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# Irradiated AGR-2 Compact 2-1-2 Examination Plan

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**Plan**

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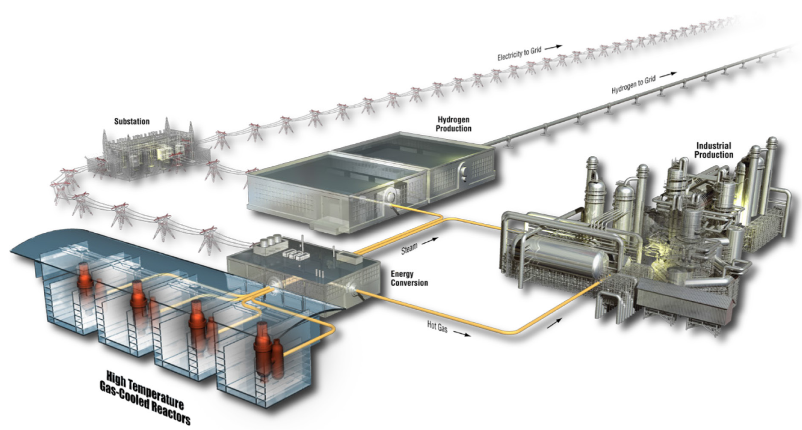


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# ACRONYMS

| | |
|---|---|
| AGR | Advanced Gas Reactor |
| CCCTF | Core Conduction Cooldown Test Facility |
| IMGA | irradiated microsphere gamma analyzer |
| INL | Idaho National Laboratory |
| ORNL | Oak Ridge National Laboratory |
| PIE | post-irradiation examination |
| SiC | silicon carbide (coating layer) |
| TRISO | tristructural isotropic (coated particles) |

# 1. INTRODUCTION

This plan describes post-irradiation examination (PIE) activities (safety testing and post-safety test analyses) to be performed by Oak Ridge National Laboratory (ORNL) on irradiated Compact 2-1-2 that was taken from the Advanced Gas Reactor (AGR) experiment, AGR-2. This work will be performed in accordance with general objectives outlined in the AGR-2 PIE plan [1] and guidance in the ORNL PIE statement of work. [2]

# 2. FUEL COMPACT DESCRIPTION

The fuel specimen contains tristructural isotropic (TRISO)-coated particles, with kernels containing a mixture of uranium carbide and uranium oxide. The specimen was irradiated in Capsule 2 of the AGR-2 test train in the B-12 position of the Advanced Test Reactor at Idaho National Laboratory (INL). [3] Table 1 shows some properties and irradiation conditions for the AGR-2 Compact 2-1-2.

Table 1. Identification and irradiation conditions for the AGR-2 Compact 2-1-2.

| Compact ID[a] | Compact Container ID | Fabrication ID | Fuel Type | Average Burnup (% FIMA[b])[c] | Fast Fluence $\times 10^{25}$ ($n/m^2$)[c] | Irradiation Temperature (°C)[d] |
|---|---|---|---|---|---|---|
| AGR-2 2-1-2 | AGR208 | LEU09-OP2-Z079 | UCO | 12.62 | 3.25 | 1219 |

a. The X-Y-Z naming convention denotes the location in the irradiation test train: capsule-level-stack.[1]
b. FIMA = fissions per initial metal atom.
c. Based on physics calculations. [4]
d. Time-averaged, volume-averaged temperature based on thermal calculations. [5]

# 3. EXPERIMENTAL OBJECTIVES

- Evaluate the time-dependent release behavior of gaseous and condensable fission products during safety testing at high temperatures in pure helium in the Core Conduction Cooldown Test Facility (CCCTF). Fission products to be analyzed include $^{85}$Kr, $^{90}$Sr, $^{95}$Zr, $^{104}$Pd, $^{106}$Ru, $^{110m}$Ag, $^{134}$Cs, $^{137}$Cs, $^{144}$Ce, $^{154}$Eu, and $^{155}$Eu. Special emphasis will be on monitoring cesium release as an indicator of silicon carbide (SiC) failure in individual particles.
- Measure the inventory of fission products outside of the intact SiC layers, but retained in the compact matrix or outer pyrolytic carbon. These measurements should be done by deconsolidation-leach analysis and burn-leach analysis of the deconsolidated particles and compact matrix debris following the safety test. If a survey of all particles within the irradiated microsphere gamma analyzer (IMGA) is performed to detect particles that released cesium, pre-burn acid leaching may be skipped to minimize particle damage prior to the survey.
- Examine individual particles deconsolidated from the as-irradiated compact to quantify retention of specific gamma-emitting fission products (including $^{95}$Zr, $^{106}$Ru, $^{110m}$Ag, $^{134}$Cs, $^{137}$Cs, $^{144}$Ce, and $^{154}$Eu) and to identify anomalous particles, especially those with a below-average cesium inventory indicative of SiC failure. An important objective of identifying these low-cesium particles and measuring their remaining $^{134}$Cs inventory is to determine if their missing $^{134}$Cs inventory is sufficient to account for the $^{134}$Cs measured on the CCCTF's internal components and in the compact matrix. This $^{134}$Cs accounting is helpful in enumerating particles with failed SiC.
- Perform microanalysis on selected particles to better understand the correlation of particle microstructure with fission product retention. Microanalysis of any particles with a low $^{137}$Cs/$^{144}$Ce ratio is of particular interest if the compact released cesium during safety testing; these particles should have the highest priority for examination. Based on specific results and discussions with the INL AGR PIE Technical Lead, these particles may be sent to INL for additional microanalysis.
- Archive the remaining particles for possible later use.

