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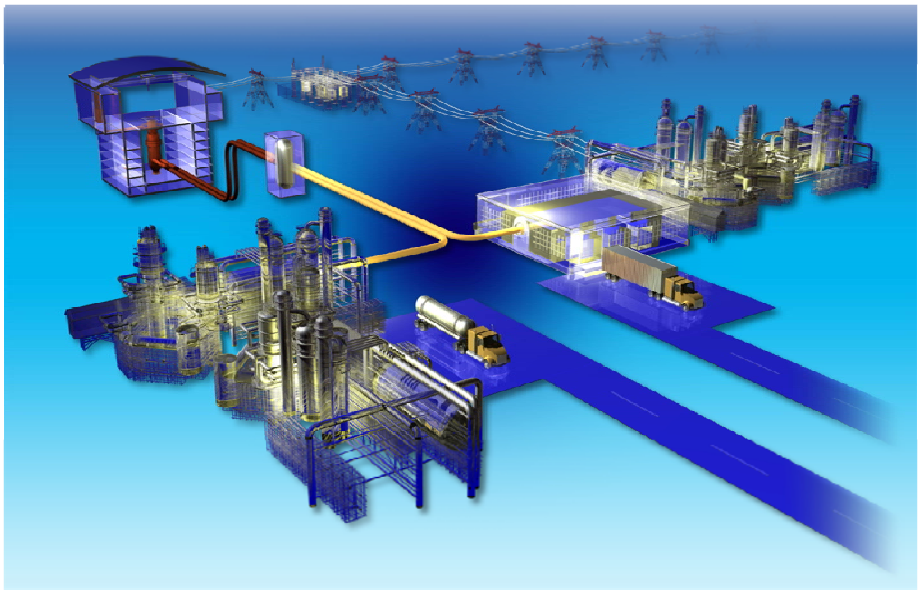
Post-Irradiation Examination Plan

Project No. 29412, 23843, 23841

AGR-1 Post-Irradiation Examination Plan



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REVISION LOG

Rev.	Date	Affected Pages	Revision Description
0	7/22/08	All	New document.
1	03/29/2010	Various	Update to AGR-1 irradiation and shipment status in Section 1.2.3. Major changes to the PIE logic in Section 2, including revised Figure 4 and addition of new Figure 5. Revision to details of test train shipment in Section 3.1. Eliminate previous section on neutron radiography. Addition of new Section 4.3 (Leadout Disassembly). Update to description of metrology system in Section 4.4. Rearrange various subsections in Section 6. Update to description of graphite holder gamma scanning in Section 6.1, including new Figure 12. Addition of Section 6.2 on compact gamma scanning. Changes to work scope for thermocouple examination (Section 6.4.2). Edit Section 6.5 to include only thermal conductivity measurements on compacts. Change scope in Section 6.6 to include quantitative inventory measurements of graphite holders and graphite spacers. Additional details added to compact deconsolidation discussion, including the option to deconsolidate half-compact segments. Edit PIE logic for leach-burn-leach analyses in Section 6.9: eliminate “compact screening” activity using 4 compacts from each capsule and replace with an LBL test on a single compact from Capsules 1—5 (2 compacts from Capsule 6). Add details on destructive burnup measurements to Section 6.11. Add details of IMGA analysis to Section 6.12. Add accident test matrix (Table 7) in Section 6.14. Add Section 6.16 on approach to compact allocation. Add Section 7 on PIE activity prioritization. Revise tentative PIE schedule in Section 8. Add Section 10.1 on PIE data management. Remove discussion of coincident gamma spectroscopy in Appendix B. Additional minor content and grammatical corrections throughout.

SUMMARY

The AGR-1 Irradiation Experiment is the first in a series of test irradiations for the Next Generation Nuclear Plant Fuel Development and Qualification Program. At the conclusion of irradiation in the Advanced Test Reactor, the AGR-1 test train will be removed from the reactor and shipped to the Hot Fuel Examination Facility at the Materials and Fuels Complex, located at Idaho National Laboratory (INL), for nondestructive examination and disassembly, followed by extensive post-irradiation examination (PIE) at INL and Oak Ridge National Laboratory. The PIE for this experiment will focus on:

1. Assessing the performance of the multicapsule instrumented test train and components
2. Evaluating the fission product retention of the fuel during irradiation and during post-irradiation accident testing
3. Characterizing the compacts and individual particles to observe the condition of the matrix material, kernels, and coatings and to document any concerns.

The AGR-1 test includes four different TRISO-coated particle fuel types (a baseline and three variants), each possessing slightly different properties for one of the particle coating layers due to controlled variations in the fabrication parameters. Comparisons of the different fuel types will be an important aspect of this PIE effort, providing a better understanding of the coating traits that lead to optimal performance and aiding in the selection of a reference fuel for subsequent fuel qualification irradiation experiments. The PIE for the AGR-1 experiment will include the following key activities:

- Test train inspections and nondestructive analyses to determine the overall condition of the test train exterior and the condition and location of internal components
- Test train disassembly, extraction of fuel and other interior components (including the graphite fuel holders, melt wires, and flux wires), and evaluation of test train performance by characterization of thermocouples, melt wires, and flux wires
- Dimensional measurements of the fuel compacts and graphite holders
- Measurement of fuel compact burnup and selected fission product inventories
- Post-irradiated fission metals release analysis by measurement of fission metal inventories on metal capsule components and gamma scanning of graphite fuel holders
- Deconsolidation of compacts to provide particles for subsequent analyses and leach-burn-leach analysis to quantify SiC failure fractions and evaluate fission product inventories in the compact matrices
- Microanalytical characterization of fuel compacts and particles using optical metallography, scanning electron microscopy, and electron probe microanalysis to investigate fuel microstructures, the condition of coatings, and fission product migration within in the fuel
- Accident testing to investigate release of selected fission products (including radioisotopes of Ag, Cs, I, Sr, Te, and Eu) at elevated temperatures in pure helium

- Irradiated microsphere gamma analysis (IMGA) to measure fission product inventories and evaluate fission product retention for individual particles.

These experiments will provide the program with early data on uranium oxycarbide particle fuel performance and on the fundamental effects of irradiation and post-irradiation heating on fuel properties. The results will indicate if the program's current approach to fuel fabrication has been successful in producing high quality fuel that exhibits good irradiation and accident performance.

This document presents the plan for PIE of the AGR-1 experiment and the general flow of PIE activities along with detailed descriptions of anticipated tasks.

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FIGURES

TABLES

ACRONYMS

AGR	Advanced Gas Reactor
ATR	Advanced Test Reactor
ASTM	American Society for Testing and Materials
CCCTF	Core Conduction Cooldown Test Facility
DTC	Dry Transfer Cubicle
ECAR	engineering calculation and analysis report
EDMS	Electronic Document Management System
EDS	energy-dispersive x-ray spectroscopy
EPMA	electron probe microanalysis
FACS	Fuel Accident Condition Simulator
FIMA	fissions per initial metal atom
GT-MHR	Gas Turbine – Modular Helium Reactor
HFEF	Hot Fuel Examination Facility
HPGe	high purity germanium
ICP-MS	inductively coupled plasma-mass spectrometry
IMGA	irradiated microsphere gamma analysis
INL	Idaho National Laboratory
IPyC	inner pyrocarbon
LBL	leach-burn-leach
MFC	Materials and Fuels Complex
NGNP	Next Generation Nuclear Plant
NRAD	neutron radiography
OPyC	outer pyrocarbon
ORNL	Oak Ridge National Laboratory
PGS	Precision Gamma Scanner
PIE	post-irradiation examination
PNNL	Pacific Northwest National Laboratory
R/B	release-to-birth ratio
SEM	scanning electron microscope
TC	thermocouple
TDO	Technology Development Office
TIMS	thermal ionization mass spectrometry

TRIGA	Training Research Isotope General Atomics
TRISO	tri-isotropic
UCO	uranium oxycarbide
VHTR	very high temperature reactor
WDS	wavelength-dispersive x-ray spectroscopy

1. INTRODUCTION

1.1 Background

The Next Generation Nuclear Plant (NGNP) Fuel Development and Qualification Program[a] was established to perform the requisite research and development on coated particle high-temperature gas reactor fuel to support deployment of a very high temperature reactor (VHTR), which has been selected as the reactor concept for the NGNP project. The overarching goal of the program is to provide a baseline fuel qualification data set to support licensing and operation of a VHTR. To achieve these goals, the program includes the elements of fuel fabrication, irradiation, post-irradiation examination (PIE) and accident testing, fuel performance, and fission product transport (Petti et al. 2008).

Eight fuel irradiation experiments are planned in the Advanced Test Reactor (ATR) at Idaho National Laboratory (INL). These experiments are intended to provide data on fuel performance under irradiation, support fuel process development, qualify the fuel for operating and accident conditions, provide irradiated fuel for accident testing, and support the development of fuel performance and fission product transport models. The first of these irradiation tests, AGR-1, began in the ATR in December of 2006. This experiment is intended to act as a shakedown test of the multicapsule design and to provide early data on fuel performance that will be used in fuel fabrication process development. This test will also provide samples for post-irradiation accident testing, where fission product retention of the fuel at high temperatures will be experimentally measured. The AGR-1 fuel, test train, and experiment description are presented in the AGR-1 Test Plan (Maki 2006). The test objectives and success criteria for AGR-1 are discussed by Kendall (2006).

1.2 AGR-1 Irradiation Experiment

1.2.1 AGR-1 Fuel

The kernels for the AGR-1 fuel are made of low-enriched uranium oxycarbide (UCO). Kernel diameters are approximately 350 µm with a U-235 enrichment of approximately 19.7%. The detailed characterization data of the kernel fabrication lot used in the AGR-1 fuel are given in the Data Certification Package (BWXT 2005). The kernels are coated successively with a porous carbon buffer (~100 µm thick), an inner pyrolytic carbon (IPyC) layer (~40-µm thick), a SiC layer (~35-µm thick), and an outer pyrolytic carbon (OPyC) layer (~40-µm thick). The total fuel particle diameter is ~800 µm.

The AGR-1 irradiation experiment includes a baseline fuel as well as three different fuel variants. Each of the fuel variants represents a particular deviation in the processing parameters of either the IPyC or SiC coating layers compared to the baseline fuel. The variants are included in order to explore areas of uncertainty in the fuel processing/performance relationship and enhance the prospects of successful performance by at least one fuel type. The baseline fuel properties were established based on extensive reviews of U.S. and German coated particle fuel performance data, with the objective of maximizing the prospects of successful performance during irradiation and accident testing. The primary uncertainties in achieving successful performance of the baseline fuel were associated with permeability and dimensional stability of the dense pyrocarbon layers and with metallic fission product permeability of the SiC layer. The changes in the coating deposition conditions relative to the baseline result in differences in coating microstructures and densities, which could influence particle performance during irradiation and accident testing. The specific changes in baseline and variant fuel coating procedures and the effect on coating properties are as follows:

a. Known previously as the Advanced Gas Reactor (AGR) Program.

- *Baseline*. Because of its excellent irradiation performance with UO_2 kernels, coating process conditions used to fabricate historic German fuel were chosen as the starting point for the baseline fuel. Parametric studies refined these conditions for the specific coater to be used to coat AGR-1 fuel.
- *Variant 1*. The IPyC coating temperature was increased relative to the baseline process (from 1265 to 1290°C) for this variant. This change is expected to enhance the irradiation dimensional stability of the pyrocarbon, but with increased permeability and resulting uranium dispersion. Also, the IPyC layer density is slightly lower than the baseline density.
- *Variant 2*. The IPyC coating gas fraction was increased relative to the baseline process (from 0.3 to 0.45) for this variant. This change is also expected to enhance the irradiation dimensional stability of the pyrocarbon but without significantly increasing uranium dispersion. Also, the IPyC layer density is slightly higher than the baseline density.
- *Variant 3*. The carrier gas composition for the SiC layer deposition was changed from 100% hydrogen to a 50% argon-50% hydrogen mixture and the deposition temperature was lowered relative to the baseline (from 1500 to 1425°C). This change is expected to reduce the potential for SiC defects resulting from uranium dispersion and provide a variation in SiC microstructure that may be less permeable to metallic fission products.

Selected properties of the baseline and variant fuel forms are given in Table 1 (Maki 2006). Detailed processing parameters and characterization data for the particle coatings of the baseline and variant fuel forms have been given in the final fuel data packages (Hunn and Lowden 2006a–d).

Table 1. Selected properties of baseline and variant particles used in AGR-1 experiment (Maki 2006).

Property	Specified Range for Mean Value	Actual Mean Value ± Population Standard Deviation			
		Baseline	Variant 1	Variant 2	Variant 3
Buffer thickness (μm)	100 ± 15	103.5 ± 8.2	102.5 ± 7.1	102.9 ± 7.3	104.2 ± 7.8
IPyC thickness (μm)	40 ± 4	39.4 ± 2.3	40.5 ± 2.4	40.1 ± 2.8	38.8 ± 2.1
SiC thickness (μm)	35 ± 3	35.3 ± 1.3	35.7 ± 1.2	35.0 ± 1.0	35.9 ± 2.1
OPyC thickness (μm)	40 ± 4	41.0 ± 2.1	41.1 ± 2.4	39.8 ± 2.1	39.3 ± 2.1
Buffer density (mg/m^3)	0.95 ± 0.15	1.10 ± 0.04	1.10 ± 0.04	1.10 ± 0.04	1.10 ± 0.04
IPyC density (mg/m^3)	1.90 ± 0.05	1.904 ± 0.014	1.853 ± 0.012	1.912 ± 0.015	1.904 ± 0.013
SiC density (mg/m^3)	≥3.19	3.208 ± 0.003	3.206 ± 0.002	3.207 ± 0.002	3.205 ± 0.001
OPyC density (mg/m^3)	1.90 ± 0.05	1.907 ± 0.008	1.898 ± 0.009	1.901 ± 0.008	1.911 ± 0.008
IPyC anisotropy[a] (BAF)	≤1.035	1.022 ± 0.002	1.014 ± 0.001	1.023 ± 0.002	1.029 ± 0.002
OPyC anisotropy (BAF)	≤1.035	1.019 ± 0.003	1.013 ± 0.002	1.018 ± 0.001	1.021 ± 0.003
IPyC anisotropy post compact anneal (BAF)	Not specified	1.033 ± 0.004	1.021 ± 0.002	1.036 ± 0.001	1.034 ± 0.003
OPyC anisotropy post compact anneal (BAF)	Not specified	1.033 ± 0.003	1.030 ± 0.003	1.029 ± 0.004	1.036 ± 0.002
Sphericity (aspect ratio)	Mean not specified[b]	1.054 ± 0.019	1.056 ± 0.019	1.053 ± 0.019	1.055 ± 0.018
Particle diameter[c] (μm)	Mean not specified	799.7	804.0	798.3	795.1
Particle mass (g)	Mean not specified	7.27×10^{-4}	7.33×10^{-4}	7.24×10^{-4}	7.26×10^{-4}

a. Specification does not apply to Variants 1 and 2.
b. Critical region is specified such that ≤1% of the particles shall have an aspect ratio ≥1.14.
c. Based upon mean average particle measurements, not sums of mean layer thicknesses.

After fabrication, the fuel particles were formed into right cylindrical compacts using a matrix composed of a thermosetting carbonaceous material. The compacts are nominally 12.3 mm in diameter and 25 mm long (Figure 1). Prior to compacting, the particles were overcoated with approximately 165 μm of the matrix material to prevent particle-to-particle contact and achieve the desired volume fraction of fuel particles in the compact. Each compact was fabricated to have a fuel-free carbon end-cap ~1.5-mm thick on both the top and bottom to prevent damage to the embedded particles from contact during handling or irradiation. There are roughly 4,100 fuel particles in each compact. The total compact mass is roughly 5.3–5.6 g with a mean uranium loading of approximately 0.9 g per compact. Detailed characterization data for the compacts has been given previously (Hunn et al., 2006a–d). Selected properties of the baseline and variant fuel compacts are given in Table 2.

Figure 1. AGR-1 fuel compact.

Table 2. Selected properties of AGR-1 baseline and variant compacts.

