall be retained. Non-metallic components from each capsule shall be individually packaged. The same type of metallic components from the same capsule shall be packaged together. For example, all through-tubes from the same capsule shall be packaged together. It may be difficult to separate gas lines from TCs; thus, TCs and gas lines may be packaged together.

Through-tubes, gas lines, and TCs were brazed into the capsule heads. The through-tubes were press-fitted into the capsule floor (bottom of the capsule). Once the head has been cut to separate it from the capsule shell, pulling on the head may be sufficient to pull the through-tubes, gas lines, and TCs out of the graphite holder in the capsule. It may be necessary to drill the through-tubes out from the floor so they can be pulled out with the head. The SPNDs and the titanium SPND spacer tubes (all of which were housed inside the through-tubes) may be discarded. Care shall be taken to avoid damage to the graphite holder. If items such as gas lines and TCs do not readily come out of the holder, they may be cut and left in the holder.

Using great care, the graphite holder shall be removed from the capsule shell in a manner that retains its orientation. The irradiated graphite fuel holder and compacts may be fragile, and therefore easily broken during handling operations. The disassembly tools have been designed to minimize the potential

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of damaging these fragile components. To the extent practicable, the components will be handled in a horizontally-supported position and sliding motions (rather than grasping and lifting) will be used. With the holder removed, the interiors of the capsule shell and heads will be photographed to the extent possible (see [Marshall 2019a] for requirements) and then retained for leach analysis. The compacts will be removed from the graphite holder. The top/bottom orientation and identity (i.e., capsule number, stack within the graphite holder, and level within the stack) of all compacts shall be retained as the compacts are removed from the graphite holder, inspected, and stored in metal containers. With the compacts removed, the graphite holder top, bottom, inner, and outer surfaces will be inspected and photographed.

Each fuel compact will be placed in a labeled, pre-weighed container. To the extent practicable, any loose fragments or chips associated with the compact will also be loaded into the container. The loaded container then will be weighed to the nearest milligram to determine the weight of the contents. Each graphite holder and any associated fragments will be placed in a labeled, pre-weighed container after separation of the upper head assembly and after unloading all compacts. The loaded holder container will then be weighed to the nearest gram or better.

Table 7. Components of each capsule other than the compacts, graphite holders, TCs, gas lines, and through-tubes. All components listed here *except* for the SPND spacer tubes must be retained.

Drawing and Part No.	Description
DWG-604661	<b>Capsule 1</b>
29	Wave spring (ring), 2" OD Inconel®
13	Top Grafoil disk, 2.5" OD
12	Top graphite spacer, 2" OD
11	Bottom Grafoil disk, 2.5" OD
10	Bottom zirconia insulator, 2" OD
9	Bottom zirconia spacer (ring), 2.5" OD
DWG-604662	<b>Capsule 2</b>
15	SPND spacer tube, 7/16" semicircular, titanium
13	Top Grafoil disk, 2.5" OD
12	Top zirconia spacer, 2" OD
11	Bottom Grafoil disk, 2.5" OD
10	Bottom zirconia spacer, 2" OD
DWG-604663	<b>Capsule 3</b>
13	Top Grafoil disk, 2.5" OD Top
12	zirconia spacer, 2" OD Bottom
11	Grafoil disk, 2.5" OD Bottom
10	zirconia spacer, 2" OD
9	Top heater (disk), tungsten, 1.2" OD
8	Bottom heater (disk), tungsten, 1" OD

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Drawing and Part No.	Description
DWG-604664	<b>Capsule 4</b>
15	SPND spacer tube, 7/16" semicircular, titanium
13	Top Grafoil disk, 2.5" OD
12	Top zirconia spacer, 2" OD
11	Bottom Grafoil disk, 2.5" OD
10	Bottom zirconia spacer, 2" OD
DWG-604665	<b>Capsule 5</b>
13	Top Grafoil disk, 2.5" OD
12	Top zirconia spacer, 2" OD
11	Bottom Grafoil disk, 2.5" OD
10	Bottom zirconia spacer, 2" OD
OD: outer diameter	

#### 2.4.2.2 *Special Considerations for Capsule 1*

In-pile fission gas measurements indicate significant TRISO coating failure in Capsule 1 (see discussion in Section 1.4.4). There are a variety of postulated causes for this (see Section 1.4.4). The condition of the fuel within the capsule prior is currently unknown. The planned PGS and neutron radiography of the intact capsule prior to disassembly may provide information on whether the fuel could have shifted. It is possible that the fuel compacts will be more difficult to retrieve from the Capsule 1 holder than in the other capsules. If the compact holder and the compacts came into hard contact as a result of irradiation-induced dimensional change, it may be impossible to push the compacts out of the holder. Cutting the holder to remove an entire stack of compacts at once may be an option. Cutting or scoring portions of the holder may enable breaking up the holder to remove the compacts. Portions of the compacts/holder could be cut and mounted in epoxy for ceramography without first removing the compacts from the holder. The approach and methods used for Capsule 1 should be conducted with great care in order not to damage the fuel compacts or lose portions of the graphite holders.

Any and all visual observations and/or detailed analyses of Capsule 1 and its components could prove useful. Some proposed mechanisms of fuel degradation in Capsule 1 involve the Neolube applied to the inside of the capsule shell, failure of the thermocouple sheaths, oxidation of the fuel and capsule internals (including the zirconium oxygen getter on the gas inlet line), dimensional change of the fuel compacts or holder, damage to the capsule shell, damage to the fuel caused by contamination from the material used in the welds and/or brazed joints, higher-than-expected irradiation temperatures, fuel damage during test train disassembly, etc. The gas flow obstructions in Capsule 1 are of interest; therefore, attempts to recover and inspect the Capsule 1 gas lines shall be made. This includes the Capsule 1 gas lines in the plenum between Capsules 1 and 2.

#### 2.4.3 **Dimensional Metrology of Compacts and Holders**

The dimensions of the compacts and graphite holders will be measured, and those measurements will be used to update the thermal analyses of the AGR-5/6/7 experiment. These measurements will also be compared to assumptions made in the thermal analysis about the irradiation-induced dimensional change of the compacts and holders. The requirements for the measurements are given in (Marshall 2019a). The compact lengths shall be measured at two locations. The second length measurement shall be made by

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rotating the compact 90 degrees about its long axis from the first length measurement. Compact diameters shall be measured at three different axial locations. At each axial location, the diameters will be measured at two different azimuthal orientations, separated by 90 degrees. The outer diameter and inner diameters of the graphite holders, including the diameters of the fuel compact stack holes (excluding the through-tube holes) shall be measured. Graphite holders were measured in pre-irradiation characterization, and PIE measurements shall be performed at the same locations as the pre-irradiation measurements (see Palmer 2018 and its associated attachments).

## 2.5 Gamma Scanning of Graphite Holders

All AGR-5/6/7 graphite holders (including the inner and outer holders employed in Capsule 3) shall be gamma scanned using the HFEF PGS system. Initial axial scans will be performed to determine the total inventory of gamma-emitting fission products in the holders. A scan over a wide energy spectrum to identify all gamma emitters is desirable, and isotopes of particular interest are Ag-110m, Cs-134, Cs-137, Eu-154, and Eu-155. Ru/Rh-106 and Ce/Pr-144 are also of interest; however, in prior experiments, they were not found in significant quantities in the graphite holders. PGS scans must cover the entire holder in order to measure a complete inventory of gamma emitters. If the initial scans determine an axial location(s) of elevated fission product activity, PGS tomographic scans at that location may be performed. Elevated cesium activity, for example, in a graphite holder in the vicinity of a particular fuel compact may indicate a SiC or TRISO layer failure in a particle(s) within that compact. That compact can then be targeted for additional PIE. The counting times may be adjusted by the PGS engineer to be sufficiently long to measure isotopes of interest or establish suitably-low minimum detectable activities; however, the counting times should not be excessively long that schedules are adversely impacted.

Significant in-pile release of Ag-110m through intact coatings has been known to occur. Releases are particularly noticeable at temperatures equal to or greater than approximately 1100°C, and historically, much of the Ag-110m released from the fuel particles is held up in the graphite holders. Small but detectable in-pile releases of Eu-154 and Eu-155 (and also beta-emitting Sr-90) through intact coatings also begin to occur after long times at high-irradiation temperatures (>1200°C). These isotopes of Eu may also be held up in the graphite holders and could be detected by PGS.

## 2.6 Precision Gamma Scanning of Fuel Compacts

All compacts shall be gamma scanned using PGS to quantify the gamma-emitting fission product content. Isotopes of particular interest include Ag-110m, Cs-134, Cs-137, Eu-154, Eu-155, Ru/Rh-106, and Ce/Pr-144. The fission product inventories will be compared with predicted inventories from the as-run physics simulations of the AGR-5/6/7 irradiation to determine if any significant release occurred during the irradiation. Noticeable in-pile release through intact coatings is particularly relevant for Ag-110m at irradiation temperatures equal to or greater than approximately 1100°C. It is common to find Ag-110m releases significantly greater than 10%. Experience with the AGR-1 compacts (Harp 2014) demonstrated that, while quantification of most fission products can be accomplished with a relatively short live time (10 minutes per increment), quantification of Ag-110m inventory in the compacts requires much longer (several hours per increment) due to the lower inventory and gamma-ray yield for energies other than 657.5 keV. Therefore, counting times will need to be sufficient to quantify Ag-110m in the compacts, unless the scan time necessary to quantify very small amounts of Ag-110m becomes prohibitive.