# 4. SCOPE OF WORK

## 4.1 Receipt Inspection

The compact shipping drum will be unpacked and an individual aluminum compact storage container removed. The compact will be removed from the storage container and inspected for any damage prior to proceeding with subsequent analysis. The condition of the compact and any features of interest will be photographically documented.

## 4.2 Furnace Testing

The compact will be heated in pure helium in the CCCTF to a maximum temperature of 1800°C; Figure 1 and Table 2 show the temperature profile for the safety test. The temperatures in the profile should be maintained within the accuracy limits of the CCCTF furnace thermocouples and control software. The hold duration at 1800°C will be based on observed fuel performance. A shorter test may be called for if significant fuel failure is observed early in the test, which is indicated either by online fission gas release measurements or preliminary gamma measurement of the released cesium activity on the CCCTF deposition cups. In the absence of significant early fuel failure, the minimum hold time at 1800°C will be 300 hours. However, a longer hold time may be considered to provide more time for possible coating layer failure or diffusion through intact SiC layers and measurable reduction of the retained particle inventory for diffusing radioisotopes. Radioisotope release will be monitored throughout the test and the amount will help determine the duration of the test. Changes to final heating times or any other test changes will be made in consultation with the INL AGR PIE Technical Lead.

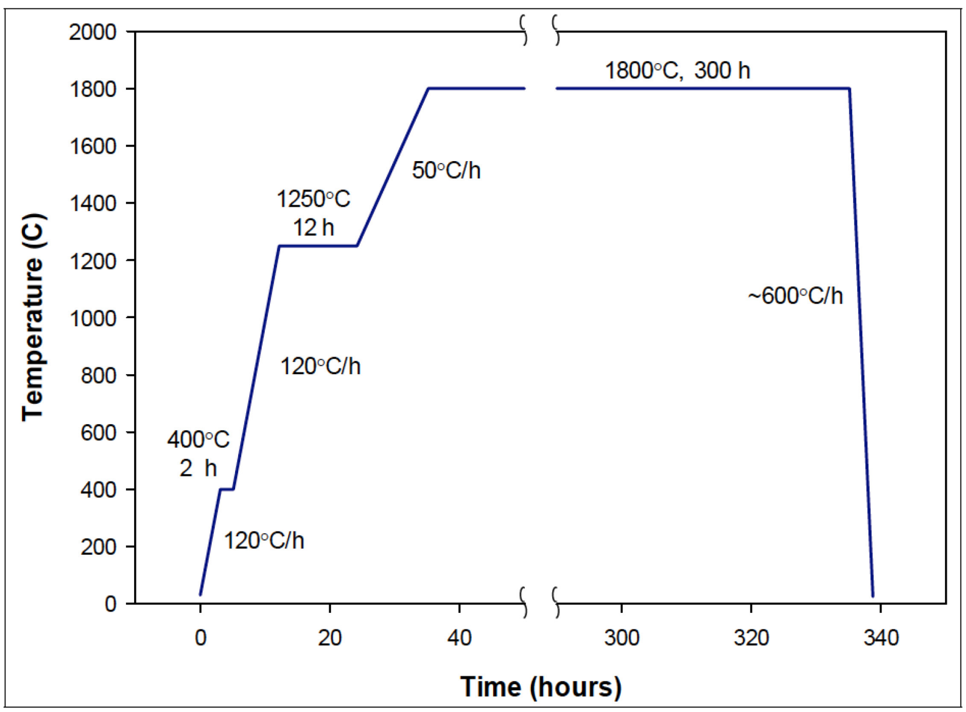


Figure 1. Temperature profile for the safety test of Compact 2-1-2. Elapsed time and ramp rates are approximate.