Property	Specified Range for Mean Value	Actual Mean Value ± Population Standard Deviation			
		Baseline	Variant 1	Variant 2	Variant 3
Compact mass (g)	Not specified	5.4789	5.3371	5.3736	5.5930
Mean uranium loading (g U/compact)	0.905 ± 0.04	0.917	0.915	0.904	0.912
Diameter[a] (mm)	12.22 – 12.46	12.36 ± 0.01	12.36 ± 0.01	12.36 ± 0.01	12.34 ± 0.01
Length[a] (mm)	25.02 – 25.40	25.066 ± 0.080	25.123 ± 0.030	25.077 ± 0.065	25.227 ± 0.037
Number of particles per compact[b]	Not specified	4154	4145	4095	4132
Particle volume packing fraction[b] (%)	Not specified	36.99	37.42	36.26	36.04
Effective overall compact density[b] (mg/m^3)	Not specified	1.822	1.771	1.786	1.854
Compact matrix density[b] (mg/m^3)	Not specified	1.297	1.219	1.256	1.344
U contamination fraction (g exposed U/g U in compact)	$\leq 1.0 \times 10^{-4}$	3.6×10^{-7}	2.8×10^{-7}	2.6×10^{-7}	1.3×10^{-7}
Defective SiC coating fraction[c]	$\leq 2.0 \times 10^{-4}$	$\leq 1.3 \times 10^{-4}$	$\leq 6.0 \times 10^{-5}$	$\leq 9.6 \times 10^{-5}$	$\leq 6.0 \times 10^{-5}$
Defective IPyC coating fraction[c]	$\leq 2.0 \times 10^{-4}$	$\leq 6.0 \times 10^{-5}$	$\leq 6.0 \times 10^{-5}$	$\leq 6.0 \times 10^{-5}$	$\leq 6.0 \times 10^{-5}$
Defective OPyC coating fraction[c]	$\leq 1.0 \times 10^{-2}$	$\leq 7.2 \times 10^{-4}$	$\leq 1.7 \times 10^{-3}$	$\leq 7.2 \times 10^{-4}$	$\leq 7.3 \times 10^{-4}$

a. Allowable range corresponding to upper and lower critical limits specified with no compacts exceeding the limits that require 100% inspection of all compacts.
b. Value derived from other characterized properties.
c. Upper limit at the 95% confidence level.

1.2.2 AGR-1 Irradiation Test Train

The AGR-1 test train consists of six irradiation capsules, each approximately 1.4 inch (36 mm) in diameter and 6 inches (152 mm) long and each containing a total of 12 fuel compacts in three stacks (Figure 2). The capsules consist of a graphite fuel holder with holes machined for insertion of fuel compacts, thermocouples (TCs), encapsulated melt and flux wires, and molybdenum through-tubes to allow gas lines and TC leads to pass through to the other capsules in the test train. The graphite fuel holders contain boron carbide (B_4C) as a burnable poison to offset U-235 depletion and provide a more uniform particle power level throughout the experiment. The orientation of the compact stacks in the irradiation capsule places Stacks 1 and 3 closer to the reactor core than Stack 2 (see Figure 2), which would result in much higher neutron fluxes in Stacks 1 and 3. To counteract this effect, a combination of hafnium and stainless steel shrouds surround the graphite holder to provide a more uniform neutron flux during the experiment. The entire assembly is encapsulated in a stainless steel outer shell.

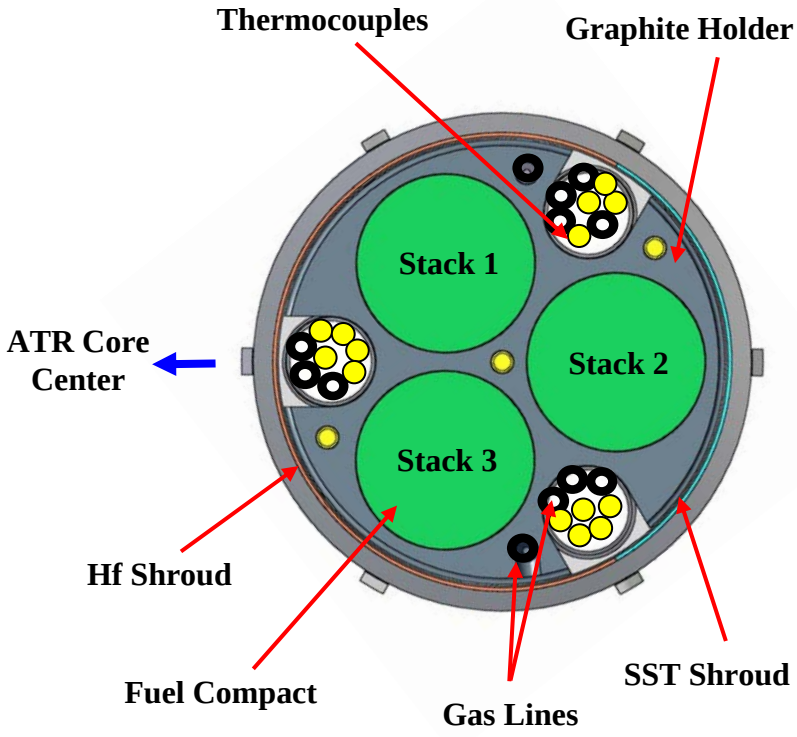

Figure 2. Radial cross-section schematic of an AGR-1 irradiation capsule (Maki 2006).

A total of six independent capsules are used in the AGR-1 test train, with each capsule containing only one type of fuel (baseline or variant). The capsules are stacked end-to-end and welded together to form the fueled portion of the test train. The relative location of each capsule in the test train and the type of fuel in each are shown in Table 3. Each capsule is supplied with an inert sweep gas mixture of helium and neon. Because of the very different thermal conductivities of the gases, varying the gas mixture can act to manage the temperature in the capsule. The sweep gas from each capsule is routed to a detector that measures the quantity of fission gas present in the effluent. This provides a means of monitoring the integrity of the fuel throughout the irradiation. In addition, He-3 gas can be used as the sweep gas mixture to limit power spikes that might occur during occasional high power cycles in the ATR. The AGR-1 test

train is described in further detail in the AGR-1 Test Plan (Maki 2006) and a detailed description of test train assembly is given in the Engineering Work Instructions (Palmer 2006).

Table 3. Capsule sequence in test train and fuel variant in each capsule.

Capsule	Fuel variant
Capsule 6 (top)	Baseline
Capsule 5	Variant 1
Capsule 4	Variant 3
Capsule 3	Baseline
Capsule 2	Variant 2
Capsule 1 (bottom)	Variant 3

A numbering system has been developed to uniquely identify each compact in the test train. This is based on the specific capsule, level, and stack number. Figure 3 identifies the stack and position (or level) numbers in a particular capsule. For example, Compact 6-4-1 refers to the compact in Capsule 6 at the top (Level 4) of Stack 1.

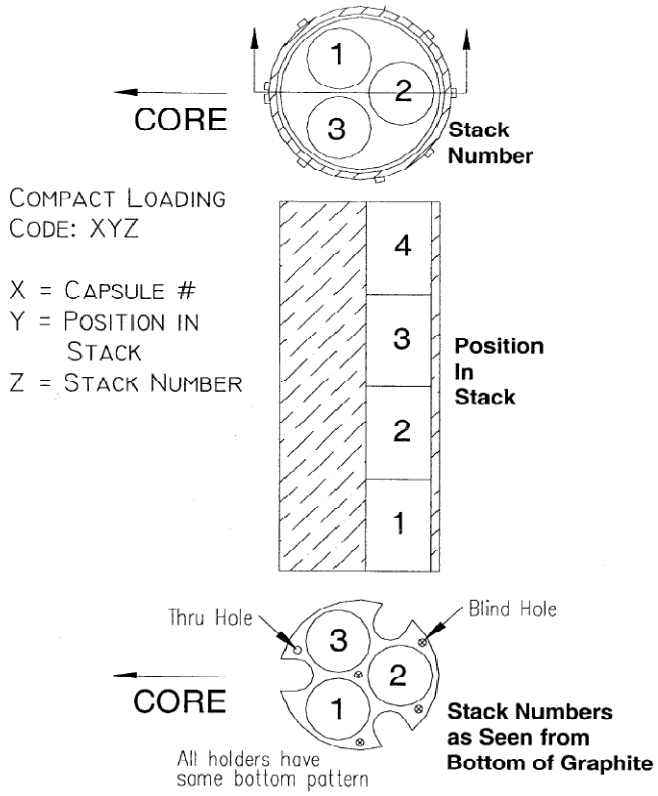

Figure 3. Numbering scheme for AGR-1 compacts.

The TCs used in the test train are a combination of commercial Type N and experimental Mo-Nb TCs fabricated and tested at INL. The TCs are inserted into holes drilled in the graphite fuel holder at various locations. Capsules 2–5 have three TCs each, while the top capsule (Capsule 6) has five TCs and the bottom capsule (Capsule 1) has two TCs. It is difficult to predict the longevity of these TCs under the

irradiation conditions expected in the AGR-1 test train. This is especially true of the Mo-Nb TCs, since these are experimental in nature. Therefore part of the PIE of the test train and capsules will be to examine the TCs to the extent possible and determine failure modes.

Each capsule contains a melt wire package containing two pure beryllium wires, which are encapsulated in vanadium and placed in a hole drilled at the centerline of the graphite holder. These will be used to indicate if the temperature of the capsule (at the location of the melt wires) exceeded 1287°C. Each capsule also contains three different flux wires (pure Fe, V-0.1%Co, and pure Nb), all of which are encapsulated in sealed vanadium tubes and placed around the periphery of the graphite holder. The measured activity in the wires after irradiation will be used to calculate the neutron fluence for the different neutron energy ranges covered by the three flux wires.

1.2.3 AGR-1 Irradiation

The AGR-1 irradiation was conducted in the B-10 position of the ATR. The irradiation test condition requirements for the AGR-1 experiment, which are specified in the AGR-1 Test Specification (Maki 2004), are listed below.

- The instantaneous peak temperature for each capsule shall be ≤1400°C
- The time-averaged, peak temperature for each capsule shall be ≤1250°C
- The time-averaged, volume-averaged temperature for each capsule shall be 1150 +30/-75°C
- The minimum compact-averaged burnup for each fuel compact shall be >14% fissions per initial metal atom (FIMA)
- The compact-averaged burnup goal for the majority of the fuel compacts should be >18% FIMA
- The maximum peak fast neutron fluence for each fuel compact shall be $<5 \times 10^{25}$ n/m^2, E>0.18 MeV
- The minimum peak fast neutron fluence for each fuel compact shall be $>1.5 \times 10^{25}$ n/m^2, E>0.18 MeV
- The instantaneous peak power per particle shall be ≤400 mW/particle.

The AGR-1 irradiation was completed on November 6, 2009 after a total of 620 effective full power days in the reactor. The test train was then capped and moved to the ATR canal to await shipping preparations. The test train was cut to remove the upper curved portion of the leadout section and then transferred to the Dry Transfer Cell where the leadout section immediately above Capsule 6 was removed in preparation for loading into the GE-2000 cask for shipment to the Materials and Fuel Complex (MFC) in early March 2010. The test train will arrive at MFC approximately 4 months after the end of the irradiation.

1.3 AGR-1 PIE

The primary objectives of the AGR-1 test are (Kendall 2006, Petti et al. 2005):

- Perform shakedown testing of the multi-capsule instrumented lead test train
- Provide early irradiation performance for baseline and variant fuel to help develop a fundamental understanding of the relationship between fabrication processes, fuel properties, and irradiation performance
- Possibly support the selection of a reference fuel.

In accordance with these objectives for the irradiation experiment, the primary objectives of the PIE will be to:

- Assess the overall performance of the test train and components and provide data to verify the test train thermal analyses
- Evaluate the fission product retention of the fuel during the irradiation and during post-irradiation accident tests
- Characterize the compacts and individual particles to assess the condition of the matrix material, kernels, and coatings and document any concerns.

For this initial fuel irradiation experiment, the Program would like to demonstrate the following for at least one fuel type:

- Low in-reactor fission gas release (release-to-birth ratio [R/B] $\leq 4 \times 10^{-6}$) as measured during irradiation by sweep gas analysis
- Low release during irradiation (as measured during PIE) of iodine and fission metals (e.g., isotopes of strontium, silver, cesium, and europium)
- Little or no kernel migration
- Minimal corrosion and good structural integrity of the coatings
- Compact matrix stability and integrity
- Minimal fission product release from fuel compacts under high temperature accident conditions (at least 1600°C in an inert gas atmosphere).

These performance measures will provide confidence that the fuel fabrication has met the standards of high quality fuel, the UCO concept is controlling CO pressure buildup and the amoeba effect, the UCO-based fuel has satisfactory iodine and metallic fission product retention under normal operation and accident conditions, and it will confirm the Program's thinking with regard to the coating properties that are critical for good irradiation performance.

This plan describes the specific activities that will be part of the AGR-1 PIE and accident testing, beginning with the transfer of the test train from ATR to the INL Hot Fuel Examination Facility (HFEF) located at the INL MFC. The work is designed to accomplish the objectives described above and meet the AGR-1 PIE test requirements (Demkowicz 2006).

2. AGR-1 PIE WORK FLOW

The preliminary priority for analysis of the six AGR-1 capsules is given in Table 4. Compacts from Capsule 6 will be analyzed first on a limited scale in order to perform a shakedown of many of the PIE methods, including compact ceramography, leach-burn-leach, particle inspections, accident testing, and IMGA. Full PIE will then proceed on the remaining capsules using the prioritized scheme in Table 4. The main interest will be on fuel that exhibits the best performance during irradiation and PIE in order to support reference fuel selection. Analysis of poorly performing fuel in order to identify causes of failure will be a secondary priority.

The prioritization in Table 4 is based on the nature of the coating variants and the final results of the irradiation experiment (e.g., all capsules have exhibited very low R/B values, with values of the various capsules essentially indistinguishable from one another). Priority is given to Capsule 4, which contains the Variant 3 fuel, followed by Capsule 3 which contains Baseline fuel. Since zero particle failures have been observed during the irradiation, the initial focus will be on the performance of SiC in retaining metallic fission products, and therefore the comparison between Capsules 3 and 4 is of greatest interest. In addition, analysis of pre-irradiated SiC microstructures of the fuel variants reveals that while Variant 2 fuel exhibits a SiC microstructure similar to the Baseline, Variant 1 has a larger SiC grain size (believed to be a consequence of the lower IPyC density in this variant). Therefore analyis of Variant 1 fuel (Capsule 5) will be the next priority, and Capsule 2 (Variant 2) will follow. Capsule 1 and the remaining compacts in Capsule 6 will be the lowest priority, as they are the same as the fuel in Capsules 3 and 4, respectively, but were irradiated to lower burnups and at lower temperatures. This overall prioritization and PIE work flow is subject to change depending on the data acquired as PIE proceeds.

Since a primary goal of AGR-1 PIE is to perform relative comparisons between the fuel variants and determine the variant with the best accident test performance in order to support reference fuel selection, PIE experiments from the different capsules will not be performed in a strictly serial manner but will be staggered so that results from multiple capsules can be compared during early testing. Section 6.16 discusses the allocation of compacts for specific accident tests based on the need to identify the effect of various compact attributes (e.g., fuel type and irradiation conditions) on accident performance.

Table 4. Analysis priority for AGR-1 capsules.

Priority	Capsule
1	Capsule 6 (methods shakedown)
2	Capsule 4
3	Capsule 3
4	Capsule 5
5	Capsule 2
6	Capsule 1
7	Capsule 6 (remaining compacts)

After initial disassembly, compact dimensional measurements, and compact gamma scanning, the number of compacts for each type of destructive examination is outlined in Table 5.

Table 5. Numbers of compacts for PIE experiments.

Capsule 6	Ceramography	1 compact (INL) 1 compact (ORNL)
	Deconsolidation/LBL	1 compact (INL) 1 compact (ORNL)
	Accident Testing	1–2 compacts (INL) 1–2 compacts (ORNL)
	Remaining compacts archived for later analysis based on progress and results from Capsules 2 through 5.	
Capsules 1–5	Ceramography	1 compact (INL)
	LBL	1 compact (INL)
	Accident Testing	Up to 10 compacts split between INL and ORNL

The tentative flow of AGR-1 PIE activities is depicted in Figures 4 and 5. The chart includes all PIE activities from initial inspection and disassembly of the test train through detailed PIE on fuel compacts from each capsule. Specific sections of this plan, indicated in red on the chart, describe each activity in further detail.