After hundreds of days at high-average irradiation temperatures, the compacts in Capsule 3 may have released enough Eu-154 and Eu-155 that gamma scanning the compacts in the PGS, and comparing the results to the predicted Eu-154 and Eu-155 production could provide evidence of this release.

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The inventory of Cs-134 and Cs-137 will be used to determine the burnup of the compacts, and these values will be compared to as-run physics simulations. The procedure for determining burnup from the spectrometric data was described for the AGR-1 fuel compacts in ECAR-1682 (Harp 2014).

## 2.7 Capsule Fission Product Analysis

All major capsule components will be analyzed to determine the total inventory of fission products released from the fuel compacts, a key indicator of in-pile fuel performance. This will include an analysis of gamma-emitting fission products Ag-110m, Cs-134, Cs-137, Ce-144, Eu-154, and Eu-155, beta-emitting Sr-90, and other non-gamma-emitting fission products of interest (such as isotopes of Pd). The experimental methods will be similar to those used for the AGR-1 capsule components (Demkowicz et al. 2013). The results will be compared to predicted fission product inventories to determine the fractional inventories released from the fuel compacts. The data will be compared among capsules to assess the relative level of performance of the AGR-5/6/7 fuel at different burnups and irradiation temperatures. Requirements for the retention, packaging, and labeling of the capsule components are given in (Marshall 2019a) and were discussed briefly in Section 2.4.2.

### 2.7.1 Graphite Holders

Once as many gas lines and TCs as possible have been removed from the graphite holders, the fuel compacts removed, and the holders inspected (Section 2.4.2), the graphite holders will be subjected to precision gamma scanning (Section 2.5). After completion of this work, the graphite holders will be transferred to the Analytical Laboratory (AL) at MFC for additional gamma counting using the spectrometers at AL, which have lower minimum detectable activities than PGS. The holders are too large for pneumatic transfer from HFEF to AL; thus, the holders shall be transferred intact from HFEF to AL using the Dry Active Waste (DAW) cask or a similar manual means. In AGR-3/4, the Cell 4 spectrometer was used to count the large Sink Rings (Stempien et al. 2018a), and a similar process could be used for AGR-5/6/7 graphite holders. After gamma counting the intact holders at AL, the holders will be individually oxidized in air, leached with acid, and the leachate analyzed for Sr-90, uranium, plutonium, and possibly isotopes of Pd. The oxidation and leaching steps will be carried at Cell 5.

### 2.7.2 Metal Capsule Hardware

Metal capsule hardware that will be analyzed for fission products includes the stainless-steel capsule shells, heads, floors, TCs, gas lines, and through-tubes. Also included in this category are the tungsten heaters from Capsule 3 and the Inconel wave spring from Capsule 1. (The SPNDs and SPND spacer tubes are to be discarded as described in Section 2.4.2 and will not be analyzed.) Given the significant activation of stainless-steel, the capsule shells, heads, floors, and through-tubes shall be moved from the HFEF main cell (argon atmosphere) to the air “decon” cell for leaching. Smaller items, such as the TCs and gas lines, may be leached either in HFEF or at AL. The Capsule 3 Inconel wave spring may be leached either at HFEF or at AL. The tungsten heaters will be leached at HFEF or at AL if they can be rabbited from HFEF to AL. Stainless-steel items from the same capsule may be leached together in the same beaker. If multiple items are combined for simultaneous leaching, detailed notes of which items were combined must be kept. Gas lines and TCs from the same capsule may be leached together. Other dissimilar items should be leached separately. Requirements for performing the metal hardware leaching are given in (Marshall 2019a), and requirements for the equipment used to accomplish the leaching are given in (Marshall 2020c).

### 2.7.3 Ceramic and Graphite Disks, Spacers, and Insulators

Each capsule had a variety of Grafoil<sup>®</sup> and/or graphite and/or zirconia disks, spacers, and insulators at the tops and bottoms of the graphite holder and fuel compact stacks. These were listed in Table 7. Each of these items shall be individually packaged and individually gamma counted either at the HFEF Out-of-

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cell Gamma (HOG) station or after transferring to AL. After gamma counting, the zirconia items from the same capsule will then be combined and leached in acid, and the leachate will be analyzed for gamma emitters, Sr-90, uranium, and plutonium. After gamma counting, the graphite and Grafoil® items from the same capsule will be combined and oxidized in air at AL. The residual ash remaining from this oxidation will be leached and the leachate analyzed for gamma emitters, Sr-90, uranium, and plutonium. In the leachate solutions generated from these processes, Pd may be analyzed using mass-spectrometry.

## 2.8 Shipping Fuel Compacts to ORNL

Select compacts will be shipped from INL to ORNL for PIE. These compacts may be selected based on the calculated as-run irradiation temperatures, burnups, and fast fluences. The results of graphite holder PGS-scanning and gamma tomography may also suggest that certain compacts are likely to have SiC layer and/or TRISO-coating failures. Those compacts could be sent to ORNL so that deconsolidation and Irradiated Microsphere Gamma Analyzer (IMGA) analysis can be employed to locate and/or quantify the particle(s) with the failures. PGS analysis of the compacts themselves may also reveal interesting behavior that could make a compact a candidate for shipment to ORNL. The potential for reirradiation-heating tests to screen compacts with TRISO failures is being explored (see Section 2.15.6), and that process may also be used to select compacts for shipment to ORNL.

## 2.9 Fuel Compact Ceramography and Microscopy

Cross sectioning followed by ceramographic preparation and microscopy of fuel compacts enables the observation of fuel particles within the compact while retaining information about the location of those particles within the compact. This process enables observation of potential spatial gradients in fuel particle morphology. It also enables examination of the morphology of the fuel compact graphitic matrix that surrounds the particles. Primary features for investigation include: cracks in the compact matrix, fuel kernel swelling and porosity, kernel migration, buffer layer degradation and densification, corrosion of the SiC layer by fission products, and fractures in the TRISO coating layers and delamination between them. Migration of fission products will also be examined where practical.

The basic approach used for sample preparation and ceramographic examination will be similar to that used for the irradiated AGR-1 and AGR-2 compacts (Ploger et al. 2012, Rice et al. 2016). AGR-3/4 compact ceramography was further simplified in that the compacts were sectioned only a single time by cutting along their axial centerlines (Stempien and Schulthess 2020). This type of simplified approach may also be employed. Following the AGR-1 and AGR-2 approach, selected compacts will be sectioned axially and radially, then mounted and polished. If extensive damage is observed at the saw-cut surface of the compact (for example, extensive removal of embedded particles from the cut surface), then the samples should be ground past the damaged layer so that as many intact particle cross sections as possible can be exposed on the final polished surface. Samples may be cut and mounted as slices to diminish radiation dose rates for certain analyses. However, even relatively thin cross-sectional samples of fuel compacts will be highly radioactive (~1,000 R/hr at contact for one-tenth of a compact); therefore, analytical instruments must be heavily shielded to accommodate them.

Compacts from Capsule 1 are prime candidates for compact ceramography owing to the likelihood of TRISO failure in these compacts, based on the in-pile fission gas measurements. One hypothesis is that Capsule 1 compacts could have come into hard contact with the graphite holder (via irradiation-induced dimensional changes in the compact matrix or the graphite holder), causing mechanical damage to the particles. Innovative ceramographic preparation may be required for Capsule 1 compacts if the compacts cannot be pushed out of the graphite holders. If necessary, portions of the Capsule 1 holder/compact could be prepared for ceramography with the compacts still inside the holder. Capsule 3 compacts would also be of interest, owing to their high-irradiation temperatures. The lowest irradiation temperatures are predicted to be in Capsule 5, and ceramography of Capsule 5 compacts would allow comparisons between

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high- and low-irradiation temperatures. Besides Capsule 1, Capsule 5 was the only other capsule with 40% packing fraction fuel. Ceramography of Capsule 5 fuel compacts would also enable observation of the graphitic matrix in these high-packing fraction compacts.

Some fuel compacts may be sectioned for ceramography after safety/heating testing. The defect types to be investigated are the same as those before safety testing, although their frequency and severity may increase appreciably at safety testing temperatures. Any compacts to be used for this purpose will be selected following safety testing.

### **2.10 Compact Deconsolidation-Leach-Burn-Leach**

Certain as-irradiated compacts (and at least some safety/heating-tested compacts, discussed in Section 2.15) will be selected for deconsolidation-leach-burn-leach (DLBL) analysis. The objectives of DLBL are to:

- Disintegrate the compact matrix and liberate particles
- Determine the inventory of fission products in the compact outside of the SiC layer (i.e., in the OPyC layer and matrix)
- Determine the number of failed particles with exposed fuel kernels, in which all three TRISO coatings have failed (determined by analysis of the pre-burn leach solutions)
- Determine the number of particles with a failed or defective SiC layer, but intact inner- or outer-pyrolytic carbon layers (determined by analysis of the post-burn leach solutions).

The four basic steps of this process are outlined below.