Table 2. Temperature profile for safety test of AGR-2 Compact 2-1-2.

| Approximate Elapsed Time (hours)[a] | Temperature (°C) |
|---|---|
| 0.0 | 30 |
| 3.1 | 400 |
| 5.1 | 400 |
| 12.2 | 1250 |
| 24.2 | 1250 |
| 35.6 | 1800 |
| 335.6[a] | 1800 |
| 338.6[a] | 30 |

a. Actual hold time may be modified depending on test results.

The first deposition cup will be exchanged near the end of the 1250°C hold cycle. A second cup will be exchanged once the furnace temperature reaches the target of 1800°C. The deposition cups will then be exchanged approximately once every 12 hours for the next 36 hours (i.e., three more cups after the first cup change at 1800°C). After 36 hours (i.e., four cup changes at 1800°C), the cups will be changed once every 24 hours for the remainder of the safety test. The actual exchange interval may deviate from this schedule based on results from the early test stages, but should not be less than 6 hours or more than 18 hours for the initial 36-hour period at 1800°C or more than 30 hours for the remainder of the test.

The cups will be examined to determine the inventory of deposited gamma-emitting radionuclides (such as $^{95}Zr$, $^{106}Ru$, $^{110m}Ag$, $^{134}Cs$, $^{137}Cs$, $^{144}Ce$, $^{154}Eu$, and $^{155}Eu$) and any other radionuclides detected. In practice, preliminary analysis will be conducted by performing a gamma scan as soon as possible after the cup is removed from the furnace to provide rapid feedback about progress of the safety test. This feedback will allow changes to be made to the test if necessary. After completion of the test, the cups may undergo additional gamma analysis prior to leaching. Analysis of the leach solutions includes measurement of $^{90}Sr$ and $^{104}Pd$ and may include additional analysis for gamma-emitting isotopes.

Reported data will include the isotopic inventory on the deposition cups and the inventory measured in the CCCTF fission gas traps as a function of test time. Conversion to fractional released inventory will be performed using compact as-run inventory calculations.[4]

## 4.3 Post-Test Analysis

Post-test characterization activities for the compact will be determined following the safety test, based on the data obtained. The baseline post-test plan will focus on accounting for fission products not collected by deposition cups and examining the performance of individual particles.

Some of the fission products (such as europium and strontium) are poorly collected by the deposition cups, and significant fractions are collected in the graphite holder or tantalum can and gas inlet line. Collection efficiency of the deposition cups must be determined to estimate the total time-dependent release of each fission product throughout the safety test. After the graphite fuel holder containing the compact has been removed from the furnace and returned to the main hot cell, the fuel compact will be removed from the holder and temporarily stored in a labeled container until deconsolidation leaching is performed. The holder will be oxidized in air and the resultant ash leached to determine the amount and identification of the fission products held up in the holder. The tantalum furnace can and tube will be initially gamma scanned, leached with acid (optionally augmented with ball milling), and gamma scanned again after leaching. If needed, gamma analysis of the leachant will be scaled by the tantalum leaching efficiency calculated from the pre and post-leach gamma scans to determine the fraction of each fission product collected on the tantalum. Alternately, historical leaching data may be used in lieu of tantalum leaching to develop correlations between the measured gamma inventory (in a specific geometry) and the actual inventory on the tantalum can and gas inlet line. The combined analysis of all furnace internals will be used to estimate deposition cup collection efficiency for final determination of fission product release during the safety test.

The fuel compact will be electrolytically deconsolidated to break up the matrix material and free the TRISO fuel particles. After electrolytic deconsolidation, select stages of the leach-burn-leach analysis process will be performed. The standard hot cell leach-burn-leach process involves two pre-burn acid leaches in a Soxhlet extractor, followed by further digestion of the matrix material in boiling acid to help remove any matrix residue from the TRISO particles and break up the matrix debris. The matrix debris is separated from the particles by washing through a sieve and subjecting it to a 750°C burn, followed by two post-burn leaches. The leach solutions are analyzed for uranium and fission products. After washing and drying, particles are transferred to the IMGA cubicle for gamma scanning prior to burn-leach analysis of the particles. The pre-burn leach process sometimes results in destruction of particles with a failed-SiC layer via fracture of the outer pyrocarbon layer and subsequent leaching of the kernel. Although it is still possible to enumerate these failed-SiC particles by determining the number of leached kernels, recovery of intact failed-SiC particles is preferred because detection of these particles with IMGA allows for further microstructural analysis. If an IMGA survey is planned, pre-burn acid leaching may be skipped to minimize particle damage prior to IMGA survey.

Deconsolidated particles will be inspected and imaged using the particle micromanipulator in the IMGA cubicle to assess overall condition and identify features of interest (such as cracked coatings or coating fragments). Abnormal particles or coating fragments may be selected for further examination.