In the flow chart, all activities after test train disassembly apply to each individual capsule. Test train inspection and disassembly will take place in the HFEF. The PIE of the AGR-1 fuel compacts will take place in facilities at both INL and Oak Ridge National Laboratory (ORNL). Flow chart elements in blue in Figure 4 indicate activities performed at INL, elements in green are performed at ORNL, and blue/green elements will be performed at both laboratories. The preferred approach will be to divide compacts from a single capsule for experiments at both laboratories (including accident testing, irradiated microsphere gamma analysis, and compact/particle microanalyses), to provide good verification of uniformity for experimental methods at the different facilities and to avoid a systematic experimental bias at either laboratory from affecting the results from an entire capsule.

Selected compacts can be screened for failures by short-duration heating tests (see Section 6.15 for details). This could include compacts identified as potentially having failed coating layers during gamma scanning of the empty graphite fuel holders. The program may then choose to focus on compacts that exhibit significant failures for subsequent analysis in order to locate failed particles and perform microstructural characterization. The probability of locating failed particles for microanalyses will depend on the observed failure fractions and the number of particles analyzed with IMGA.

Compacts not utilized for PIE or safety testing experiments before the beginning of PIE on the AGR-2 irradiation experiment will be archived in dedicated storage bins in the HFEF Main Cell. These will be available for later examination by the program if desired, including additional comparative tests between AGR-1 compacts and fuel from subsequent irradiation experiments.

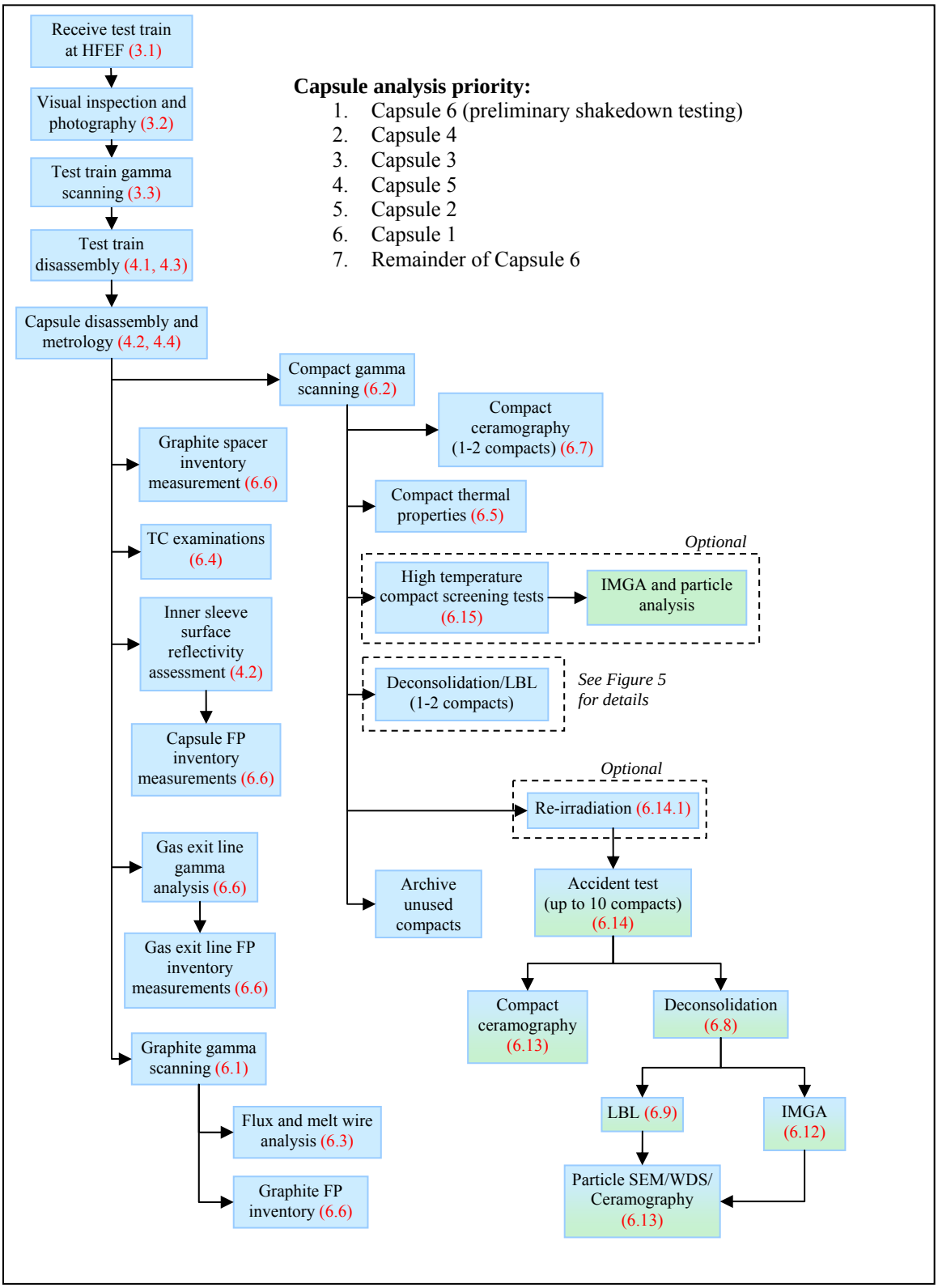

Figure 4. AGR-1 PIE flow chart.

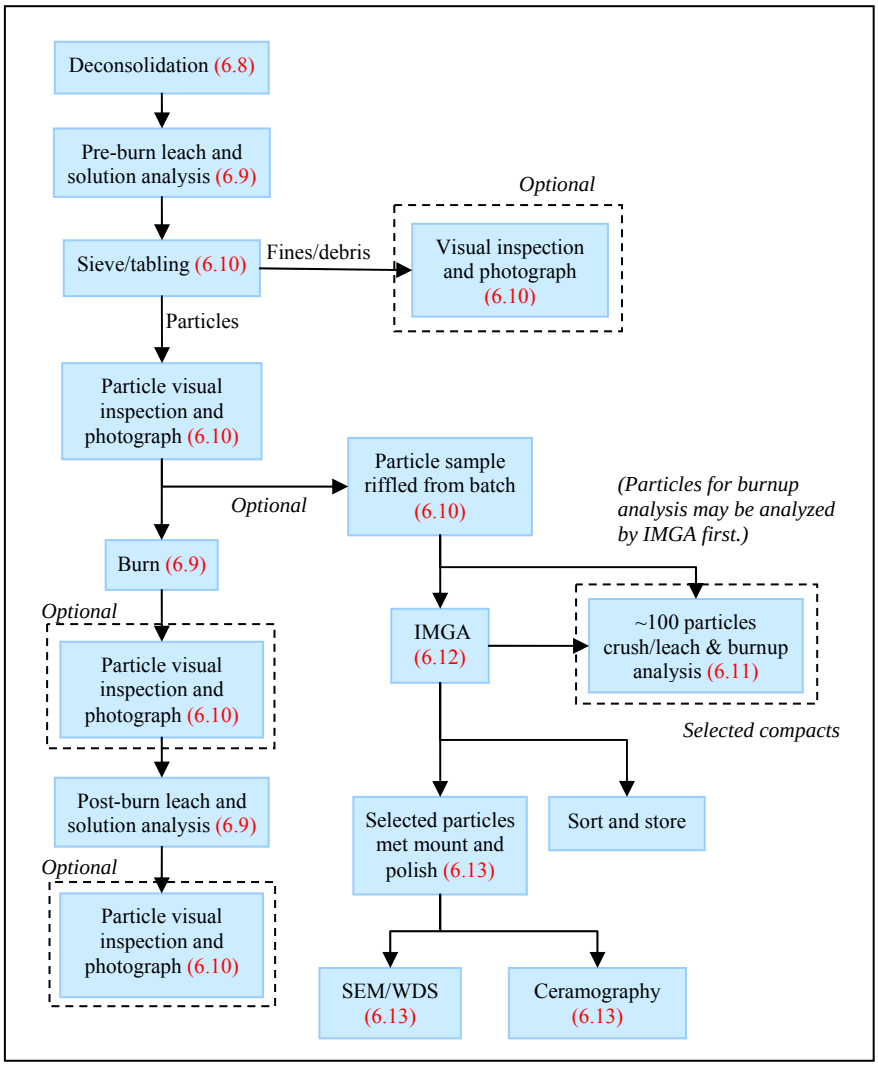

Figure 5. Flow chart for leach-burn-leach tests and particle analyses.

3. TEST TRAIN RECEIPT AND INSPECTION

3.1 Cask Transfer from ATR to HFEF

After removal from ATR and a decay time in the reactor water canal of approximately 4 months, the test train will be loaded into a GE-2000 shipping cask for transfer to the HFEF. Shipping preparations for loading and unloading the GE-2000 cask are discussed below. Since the GE-2000 cask cavity is only 54 inches deep, the test train will be shortened and two separate sections (the fueled lower portion and an approximately 1 meter section of the test train "leadout") will be transported to HFEF. The logistics of shortening the test train require cutting it twice.

The first cut will facilitate transporting the test train into the shielded ATR Dry Transfer Cubicle (DTC; a shielded hot cell with remote handling capabilities) while keeping the experiment dry and operators shielded. ATR personnel will manually cut off the top curved portion of the test train lead-out tubing while the fueled portion of the experiment is maintained underwater in the canal for shielding purposes. Care will be taken to keep the cut portion of the lead-out well above the canal water level to prevent water from entering the experiment during this handling activity. This cutting operation will configure the test train to fit into the ATR facility cask, which is specifically designed to mate to the DTC. After the first cutting, the test train will be loaded into the ATR facility cask and lowered from the cask into the DTC by opening the bottom door of the cask.

The second cut, made within the DTC, will size the fueled portion of the test train to fit within a shielded liner designed to fit into the shipping cask and shield operators from excessive radiation exposure during dry cask loading operations. The fueled portion will be placed inside the liner, and a lid will be secured on the liner. ATR personnel will then push the shielded liner containing the test train out of the DTC and dry load the shipping cask. The cask will then be transported by truck to the HFEF hot cells. Approximately 1 meter of the capsule lead-out, which includes the gas lines and thermocouple leads above Capsule 6, will be retained for a separate shipment to MFC using similar equipment and procedures.

HFEF routinely receives the GE-2000 shipping cask, so standard procedures will be used to mate the cask to the hot cell and open the cask. The test train will be removed from the cask and transferred to a shielded window location within the HFEF where the test train will be externally inspected and disassembled for PIE.

3.2 Photo-visual Inspection of Test Train

After unloading from the shipping cask, the exterior of the intact test train will be photo-visually inspected to identify any significant damage or degradation. The entire test train will be inspected and photographed in segments at a macroscopic scale (approximately 6-inch field of view). Fine features of interest, such as the weld seams, will be photo-visually inspected with an approximately 3-inch field of view. A high-resolution digital camera will be used to enable resolving features as small as approximately 250 μm. A procedural checklist will be employed to ensure that all important features are examined, and all significant observations will be entered in an electronic inspection log sheet as permanent records to accompany digital photographs.

3.3 Gamma Scanning of Test Train

The intact test train will be examined by precision (isotopic) gamma scanning for information on both the migration of fission products and the shifting of fuel compacts within the capsules. Regions of interest on the vertically oriented test train will be raised in front of the scanner collimator slit in vertical increments equal to the adjustable slit height. Scans adjacent to the nominal fueled regions for

nonmigratory fission products will indicate whether deterioration of graphite spacers allowed fuel compacts to shift axially. Scans outside the nominal fueled regions will be performed to determine which fission products (if any) have relocated into the gas exit lines and the regions between the upper head and tops of the fuel stacks in each capsule. Potentially migratory long-lived fission products that may be monitored include Ag-110m, Cs-134, Cs-137, and Eu-154. The ability to detect these fission products may be limited by competition with the Compton scattering from the capsule material. The major goal of this task is to provide an early indication of the release and migration of metallic fission products, if any, so that capsule testing priorities may be established.

4. DISASSEMBLY, INSPECTION, AND DIMENSIONAL MEASUREMENT

4.1 Test Train Disassembly

The test train will be disassembled by separating each capsule from the test train and then opening each capsule separately. The capsules will be separated by making circumferential cuts at the weld joint locations where the capsules were joined. The separated capsules will then be intact much as they were prior to the final assembly process to join the capsules when originally building the test assembly. A single AGR-1 capsule is shown in Figure 6.

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 is necessary because the gas lines and TCs of each of the lower capsules are routed in the through-tubes of capsules above it in the test train. Thus, the top capsule must be cut first to allow pulling each capsule free of the gas lines and TCs coming from the capsules below it.

Figure 6. A single assembled AGR-1 capsule.

Figure 7 shows where the cut will be made to separate the capsules from each other and where the cut will be made to remove the capsule head, thereby providing access to the capsule contents. These selected cutting locations are where the capsule head was welded to the capsule body and where the capsules were welded together to form the test train. Cutting in these locations eliminates the risk of cutting into the test components within the capsule since the capsule head serves as a “backing ring” for both welds. This backing ring will also prevent the pressure from the tubing cutter from leaving a lip on the inside diameter of the capsule body that could prevent the graphite holder from sliding out. The circumferential cuts will be made using a commercial-grade tubing cutter that has been modified for remote handling operation (depicted conceptually in Figure 8). The tubing cutter uses a lathe-type bit designed to leave any lip or burr on the capsule head portion of the cut, rather than on the capsule body.

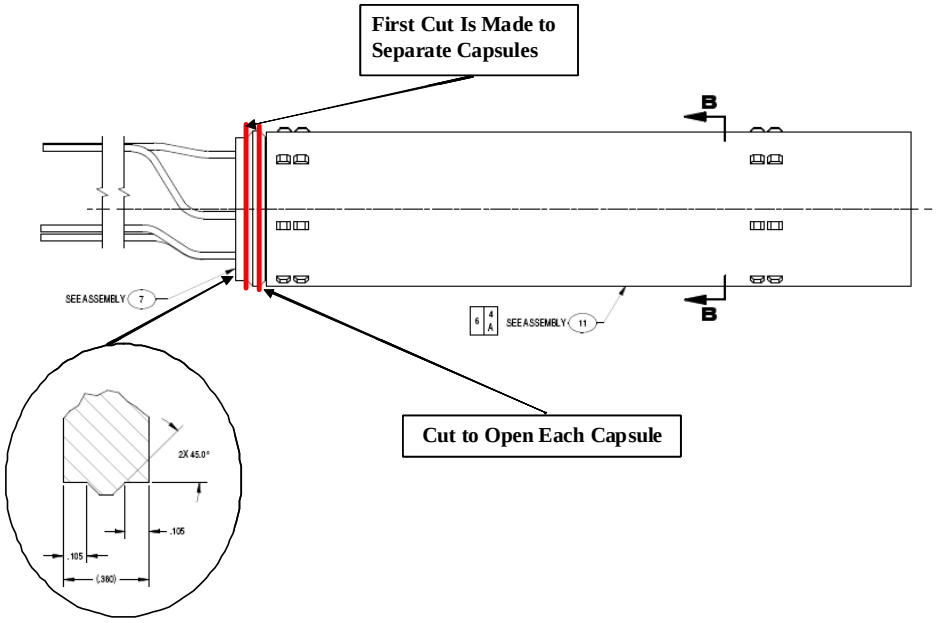

Figure 7. Sketch of capsule showing locations for circumferential cuts.