1. The deconsolidation process involves the electrolytic oxidation at ambient temperature of the carbonaceous binder in the compact matrix. In the process, the compact—the anode in the electrochemical circuit—is suspended in nitric acid solution (the electrolyte) while a direct current is applied between the compact and the cathode, which is suspended in the electrolyte solution. The total power applied to the compact is maintained below 10 watts throughout the process in order to avoid damage to particles. This results in oxidation of the matrix (without significant oxidation of the OPyC) and disintegration of the compact, liberating free particles. The deconsolidation solution may be analyzed separately for inventory of actinides and fission products, or it may be used in the first pre-burn leach and analyzed following that step.
2. The pre-burn leach is used to dissolve most of the actinides and fission products in exposed kernels (i.e., particles with all three coating layers breached) or outside intact particles due to either uranium contamination or release through one or more intact coatings. The leach is performed in hot nitric acid for a period of approximately 24 hours using a Soxhlet extraction apparatus. The process for AGR-5/6/7 will be similar to that used for AGR-1 and AGR-2 at INL (Demkowicz et al. 2012a, Stempien 2020) and ORNL (Hunn et al. 2013, Hunn et al. 2018). This pre-burn leach is performed twice—with additional leaches if necessary—to ensure that all analytes have been effectively leached from the deconsolidated material. Each of the solutions are analyzed for actinides and fission products.
3. The burn step, performed at 750°C in air, oxidizes the carbon residue from the matrix and all exposed pyrolytic carbon coatings, including the inner pyrolytic carbon and buffer layer of particles with a defective SiC coating, but with otherwise intact carbon coatings. This step exposes the fuel kernel in those particles with a failed SiC (but with intact pyrolytic layers) to the subsequent post-burn leach.

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4. The post-burn leach dissolves the fission products and uranium in the exposed fuel kernels exposed by the burn step and allows for a calculation of the number of equivalent particles with defective or failed SiC coatings. It also dissolves any actinides or fission products from the oxidized carbon material that were not dissolved prior to the burn step. The post-burn leach is performed in the same manner as the pre-burn leach and is also repeated a second time. Each of the solutions are analyzed for actinides and fission products.

Minor variations on the procedure outlined above may be performed based on the specific needs for subsequent particle analysis. For example, if particles with an intact OPyC layer are required for irradiated microsphere gamma analysis, the process may be interrupted after the pre-burn leaches. Some or all of the particles may then be sieved to remove the matrix debris, and an additional boiling step may be performed on the particles to remove any additional matrix material in order to facilitate particle handling during gamma analysis (see description in Hunn et al. 2013). Another use for counting all particles via IMGA after pre-burn leaching has been completed is that IMGA can be used to find and recover particles with failed SiC layers that can be further examined. Without IMGA counting after the pre-burn leaches, particles with failed SiC layers are likely to be lost because the pyrocarbon layers will be oxidized during the burn step, and the kernels will be leached during the post-burn leaches. There was one notable exception in AGR-2 where a portion of the failed SiC layer from a particle with a SiC layer failure was indeed recovered for additional analysis (Hunn et al. 2018).

If there are no particles with failed TRISO coatings, nor failed SiC layers in the compact (and therefore no kernels exposed to acid dissolution during the DLBL process), the cumulative inventory of actinides and fission products found in the DLBL solutions can be attributed to the original uranium contamination in the matrix, plus the contribution from any fission products released from the intact particles. Since the average level of uranium contamination in the compacts is known from as-fabricated fuel analysis, the DLBL data can be used as a measure of fission products released from intact particles that were retained in the compact.

In-pile fission gas measurements have historically been used to estimate the number of TRISO failures in-pile. The target in-pile TRISO failure rate for nominal operating conditions is 2E-4, or lower, at 95% confidence. PIE is used to collect additional data to support the in-pile data. Post-irradiation gamma scanning is used to find any locations within the graphite compact holders with elevated activities of radiocesium (cesium itself is an indicator of SiC failure, but not necessarily TRISO failure) so that the compacts in proximity to those regions of elevated cesium activity can be selected for DLBL to find the particle(s) with defective SiC layers and/or measure the number of particles with TRISO failures. AGR-5/6/7 Capsule 1 did not have fission gas measurements for the final portion of the experiment following the onset of particle failures; however, the fission gas measurements that were obtained during Cycle 166A indicated significant numbers of TRISO failures. As a result, it may be useful to perform as-irradiated DLBL of a certain number of Capsule 1 compacts to estimate TRISO and SiC failure fractions in Capsule 1. The axial temperature variation within Capsule 1 is larger than the azimuthal variation; therefore, to determine if temperature variations played a role in Capsule 1 failures, one possible course of action would be to examine compacts within the same stack. Exams would start at the top of the stack, where the temperatures were highest, and move downward, or vice versa.

## 2.11 Destructive Burnup Measurements

In addition to the burnup determined from non-destructive compact gamma scanning (see Section 2.6), selected compacts will be analyzed for burnup using destructive methods. This will enable comparison to the burnups calculated from physics simulations. In AGR-1, a method called the “Fission Product Monitor – Residual Heavy Atom” technique was used (Harp et al. 2014). This method is based on end-of-life inventories of certain actinides and fission products in the fuel and the respective fission yields. This technique uses the following isotopes: La-139, Ce-140, Ce-142, Pr-141, Nd-145, and Nd-146.

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In AGR-2, destructive burnup measurements were made at ORNL using a method based on the ASTM E321-96(2012) standard where isotopes of U, Pu, and Nd were measured (Montgomery et al. 2018, Hunn et al. 2019b). In each of the methods, particles from deconsolidated compacts were pulverized, oxidized in air, and leached in acid. The leachate was then analyzed using methods based on inductively-coupled-plasma mass-spectrometry (ICP-MS).

It is expected that three or four compacts will be used for burnup measurement. Compacts will be selected to cover the range of burnups predicted in AGR-5/6/7. It would be advantageous to perform burnup measurements on compacts that are also being used for other purposes such as DLBL, particle microscopy, etc.

## 2.12 Particle Inspection and Gamma Counting

TRISO particles will be recovered from the deconsolidation process discussed in Section 2.10. Individual particles from deconsolidation will be visually inspected, imaged, and gamma counted. Gamma counting will establish the inventories of gamma-emitting fission products in the fuel particles. Gamma-counted particles will be compared with one another and to the calculated inventories of gamma-emitting fission products from as-run physics simulations. These counts and subsequent comparisons can be used to screen particles for further analysis, such as X-ray tomography, microscopy, or longer gamma-counting times.

One of the primary objectives is to screen particles from selected compacts to locate particles with abnormal fission product release indicative of defective or failed coatings. Particles with failed SiC layers are particularly well-suited to recovery from deconsolidation, screening by gamma counting, and additional exams. This is typically accomplished by examining the Cs-137 inventory in the particles because Cs is well retained by intact SiC, but it is significantly released through a defective or failed SiC layer, even if one or both PyC layers remain intact. Since cerium is relatively immobile in the kernels, the ratio of Cs-137 to Ce-144 activity is a useful metric to screen for particles with abnormally low Cs inventory because it adequately adjusts for variations in initial fissile content in the kernel (due to variation in stoichiometry, density, or total kernel volume) and burnup among the particles. A particle with an abnormally low Cs-137/Ce-144 ratio most likely has released a significant inventory of Cs. A particle that has a typical ratio of radiocesium to Ce-144, but a low absolute amount of each, is indicative of a particle with less fissile material at the time of fabrication. One potential cause of this is a particle with an undersized kernel.

As there are a large number of particles in a single compact (approximately 3,400 in the 40% packing fraction compacts and 2,200 in the 25% packing fraction compacts) the counting time for a single particle must be relatively short to enable all particles to be analyzed in a reasonable timeframe (generally several weeks). Previous experience with irradiated AGR-1 particles indicates that the Cs-137 and Ce-144 activities can usually be measured with 50–100 second count times. For compacts where it is desirable to examine every particle, this task will be performed using the IMGGA at ORNL, which was used effectively during the AGR-1 and AGR-2 PIE campaigns. As the elapsed time following irradiation increases, decay of Ce-144 may render quantification with gamma spectrometry infeasible. In such cases, Ru-106 can be used instead since it has a longer half-life and is also primarily retained in the kernel, similar to Ce.

A second objective is to analyze the fission product retention of other key gamma-emitting fission products, including Ag-110m and Eu-154. Since these fission products have relatively low fission yield (and therefore have lower inventories compared to Cs-137 and Ce-144) and also often have lower gamma-ray yields, longer counting times are required, and only a subset of particles (on the order of tens to one hundred particles) will be analyzed using long counts. As time progresses, Ag-110m activities will steadily decrease from radioactive decay, and longer count times may be required to adequately measure

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Ag-110m content. The measured inventory of each specific fission product analyzed will be compared to the predicted inventory and normalized based on the relative inventory of Cs-137. Cs-137 is used here because it has a high fission yield, long half-life, its inventory is fairly linear as a function of burnup, and it is very well retained as long as the SiC layer remains intact. This activity will be performed on a subset of particles from selected compacts using the IMGA at ORNL and multiple spectrometers at INL. INL has two HOG gamma stations at HFEF and two gamma stations at AL. The Cell 4 spectrometer at AL can accept one sample at a time. The recently installed AL Cell 5 spectrometer has a six-position sample carousel that can automatically count six different samples sequentially.