The need for a full gamma survey of the particles with IMGA will be based on the preliminary safety test results. If cesium or krypton release indicates the probable presence of particles with failed SiC or failed TRISO (i.e., failure of all three dense layers), respectively, then a full IMGA survey will be performed. A full survey may also be performed with the approval of the INL AGR PIE Technical Lead if it is determined appropriate and beneficial to the post-safety test analysis. If a full gamma survey is performed, a short counting time (i.e., typically 50 to 100 seconds) will be used to measure $^{137}$Cs and $^{144}$Ce and sort out particles with a low cesium-to-cerium ratio or low-cerium content. A below average $^{137}$Cs/$^{144}$Ce ratio is indicative of significant cesium release during irradiation due to failed SiC or failed TRISO. Below average $^{144}$Ce content may indicate abnormal kernels or general fission product loss that could possibly be related to failed TRISO.

After completion of the full IMGA survey (or after particle inspection if the full IMGA survey is not performed), an archive sample of about 10% of the particles will be riffled out and the remaining 90% of the deconsolidated TRISO particles will be returned to the main cell for burn-leach analysis. Similar to the matrix burn-leach, particles will be burned at 750°C to remove the exposed carbon and to oxidize the metallic fission products not previously leached. Burned-back particles will then be leached two more times in a Soxhlet extractor. Analysis of the burn-leach solutions will be performed to detect uranium and fission products not leached before the burn, including any exposed kernels from particles with defective SiC not separated out during IMGA analysis. After burn-leach, the burned-back particles will be washed, dried, and archived.

All particles sorted out during full gamma survey with the IMGA due to abnormal $^{137}$Cs or $^{144}$Ce inventory will undergo a longer count time (i.e., typically 4 to 6 hours) to measure the radioisotopic inventory (i.e., $^{95}$Zr, $^{106}$Ru, $^{110m}$Ag, $^{134}$Cs, $^{137}$Cs, $^{144}$Ce, and $^{154}$Eu) and determine distributions for $^{154}$Eu and $^{110m}$Ag. Long-count IMGA analysis will also be performed on a random riffled sample of about 50 particles.

After IMGA analysis, particles of interest will be selected for materialography and/or x-ray imaging, especially any particles with a below average $^{137}Cs/^{144}Ce$ ratio or low overall radioisotopic inventory that may be indicative of failed SiC or failed TRISO. Materialography may include optical imaging, scanning electron microscopy imaging, and/or elemental analysis of polished cross sections. Based on specific results and discussions with the INL AGR PIE Technical Lead, individual particles may be sent to INL for additional microanalysis.

In addition to identifying particles that released cesium due to failed SiC, another goal of this PIE is to identify any mechanisms that may explain why some particles release silver and europium to a greater extent than others. Features of interest will include the structure of the coatings and kernels, particularly changes induced by radiation or safety testing. Also of interest are the location and composition of fission products or actinide inclusions within the coating layers, primarily in the inner pyrolytic carbon and SiC layers.

## 4.4 Data Acquisition, Analysis, and Reporting

A compact PIE report will be prepared and include a description of experiments performed and all relevant data acquired. Overall data to be reported will include the following:

- Analysis results of furnace internals and a final deposition cup efficiency determination
- A compact fractional inventory of fission products released during the safety test, based on as-run inventory calculations[4]
- Compact fractional fission product inventories outside of SiC, as determined by compact deconsolidation and leach-burn-leach, based on as-run inventory calculations[4]
- Results of a particle inspection and IMGA gamma analysis of the individual particles
- X-ray and materialographic images, including detailed analysis of particles with low cesium retention
- Discussion of any unusual particle, kernel, or coating behavior that may be linked to fission product release.

# 5. QUALITY ASSURANCE

PIE activities performed at ORNL shall be performed in accordance with the AGR-2 PIE plan, applicable ORNL procedures, and the ORNL quality assurance plan for nuclear research and development activities[6] to meet INL quality assurance requirements specified in SOW-11467.[2]

# 6. REFERENCES

1. PLN-4616, “AGR-2 Post-Irradiation Examination Plan,” Revision 0, December 2013.
2. SOW-11467, “Statement of Work for AGR-2 PIE at Oak Ridge National Laboratory,” Revision 3, October 2016.
3. INL/EXT-14-32277, *AGR-2 Irradiation Test Final As-Run Report*, Revision 2, August 2014.
4. ECAR-2066, “JMOCUP As-Run Daily Depletion Calculation for the AGR-2 Experiment in the ATR B-12 Position,” Revision 2, April 2014.
5. ECAR-2476, “AGR-2 Daily As-Run Thermal Analyses,” Revision 1, August 2014.
6. QAP-ORNL-NR&D-01, *Quality Assurance Plan for Nuclear Research and Development Activities Conducted at the Oak Ridge National Laboratory*, Oak Ridge National Laboratory, Revision 0, May 2013.