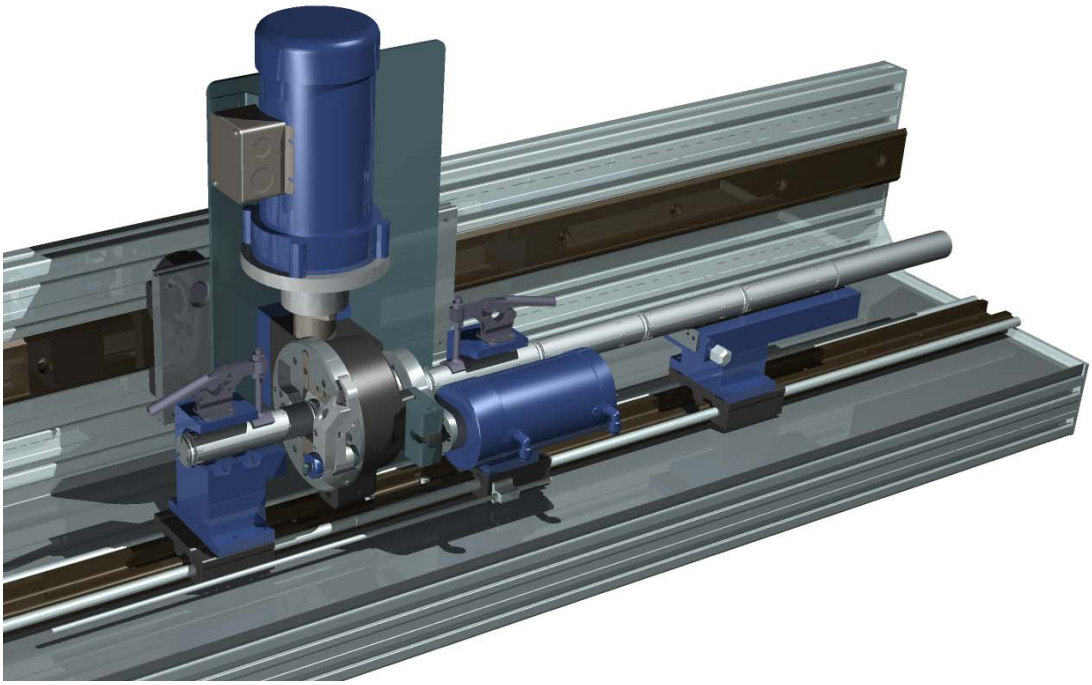

Figure 8. Portable tubing cutter concept for disassembling the AGR-1 test train.

Each capsule will be examined as it is separated from the test train. Along with outer capsule regions, exposed metallic capsule components (top and bottom caps, gas lines, and braze joints) will be photo-visually inspected at a macroscopic scale to identify any degradation such as evidence of chemical reactions between components, cracking, or failure of the braze joints.

4.2 Capsule Disassembly

Following outer capsule inspection and prior to cutting the capsule head, the following cuts will be made:

- TC leads above the capsule head (these will vary in length depending on the capsule; lower capsules will have longer leads) will be cut and discarded
- The exhaust gas lines from each capsule will be cut, labeled, and saved in designated containers for potential leaching activities to assess fission product migration behavior.

Hot cell personnel will use the tubing cutter to cut the capsule heads at the location shown in Figure 7, allowing access to internal capsule components. Special tools, which move on slide rails shown conceptually in Figure 9, will be used to handle the capsule components after the cuts have been made. These tools have been custom designed to handle and remove the components.

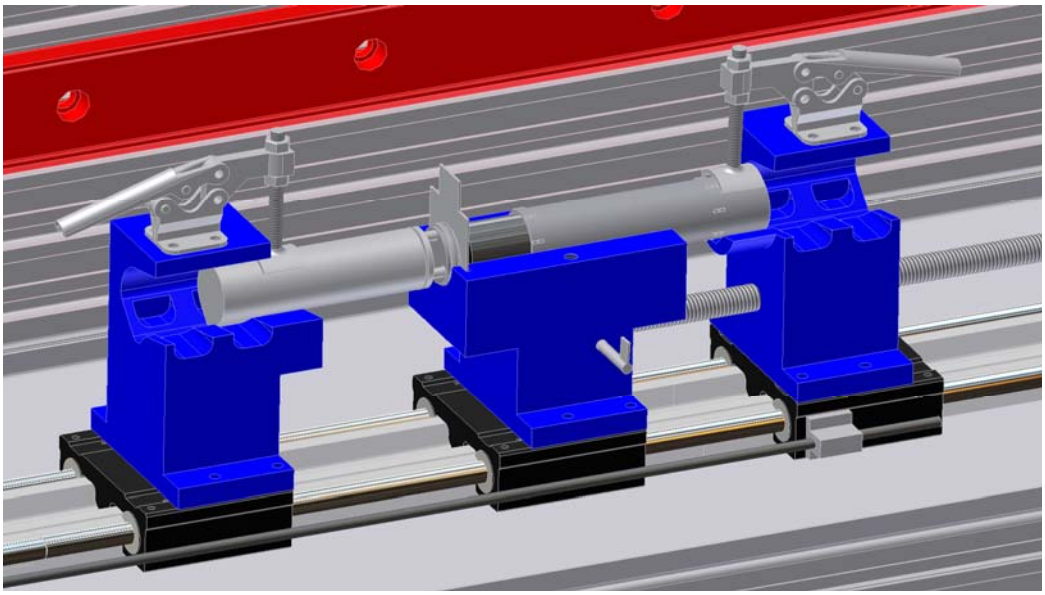

Figure 9. Conceptual drawing of capsule disassembly tools for removing the capsule shell.

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 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. A force gauge will be used to measure any force applied to push the compacts out of the graphite holder. A tentative limit of 10 lb_f for a single compact stack has been estalished in order to avoid damage to the compacts. If the compacts do not come out with this force, alternative tools will be used to score and “crack” the graphite holder to free the compacts.

Efforts will be made to minimize contamination of the capsule components from radionuclides present in the HFEF hot cell. To the extent possible, components will be measured, inspected, and

photographed concurrent with the disassembly activities to minimize the handling and sample contamination potential.

The tools will be used to remove the following capsule components:

1. Capsule body shell (1)
2. Capsule head with through tubes (1)
3. Graphite fuel holder (1)
4. Fuel compacts (12)
5. TC ends inside the graphite fuel holder (2–5 per capsule)
6. Melt wire packages (1)
7. Flux wire packages (3)
8. Gas exit line (1)
9. Graphite and Grafoil® spacers, top and bottom (4 total).

Capsule disassembly operations will be documented by digital photography and videography. Entire components will be inspected and photographed at a macroscopic scale, while fine features of interest such as cracks and corroded areas will be photo-visually inspected at a close scale. Components will be rotated as necessary to document all exterior surfaces. A high-resolution digital camera will be used to resolve features as small as approximately 250 μm. These high-resolution close-up images will facilitate selecting any regions for subsequent microscopic examinations. During component inspections, particular attention will be paid to crack formation, spallation, delamination, carbide formation, abrasion, and any other anomalous behavior. The interior surface of each capsule's stainless steel sleeve will be examined for any discoloration. Graphite holders will be inspected upon extraction from each capsule and after removal of the upper head assembly and fuel compacts to document any incremental damage during these separation steps.

All removed capsule components will be labeled and cataloged to preserve the identity of the component and the location within the test train from which the component was removed. The capsule number, level, and stack numbers will be recorded for each fuel compact so that it can be cross referenced to the originally assigned ORNL identification number.

Each fuel compact will be placed in a labeled, preweighed container. To the extent practicable, any loose fragments and fines 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, preweighed container after separation of the upper head assembly and after unloading all compacts. The loaded holder container will then be weighed to the nearest milligram.

4.3 Leadout Disassembly

As described in Section 3.1, the 1-meter-long section of the capsule lead-out that was immediately above Capsule 6 will be received separately from the lower test train portion. Since both ends of the leadout were cut with a guillotine-type cutter, the ends will be severely crimped, which will not allow easy removal of the gas lines and thermocouples. A pipe cutter will be used to remotely cut the leadout near the middle of the section, allowing the removal of both ends of the leadout tube from the gas lines and thermocouple sections. The gas lines will be identified by the fact that each has a Swagelok tube coupling. The leadout gas lines were not marked during the pre-irradiation test train assembly activities,

so identifying gas lines from a specific capsule will not be possible. The gas lines will be gamma scanned to determine if activity is present. Gas lines with measurable activity will be assumed to be exhaust lines and will be kept for leach analysis to give information concerning fission product transport. The lines that do not have activity will be discarded, along with the thermocouple lengths removed from the leadout section. The retained lines will be coiled with the tool (shown in Figure 10) so they will fit in the pneumatic transfer rabbit used to transfer samples to the Analytical Laboratory.

Figure 10. Tool used to coil gas lines for pneumatic transfer to the Analytical Laboratory.

4.4 Dimensional Metrology of Internal Components

Low uncertainty dimensional measurements will be made during PIE on the length and diameter of fuel compacts and graphite holders, the diameter of the three holes in each holder after removal of compacts, and the inner diameter of each stainless steel capsule sleeve. Irradiation-induced length and diameter changes on carbonaceous fuel compacts and graphite holders will be used to validate assumptions on these materials used in computer models. Dimensional changes are also important for assessing radial heat transfer between fuel compacts and graphite holders and between graphite holders and stainless steel capsules. Measurements must be made with a combined uncertainty no larger than ±0.001 inch (±25 μm) and with a resolution no larger than 0.0005 inch (12.5 μm) to reliably quantify dimensional changes as small as those observed during past PIE of similar components from comparable high temperature, gas reactor-related irradiation experiments. At a minimum, diameter measurements will be taken at top, middle, and bottom elevations of each component. Exterior diameter measurements will also be made at three azimuthal orientations to enable assessments of ovality.

Experience indicates that AGR-1 fuel compacts could be very fragile, and the mechanical strength of AGR-1 graphite holders after irradiation is speculative due to the thin ribs with this design. Because the possibility of damage from contact probes during PIE cannot be discounted, a non-contact metrology method will be used wherever practicable. Lengths and exterior diameters of fuel compacts and graphite holders will be obtained with a custom vision measurement system shown in Figure 11. This system features a shielded digital camera (6.6 megapixels) and a telecentric lens for producing high-resolution

images with virtually no distortion, plus measurement software proven for image analysis on cylindrical objects. Images produced by this system also will be used as inspection photographs where appropriate.

Inner diameters of graphite holder holes and stainless steel capsule shells will be measured by commercial bore gauges. Both gauges use 3-point probes that can be retracted by master-slave manipulators using custom fixtures which also maintain probe shaft alignment with hole centerlines. Diameter values from conventional dial indicators will be read through the hot cell window. Fiducial marks on the extension shafts will indicate the depth inside the components at which diameters are measured.

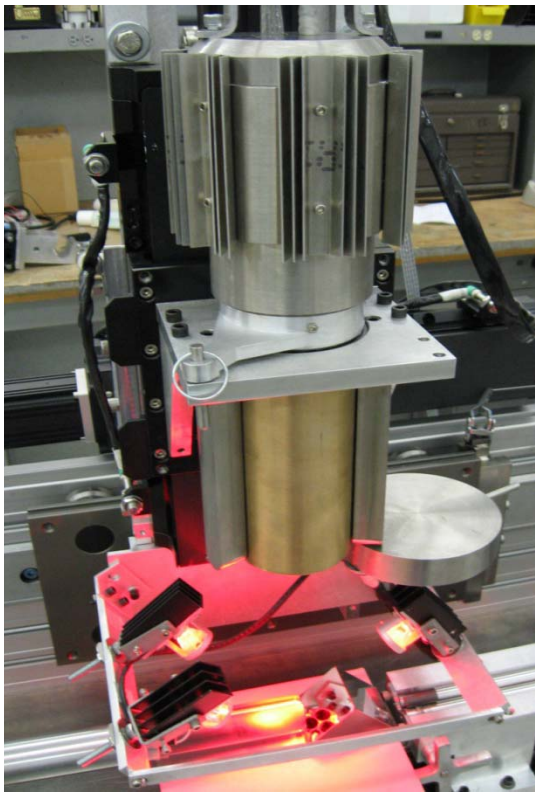

Figure 11. Shielded metrology camera and lens mounted on vertical and horizontal stages, with light-emitting diode illumination.

5. SHIPPING COMPACTS TO ORNL

During the course of the AGR-1 PIE campaign, selected compacts will be shipped to ORNL for parallel PIE work. The shipments will be made in a Type B cask licensed by the Nuclear Regulatory Commission or the Department of Energy. All shipments will be made in full compliance with U.S. Department of Transportation shipping regulations found in 49 CFR 173 Subpart 1, "Shippers - General Requirements for Shipments and Packaging for Class 7 (Radioactive) Materials," and 10 CFR 71, "Packaging and Transportation of Radioactive Material." Because the compacts contain accountable nuclear material, the shipments will be coordinated between INL and ORNL by the Safeguards and Accountability organizations of both laboratories.

Details will be coordinated with ORNL prior to these shipments, and the specific scheduling will be determined as PIE proceeds. It is expected that multiple casks that can contain one or more irradiated compacts will be available, allowing shipment of compacts in small batches. The initial shipment will contain compacts from Capsule 6 to be used at ORNL for PIE process shake-down testing in parallel with work at INL. After this, compacts from each AGR-1 capsule will be sent to ORNL in separate shipments after they are removed during capsule disassembly and initial inspection and measurements are completed.

6. PIE ACTIVITIES

6.1 Gamma Scanning of Graphite Holders

Each empty graphite holder will be scanned for fission products that may have escaped fuel particles and collected in the graphite. Locally high fission product concentrations in graphite holders will be mapped in an attempt to identify which compact released them for input to destructive compact PIE. Potentially migratory long-lived fission products that may be detected by the HFEF Precision Gamma Scanner (PGS) include Ag-110m, Cs-134, Cs-137, and Eu-154. Gamma scanning will be performed before extracting flux wires and melt wires to maintain holder integrity, but no interferences from neutron activation products are anticipated.

The strategy that has been developed to map fission product "hot spots" is to first rotate each holder in front of a vertically oriented PGS collimator at the four heights corresponding to compact rows for a relative determination of the hottest elevation. This elevation then will be scanned in a triangular grid pattern consisting of 12 laterally adjacent scans with a 0.1 inch (2.5 mm) collimator slit at each of three rotational orientations. Counts for each isotope of interest from the three scans that intersect at each grid triangle will be multiplied for enhanced contrast. Colors will be semi-quantitatively assigned to the multiplied scan counts based on results from Ag-110m, Cs-137, and Eu-154 calibration sources inserted in a surrogate holder (forming a standard that also will confirm spatial resolution). Special software has been developed for this purpose, and an example of the output is shown in Figure 12.

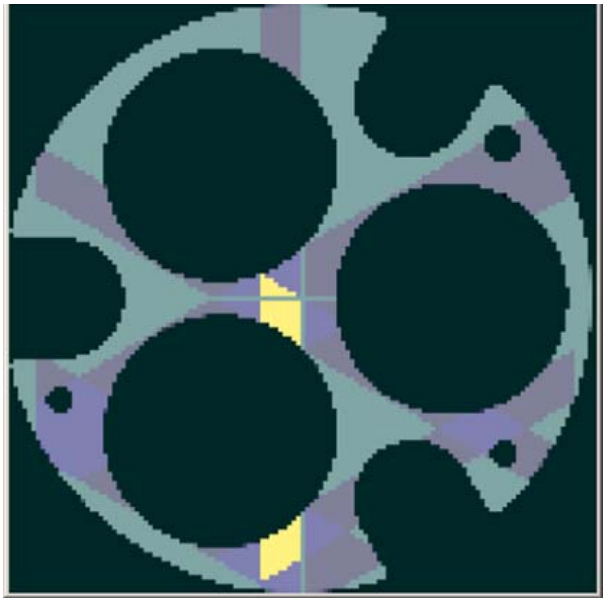

Figure 12. Graphite holder cross section with two simulated fission product hot spots.

6.2 Gamma Scanning of Compacts

The irradiated AGR-1 compacts will be characterized with gamma spectroscopy to determine inventories of key fission products (e.g. Ag-110m, Cs-137) and to measure burnup by taking the ratio of specific fission products such as Cs-137/Cs-134 (see Section 6.11 for further detail on burnup determinations). The spectrometer to be used for this activity uses a high-purity germanium (HPGe) detector and has a 7/8 inch wide collimator with an adjustable slit width of 0 to 0.1 inch. Compacts that will undergo high temperature accident testing will be gamma scanned along the entire axial length so

that the entire compact volume is counted. The step size will depend on the collimator slit width; the preliminary plan is to use a slit width of 0.1 inch. This data will be used to determine whole-compact fission product inventories as well as axial burnup profiles. In order to reduce the duration of the time-intensive gamma scans, the remaining compacts may be gamma scanned only at selected axial locations (without obtaining complete axial coverage) to collect data for burnup calculations for comparison with destructive burnup measurements and to support verification of as-run code calculations. The compacts will be gamma scanned inside of their aluminum storage containers. This activity will also include gamma counting the non-fueled end caps on selected compacts to provide information on the inventory of fission products in the compact matrix.