Particle gamma counting will be performed on fuel after irradiation to examine in-pile fission metals retention. Individual particle gamma-counting will also be performed after heating tests to examine the temperature dependence of fission metals retention. The analysis will be performed on particles following deconsolidation-leach or full DLBL analysis. Any particles of interest identified during gamma counting can be selected for detailed microanalysis as described in the following sections of the plan. This may include particles with low Cs-137, which will be examined in an attempt to identify the cause of the defective or failed SiC layer. It may also include particles sorted based on their relative level of Ag-110m retention.

### 2.13 X-ray Imaging of Particles and/or Compacts

ORNL has utilized X-ray and X-ray Computed Tomography (CT) of individual TRISO particles to look for cracks in the SiC layer of particles that were suspected of SiC failures based on the results of counting on IMGA (see Section 2.12). INL has recently acquired a similar capability. Particles deconsolidated from compacts (see Section 2.10) could be subject to X-ray imaging before or after IMGA for a variety of purposes. One purpose might be to observe the integrity of the TRISO coatings if a particle is suspected of having SiC failure. Another potential use of X-ray CT is to determine the volumes of the kernel and/or TRISO coating layers, such as the buffer, so that irradiation-induced swelling of the kernel and shrinkage of the buffer could be quantified. These are common parameters in fuel performance models.

Work at INL has been conducted on X-ray CT of six unirradiated AGR-5/6/7 fuel compacts (Kane and Marshall 2018). This work was effective in determining kernel sizes, defects, and kernel-to-kernel distances. Development work at INL is currently underway to evaluate the efficacy of X-ray CT on irradiated compacts. One of the goals of the development work is to determine if any of the TRISO coatings can be effectively imaged on particles that are still in a compact. If possible, such a technique could be employed on Capsule 1 compacts (or others) to look for signs of TRISO failure. If it proves difficult to image TRISO coatings in irradiated compacts, compact X-ray CT would still be useful for measuring kernel swelling in the particles in a compact.

### 2.14 Fuel Particle Microanalysis

Based on the experimental results of several of the preceding PIE tasks, individual particles or groups of particles will be selected for microanalysis using an array of characterization methods. The general objective of these fuel particle analyses is to characterize the morphology of the kernel and TRISO coatings. Elements of morphology such as fuel kernel porosity, kernel migration, buffer layer irradiation-induced dimensional changes, buffer fracture, fractures in the TRISO coating layers, delaminations between coating layers, existence of fabrication defects affecting irradiation performance, reaction of fission products (such as palladium) with the silicon carbide layer, and deposition or residual clustering of fission products outside the kernel. Because these analyses will be performed after particle gamma counting in certain cases, the gamma-counting results can be factored into particle selection. In such cases, one important aspect will be relating gamma-counting results on release of metallic fission

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products to deterioration of the SiC layer or the presence of other defects. Microstructural features observed during PIE should also be compared to pre-irradiation microstructures.

This activity will primarily involve mounting one or more particles in epoxy, grinding to near the particle midplane (or alternatively, grinding to the particular plane of interest), and polishing the surface to a sufficient quality to observe the features of interest. Prior to this sample preparation, selected particles may be analyzed nondestructively using X-radiography and three-dimensional tomographic reconstruction. This can allow specific features within the particles (including coating fractures, delaminations, and SiC defects) to be observed in-situ. This information can then be used to focus subsequent mounting and polishing so that specific features of interest can be revealed for detailed microanalysis. Particle X-ray analysis will be performed using the system developed at ORNL and used successfully on irradiated AGR particles (Hunn et al. 2013). Recently, INL has installed a new system for X-ray tomography of irradiated fuels and samples at the Irradiated Materials Characterization Laboratory (IMCL). This system could also be utilized for analysis of individual fuel particles. A methodology must be developed for effectively holding the particles in the INL X-ray system and for mounting and polishing the particles following X-radiography. To the extent possible, the methods employed for X-radiography of irradiated particles at ORNL should be employed at INL as well.

A basic analysis of polished-particle cross sections will be accomplished with optical microscopy. This will consist of a general inspection of the particle morphology, including kernel swelling and buffer densification, along with coating layer fractures and delaminations. High-resolution digital images of the specimens will be acquired. The frequency of occurrence of certain features (i.e., buffer fracture, IPyC-SiC delamination, layer cracking/tearing) will be noted.

A scanning electron microscope (SEM) will be used to perform more detailed, higher resolution examinations of the particle cross sections. This technique will be used to inspect the entire particle cross section for features of interest, including coating damage or fracture, evidence of fission product reaction with silicon carbide, and coating delaminations. Using appropriate spectroscopic techniques, elemental analysis will be used to identify clusters of actinides or fission products in the kernel, coating layers, and at interfaces of these layers. The elemental data can provide important information about the migration of fission products through the coating layers and reaction of fission products with silicon carbide. The SEM and energy-dispersive X-ray spectroscopy (EDS)/wavelength-dispersive X-ray spectroscopy (WDS) analyses may be used to further focus any subsequent examination to particular areas of interest on the particle cross section. Both INL and ORNL have SEMs with EDS and WDS capabilities for analyzing irradiated fuel. An electron probe microanalyzer (EPMA) at INL employs an array of WDS spectrometers for quantifying the elemental composition of small volumes. When used with standards, the absolute amounts (rather than just the relative amounts) of isotopes can be quantified.

A transmission electron microscope (TEM) or scanning-transmission electron microscope (STEM) may be used to examine small areas of the particle cross section at very high magnification to understand microstructural behavior down to the nanometer scale. Specimens from a particular location on a polished-particle cross section can be easily prepared using a focused ion beam (FIB) to produce an electron-transparent lamella for examination with the TEM. The TEM analysis will be coupled with elemental analysis, such as EDS or electron energy loss spectroscopy (EELS), to identify the elemental constituents within the observed microstructure. This will allow the location of various fission products or actinides within the microstructure to be observed, providing further information on elemental transport through the coatings. FIB sample preparation and TEM/EDS/EELS analysis can be performed using instruments at the INL Electron Microscopy Laboratory (EML), IMCL, and the Center for Advanced Energy Studies.

Electron backscatter diffraction (EBSD) could be used to characterize the crystallographic orientation of grains and the grain boundary character within the SiC layer in order to aid the interpretation of

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observed fission product release behavior in the particles. In particular, the diffusive release of fission products through intact SiC may be related to the nature of the grain boundaries. Additional advanced characterization methods (such as atom probe tomography and slice-and-view) could be employed to examine selected samples to provide further detail. It is expected that these types of analyses will contribute to the understanding of fission product transport throughout the kernel and TRISO coatings.

## 2.15 Heating Tests

Here the term “heating test” broadly refers to any type of test in which fuels are heated after irradiation. The term “safety test” is more specific, and has been used previously to refer to the post-irradiation heating of intact fuel compacts to high temperatures (1600–1800°C) under helium atmospheres to determine fission product retention and the statistical rates of SiC layer and TRISO coating failure under high-temperature, conduction-cooldown accidents. Other types of heating tests may also probe phenomena such as fission product release rates over a range of nominal reactor temperatures and accident temperatures. In that case, the focus is on observing temperature-dependent fission product transport, not on observing TRISO coating failure rates. “Oxidation testing” refers to post-irradiation heating in oxidizing atmospheres containing air or moisture.

### 2.15.1 Safety testing

#### 2.15.1.1 Safety Test Description

Select fuel compacts will be safety tested under conditions characteristic of conduction-cooldown accidents (i.e., under helium atmospheres and temperatures generally from 1600 to 1800°C). The tests will assess fuel particle SiC layer and TRISO coating resilience and fuel fission product retention. The facilities to be used for this activity are the Fuel Accident Condition Simulator (FACS) furnace system at INL (Demkowicz et al. 2012b) and the Core Conduction-Cooldown Test Facility (CCCTF) at ORNL (Baldwin et al. 2012). Each of these facilities enables irradiated fuel compacts to be heated while releases of fission gases and condensable fission products are measured.

Fission gases (particularly Kr-85) released from the compacts will be carried from the furnace in the helium sweep gas and collected in cryogenically cooled traps that will be continuously monitored with gamma spectrometers throughout the tests. Water-cooled condensation plates (FACS furnace) and deposition cups (CCCTF) will be used to collect condensable fission products. These plates and cups are exchanged at pre-determined intervals during the test (approximately 12 to 24 hours) to get time-dependent condensable fission product release information. The plates and cups will be gamma counted in a controlled geometry to quantify the activity of gamma-emitting fission products (including Ag-110m, Cs-134, Cs-137, Eu-154, and Eu-155). The plates and cups will then be leached with acid to transfer all deposited fission products into solution, which will then be analyzed with ICP-MS to quantify the inventory of non-gamma-emitting fission products and/or actinides. Aliquots of the leach solutions will be treated with an ion-exchange resin to selectively extract strontium, followed by subsequent analysis of the beta-emitting Sr-90 inventory (e.g., with gas flow proportional counting or liquid scintillation).

The measured inventory of fission products will be compared to the predicted inventory in the fuel compact based on as-run AGR-5/6/7 physics simulations to calculate the fraction of the predicted inventory released from the fuel during the safety test. This will also involve a collection efficiency factor, which is the fraction of a particular element released from the fuel that is deposited on the plate or cup (the remaining fraction being deposited on other internal furnace components). This collection efficiency will either be determined from previous furnace testing or by measuring the total amount of each fission product deposited on the cups and furnace internals during a specific safety test.

The majority of safety tests will consist of an isothermal hold at the target temperature. Typical maximum temperatures of 1600–1800°C will be used, but other temperatures could also be employed.

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The temperature profiles for 1600, 1700, and 1800°C isothermal tests are shown graphically in Figure 4. The temperature profile for the isothermal tests will involve the following steps; however, these steps may be altered, re-ordered, or omitted depending on the goals for a given test.

1. Ramp to ~400°C at a rate of ~120°C/h and hold for sufficient time to eliminate adsorbed water from the fuel (typically 2 hours).
2. Ramp to the representative fuel operating temperature (e.g., 1250°C) at a rate of 120°C/h and hold for ~12 hours to establish thermal equilibrium in the fuel compact.
3. Ramp up to the target test temperature at a rate of 50°C/h<sup>a</sup>.
4. Nominal hold time at the test temperature will be 300 hours, although tests can be shortened (for example, in the case of excessive particle failures observed in the early stages of the test based on fission gas release) or lengthened as necessary.

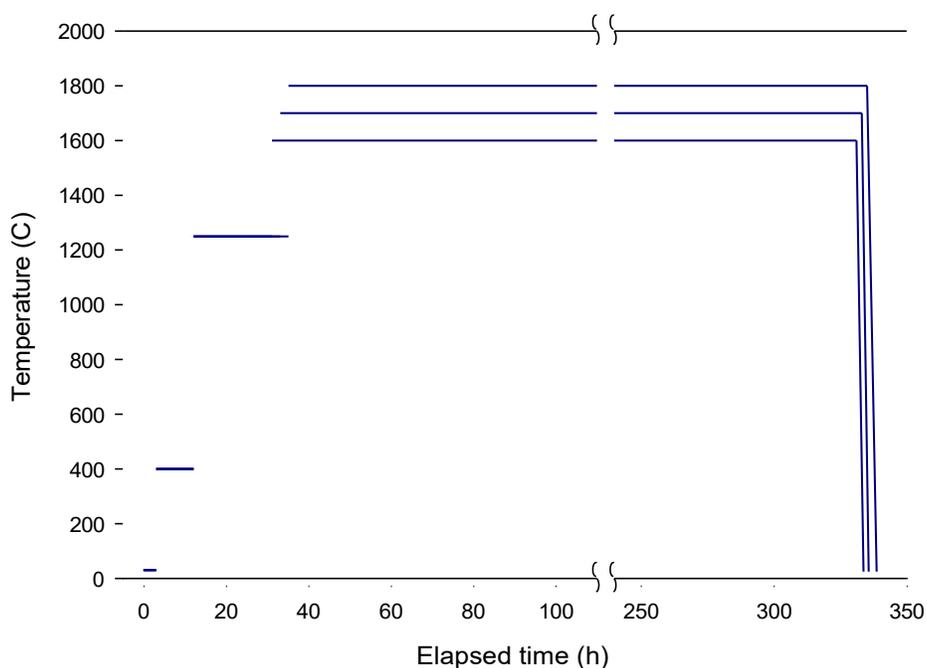


Figure 4. Temperature profiles for isothermal safety tests at maximum temperatures of 1600, 1700, and 1800°C.

### 2.15.1.2 Numbers of Particles and Compacts Required

In safety testing, the number of compacts that must be tested depends on the accident failure fraction that the AGR program wants to establish with adequate statistical basis. For reference, historic reactor design specifications have established a TRISO failure fraction of  $\leq 6E-4$  at 95% confidence during reactor accidents with peak fuel temperatures of  $\leq 1600^\circ\text{C}$ , and  $\leq 2E-4$  at 95% confidence during normal operation. Table 8 gives the number of particles needed to demonstrate a certain failure fraction at 95% confidence based on the number of failures observed in all of the heating tests. The table also shows how

<sup>a</sup> The heating rate of 50°C/h was determined based on a simple analysis of previous predictions for peak core temperatures of the 350 MWt MHTGR (Department of Energy 1986) and 600 MWt GT-MHR (General Atomics 1994) during depressurized core conduction cooldown. A rate of 50°C/h corresponds to roughly the maximum heating rate in either scenario once the temperature exceeds 1250°C.

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many compacts would be needed to achieve that many particles, depending on whether they are 25% or 40% packing fraction compacts.

Table 8 shows that with zero particle failures, testing of at least 5,000 particles (two to three compacts) is needed to confirm a failure fraction of  $\leq 6E-4$  at 95% confidence. To demonstrate a failure fraction three times lower, 15,000 particles are needed with zero observed failures. For a failure fraction 10 times lower, 50,000 particles are needed with zero observed failures. Increasing the number of particle failures observed in the tests incrementally increases the total number of particles that need to be tested to demonstrate a particular failure fraction at 95% confidence.

In order to demonstrate TRISO failure fractions  $\leq 6E-4$  with some margin. A failure fraction of  $\leq 2E-4$  can be demonstrated by testing 15,000 particles if zero failures are observed. If somewhere between one and five TRISO failures are observed, then the number of particles that must be tested ranges from 24,000 to 53,000; therefore, the plan is to test a minimum of 15,000 particles, up to about 53,000 particles. Depending on the compacts chosen for these tests (i.e., whether they are of the 25% or 40% packing fraction variety), between five and 23 compacts must be tested.

Additional testing can show that the failure fraction is considerably lower. For example, between AGR-1 and AGR-2, approximately 45,804 particles were tested at 1600°C across 12 different compacts with zero TRISO coating failures. This equates to a TRISO failure fraction of  $\leq 6.6E-5$  at 95% confidence from binomial statistics. In the same tests, three SiC layer failures occurred, resulting in an observed SiC layer failure fraction of  $6.5E-5$ , which equates to a SiC failure fraction of  $\leq 1.7E-4$  at 95% confidence. (The AGR program has not targeted as specific SiC layer failure rate to demonstrate.) If no TRISO failures are observed at 1600°C, between 14 and 21 AGR-5/6/7 fuel compacts (depending on how many 25% or 40% packing fraction compacts were tested) would have to be tested to demonstrate the same TRISO failure rate as the combined population of AGR-1 and AGR-2 tests ( $\leq 6.6E-5$  at 95% confidence). To demonstrate the same acceptably-low TRISO failure rates as AGR-2 at 1600°C ( $\leq 2.4E-4$  at 95% confidence), between four and six AGR-5/6/7 compacts would have to be tested.

Table 8. Number of fuel compacts and fuel particles that must be tested/examined to demonstrate three potential ranges of failure fraction and how the required number of particles/compacts changes with the number of observed failures.

Failure fraction at 95% confidence	# of failures observed	# of particles to be tested	# of Capsule 1 and/or 5 compacts, 40% particle packing fraction	# of Capsule 2, 3, and/or 4 compacts, 25% particle packing fraction
$\leq 6E-4$	0	5,000	2	3
	1	8,000	3	4
	5	17,500	6	8
$\leq 2E-4$	0	15,000	5	7
	1	24,000	8	11
	5	53,000	16	25
$\leq 6E-5$	0	50,000	15	23
	1	79,000	24	36
	5	175,000	52	80

### 2.15.1.3 Compact Selection

In addition to determining TRISO failure rates at 1600°C, it is also desirable to test compacts with a broad range of burnups and irradiation temperatures to determine if or how those factors affect performance. Testing fuel performance margins using tests at temperatures  $>1600^\circ\text{C}$  are also important.

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Specific compacts will be selected for safety testing based on irradiation history (including burnup, fast fluence, and temperature taken from the final as-run AGR-5/6/7 physics calculations) and on early PIE data including analysis of the irradiation capsule components (Sections 2.5 and 2.7) and gamma scanning of the compacts (Section 2.6). A preliminary estimate of the number of compacts from each AGR-5/6/7 capsule that will be safety tested is presented in Table 9.

To construct Table 9, it was assumed that the target TRISO failure fraction to demonstrate is  $\leq 2E-4$  at 95% confidence. This was done so that there would be some margin to the maximum-acceptable TRISO failure fraction of  $\leq 6E-4$  at 95% confidence. Another assumption was that between one and four TRISO failures would occur during 1600°C testing, necessitating that between roughly 24,000 and 40,000 particles be tested at that temperature. Based on AGR-1 and AGR-2 safety test results, this is a conservatively-high assumption on the number of TRISO failures. If no TRISO failures are observed in 1600°C testing, the number of tests could be reduced as long as the program needs to explore the effects of burnup and/or irradiation temperature can still be met.