6.3 Melt and Flux Wire Analysis

To determine neutron fluence and monitor temperatures achieved during AGR-1 irradiation, each graphite fuel holder in the six test assemblies in the AGR-1 test train is instrumented with flux and melt wire packages. Each fuel holder contains three flux wires and one melt wire, for a total of 18 flux wires and six melt wires for the test train. The flux and melt wire packages will be removed from the graphite fuel holders and packaged to preserve their condition, identity, and purity as described below. The flux wire packages will be analyzed for a determination of neutron fluence by gamma spectroscopy at Pacific Northwest National Laboratory (PNNL). The melt wires will be inspected for evidence of melting of the beryllium indicator wires at INL.

6.3.1 Description of the Flux and Melt Wire Packages

Each graphite fuel holder contains a cobalt-vanadium (1% Co-V), an iron (Fe), and a niobium (Nb) flux wire package, and one beryllium (Be) melt wire package containing two Be wires. All the flux and melt wires are encapsulated in sealed vanadium tubes that are nominally 5 to 9 mm long, depending on wire type. One each of the Co-V, Fe, and Nb flux wire packages are embedded at the periphery, and the one melt wire package is in the radial center of the graphite fuel holder. All four packages were inserted into axial mounting holes drilled from the bottom of the fuel holder.

The neutron fluence at each graphite fuel holder will be determined from the activity of the flux wires. The pertinent nuclear reactions and the characteristic emissions for each flux wire type are given in Table 6. The activity of the flux wires will be determined by direct gamma counting, without opening the packages. Of the two Nb reactions, the Nb-93m is of greater interest because it yields the fluence of neutrons with >180-keV energy. Because Nb-93m decays by emission of a relatively low energy gamma, its measurement will require dissolution of the Nb flux wire and scintillation counting of the resultant solution. The vanadium components do not contribute significantly to the activity of the packages, for the vanadium activation products have relatively short half-lives—generally minutes or less.

Table 6. Characteristic nuclear reactions and gamma emissions for the flux wires.

Flux wire	Nuclear reaction	Threshold	Product half-life	Gamma emission, MeV (yield)
1% Co-V	Co-59 (n,gamma) Co-60	thermal	5.27 y	1.173 (100%) 1.332 (100%)
Fe	Fe-54 (n,proton) Mn-54	1.0 MeV	312 d	0.835 (100%)
Nb	Nb-93 (n,gamma) Nb-94		2×10^4 y	0.702 (100%) 0.871 (100%)
	Nb-93 (n, n') Nb-93m	0.18 MeV	16.1 y	.031 (100%)

6.3.2 Retrieval of Flux Wire and Melt Wire Packages

After dimensional measurement and gamma spectrometry (described in Section 6.1) of the graphite holder is completed, the flux and melt wire packages will be retrieved from the graphite holder. A picture of a flux wire package being inserted during the capsule assembly process is shown in Figure 13. Melt wire packages are similar in appearance to the flux wire packages. The packages must be retrieved whole and without loss of integrity to ensure that none of the irradiated material is lost or contaminated. It is anticipated that retrieval of the melt wire packages will be more difficult than retrieval of the flux wire packages due largely to the snug fit of the melt wire capsules in the mounting holes.

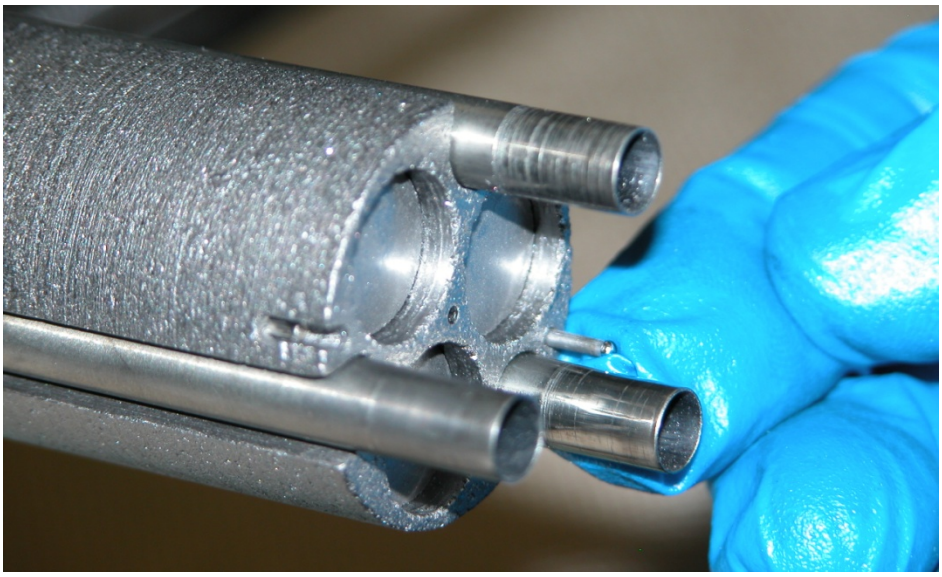

Figure 13. Flux wire package being inserted during capsule assembly activities.

The first attempt to remove the packages will be done by gently tapping the graphite fuel holder to dislodge the packages, exposing them sufficiently for extraction. If they will not come out, the graphite holder will be cut to facilitate retrieval of the flux and melt wire packages. A core drill fixture has been designed to facilitate extraction of the flux and melt wire packages.

Some protective graphite can be left around the packages since it will not interfere with the analysis of either the flux wire or melt wire packages. These operations will be documented by digital photography. Each graphite holder will be photo-visually examined after flux and melt wire extraction for permanent records of any holder damage during the extraction process. Exterior surfaces of the vanadium containers will be inspected and documented at a close-up scale to record any evidence of vanadium-carbon reaction.

After removal, all three flux wire pacakges from a capsule will be placed in a single labeled, radiologically clean container while the melt wire package from each capsule will be sealed in a separate labeled, radiologically clean vial to prevent loss of material and to minimize contamination. If the retrieved packages are embedded in graphite, researchers will not be able to read the identification number stamped on each package. Furthermore, there is a small possibility of confusing identification numbers during extraction, due to a redundancy in identifiers discussed in Appendix A. However, each of the three different flux wires can be identified based on its unique gamma emissions during analysis, so it is not necessary to identify the individual flux wires from a capsule.

6.3.3 Analysis of the Flux and Melt Wire Packages

The analysis of the flux wire packages requires the gamma counting of the packages for the Co-60, Mn-54, and Nb-94 content, and scintillation counting for the Nb-93m content of the irradiated packages, as indicated in Table 4 above. The neutron fluence values are calculated from the counting data, flux wire mass, the calculated effective neutron absorption cross section, the neutron energy spectrum of the reactor core, and the operating power history of the reactor. The gamma counting is done dry and nondestructively, with no sample preparation other than the proper mounting of the samples to preserve the material and its purity and to provide a fixed sample measurement geometry. The analysis for Nb-93m involves liquid scintillation because of the very low-energy gamma emissions and requires the dissolution of the contents of the Nb package in a scintillation cocktail. For the fluence analyses to be valid, the flux wire packages must be supplied intact, with no loss of contents, and sealed in clean protective vials or counting cards. The packages do not have to be cleaned of adherent graphite or carbide because the carbon will not interfere with the sample preparation or counting processes.

The flux wire packages will be analyzed by PNNL, the fabricator of the flux and melt wire packages. Both laboratories will reduce the counting data to neutron fluence values. INL will provide the neutron energy spectrum and irradiation history for data reduction.

The analysis of the Be melt wires requires the determination of whether or not the two Be wires in the vanadium package have melted. This requires simply the determination of the presence or absence of two free-standing wires in the Be packages. An initial check will be made by cutting open the end of the melt wire packages and determining if intact Be wires drop out. If this is unsuccessful, the opened packages will be analyzed using metallographic sample preparation and analysis methods with back-potting to stabilize the Be wires. This method involves potting the melt wire package (and any adherent graphite) in epoxy, grinding the mount just enough to open the package, then filling the void space in the package by vacuum impregnation of a low viscosity resin (back-potting). The samples will be ground and polished in stepwise increments to expose the condition of the two Be wires inside of the package.

6.4 Thermocouple Analysis

The AGR-1 test train is instrumented with 19 TCs embedded in the graphite fuel holders for monitoring the fuel temperature during the irradiation. The TCs consist of eight Type N and 11 Mo-Nb. Capsules 2 through 5 each contain two Mo-Nb and one Type N TC; Capsule 1 contains one of each type, and Capsule 6 contains two Mo-Nb and three Type N TCs.

6.4.1 Description of the Thermocouples

The Type N TCs were supplied by Idaho Laboratories, Inc. They consist of an Inconel 600 sheath (76 wt% Ni, 15.5% Cr, 8.0% Fe, and 0.5% Mn), MgO insulation, and a nicrosil/nisil junction (14.2 wt% Cr, 1.4% Si, balance Ni; 4.4% Si, 0.1% Mg, balance Ni, respectively). The Inconel sheath has a 0.062-inch outer diameter, and the TC wires are 0.010 inch in diameter. Because of concern over Ni and Fe migration at irradiation temperatures from the Inconel sheath into the graphite of the fuel, plus attack of the SiC coating of the fuel particles, the Type N TCs were housed in a Nb-1% Zr protective sleeve with 0.085-inch outer diameter and 0.010-inch wall thickness. Capsule 6 differs from the other capsules in that the bottom ends (2-foot length) of the Type N TCs are sheathed in Mo and have Al_2O_3 insulation; these three TCs have 0.094-inch outer diameters and are embedded directly in the graphite fuel holder.

The Mo-Nb TCs were fabricated at INL. They consist of an Nb-1.0 wt% Zr sheath, HfO_2 insulation, and a KWMo/Nb-1% Zr junction. (The KWMo consists of molybdenum doped with potassium, tungsten, and silicon.) The Nb-Zr sheath has a 0.062-inch outer diameter, and the TC wires are 0.010 inch in diameter. The Mo-Nb TCs were seated directly in the graphite fuel holders without a protective sleeve.

6.4.2 Thermocouple Examination

As of October 2009, all eight TCs in Capsules 1, 2, and 3 had failed. In addition, one of the TCs in Capsule 4 had failed and one of the Mo-Nb TCs in Capsule 5 broke during assembly of the test train. The ten failed TCs have exhibited a range of behaviors indicating open circuits, virtual junctions, and performance changes that could be due to flaws induced by their fabrication (e.g., changes in resistivity, possibly due to brazing).

The primary objective of the TC analysis is to identify chemical interactions between the various components—in particular the outer sheath/sleeve and the graphite, which could have implications for fuel coating interactions. Several candidate TCs from the test train will be chosen for characterization at the end of the irradiation. This should also include an analysis of the graphite holder in the vicinity of the TC well. The leading candidate TCs for analysis would be those in the hottest capsules (Capsules 3 and 4), since these would represent the most severe in-service conditions. This analysis is a relatively low priority for the AGR-1 PIE and will be pursued on a limited basis as permitted by schedule and budget.

Optical metallography of TC and sheath/sleeve cross-sections will be used to look for evidence of any chemical reaction, such as the formation of carbides and intermetallic compounds. The sections will be potted, ground, polished, and the microstructure examined by optical microscopy. The grinding phase may require the physical stabilization of the TC components in the sample by back-potting with a fluid resin to fill the voids and stabilize the insulation and wires. If warranted, the samples may also be analyzed by scanning electron microscope (SEM) and energy-dispersive x-ray spectroscopy (EDS) or wavelength dispersive x-ray spectroscopy (WDS) to acquire elemental maps of features of interest, to identify reaction products, and to determine the reaction mechanisms responsible.

6.5 Thermal Conductivity

The bulk thermal conductivity of selected fuel compacts will be measured. Thermal conductivity data will be acquired by measuring the axial temperature distribution across a thermally insulated specimen that is heated on one end. Data across a temperature range of approximately 25–800°C will be examined, depending on the capabilities of custom measurement equipment still being developed. These data will be used to refine test train thermal analyses for AGR-1 and future irradiations.

6.6 Capsule Deposited Fission Products

After each capsule has been disassembled, the interior metal surfaces will be analyzed for the presence of fission products such as Ag, Cs, Eu, I, and Te that may be released from the fuel during irradiation. This analysis will include the stainless steel capsule body, the stainless steel sleeve inside each capsule, the molybdenum through-tubes, steel upper end caps, and the gas exit lines. The analysis will provide information on the quantities of released fission products in each capsule and the extent of migration outside of the compacts and graphite holders. The data will help to determine the fractional release of fission products from the fuel during irradiation.

Selected empty capsules and through-tube assemblies (including upper end caps) will be immersed in an acid solution to leach off deposited fission products. The leach solutions will receive a radioassay to establish the concentrations and absolute amounts of detected radionuclides. Results will be compared to calculated, decay-corrected inventories to determine the fractional inventories deposited. Results also will be compared among capsules for any inferences on performance of the various fuel types in the AGR-1 test train. Additional information will be obtained on effects of differences in burnup and temperature (especially between Capsules 1 and 6 and the remaining higher temperature, higher fluence capsules).

Limited investigations will be conducted on radionuclide deposition in gas exit lines. Gross and/or isotopic gamma scans will identify the region(s) in which detected fission products deposited while in the

ATR. Depending on the results, an acid flush may be performed to leach out the deposited fission products. Radioassay of the leach solution will determine the approximate fractions of decay-corrected inventories present.

After qualitative mapping on the PGS (Section 6.1), the graphite holders (along with their adjacent graphite spacers and Graphoil disks) will be transported to the MFC Analytical Laboratory in clean containers for quantitative counting. Isotopes of particular interest are Ag-110m, Cs-134, Cs-137, and Eu-154. The spectrometer used will be calibrated with a Eu-152 source, which has well-established gamma peaks across the energy range of interest. Here an entire holder can be counted at once due to the absence of a narrow collimator. However, calculations indicate that the 1% Co-V flux wire and a Nb thermocouple sleeve will present too much gamma activity from neutron activation to be safely handled. Consequently, both of these components will be extracted (with a special coring tool if necessary) before the holders leave HFEF (see Section 6.3.2 on flux wire extraction). This data together with the results from metal capsule component analysis will provide the fractional release of fission products from the fuel during irradiation.

6.7 Micro-scale Analyses of Fuel Compacts

After irradiation and accident testing, selected compacts will be analyzed in cross-section at the microscopic scale to assess localized effects of ATR irradiation on the compact matrix and embedded fuel particles. Primary features for investigation include cracks in the compact matrix, fuel kernel porosity and migration, buffer layer degradation, corrosion of the SiC layer by fission products, fractures in the tri-isotropic (TRISO) coating layers and delaminations between them, and deleterious interactions between the carbonaceous matrix and the outer pyrolytic carbon layer. Migration of fission products within particles and from kernels into the matrix will also be examined where practical. Selected compacts will be sectioned axially and/or radially, mounted, and polished. 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), so analytical instruments must be heavily shielded to accommodate them.

Some fuel compacts will be sectioned for micro-scale analyses after accident testing. The defect types to be investigated are the same as those before accident testing, although their frequency and severity are expected to worsen appreciably at accident testing temperatures.

Compact cross sections can be reground and repolished to expose multiple planes for examination when appropriate. Shallow regrinds can provide three-dimensional information on effects within individual particles or their immediate matrix surroundings. Deeper regrinds can expose new particles plus facilitate surveying effects of interest in different portions of a compact. Considerable care is required to prevent dislodging loose kernels or coating fragments in the process, which could scratch mount surfaces and force extensive rework.

Most key features anticipated in compact cross sections can be investigated by ceramography on shielded metallographs, typically at magnifications from 20X to 1000X. Microstructures are often enhanced by proper choice of illumination (bright field, dark field, polarized light, etc.). Digital images can be processed by computer software for aspects such as porosity, nodularity, and particular colors or gray levels. These versatile capabilities are expected to expedite detection and characterization of some features of interest across entire compact cross sections.

Once cross sections have been surveyed by ceramography, specific areas can be investigated further by additional techniques. A SEM can provide images at much higher magnifications than possible on an optical metallograph. EDS can identify the primary elemental constituents (heavier elements only on some instruments), measure concentrations above ~0.5% (depending on radiation background), and

produce qualitative concentration maps. WDS—also performed using a SEM—improves elemental detection thresholds by at least a factor of 10 and provides information on lighter elements (excepting hydrogen and helium).

6.8 Compact Deconsolidation

The deconsolidation of compacts is a treatment process whose purpose is to free individual fuel particles from the matrix binder by disintegrating the carbonaceous matrix of compacts. The deconsolidation process is used to provide loose fuel particles for other PIE tasks such as IMGA, particle microanalyses, and burnup measurements. It is also used as a first step in the LBL process to improve the efficiency of the first leach step.