There are other considerations in formulating Table 9. One consideration is whether or not Capsule 1 compacts can be used for safety testing. The number of tests without parentheses assumes that Capsule 1 compacts can be used for safety testing. Values in parentheses assume that Capsule 1 compacts *cannot* be used for safety testing. Early PIE may determine that no Capsule 1 compacts can be used for safety testing due to the significant level of TRISO failures in that capsule. In that case, compacts from other capsules will be substituted in place of Capsule 1 compacts. Given the significant in-pile fission gas releases from Capsule 1, it would be difficult to nondestructively distinguish compacts with fully intact fuel from compacts with some SiC and/or TRISO failures. Even a compact with fully intact particles could have significant matrix inventory from fission products that were released from other fuel compacts in the capsule that did experience particle failures. Significant PIE and/or screening analyses (see Section 2.15.5) could be attempted to establish if any Capsule 1 compacts are viable for safety testing.

If some or all compacts from Capsule 1 are not suitable for determining safety-test failure rates due to in-pile SiC and/or TRISO failures, then some or all of the 40% packing fraction compacts used for safety testing will have to come from the relatively cold, low-burnup Capsule 5 (TAVA < 800°C). Capsule 5 compacts are also of interest to compare to model predictions that indicate less pyrocarbon creep (and possibly more pyrocarbon cracking) at low irradiation temperatures. Capsule 1 was predicted to have many of its 90 compacts with irradiation temperatures in the ~1100–1250°C range typical of nominal HTGR conditions. Outside of this capsule, few other compacts will have had irradiation temperatures in this range. As of this writing, preliminary temperature analysis suggests that approximately 17% of the Capsule 3 particles (about four compacts worth) have average irradiation temperatures in the ~1100 to 1250°C range. Therefore, these four Capsule 3 compacts would be prime candidates for safety testing in place of Capsule 1 compacts. Substituting Capsule 3 compacts in place of Capsule 1 compacts would cover the ~1100 to 1250°C range; however, additional Capsule 5 compacts would need to be tested to make up for the fact that Capsule 1 compacts are the only other 40% packing fraction compacts.

Another consideration is whether there are any behaviors related to fuel particle performance in the 40% packing fraction compacts that are different than in the 25% packing fraction compacts. The TRISO particles used in the two varieties of compacts are from the same composite lot (see Section 1.4.1), but as-fabricated fuel characterization has determined that the two packing fractions are different in that the exposed kernel fraction was higher in the 40% packing fraction compacts after the compacting process (Marshall 2020b, Hunn et al. 2019c). As long as the observed behaviors of the fuel particles in the 25% and 40% packing fraction compacts are similar (i.e., that there are no inherently different behaviors of the particles in the 40% packing fraction compacts), it seems reasonable to determine the TRISO and SiC failure fractions by considering the particles in the 25% and 40% packing fraction compacts to be from the same population. In other words, safety tests can interchangeably utilize both packing fractions for the purpose of establishing TRISO particle failure statistics.

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Except for the fuel predicted to have irradiation temperatures between approximately 1100 and 1250°C, many of the rest of these Capsule 3 compacts are expected to have high burnups and high average and peak irradiation temperatures in excess of what are considered nominal HTGR fuel temperatures. Capsule 3 is considered AGR-7, the high-temperature margin test. These compacts are less appropriate for gauging nominal fuel performance and more appropriate for establishing performance margins. It is anticipated that TRISO failures will occur during some of the safety tests of the hot Capsule 3 fuel.

Capsule 2 fuel consists of 25% packing fraction, relatively low-irradiation temperatures, and high burnup. Thus, these compacts are useful for testing the safety performance of low-irradiation temperature, high-burnup fuel. Capsule 4 fuel is also 25% packing fraction with similar irradiation temperatures to Capsule 2 at slightly lower burnup. This makes safety testing Capsule 4 fuel less of a priority, and some of the tests specified in Table 9 to use Capsule 4 fuel could be substituted with additional tests of Capsule 2 fuel.

Besides proving failure statistics at 1600°C, it is also desirable to determine the temperature margins to fuel failure. Table 9 currently calls for two tests at 1700°C and six tests at 1800°C. These tests could serve as margin tests and could be compared to the AGR-1 and AGR-2 tests at  $\geq 1700^\circ\text{C}$ . Safety testing the hotter compacts from Capsule 3 is of interest for establishing performance margins. Based on results from AGR-1 and AGR-2 testing, no TRISO failures were observed below 1800°C. There is an option to replace some of the AGR-5/6/7 1600°C tests with 1700°C tests to demonstrate TRISO failure statistics at higher temperatures. A small number of short-duration tests at temperatures  $>1800^\circ\text{C}$  shall also be considered.

To reduce the number of tests conducted at 1600 and 1700°C, an attractive option is to perform tests of multiple compacts at once to increase throughput for gathering failure rates. Tests of up to three compacts at a time have been conducted previously in CCCTF and FACS.

Table 9. Preliminary estimates of compacts for safety testing. Assumes 25% and 40% packing fraction compacts can be used interchangeably. Numbers of compacts tested could be adjusted as data are acquired. Numbers in parentheses are the number of tests if Capsule 1 compacts cannot be used for safety testing. Approximate irradiation conditions for each AGR-5/6/7 capsule based on plots from pre-test predictions in (Collin 2018b).

Capsule	Packing Fraction (%)	TA Peak (°C)	TAVA (°C)	TA Min (°C)	Burnup (% FIMA)	Fast Fluence ( $10^{25}$ n/m <sup>2</sup> , E>0.18 MeV)	1600°C Safety Tests	1700°C Safety Tests	1800°C Safety Tests
1	40	1350	1100	760	11.5	6.3	6 (0)	–	2 (0)
2	25	1000	910	710	18.0	6.8	2 (2)	–	1 (1)
3	25	1480	1380	1060	18.0	7.2	3 (4)	2 (2)	2 (3)
4	25	990	910	760	16.5	6.0	2 (2)	–	–
5	40	890	780	600	10.8	3.4	2 (8)	–	1 (2)
Totals:							15 (16)	2 (2)	6 (6)

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### 2.15.2 Temperature Transient Safety Testing

A temperature-versus-time profile had been calculated for a conduction-cooldown event in the German Arbeitsgemeinschaft Versuchsreaktor (AVR) pebble-bed reactor. This profile had a peak temperature of 1600°C. Schenk et al. tested irradiated UO<sub>2</sub> fuel pebble AVR-91/31 using this profile where the temperatures had been shifted up to give a peak temperature of 1695°C (Schenk et al. 1993). Transient tests using this same temperature profile were performed with three AGR-1 (Stempien et al. 2016b) and three AGR-2 (Hunn et al. 2019a) compacts for a total of about 21,900 particles. The AGR-1 and AGR-2 test results showed that no SiC layers or TRISO particles failed, and the fission product release rates at a particular temperature during the transient were consistent with results from isothermal tests at corresponding temperatures. Based on the low observed fission product releases, the temperature variation did not stress the AGR fuel any more than an isothermal test at 1600 or 1700°C. Neither AGR-1 nor AGR-2 experienced the higher TRISO failure rates observed during the temperature transient test of the German UO<sub>2</sub> sphere AVR-91/31. Therefore, fuel testing in temperature transients will not be repeated using AGR-5/6/7 fuel in order to conserve resources for higher-priority activities.

### 2.15.3 Loose Particle Testing to Measure Fission Product Releases from Particles

The Furnace for Irradiated TRISO Testing (FITT) has been used at ORNL for heating loose AGR-2 particles in inert atmospheres at temperatures from 1150 to 1600°C for times ranging from 100 hours to 1500 hours. These tests were intended to measure releases directly from the particles rather than releases from compacts, which are a function of release/retention in the particle/compact matrix composite system. Particles were counted via IMGA both before and after the heating period to determine releases of gamma-emitting fission products. Ag-110m and Eu-154 were of particular interest because they can be released from intact TRISO particles. Data were obtained for Eu-154; however, few data were obtained for Ag-110m release from these loose AGR-2 particles. Given that Ag-110m has a half-life of about 250 days, FITT testing to probe its release from AGR-5/6/7 loose particles will need to be performed before significant decay of Ag-110m has occurred. In the course of this testing, Eu-154 releases from AGR-5/6/7 loose particles will also be measured. The times and temperatures used for AGR-2 testing shall be considered in developing a test matrix for FITT testing of AGR-5/6/7 particles.

### 2.15.4 Oxidation Testing

Two accident scenarios in HTGRs include the ingress of air (from a break in the system pressure boundary followed by depressurization and air infiltration through the break) and moisture ingress (from a steam generator tube break and in-leakage of water from the steam generator). Whether or not these accidents would be considered part of the reactor design-basis may depend on a number of variables including the specifics of a given plant design and the judgement of the nuclear regulator (e.g., U.S. Nuclear Regulatory Commission). The Air/Moisture Ingress Experiment (AMIX) facility is under construction at INL to test irradiated fuels under oxidizing conditions containing air or water vapor. A detailed oxidation test plan for irradiated fuels and graphite materials is given in (Stempien 2019). PLN-5934, Rev 0 calls for 11 tests of AGR-5/6/7 fuel compacts, seven in moisture and four in air (Stempien 2019). Additional oxidation tests in AMIX are planned for samples from earlier AGR experiments (e.g., AGR-3/4). Some oxidation tests of loose AGR-2 particles in air and mixtures of air and helium are planned for the end of fiscal year 2020 and in fiscal year 2021 in FITT at ORNL. Additional testing may be performed in FITT in order to inform and focus the AMIX test matrix.