Therefore, deconsolidation will be used prior to leach-burn-leach (LBL) determinations of fuel failure, destructive analysis of burnup, IMGA, and the SEM analyses to support the evaluation of coating failures and microstructural changes in fuel particles following irradiation and following the high temperature accident tests. Initially one compact from Capsules 1–5 and two compacts from Capsule 6 will be deconsolidated as part of the LBL screening analysis (Section 6.9). In addition, selected compacts will be deconsolidated following accident tests to support LBL, particle inspections, and/or IMGA. For certain analyses (e.g., burnup measurement, Section 6.11) it will be important to perform a stepwise deconsolidation to obtain particles from a specific axial segment instead of the entire compact. In these cases, half-compact segments will be deconsolidated, the process stopped so that the particles can be recovered, and the remaining compact length measured to determine the fraction of the compact that has been deconsolidated.

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 (approximately 15 watts power) is applied between the compact and the cathode, which is suspended in the electrolyte solution.

The processing and analysis of the deconsolidated material depends on the end use. If the purpose of the deconsolidation is to generate loose fuel particles for IMGA, particle analysis, or burnup measurements, the deconsolidated particles and the carbonaceous filler material will be filtered from the electrolyte, inspected, and sieved or sorted for later analysis. The electrolyte solution will be analyzed for fission product content. Depending on the end use, the cleaning process may need to be monitored by visual inspection for the degree of separation of debris and fragments from whole particles. If the deconsolidation is the preparatory step for LBL, the entire mixture (electrolyte and solid residue) is transferred to the LBL system for the first leach.

6.9 Leach-Burn-Leach of Compacts

The purpose of the LBL process for fuel compacts is primarily to determine:

- The inventory of fission products in the compact matrix (determined by analysis of the pre-burn leach solution provided no failed particles are present)
- The number of failed fuel particles with exposed fuel kernels, in which all three coatings have failed (determined by analysis of the pre-burn leach solution)
- The number of fuel particles with a failed SiC layer but intact inner and outer pyrolytic layers (determined by analysis of the post-burn leach solution).

The pre-burn leach solution will contain contributions from uranium contamination in the particle coatings and the resulting fission product contamination and fission products released by diffusion from intact fuel particles. The LBL process cannot distinguish between fission products resulting from these

sources and from exposed kernels of failed fuel particles. However, the inventory of fission products from uranium contamination and the inventory of diffusively released fission products in the compacts after irradiation are expected to be at least an order of magnitude lower than the inventory in one exposed fuel kernel, with the possible exception of silver[b].

To efficiently process the fuel compacts, the LBL process will be performed with deconsolidation as the preliminary step after which the pre-burn leach is performed on the mass of deconsolidated particles and carbon debris. The LBL process consists of three steps:

1. An initial acid leach to dissolve the uranium and fission products in exposed kernels (i.e., kernel with all three coating layers breached), and potentially remove the fission products in the graphite matrix that are due to uranium contamination outside the SiC layer, and the fission products released by diffusion from intact particles. If there are no failed particles, the fission product inventory should be the sum of contributions from contamination and diffusive release. Since the contamination fraction is known for the fuel (measured prior to the irradiation), this value should give an idea of the inventory of fission products diffusively released from particles and retained in the matrix. 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.
2. The burn step, performed at 750°C in an air furnace, oxidizes the carbon residue and all exposed pyrolytic carbon coatings, including the inner pyrolytic carbon and buffer coatings of particles with a defective SiC coating but 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.
3. The post-burn leach dissolves the fission products and uranium in the exposed fuel kernels, and allows for a calculation of the number of equivalent particles with defective or failed SiC coatings. The post-burn leach is also repeated a second time.

Small samples of particles (approximately 100–300) may be randomly extracted from selected LBL compacts after deconsolidation for separate testing, including destructive burnup measurements (see Section 6.11) and irradiated microsphere gamma analysis (see Section 6.12). In addition, particles may be inspected microscopically at intermediate stages of analysis (after pre-burn leach or burn steps) before completing the process.

In addition to the compacts analyzed by the LBL process following irradiation, selected compacts will be analyzed by LBL after accident testing. Compacts selected for these analyses will be determined based on the specific results of the accident tests and programmatic objectives.

6.10 Particle Inspection and Sorting

Particles for specific analyses will be selected following compact deconsolidation, the first nitric acid leach step, or particle burn-back. If particles are to be selected following deconsolidation or the first leach, they may need to be first separated from the matrix debris. The methods used will depend on the effectiveness of the deconsolidation and leach steps in liberating individual particles from the compact matrix. Wet sieving or simple tabling of dried particles and debris will be used as appropriate.

Particles and debris will then be inspected microscopically with sufficient magnification to identify coating cracks and missing coating layers, inspect loose coating fragments, and provide an overall indication of the condition of the deconsolidated particles and extent of coating damage. During this process, individual particles of particular interest or particle batches can be selected for subsequent

b. Based on previous German data, diffusively released silver retained in the matrix can reach a level that would significantly exceed a fractional value of 2.4×10^{-4}, i.e., the inventory of one AGR-1 particle in a single compact.

analysis, such as detailed SEM/WDS analysis, burnup analysis, or irradiated microsphere gamma analysis. Simple riffling equipment will also be used to subdivide the deconsolidated particles into smaller samples for analysis where it is important to obtain a random sample from a compact (e.g., burnup measurements). Coating fragments can also be selected for further analysis—such as in-depth microstructure studies if desired.

6.11 Burnup Measurements

The burnup of the AGR-1 compacts will be experimentally measured for comparison with calculated values. The burnup measurements will be made by two methods: destructive analysis per American Society for Testing and Materials (ASTM) E 321-96 (or a variant of this method), and nondestructive fission product activity ratio analysis by gamma spectrometry of whole compacts and individual particles.

Because of the expense and complexity of the ASTM method, the destructive analyses will primarily serve as "benchmark" tests to calibrate the cheaper, faster, and nondestructive fission product activity ratio method. The ASTM method will be performed on approximately four compacts selected to span the anticipated burnup range of the fuel in the test train. To establish the proportionality between burnup (as determined by the ASTM method) and the activity ratio, the same samples must be analyzed by gamma spectroscopy prior to the ASTM test. The ASTM method is an internationally accepted method for the absolute determination of fuel burnup and yields the highest accuracy, with typically about 2% relative error. The ASTM method measures the content of Nd-148 and the uranium and plutonium isotopes in the fuel, for the absolute determination of burnup as % FIMA. The method requires the dissolution of the fuel sample, removal of interfering isobaric fission products by ion exchange or ion chromatography, addition of internal calibrants (spikes), and analysis for Nd-148 and the uranium and plutonium isotopes by thermal ionization mass spectrometry (TIMS) or inductively coupled plasma-mass spectroscopy (ICP-MS).

While the standard ASTM method specifies Nd-148 as the preferred monitor radionuclide, other radionuclides can be chosen as the monitor. For the Arbeitsgemeinschaft Versuchsreaktor work, the Germans have used Cs-137 as the monitor radionuclide. While it is highly likely that the AGR-1 PIE will use Nd-148 as the monitor of choice, other radionuclides might be evaluated for suitability.

The second method involves the nondestructive gamma spectroscopy of the fuel compacts and individual particles to derive the Cs-134/Cs-137 activity ratio. Other isotopic activity ratios could be used for the burnup measurement, such as Eu-154/Cs-137, provided that the ratio consists of one isotope that is formed by two neutron events (i.e., a fission and a neutron capture event), and a second isotope that is formed by only one neutron event (i.e., fission). The activity of an isotope formed by two neutron events is proportional to the square of the thermal neutron fluence; the activity of an isotope from a single neutron event is directly proportional to the fluence. Therefore, the activity ratio is approximately linearly proportional to the irradiation neutron fluence, and the burnup of the fuel can be calculated if the thermal neutron fluence is known. The relationship between fluence and burnup is determined from a correlation curve of the activity ratio with the absolute burnup (by the ASTM method) as determined for several fuel compacts that define the expected range of burnups in the test train. Despite the need to validate the activity ratio method with the ASTM method, the nondestructive activity ratio method is advantageous because it is rapid, easy to perform, and preserves the samples for other examinations or archiving. The method does require the correction of the measured activities for isotopic decay, the relative detector efficiency as a function of emission energy, and attenuation as a function of emission energy if significantly different emission energies are used to determine the activity ratio.

Compacts will be gamma counted for burnup measurements following removal from the irradiation capsules as described in Section 6.2. Gamma spectra will be acquired for one or more axial slices of each

compact, and may include complete axial coverage of selected compacts. The activity ratio can then be derived from intensity (area) ratio of two selected emission peaks representing the desired isotopes (e.g., the peak at 662 keV for Cs-137 and 605 keV for Cs-134), after correction for decay time after the end of irradiation, detector efficiency as a function of energy, and, if necessary, self-attenuation.

The ASTM destructive test can be performed on fuel particles taken from compacts at any stage of PIE after unloading the compacts from the graphite fuel holders. However, it is preferred that the ASTM test be performed before the accident tests until the assumptions of immobility of Nd and other monitor radionuclides can be validated; for example, burnup determinations using Cs-137 as the monitor isotope may not be valid if performed after the accident tests where significant cesium has been released from the fuel. Small samples of particles (approximately 60–150, depending on the analysis needs) will be randomly extracted from selected compacts after deconsolidation, and subsets of these samples will be subjected to destructive burnup analysis. Particles for burnup analysis should come from a half-compact region where the capsule axial burnup profile has the least variation, in order to minimize the range of burnups that will be represented by the deconsolidated particle sample (see Section 6.8 for discussion of partial compact deconsoldiation)[c]. The specific compacts will be selected prior to the analysis based on review of the as-run physics calculations for the AGR-1 test train. In addition, it may be desirable to analyze selected particles with IMGA prior to the destructive burnup analysis in order to perform a measurement of burnup using the isotope ratio method on the specific particles that will be crushed for the burnup measurement. This test could also be used to verify that the selected particles have intact kernels prior to burnup analysis.

The selected particles will be crushed and the exposed kernels in the broken particles will be dissolved in hot nitric acid. The resulting solution of fission products and transuranics will be processed per the ASTM procedure and analyzed by TIMS or ICP-MS systems at the Analytical Laboratory. The analysis results may need to be corrected for neutron activation of Nd-147 in the sample and for the fast neutron component of the neutron flux.

6.12 Irradiated Microsphere Gamma Analysis

Individual particles will be gamma counted to quantify the inventories of selected fission products. The data will primarily be used to gauge the relative fission product retention in each of the analyzed particles. The data may also be used to screen particles based on radionuclide inventories prior to performing other analyses, such as destructive burnup measurements (see Section 6.11), to ensure that the kernels have remained intact after compact deconsolidation. This will be accomplished using IMGA, which positions individual particles in front of a gamma spectrometer and counts for a prescribed time period. The specific count time will be influenced by the particular radionuclides that are of interest, the burnup and age of the fuel, and the counting geometry. After gamma counting, the particles will be sorted based on the observed results. As indicated in Figure 4, this procedure will be performed on fuel after irradiation (to examine in-pile fission metals retention) and after accident testing (to examine high temperature fission metals retention). The particles for the post-irradiation IMGA experiments will be obtained from compacts deconsolidated specifically for this purpose or extracted as small samples (e.g., several hundred particles) from the compacts deconsolidated prior to LBL measurements.

Typical use will involve taking the ratio of various fission products of interest to one that is known to be relatively immobile within the kernel (such as Ce-144) to obtain the fraction of the fission product that is retained in the particle. This can provide data on whether a particle contains coating failures (e.g., by observing high Cs release resulting in a low Cs-137/Ce-144 ratio) or whether a particle has abnormally

c. Preliminary as-run physics calculations for the AGR-1 experiment indicate that the variation in burnup across some compacts in the test train can significantly exceed 1% FIMA.

low retention of various fission metals (e.g., by observing high Ag release resulting in a low Ag-110m/Ce-144 ratio).

The radionuclides to be included in the analysis for specific particles will be determined prior to the experiment based on the experimental objectives and data needs. Measurements aimed at determining fission metals release will include Ag-110m, Cs-137, and Eu-154. The number of particles to be analyzed from each compact or capsule will depend on the objectives of the analysis (searching for failed particles where failure fractions are relatively low may require counting several thousand particles) and on the radionuclides to be counted, which will dictate count times. After IMGA, individual particles of interest can be selected as appropriate for various microanalyses such as ceramography or SEM/WDS.

IMGA on relatively small numbers of particles (less than approximately 100 per compact) will be performed at INL using a manual scanning arrangement.This analysis will be focused on examining retention of fission metals in particles with intact coatings (e.g., variations in Ag-110m retention) and can also be used to gamma count particles prior to making destructive burnup measurements. IMGA will also be performed at ORNL with the capability of examining large numbers of particles in automatic mode using the Advanced-IMGA instrument. This can be used either to examine fission metals retention on a few hundred particles from a compact or to sort several thousand particles to find those with failed coatings.

6.13 Micro-scale Analyses of Fuel Particles

Based on the results of several of the preceding analysis steps, individual particles or groups of particles will be placed in shallow trays, potted, ground, and polished for analyses at a microscopic scale. Mounts could contain from one particle to ~500 particles, depending on the dose rate that can be tolerated at a particular analytical instrument. Mounts may also be reground and repolished to investigate multiple planes through the particles in order to extract additional data from the microanalyses. Mounted fuel particles may or may not have been subjected to accident testing (see PIE flow chart in Figure 4 above).

Obtaining high-quality polishing on mounts is essential for refined analyses. Grinding the mounts will involve potting the specimens, rough grinding, repotting to avoid kernel fallout, more grinding, and then polishing. A careful and tedious technique is required because irradiated particles are very friable and a loose kernel or coating fragment will damage a mount by falling out, losing the item of greatest interest or creating a large scratch.

The general objective of these fuel particle analyses is to characterize fuel kernel porosity, kernel migration, buffer layer degradation, fractures in the TRISO coating layers, delaminations between coating layers, and corrosion of the silicon carbide layer by fission products. Migration of fission products from the kernels across the TRISO layers will also be scrutinized. Because these analyses can be performed after IMGA in certain cases, IMGA results can be factored into particle selection. In such cases, one important aspect will be relating IMGA results on release of metallic fission products to deterioration of the SiC layer or the presence of microstructural defects. Furthermore, microstructural features observed during PIE can be compared to pre-irradiation microstructures.

SiC layers also will be examined in particles that did not exhibit substantial releases of fission products in-pile. In particular, corrosion of the SiC layers by fission products such as palladium, resulting in localized layer thinning, will be investigated. Statistically significant data in this regard will be used to optimize fuel performance models.

Many of the features of interest on fuel particles will be studied optically on INL and ORNL metallographs, typically at magnifications from 20X to 1000X. Shielded instruments are available at both laboratories and can accommodate mounts with large numbers of particles. High-resolution digital images will be analyzed by computer software to automate detection and characterization of key particle features

to the extent practicable. After ceramography has screened specific particles for further investigation, mounts can be transferred to other instruments for detailed elemental studies.

A SEM can provide higher resolution, more depth of field, and different contrast compared to optical metallography, and many SEMs are equipped with elemental analysis capabilities—EDS and WDS—to provide information on elemental interactions, especially at particle layer boundaries. Detection thresholds are typically at the level of one atom per thousand, so these instruments can quantify concentrations of the primary elemental constituents and can map relatively abundant fission products. However, most of the SEMs at INL and ORNL will be restricted to studying mounts with only one or two particles to limit operator and instrument dose.

Electron backscatter diffraction will also be used to characterize SiC coatings on selected particles. This technique will provide data on crystallographic orientation of SiC grains and grain boundary alignments in order to aid the interpretation of observed fission product behavior. In particular, the diffusive release of metallic fission products, especially silver, through intact SiC coatings will be related to the crystallographic texture and grain boundary characteristics of the material.

6.14 Accident Testing

6.14.1 Compact Re-irradiation

Selected fuel compacts will be re-irradiated prior to accident testing. The primary objective of this activity will be to generate short-lived I-131 (8-day half-life) in the fuel via fission such that iodine release during accident testing can be measured. Compacts will be irradiated in the core of the Neutron Radiography (NRAD) TRIGA reactor located in the basement of the HFEF facility at MFC. After re-irradiation the compacts will be quickly transferred to the fuel heating furnace installed in the HFEF Main Cell for accident tests. The time in the NRAD reactor core and the reactor power will be recorded such that the I-131 inventory in the fuel can be calculated. Only a selected number of the compacts to undergo accident testing will be re-irradiated prior to the experiments, since this step adds considerable complexity to the tests (both in the re-irradiation stage and in the analysis of I-131 on the condensation plates) and the correlations between the release of iodine and other fission products (xenon, for example) can be adequately established on a subset of accident test samples. The number of compacts that will be reirradiated is still to be determined and will be based on a number of factors, including the documented in-pile fuel performance, the temperatures of the specific accident tests, the demonstrated sensitivity of the experimental methods for measuring iodine on the furnace condensation plates, and the availability of the facilities for performing the re-irradiation. Fuel with higher R/B fractions in-pile and fuel tested at higher temperatures will be more likely to release larger amounts of iodine, which may prove to be more easily quantified.

6.14.2 Heating Tests

Selected fuel compacts will undergo testing to assess the fission product retention characteristics under high temperature accident conditions. Furnaces at INL and ORNL are available for this activity. The facilities to be used for this activity are the Fuel Accident Condition Simulator (FACS) furnace system at INL and the Core Conduction Cooldown Test Facility (CCCTF) at ORNL. The fuel will be heated at temperatures up to 1800°C in a helium atmosphere while measuring fission product releases as a function of time and temperature. Released fission gases (Kr and Xe radioisotopes) will be measured in dedicated cold traps during the heating tests. Condensable fission products, including radioisotopes of Sr, Ag, I, Cs, Eu, and Te, will be collected on water-cooled surfaces that can be periodically exchanged during the tests to obtain time-dependent release values. Note that the short-lived fission products Xe-133 and I-131 will only be measured for compacts that have been re-irradiated prior to furnace testing, and therefore will only be measured at INL.

Fission gases will be continuously monitored throughout the tests using in-line cryogenic traps and detectors. The CCCTF uses NaI detectors to monitor Kr-85 in the cryogenic traps. The INL furnace uses HPGe detectors to monitor Kr-85 (514 keV) and short-lived isotopes resulting from re-irradiation, such as Xe-133 (81 keV).

Condensation plates (FACS) and cups (CCCTF) will be exchanged at regular intervals during testing to get time-dependent condensable fission product release information during the tests. The exchange interval should be determined for each experiment based on the overall test duration. A total of 20 or 30 separate plates per test will provide a reasonable resolution for the release curves.

Both isothermal and nonisothermal heating tests will be performed for each selected fuel type. The majority of the isothermal tests will be performed in the temperature range of 1400–1800°C. Temperature profiles for the isothermal tests will involve the following general steps:

1. Ramp to ~300°C and hold for sufficient time to eliminate adsorbed water from the fuel (typically 5 hours).
2. Ramp to the representative fuel operating temperature (e.g., 1250°C) at a rate of ~3–10°C/min.
3. Hold at operating temperature for several hours to allow thermal equilibrium in the fuel compact.
4. Gradually ramp (<1°C/min) up to the target test temperature. It is expected that input from the reactor design activity, in particular the expected rate of core heating during a depressurized conduction cooldown accident scenario, will be relevant in determining the ideal ramp rates during the final heating step.
5. Hold at target temperature for desired test time, typically 100–500 hours; the hold time will be dependent on the temperature and fuel behavior during the test.

The generic temperature profile for the isothermal heating tests is summarized in Figure 14.

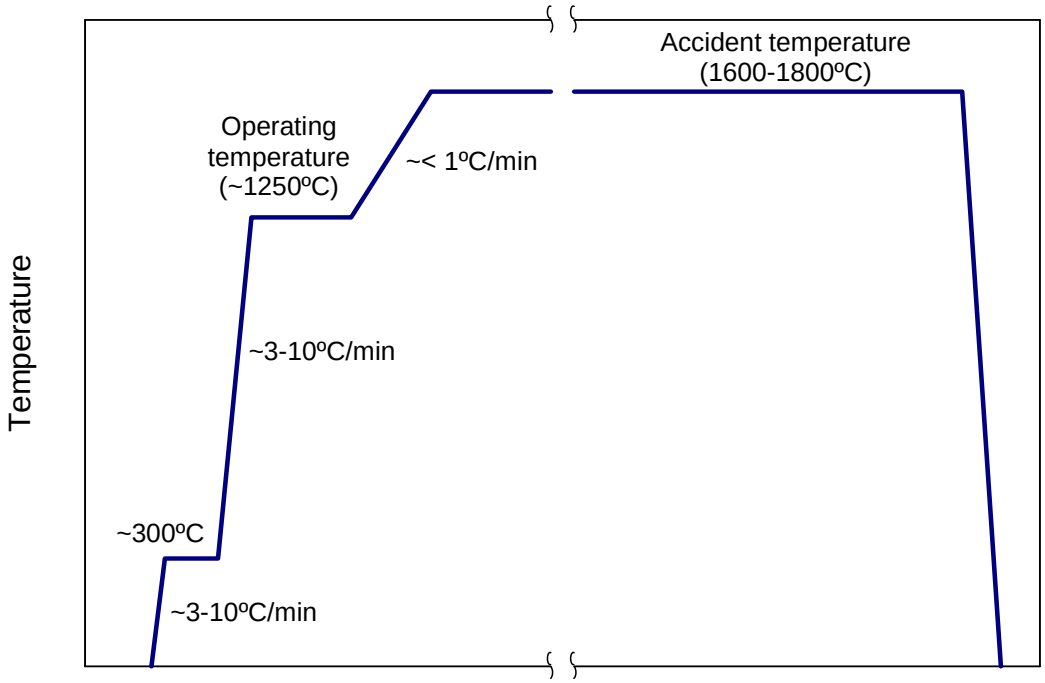

Figure 14. General temperature profile for isothermal heating tests.

Nonisothermal heating tests will be performed to more realistically simulate the peak fuel temperature-time profiles during a postulated accident scenario. The maximum temperature of these tests and specific temperature-time profile are still to be determined and will be established based on input from the NGNP reactor design and safety analysis activities.

Table 7 presents a tentative plan for the specific accident tests planned for each capsule in rough order of priority. This plan will be adjusted if appropriate based on the initial results from heating tests or other PIE data. The duration of the isothermal heating tests will be somewhat dependent on the test conditions and fuel performance during the test. Tentative durations proposed for the high-temperature portion of the test are 200–300 h. The planned test duration may be modified (to either extend or shorten the test) based on real-time data collected from the fission gas monitors.

If available program funding proves to be inadequate to support the required number of tests to complete this matrix for all AGR-1 capsules, it may be possible to run selected compacts in tandem in the same test in order to reduce the number of accident tests but maintain the overall number of particles that are subjected to high temperature tests. Consideration will have to be given to which compacts are combined in a single test, as they may represent different burnups, fast fluences, irradiation temperatures, or even fuel variants, and this will affect interpretation of test results.

Table 7. Tentative accident testing conditions for compacts from each AGR-1 capsule.

Test conditions	Comments
Isothermal test at 1600ºC	Collect initial data at 1600°C. Examine metallic fission products inventory in the matrix to determine if 1400ºC test is warranted.
Isothermal test at 1400–1500ºC	Perform primarily to measure metallic fission products in the compact matrix. Temperature can perhaps be determined based on 1600ºC test results and in-pile compact temperature.
Isothermal test at 1800ºC	Collect data at 1800°C.
Isothermal test at 1700ºC	Collect data at intermediate temperature of 1700°C for comparison with 1600ºC and 1800ºC data and to better understand the effect of accident temperature.
Additional isothermal testing at 1600, 1700, and/or 1800ºC	Test temperatures will be dictated by results from previous tests and the need to collect data for comparing effects of different fuel attributes and accident temperature on performance.
Transient test at 1600–1800°C peak temp	Transient test to simulate actual temperature profile in depressurization accident and address NRC concerns. Maximum temperature is TBD.

It may also be of interest to perform heating tests on individual particles, with gamma analysis of each particle performed (using IMGA, see Section 6.12) both before and after the experiments. This will allow the amount of fission products released from each particle to be independently measured to compare with the fission product releases measured during the accident tests. This data comparison will also provide an additional opportunity to calibrate the furnace collection efficiencies (in addition to the initial system calibrations performed during furnace testing and qualification activities).

6.14.3 Condensation plate/cup Analysis

The accident tests will release a portion of the inventory of numerous fission products, including Kr, Xe, Cs, Ag, Sr, Eu, I, and other radioisotopes, from the compacts as vapor. During the accident tests, the gaseous Kr and Xe radioisotopes will be carried from the heating furnace by the sweep gas flow and trapped in the downstream cryogenic carbon traps, where the inventory will be continuously monitored.

The vapor consisting of condensible fission products will primarily be deposited on the surfaces of the condensation plates (INL) or cups (ORNL). Some additional condensation will occur at other locations within the furnace, resulting in a collection efficiency that must be determined in the context of test furnace calibration. Each heating test will generate about 20 or more condensation plates/cups (the number will be dictated by the test duration and the sampling frequency, which will be determined prior to each test), each containing various amounts and distributions of condensed radionuclides released by the heating tests.

The primary fission products that will be analyzed on the condensation plate are Sr-90, Ag-110m, I-131, Cs-134/137, and Eu-154. The analysis method will involve gamma analysis of the dry, unprocessed condensation plates/cups, followed by dissolution of the condensed fission products, chemical separation of isotopes of interest, and analysis by appropriate methods. After extraction from the furnace, the whole condensation plate will be analyzed by gamma spectroscopy. The time between removal from the furnace and gamma analysis of the plate will be minimized to the extent possible for tests involving re-irradiated compacts in order to accommodate short-lived isotopes. This step will count the activities of the cesium isotopes (primarily Cs-134 and Cs-137), silver (primarily Ag-110m), and possibly europium (most likely Eu-154). Any other radioisotopes that are identified on the plates will also be reported and quantified, if possible.

For compacts that have been reirradiated prior to the accident test, I-131 might be measurable by direct gamma spectroscopy of the condensation plates. However, if the condensation plates also contain a high activity of Ag-110m or cesium isotopes, the resulting high Compton background may make detection and quantification of the I-131 emissions difficult, as indicated by Monte Carlo N-Particle Transport Code modeling results. If this is the case, the I-131 can be analyzed by dissolution, separation, and analysis of the iodine as described in Appendix B. The contents of the condensation plates may also be analyzed for I-129. The low anticipated activity of I-129 will require analysis by ICP-MS or TIMS, possibly with separation of iodine from the other fission products (as described in Appendix B) to minimize isobaric interferences.

Analyzing for strontium requires an analysis of Sr-90, a beta emitter. The Sr-90 analysis requires the dissolution of the fission products from the condensation plate and separation of the strontium from the other fission products using a strontium-specific ion exchange resin. The Sr-90 component of the eluted strontium solution is analyzed by liquid scintillation. Further details of the radiochemical Sr and I analysis are given in Appendix B.

6.15 Compact Screening Heating Tests

Fuel that has exhibited poor in-pile performance (as indicated by relatively high R/B values during the test irradiation), relatively high SiC failure fractions in-pile (as determined by post-irradiation LBL measurements), or high fission product inventories in the graphite holder or on the metal capsule components will undergo investigation to determine the causes of the poor behavior. In order to help identify specific compacts with significant particle failures, it may be possible for compacts to be screened by high temperature heating and simultaneous monitoring of released fission products for short durations. This will be accomplished using the fuel accident testing furnaces at INL and ORNL. Screening tests will be performed at temperatures sufficient to generate fission product releases (~1400–1600°C) for short durations (approximately one day). Fission gas releases will be monitored during the tests, and condensation plate analysis will be performed at the conclusion of the tests to determine release of fission metals. Both types of data will then be scrutinized to determine if the releases from the tested compacts indicate failed particle coatings.

6.16 Compact Allocation

A tentative allocation of compacts in each capsule for the destructive examinations has been prepared based on the irradiation histories (burnup, fast fluence, and temperature). Some factors influencing the compact selection are described below:

- A compact from each capsule with burnup and volume averaged, time averaged irradiation temperature values near the capsule mid-range value will be selected for post-irradiation ceramography. The main data objectives for post-irradiation compact ceramography are (1) a first look at irradiated particles and (2) the condition of the matrix and OPyC-matrix interactions. A compact with burnup and temperature values near the capsule mid-range will provide a representative compact for analysis, while higher burnup compacts and particles will still be available for microscopic examination as part of the post-accident test experiments.
- The compacts for post-irradiation LBL tests will in most cases be chosen from the higher end of burnup and irradiation temperature for each capsule. This will ensure that the fission product inventory in the matrix (measured as part of the pre-burn leach) is near the top of the range expected for the capsule and will therefore represent a somewhat conservative measurement. In addition, particle samples for destructive burnup measurements will be extracted from some of the post-irradiation LBL compacts, so LBL compact selection will take this into consideration as well. Specifically, at least three compacts from Stack 1 and one compact from Stack 2 are desired for a total of 4 compacts from the entire test train. Also, compacts should be selected from the portion of each capsule where the axial burnup gradient is lowest to minimize the range of burnups expected across the particle sample.
- The majority of the remaining compacts available for accident testing will be assigned to specific accident tests with the goal of clarifying the effect of various compact attributes (fuel variant and irradiation conditions) and accident test temperature on the fuel performance. Table 8 lists the important independent variables whose effect on accident behavior is to be examined in order of priority. As indicated in the table, the effect of SiC variant is the highest priority, followed by the effect of burnup, etc. In addition to the attributes listed in Table 8, the effect of any potential laboratory bias in accident test results at ORNL and INL will be examined as well.
- Compacts not used in accident tests or other experiments will be archived at the completion of AGR-1 PIE.

Table 8. Independent variables to be analyzed as part of the accident test matrix for AGR-1 compacts, listed in order of importance.

Attribute/variable
SiC variant (compares Baseline, Variant 1, and Variant 3)
Burnup
Irradiation temperature
Accident test temperature
Fast fluence
IPyC variant (compares Baseline, Variant 1, and Variant 2)

A detailed experimental matrix will be constructed once the final AGR-1 as-run physics and thermal calculations are completed and verified. The primary purpose of the matrix will be to select compacts for specific accident tests so that the effects of the variables in Table 8 on fuel performance can be best understood. A tentative allocation of compacts for the initial destructive ceramography, LBL, and

destructive burnup analysis activities has been made based on preliminary as-run data and is shown in Tables 9 through 14. In these tables, the burnup column refers to destructive burnup measurements as discussed in Section 6.11. Post-accident test analysis of compacts is not included in these matrices, as the specific compact characterization methods (LBL, IMGA, microscopy, etc.) will be influenced by the accident test results. Tables 9–14 will be updated as necessary once final as-run data are available.

Table 9. Allocation of Capsule 6 compacts for PIE experiments.

Compact	Ceramo-graphy	LBL	Burnup	IMGA	Accident test or archive	Lab
6-4-1					X	TBD
6-3-1					X	TBD
6-2-1					X	TBD
6-1-1		X		X[1]		ORNL
6-4-3					X	TBD
6-3-3	X					INL
6-2-3					X	TBD
6-1-3					X	TBD
6-4-2	X					ORNL
6-3-2		X	X	X		INL
6-2-2					X	TBD
6-1-2					X	TBD

1. All particles in compact will be gamma counted at ORNL using the Advanced-IMGA and the results compared to whole-compact gamma counting performed at INL for fission product inventory and burnup.

Table 10. Allocation of Capsule 5 compacts for PIE experiments.

Compact	Ceramo-graphy	LBL	Burnup	IMGA	Accident Test or Archive	Lab
5-4-1					X	TBD
5-3-1		X	X	X		INL
5-2-1					X	TBD
5-1-1					X	TBD
5-4-3					X	TBD
5-3-3	X					INL
5-2-3					X	TBD
5-1-3					X	TBD
5-4-2					X	TBD
5-3-2					X	TBD
5-2-2					X	TBD
5-1-2					X	TBD

Table 11. Allocation of Capsule 4 compacts for PIE experiments.

Compact	Ceramo-graphy	LBL	Burnup	IMGA	Accident test or archive	Lab
4-4-1					X	TBD
4-3-1		X		X		INL
4-2-1					X	TBD
4-1-1					X	TBD
4-4-3					X	TBD
4-3-3	X					TBD
4-2-3					X	TBD
4-1-3					X	TBD
4-4-2					X	TBD
4-3-2					X	TBD
4-2-2					X	TBD
4-1-2					X	INL

Table 12. Allocation of Capsule 3 compacts for PIE experiments.