### 2.15.5 Reirradiation Testing for Short-Lived Fission Products

An important data need for TRISO fuel performance evaluation is the behavior (i.e., retention or release from the fuel) of short-lived fission products, particularly short-lived radioiodine (I-131). Short-lived fission products decay away before the fuel can be retrieved from the test train after completion of

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the irradiation in ATR. The NRAD reactor at HFEF has been used to reirradiate loose AGR-2 fuel particles and AGR-3/4 compacts to produce measurable quantities of I-131 (half-life 8.0 days) and Xe-133 (half-life 5.2 days). The samples were then quickly retrieved and loaded into the FACS furnace so that the time and temperature-dependent release of I-131 and Xe-133 could be measured.

Intact TRISO particles retain fission gases and I-131; thus, the AGR-2 particles used for reirradiation tests had been deliberately cracked so the TRISO coatings were breached prior to reirradiation and FACS testing, allowing fission gases and I-131 to escape the particles upon heating in the FACS furnace. The AGR-3/4 fuel compacts contained 20 DTF particles that released short-lived fission products during the FACS heating tests. Online fission gas monitoring of the AGR-5/6/7 irradiation test indicates that significant TRISO failure has occurred in Capsule 1. This could make Capsule 1 compacts useful for reirradiation-heating tests to determine short-lived fission product release from the exposed kernels of the failed TRISO particles (similar to how the DTF particles were used in AGR-3/4). The highest-temperature AGR-5/6/7 capsule, Capsule 3, may also have some TRISO failures, making it useful for testing short-lived fission product releases from exposed kernels. While the number of failed TRISO particles in a compact may not be known prior to a reirradiation test, DLBL of the compacts following the test would be used to establish the number of exposed kernels in the compact. Additional testing of cracked, loose particles could be performed as necessary (with or without reirradiation) for determining FACS condensation plate and/or CCCTF deposition cup fission product collection efficiencies.

Design work is currently underway as part of a Laboratory Directed Research and Development project to install an in-pile furnace at NRAD (Riley 2020). It may be possible to install a fission gas monitoring system in conjunction with this furnace. Conversations with NRAD personnel revealed that the AGR program may have an opportunity to provide input to this design. Depending on the availability of this system, there may also be an option to use reirradiation, in-pile heating, and fission gas measurement to measure short-lived Xe-133 release directly in NRAD. The data from recent AGR-3/4 compact reirradiation heating tests indicate similar behavior between I-131 and Xe-133 releases; therefore, measuring only Xe-133 using an in-pile facility with fission gas monitoring at NRAD could be an expedient way to obtain additional data on short-lived fission product release.

As discussed in (Stempien 2019), reirradiation in NRAD will be utilized prior to oxidation testing of at least some of the loose particles and intact compacts in AMIX. This would enable determination of the release of short-lived fission products due to kernel hydrolysis and is an expected activity under PLN-5934 (Stempien 2019).

### **2.15.6 Short Heating Tests With or Without Reirradiation to Screen Compacts for TRISO Failures**

The highest priority is to determine the cause of TRISO failures in Capsule 1. A somewhat lower priority activity that could be pursued would be to determine if selected Capsule 1 compacts have no TRISO failures. Reirradiations in NRAD followed by heating tests in the FACS furnace have been performed previously. Reirradiation followed by low-temperature heating ( $\leq 1200^{\circ}\text{C}$ ) and measurement of any short-lived fission gases released from the compact could be used to screen compacts for TRISO failures. If short-lived fission gases in excess of what would be expected from as-fabricated dispersed uranium are measured from the reirradiation-heating test, that would confirm that the compact has TRISO failures. If no short-lived fission gases are measured, that would be a good indication that the compact does not contain failed TRISO particles. It may, however, contain particles with failed SiC.

Inserting a compact into NRAD and then heating it in the FACS furnace with its fission gas monitoring system (FGMS) could be used for screening; however, the throughput will be very low given the compact transfer steps in and out of NRAD and FACS. Furthermore, FACS is needed for other compact testing. One option is the installation of a furnace and FGMS at one of the NRAD beam ports.

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This would allow simultaneous reirradiation and heating of the compact so that short-lived fission gas measurements can be used to determine if a compact has TRISO failures without having to install and uninstall compacts in the NRAD core. The limitation here is the very low neutron flux at the beam ports: approximately one million times lower than in the core. Such a low flux may not produce enough short-lived fission gases for detection in FGMS.

Depending on the availability of the NRAD in-pile furnace (Riley 2020) and the inclusion of a fission gas monitoring capability with that system, there may also be an option to use reirradiation, in-pile heating, and fission gas measurements to screen some Capsule 1 fuel for TRISO failures without constructing a furnace at a beam-line or having to transfer reirradiated compacts to FACS.

A simpler approach would be to test as-irradiated Capsule 1 fuel (without a reirradiation step) in a furnace with a fission gas monitoring system similar to that used with the FACS furnace to measure Kr-85 release. The problem with this approach is that any failed particle would have had significant time in-pile at ATR to release fission gases prior to PIE, and depending on the irradiation temperature of that fuel, very little Kr-85 may remain. Recent as-irradiated AGR-3/4 fuel compact heating tests in FACS have measured Kr-85 releases ranging from 0.3% of the exposed kernel inventory after 300 hours at 1200°C to 1.6% of the exposed kernel inventory after 300 hours at 1600°C (Stempien et al. 2018b). While this is measurable Kr-85, it is significantly less than the inventory of a single AGR-3/4 particle, and is not a good indicator that there are 20 exposed kernels in each AGR-3/4 compact. Many failed particles would have to exist for significant Kr-85 to be measured in relatively low-temperature (<1200°C) post-irradiation screening tests of Capsule 1 fuel. This type of testing remains a possibility, but reirradiation screening tests where short-lived fission gases can be measured would offer vastly enhanced sensitivity for non-destructively determining the extent of failed TRISO particles in Capsule 1 compacts.

### **2.15.7 Post-Heating Test PIE**

Given the substantial number of tests that will need to be performed and the expectation that AGR-5/6/7 PIE must be completed in a timely manner, post-heating test PIE will need to be limited to selected compacts and/or scope. Table 9 lists up to 24 inert heating tests using AGR-5/6/7 compacts. Section 2.15.3 discussed 11 AGR-5/6/7 compact oxidation tests. Given that oxidation testing is a new activity, most, if not all, of those samples will undergo post-test destructive exams such as DLBL and ceramography. If safety test results are generally similar to those from AGR-1 and AGR-2, the exams after inert safety tests will be limited as much as possible while still determining if (1) intact AGR-5/6/7 TRISO fuel behaves like previous fuel from AGR-1 and AGR-2 testing and (2) failed AGR-5/6/7 SiC layers and failed TRISO coatings occur under similar conditions (i.e., prior irradiation history and safety-test temperatures) and via the same mechanisms as failures from AGR-1 and AGR-2. More extensive post-test analysis may be warranted if AGR-5/6/7 safety test results deviate significantly from AGR-1 and AGR-2.

The AGR-1 and AGR-2 PIE campaigns saw a combined zero TRISO failures and only three SiC layer failures in 1600°C safety testing of 45,800 particles. Four AGR-5/6/7 compacts with no TRISO and no SiC failure (as indicated by fission gas and condensation plate/deposition cup measurements) could be subject to post-test destructive exams including but not limited to, DLBL, IMGA, ceramography, optical microscopy, and electron microscopy. The goals of these exams would be to observe the intact fuel behavior and morphology to determine if they are similar to AGR-1 and AGR-2 fuel with similar irradiation histories that were also tested at 1600°C. Little or no post-heating test destructive exams would be performed beyond these four compacts. Destructive exams of compacts that indicated a failed SiC or TRISO coating during a heating test may also have to be limited in number and/or scope.

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**2.16 Sample Archiving and Disposal**

All AGR-5/6/7 compacts not subject to destructive analysis at INL or shipped to ORNL will be held in temporary storage in HFEF. After deconsolidation of a compact at either INL or ORNL, all particles not used for destructive analysis will be held in temporary storage at HFEF at INL or the ORNL Irradiated Fuels Examination Laboratory (IFEL). Disposition plans for the unused fuel specimens will be determined at a later date by AGR staff.

**3. LIMITATIONS OF THIS PLAN**

Attempts were made in the previous sections to capture all major activities and essential details. These were generally discussed in chronological order, but many activities may be performed in parallel, as appropriate. Some activities may require additional supporting procedures and details. It is anticipated that details of some operations and sample selections will change as data are acquired. Any destructive compact exams (e.g., ceramography, DLBL, heating tests) shall have test plans written prior to their performance. The supporting documents cited in this plan shall be considered key elements of the plan.

**4. WASTE HANDLING**

PIE activities will generate small amounts of radioactive waste (estimated at less than 10 ft<sup>3</sup> per year) that must be properly dispositioned. This waste will be generated by the disassembly, ceramography, safety testing, equipment maintenance activities, and analytical laboratory activities associated with the AGR-5/6/7 examination and analysis. Typical wastes will include short sections (< 2 meters) of 1/16- to 1/8-inch-diameter sheathed TCs and gas lines, turnings from the tubing cutter, condensation plates from the heating furnaces, pneumatic transfer rabbits, and parts replaced on the safety testing furnaces (e.g., replacement tantalum hot zone components, metal heat shields, the graphite furnace elements, and other relatively small furnace components), and analytical laboratory solids and solidified liquids. Additionally, after analysis activities of the test-train capsule components (e.g., capsule head, through-tubes, outer shell, graphite holder, and ceramic and graphite disks, spacers, and insulators) are completed, these components will be dispositioned as waste. Most of the 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. These wastes will be gathered and placed into appropriate disposal containers. At INL, these wastes will be stored in the Radioactive Scrap and Waste Facility located at MFC until final disposal arrangements can be made.