Compact	Ceramo-graphy	LBL	Burnup	IMGA	Accident test or archive	Lab
3-4-1					X	TBD
3-3-1					X	TBD
3-2-1		X	X	X		INL
3-1-1					X	TBD
3-4-3					X	TBD
3-3-3	X					INL
3-2-3					X	TBD
3-1-3					X	TBD
3-4-2					X	TBD
3-3-2					X	TBD
3-2-2					X	TBD
3-1-2					X	TBD

Table 13. Allocation of Capsule 2 compacts for PIE experiments.

Compact	Ceramo-graphy	LBL	Burnup	IMGA	Accident test or archive	Lab
2-4-1					X	TBD
2-3-1					X	TBD
2-2-1					X	TBD
2-1-1					X	TBD
2-4-3					X	TBD
2-3-3					X	TBD
2-2-3		X		X		INL
2-1-3	X					INL
2-4-2					X	TBD
2-3-2					X	TBD
2-2-2					X	TBD
2-1-2					X	TBD

Table 14. Allocation of Capsule 1 compacts for PIE experiments.

Compact	Ceramo-graphy	LBL	Burnup	IMGA	Accident test or archive	Lab
1-4-1					X	TBD
1-3-1					X	TBD
1-2-1					X	TBD
1-1-1		X	X	X		INL
1-4-3					X	TBD
1-3-3					X	TBD
1-2-3					X	TBD
1-1-3					X	TBD
1-4-2	X					TBD
1-3-2					X	TBD
1-2-2					X	INL
1-1-2					X	TBD

7. PIE ACTIVITY PRIORITIZATION

The relative priority of the PIE activities presented in this plan are presented in Table 15, with each activity assigned a "low," "medium," or "high" ranking based on NGNP program objectives. This prioritization, along with the general capsule priority outlined above in Section 0, will be used to help guide program decisions on appropriate work scope consistent with available funding.

Table 15. Relative priority of AGR-1 PIE activities.

Activity	Section	Priority
Cask transfer from ATR to HFEF	3.1	High
Test train visual inspection	3.2	Med
Test train gamma scanning	3.3	Med
Test train and capsule disassembly	4.1, 4.2	High
Capsule liner inspection	4.2	Low
Leadout disassembly	4.3	Low
Component metrology	4.4	High
Graphite holder qualitative gamma scanning	6.1	Med
Compact gamma scanning	6.2	Med
Flux wire analysis	6.3	High
Melt wire analysis	6.3	Low
Thermocouple analysis	6.4	Low
Thermal conductivity	6.5	Low
Capsule deposited fission products	6.6	Med
Microanalyses of fuel compacts	6.7	Med
Deconsolidation	6.8	High
LBL	6.9	Med
Particle inspection	0	Med
Compact burnup determination	6.11	High
IMGA	6.12	Med
Microanalyses of fuel particles	6.13	High
Re-irradiation	6.14.1	Med
Accident testing	6.14.2	High
Condensation plate/cup analysis	6.14.3	High
Compact screening heating tests	6.15	Low
Waste Handling	0	High
Reporting	0	High

8. TENTATIVE AGR-1 PIE SCHEDULE

A tentative schedule for the AGR-1 PIE activities is given in Table 16.

Table 16. Tentative high-level schedule for AGR-1 PIE.

Activity	Schedule
Ship AGR-1 test train to HFEF	Mar 2010
Initial disassembly and component metrology completed	5 months after receipt of test train at HFEF
"First look" report completed	6 months after receipt of test train
Completion of AGR-1 PIE activities	Approximately 28 months after receipt of the test train at HFEF
Completion of final AGR-1 PIE data report	4 months after completion of AGR-1 PIE

9. WASTE HANDLING

The PIE activities will generate small amounts of radioactive waste (estimated at less than 10 ft^3 per year) that must be properly dispositioned. This waste will be generated by the disassembly, metallography, furnace accident testing, equipment maintenance activities, and analytical laboratory activities associated with the AGR-1 examination and analysis. Typical wastes will include a short length of stainless steel leadout tubing, 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 furnace, pneumatic transfer rabbits, and parts replaced on the accident testing furnaces (replacement 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 (capsule head, through tubes, outer shell, graphite holder, and graphite spacers) 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 metallography 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, graphite components, and small pieces of TC. The whole compacts may have contact radiation fields as high as 10^4 R/hr 6 months after the test irradiation. The wastes associated with the fuel compacts 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 channels. Most of the waste is expected to be low-level waste or remote handled low-level waste that falls within the current waste disposal paths. The liquid waste generated by the analytic 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 activated metal will be handled; most of the waste generated will be from the metallographic and analytic tasks.

10. QUALITY ASSURANCE

The VHTR Technology Development Office (TDO) Project Execution Plan (Petti 2008a) identifies the quality assurance requirements imposed on all TDO fuel development activities. The plan stipulates that fuel development activities will be performed in accordance with LRD-13010 (2008). Specific application of quality assurance requirements to the VHTR TDO are described in PLN-2690 (Petti 2008b). The project execution plan also identifies governing documents for:

- Records Management
- Software Quality Assurance Plan
- Data Management Plan
- Configuration Management
- Personnel Indoctrination, Training, and Qualification.

Work activities associated with this plan are conducted under a quality program implementing American Society of Mechanical Engineers (ASME) NQA-1 2000 Part 1 and Subpart 2.7. Organizations or services subcontracted to support PIE quality affecting work activities will be on the INL Qualified Suppliers List for the selected activities to be performed. Activities affecting quality include, but are not limited to, procurement, handling, shipping, storing, inspecting, testing, training, data collection, records, electronic data storage, software control for software used in data analysis, and the generation of reports from collected data. ORNL will perform PIE support services in accordance with their project AGR specific quality assurance program plan, QAP-ORNL-AGR-01 (Bell 2006).

10.1 PIE Data Management

The primary product of the NGNP PIE and safety testing activities is data that will be used to support future VHTR fuel licensing efforts. The data must withstand scrutiny of NRC license application reviewers. Collecting, storing, and maintaining good data that will adequately support fuel licensing activities requires a project-wide effort that implements principles of quality control on:

- Design control
- Instructions, procedures, and drawings
- Identification and control of items
- Control of special processes
- Document control
- Records control
- Test control
- Control of measuring and test equipment
- Handling, storage, and shipping
- Disposition of nonconformances.

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, PNNL, 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 NGNP Data

Management and Analysis System (NDMAS) or to the INL Electronic Document Management System (EDMS). Primarily, the NDMAS system 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, evaluation reports, pdf documents, engineering calculation and analysis reports (ECARs). Since the 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 VHTR Program Data Management and Analysis Plan (PLN-2709, 07/23/09) details how data will be stored, controlled, categorized, and qualified.

11. REPORTING

Program staff will create reports to ensure that pertinent data from the PIE activities are available for various programmatic decisions as necessary. These will include:

- *First-look report.* This report will summarize the results of preliminary PIE activities, including: gamma scanning the intact test train, test train disassembly and inspection, compact/graphite metrology, graphite holder gamma scanning, and gamma counting of compacts along with associated burnup measurements. The availability of these data will help support subsequent test train design and fabrication as well as a revision of the AGR-1 capsule thermal analyses.
- *Annual reports.* The annual reports will summarize the PIE activities and data for the AGR-1 test train performed in each fiscal year for which the experiments are in progress.
- *Final AGR-1 PIE data report.* This report will be prepared at the completion of the AGR-1 PIE and when all data have been obtained from ongoing experiments and analyses; it will include the comprehensive description of AGR-1 PIE activities and data. This report will also include relevant conclusions based on the experimental results.

Regular input on PIE activities and experimental results will also be provided as needed for the NGNP monthly reports and weekly highlights. The NGNP Fuels PIE staff will make selected PIE data available to the NGNP database as it is generated and will participate in NGNP teleconferences and discussions to facilitate dissemination of experimental data as needed by the program.

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Appendix A

Identification and Location of Flux and Melt Wires

Each graphite fuel holder contains a cobalt-vanadium (Co-V), an iron (Fe), and a niobium (Nb) flux wire package, plus one beryllium (Be) melt wire package. One each of the Co-V, Fe, and Nb flux wire packages are embedded at the periphery of the holder and the one melt wire package is in the radial center. All four packages were inserted into axial mounting holes drilled from the bottom of the holder.

The locations of the flux and melt wires are summarized by package identity in Table A-1. Each package has an identification code stamped on the bottom. However, the identification numbers are not unique because five identifiers can correspond to two different packages (6 = Fe flux or Be melt wire; 7 = Co-V flux or Fe flux wire; H = Co-V flux or Be melt wire; K = Co-V flux or Be melt wire; and N = Fe flux or Be melt wire), as shown in Table A-1. The pairs of redundant identifiers are shown in bold colored type in the table. This lack of specificity could be a problem only for two capsules, in which two of the four packages have the same identifier. The potential therefore exists for confusion of package identity if the packages are extracted from the fuel holder at the same time and co-mingled.

Table A-1. Identities and locations of the flux and melt wire packages in the AGR-1 test train.

Fuel Capsule	Package Type	Package Identity
1	Flux, Nb	18
	Flux, Co-V	7
	Flux, Fe	3
	Melt, Be	N
2	Flux, Nb	7H
	Flux, Co-V	H
	Flux, Fe	6
	Melt, Be	H
3	Flux, Nb	VE
	Flux, Co-V	V
	Flux, Fe	2
	Melt, Be	4
4	Flux, Nb	87
	Flux, Co-V	B
	Flux, Fe	7
	Melt, Be	X
5	Flux, Nb	RU
	Flux, Co-V	K
	Flux, Fe	N
	Melt, Be	K
6	Flux, Nb	R7
	Flux, Co-V	J
	Flux, Fe	1
	Melt, Be	6

As specifically shown in Table A-1, the two packages labeled “H” are both located in Capsule 2, and the two packages labeled “K” are both located in Capsule 5; the other three sets of packages with redundant identifiers are individually distributed in the fuel capsules. Because the redundant identities involve dissimilar package types, the true identities of the packages can always be resolved at the time of analysis by the gamma signature and by the nominal lengths of the package (Co-V, 5 mm; Fe, 7 mm; Nb, 9 mm; Be, 8 mm), which indicate the type of wire within the package. Therefore the combination of package length, gamma signature, and identifying number will uniquely identify the packages used in the AGR-1 test train. Such identification might not be possible, however, if the packages remain stuck in the graphite fuel holder due to carbide formation and must be removed with some graphite still intact. Confusion in package identity could result in mistakenly sending some Be melt wire packages to the gamma counting laboratory, or some flux wire packages to the metallography laboratory for destructive sectioning. While the former error will result only in the loss of time, the latter error could result in the loss of fluence data.

Appendix B
Supporting Information on Deposition Plate/Cup Analyses

Strontium Separation and Liquid Scintillation

Failure of fuel particles during the accident tests and possibly diffusive release from intact particles may result in the release of some Sr-90, which undergoes beta decay with no gamma emissions. Analysis of Sr-90 will therefore require the dissolution of the condensed fission products on the condensation plates, separation of the strontium from the other radionuclides, and analysis of the strontium isotopes by liquid scintillation.

The quantitative separation of strontium from other radionuclides is a well-established procedure. The separation process uses a strontium-specific crown ether resin that preferentially binds strontium, while the other solution components elute through the bed. The bound strontium is purified by several washes of the bed before being eluted from the bed. The activity of the strontium is determined by liquid scintillation. This analysis will be performed both at INL and ORNL to determine activities of Sr-90 on the condensation plates (FACS) and cups (CCCTF).

Gamma Spectroscopy

The first step of the proposed analysis process consists of gamma spectroscopy of the condensation plates. The spectroscopy will be performed on the whole, dry, unprocessed condensation plate using standard HPGe detector systems. The objective of the initial gamma spectroscopy is to quantify the cesium, silver, iodine, tellurium, and europium content of the condensation plates. The gamma emissions of interest for the radioisotopes with significant potential released activity are given in Table B-1. The remaining radionuclides have low released activities, low-energy gamma emissions, or gamma intensities that are too weak to be of interest.

Each heating test will generate about 20 or more condensation plate/cup samples. It is anticipated that each element will be evolved at different rates during the course of the heating test, with differing “breakthrough” times and total release fractions that may be dependent on the temperature, fluence, and burnup history of the fuel. It is expected that each condensation plate/cup will be coated with a variable fraction of the total releasable inventory, and with a variable ratio of two or more elements comprising the sample mixture. These samples pose a challenge because they require the analysis of Sr, a beta emitter, that will be mixed in with gamma emitters. Because iodine evolves along with Ag and Cs, both having high activities, the signal-to-noise ratios for the I-131 emissions may be adversely impacted by the high Compton background produced by the Cs and Ag in the sample mixture. Initial modeling of sample spectra involving the above radionuclides has indicated that I-131 may be difficult to detect in the presence of Cs and Ag deposits, particularly as the iodine evolution tapers off and the cesium evolution rises on successive condensation plates. It is anticipated that I-131 might not be detectable on those condensation plates with high silver and/or cesium content, and that alternative methods of analysis of I-131 will be needed.

Table B-1. Principal emissions of the radionuclides of interest for fuel re-irradiated in NRAD. The emissions of interest are highlighted in bold face type.

Radionuclide of Interest	Potential Released Inventory per Failed Particle, Ci/particle	Gamma Energies of Interest, keV (% intensity)	Comments
Sr-90	1.75E-06	None	Beta emission at 546 keV
Ag-110m	2.25E-03	657.8 (96), 884.7 (71), 937 (32), 764 (23), 1384 (21), 1505 (11)	
Te-125m	3.42E-09	35 (7), 110 (0.3)	
Te-127m	6.83E-09	59 (0.19), 89 (0.08)	
Te-129m	1.13E-09	690 (6)	
I-131	9.61E-07	364.5 (82), 637 (6.8), 284 (5.4)	Potential interference from 360 keV of Te-127m
Cs-134	8.24E-05	795.9 (99), 604.7 (98), 570 (23), 1365 (3.4)	
Cs-137	8.98E-05	661.7 (85)	Sole gamma emission. Potential interference from 658 keV of Ag-110m
Eu-152	8.56E-13	344 (27), 1408 (22), 965 (15), 1113 (14)	
Eu-154	1.18E-12	1278 (37), 1000 (31), 724 (21)	
Eu-155	1.24E-12	87 (32), 105 (20)	
Eu-156	2.13E-11	1230.7 (16), 1150 (14), 1070 (11), 811.8 (9)	

Iodine Separation and Gamma Counting

If gamma counting of the I-131 inventory on the plates is not feasible, then the most practical method for I-131 analysis is to radiochemically separate the iodine from the other fission products in the mixture. The method involves the dissolution of the fission products condensed on the deposition plate, the separation of the iodine by selective sorption on an ion exchange resin, and the sequential and selective elution of the sorbed species. The "purified" iodine solutions can then be analyzed by liquid scintillation for maximum sensitivity or by gamma spectroscopy if the separation is incomplete. Regardless of the purity of the iodine separation, the method will reduce the Compton background greatly by removing most of the other radionuclides, and correspondingly improve the I-131 analysis.

The iodine separation method is based on the results from research funded by the INL Laboratory-Directed Research and Development Program in 2005. The research improved the sensitivity of I-129 analysis in environmental and geological samples. The method requires the dissolution of the fission products on the condensation plate with water or a nonoxidizing acid or salt solution to prevent the oxidation of iodine from the iodide to iodate. The resulting solution is passed through a conditioned AG1-X2 resin column to bind the iodide onto the resin. In the initial loading process, the radionuclides of cesium, strontium, and europium pass through the resin bed with no retention; subsequent washes ensure their quantitative separation from the bound iodine. Only iodine, silver, and tellurium remain on the resin bed. Tellurium, iodine, and silver are selectively and quantitatively eluted from the resin column using bisulfite, hypochlorite, and nitric acid washes, respectively. After selective elution from the resin bed, the iodine solution can be analyzed by liquid scintillation or gross gamma counting if the separation is complete, or by gamma spectroscopy if the separation from silver and tellurium is not complete. Even if the separation is not complete, the degree of purification is expected to be substantial, with a commensurate decrease in the Compton background.