The ceramography preparation work will involve cutting, slicing, grinding, and polishing activities that create small volumes of highly radioactive wastes, including the grinding and polishing residuals and the unused portions of the fuel compacts and/or particles. The whole compacts may have contact radiation fields as high as 10<sup>4</sup> R/hr 6 months after the end of the irradiation test. The wastes associated with the fuel compact analysis and the residual compact material will be disposed of after analysis activities are complete. INL Safeguards personnel must be notified and authorize disposition activities of the accountable fuel materials, including analytical and residual material wastes, since they contain accountable materials.

ORNL plans to handle the waste generated by this work through the normal laboratory waste disposal protocols. Most of the waste is expected to be low-level waste or remote-handled low-level waste that falls within the current waste disposal pathways. The liquid waste generated by the analytical tasks will be handled by the normal channels, either by direct disposal to the liquid waste system, drying and disposal as solid waste, or grouting, if necessary. The remaining compacts, if any, will be dispositioned as spent nuclear fuel. Since the test train and capsule disassembly work will be done at INL, very little

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activated metal will be handled at ORNL; most of the waste generated will be from the ceramographic and analytical tasks.

## 5. QUALITY ASSURANCE

### 5.1 Overarching Descriptions and Documents

All activities within the ART AGR Fuel Development and Qualification Program are to be performed according to the requirements identified in the INL Quality Assurance Program Description (Jensen 2018) and ART Quality Assurance Program Plan (QAPP) (Sharp 2020). Among other regulatory and requirements documents, the QAPP states that all activities are to be conducted in accordance with the American Society of Mechanical Engineers (ASME) Nuclear Quality Assurance (NQA)-1-2008/1a-2009. 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 the generation of reports from collected data.

ORNL will perform PIE support services in accordance with their AGR-specific QAPP, QAP-ORNL-NR&D-01 (Vance 2018).

### 5.2 Data Management

INL is responsible to maintain the record copy of all data associated with the PIE and safety testing activities. This 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 the desired data formats. PIE and safety testing data that will be kept as project records will be transferred from their original source to either the Nuclear Data Management and Analysis System (NDMAS) or the INL Electronic Document Management System (EDMS). Primarily, NDMAS will be the data storage forum for machine readable data (e.g., database, spreadsheet, or tab delineated), and EDMS will be the storage forum for other types of information including pictures, reports, Portable Document Format (PDF) documents, technical evaluations (TEVs), and engineering calculation and analysis reports (ECARs). Since NDMAS will have provisions that allow access to the data outside of the INL computer firewall, data that would normally be stored on EDMS may be moved to NDMAS to allow access by users outside INL. The NDMAS 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.

## 6. REPORTING

Program staff will create reports pertaining to results from AGR-5/6/7 PIE to ensure that pertinent data from the PIE activities are available for various programmatic decisions. These will include:

- *Test Train Inspection, Disassembly, and Metrology Report.* This report will summarize the results of preliminary PIE activities, including: exterior visual inspection of the intact test train, gamma scanning and NRAD of the intact test train, test-train disassembly and inspection, and compact and graphite holder metrology. The availability of these data supports revision of the AGR-5/6/7 as-run thermal analyses and PIE decisions regarding sample use and priorities.
- *Topical reports.* Topical reports will be prepared to provide details on specific components of AGR-5/6/7 PIE. These will include topical reports summarizing the destructive PIE performed on specific compacts and safety and oxidation testing results. It is envisioned that a report will be prepared for

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each compact that undergoes destructive PIE. Multiple compacts or topics may be combined in a single report for comparison purposes or if there is another good reason for grouping them together. Other report topics may include:

- Results of gamma scanning the fuel compacts and the graphite compact holders.
  - Results of compact ceramography.
  - AGR-5/6/7 fission product mass balance of the fission products measured on irradiation capsule components outside of the fuel compacts themselves.
  - Observations and analysis of Capsule 1.
- *Periodic meetings, teleconferences, and conferences.* Regular input on PIE activities and experimental results will also be provided as needed for the AGR monthly and quarterly reports. The AGR Fuels PIE staff will make selected PIE data available to the NDMAS database as it is generated and will participate in biweekly teleconferences, fuels program meetings, teleconferences, videoconferences, and annual program review meetings to facilitate dissemination of experimental data as needed by the program and to discuss relevant issues.
  - *Final AGR-5/6/7 PIE data report.* This report will be prepared at the completion of the AGR-5/6/7 PIE and when all data have been obtained from ongoing experiments and analyses. It will include data summaries taken from the relevant topical reports and present the pertinent conclusions from the AGR-5/6/7 PIE.

## 7. PRELIMINARY PIE SCHEDULE

The following schedule is preliminary. It does not account for availability of common resources used between AGR-2, AGR-3/4, AGR-5/6/7, and other programs at MFC. Some of the expected major reports are listed, but this is not an exhaustive list. Activities may be added or subtracted from this schedule. The preliminary start and end dates for a given activity may change.

Table 10. Preliminary AGR-5/6/7 PIE schedule.

Date Start	Date End	Activity	Notes
February 2018	July 2020	Irradiation	
August 2020	December 2020	Cooling in ATR canal and preparations for shipping	
January 2021	January 2021	Shipment(s) to HFEF at MFC	Likely the test train will be sectioned between Capsules 2 and 3 and sent to HFEF in two shipments.
January 2021	February 2021	Test-train exterior inspection and photography	Assume first test section comes mid-January.
February 2021	March 2021	Test train PGS	Do one section of test train in Feb, and one section in March. Do all the test-train capsules.
March 2021	April 2021	Test train NRAD	May only scan Capsule 1 in NRAD. Could decide to separate Capsule 1 from the others to send only Cap 1 down to NRAD.

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Date Start	Date End	Activity	Notes
April 2021	April 2021	Test-train disassembly	
April 2021	June 2021	Capsule disassembly	Items removed from the capsules could be gamma scanned and leached or burn/leached after each capsule is completed. <i>Assumes no difficulties in removing/recovering Capsule 1 compacts.</i>
June 2021	June 2021	Compact metrology	
June 2021	August 2021	Graphite holder metrology	
May 2021	December 2021	Compact PGS	As soon as compacts complete both PGS and metrology, some could be shipped to ORNL, and some could be examined further at INL. Assumes 1 to 2 weeks to scan a set of 12 compacts.
August 2021	February 2022	Graphite holder PGS	Will likely need to push this end date out given each holder may take 2 weeks to scan, compact scanning will also be occurring, and we cannot assume the PGS will be 100% available for AGR during this time frame.
October 2021	June 2022	PGS Report	PGS report to include results from compact and graphite holder scans. Could think about splitting PGS report into two reports: compacts and holders.
August 2021	February 2022	AGR-5/6/7 Disassembly and Metrology Report	
October 2021	November 2021	Fuel compact shipment to ORNL	Could begin as soon as PGS and metrology and inspection are completed, perhaps closer to July.
July 2021	November 2021	Non-metallic capsule components gamma counting	
July 2021	August 2021	Capsule metal components leaching	
July 2021	August 2021	Capsule ceramic components leaching	
July 2021	December 2021	Capsule graphite spacers and felts burn-leach	Includes all lab work, but data review/transmission may take longer.
September 2021	June 2022	Compact reirradiation screening -OPTIONAL	Might be able to screen at least some Capsule 1 compacts for broken TRISO.
October 2021	July 2022	AGR-5/6/7 Fission Product Mass Balance Report	Includes time from starting the report to getting all of the AL data and finishing the report. Report can be started with PGS data of holders and some gamma of capsule components.
October 2021	June 2022	Fuel compact ceramography	
July 2022	January 2023	Fuel compact ceramography Report	
October 2021	February 2026	Fuel compact as-irradiated DLBL	Estimate includes DLBL hot cell work, IMGA and radioanalytical work.

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Date Start	Date End	Activity	Notes
April 2022	February 2026	Destructive burnup analysis	Includes all analysis and report. Assumes use of compacts undergoing as-irradiated DLBL.
December 2021	February 2026	As-irradiated fuel particle micro analysis at ORNL	
November 2021	December 2024	Fuel compact inert heating tests at INL	Includes time to receive FGMS and condensation plate results. Does not include time to do any post-test PIE on compacts.
January 2022	December 2024	Fuel compact inert heating tests at ORNL	Includes time to receive FGMS and condensation plate results. Does not include time to do any post-test PIE on compacts.
August 2022	December 2025	Compact oxidation tests	Does not include any post-test compact destructive exams.
January 2022	December 2025	Post-safety-test compact/particle exams primarily ORNL	
September 2022	March 2026	Post-oxidation test compact/particle exams	
October 2025	December 2026	Final AGR-5/6/7 PIE Report	

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