WHC TESTING PROCEDURES IN SUPPORT OF GEOLOGICAL REPOSITORIES

Title: SAMPLE PREPARATION FOR SPENT FUEL OXIDATION TESTING USING A DRY BATH HEATING SYSTEM

Proc. No. SFO-1-1 Rev. No. 0 Issue Date 2/XX/86

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RECORD OF REVISIONS

Refer to Procedure HTA-1-1 for instructions regarding revision of procedures and use of this page.

Rev. No.	Date	Summary of Revisions
0	2/XX/86	Initial issue.

SAMPLE PREPARATION IN SPENT FUEL OXIDATION TESTING USING A DRY BATH HEATING SYSTEM

1.0 OBJECTIVE

This procedure provides instructions for preparing characterized samples to be used in measuring the rate of oxidation of spent LWR fuel for long durations at low temperatures. A dry bath heating system is used in which the fuel specimen is exposed to an air atmosphere with a known moisture content. Work performed under this procedure shall be in compliance with the quality assurance requirements of the approved sponsor request for services for the current fiscal year.

NQA-1 criteria applicable to this procedure are implemented through HEDL-MG-100, Quality Assurance Manual, and BQA-1-1, "Quality Assurance Program Plan."

2.0 APPLICABILITY

This procedure applies to the staff members of Westinghouse Hanford Company who are preparing characterized samples for spent fuel oxidation studies. These people are responsible for sample preparation and characterization by x-ray diffraction, SEM analysis, and ceramographic evaluation.

3.0 DEFINITIONS

3.1 Equipment

- Cutoff wheel.
- Beakers, Pyrex*.
- Tweezers, stainless steel.
- Petri dishes, glass.

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*Pyrex is a registered trademark of Pittsburgh Corning, Pittsburgh, PA.

- Rules, mm, steel, 6-in. long.
- Vials, cappable, for fuel transfer to "I" Cell.
- Photography equipment, for taking pictures through the cell window.
- Balance, two-place or better accuracy.
- Mortar, Diamonite, plattner-style, with zircaloy disc faces.
- Hammer, steel or lead.
- Mesh seives, Tyler screen scale.
- Seive shaker.
- Cask, for transporting fuel specimens between cells in 327 Building and between 327 and 325 Buildings.

3.2 Materials

- Turkey Point PWR spent fuel, from Fuel Assembly B17.
- BWR and PWR spent fuel, as supplied by MCC.
- Wipes, paper.
- Acetone.

3.3 Definitions of Terms

- Rod Segment -- Cut piece of fuel rod as received from source.
- Rod Section -- Piece of rod segment cut specifically for defueling.
- Fuel Fragment -- Piece of fuel fractured in reactor and removed from cladding.
- Sample Preparation Campaign -- Preparation of fuel all at the same time from a single source.
- Fragmented Sample -- Sample consisting of fragments of fuel.
- Pulverized Sample -- Sample consisting of fuel that has been reduced in size by mechanical processing after it was removed from cladding.
- Fuel Fraction -- All pulverized fuel in a particular size range.

4.0 RESPONSIBILITIES

4.1 Management

First-line management is responsible for ensuring that personnel performing activities under this procedure are qualified. In addition to technical skills, personnel are to be trained in safety and emergency procedures. Lead managerial responsibility lies with the Manager of Waste Package Engineering.

4.2 Principal Investigator

The principal investigator is responsible for conducting the Dry Bath Oxidation Experimentation and for making and documenting appropriate modifications to the procedure as occasion arises. All programmatic procedural changes shall have the concurrence of the Task Leader. The Principal Investigator is also responsible for ensuring that all required test and measuring equipment are maintained in calibration, for maintaining all records, for reviewing the experimental data, and for ensuring their quality.

4.3 Hot Cell Technicians

The hot cell technicians shall provide spent fuel specimens when required for experimental measurements. They shall perform the actual sample preparation and characterization as directed by the Principal Investigator. They shall maintain records pertinent to sample characterization and preparation. They shall also appropriately dispose of spent fuel materials that are no longer needed. They shall be qualified radiation workers, as defined in HEDL-WHAN-M-8.

4.4 Quality Assurance Representative

A WHC Quality Assurance staff member shall monitor activities performed according to this procedure in conformance with BQA-1-1.

4.5 Task Leader

The Task Leader shall be responsible for checking that samples are prepared and characterized according to the approved Dry Bath Spent Fuel Oxidation test plan and for making changes to the test plan with the concurrence of the sponsor. The Task Leader and the Principal Investigator may be the same person.

5.0 SAFETY

All normal safety practices prescribed in HEDL-WHAN-M-11, Industrial Safety Manual, shall be adhered to for any of the listed procedures described below. Radiation Work Procedures shall be prepared as required in HEDL-WHAN-M-8, Radiation Protection Procedures.

6.0 REQUIREMENTS/PROCEDURES

6.1 Removal of Fuel from Rods

Step 1) Transfer rod segments to cell for cutting, as specified by Principal Investigator.

Step 2) Check rod segment container identification, record identification in notebook, and remove rod segment from container. Only one rod segment is to be out of its container at any one time.

Step 3) Cut rod segment according to approved cutting diagram into sections 4 in. to 8 in. long using a cutoff wheel with no lubricant.

Step 4) Decontaminate the outside of the rod segments' cladding with a paper wipe and acetone.

Step 5) Transfer rod segments in properly shielded container to the cladding slitting cell.

Step 6) Slit cladding and pull it away from the fuel.

Step 7) Place fuel fragments in Pyrex beaker by dumping the fuel out of the cladding or by using stainless steel tweezers. NOTE: All fuel from a particular sample preparation campaign can be comingled. Fuel may be temporarily stored in an identified container.

6.2 Preparation of Fragmented Samples

Step 1) The Principal Investigator or his designate shall randomly choose 30 to 60 fragments from the comingled fuel prepared in Section 6.1 to make a sample. The number of fragments should be chosen so that the weight of a sample is ~10 g. The number of samples shall also be designated by the Principal Investigator.

Step 2) Place the fragments in a glass petri dish along with a mm rule. With fuel spread out, photograph fuel and ruler at 1.5X to 3X.

Step 3) Count and record the number of fragments.

Step 4) Number the samples as described in the test plan. Label the photograph with sample number.

Step 5) Place fragments of fuel in a covered and labeled sample container and transfer in a properly shielded cask to the 327 Building "I" Cell.

6.3 Preparation of Pulverized Samples

Step 1) Pour remaining fuel into stainless steel screen stack. The screen sizes shall be specified by the Principal Investigator. Screen remaining fuel from Section 6.2.

Step 2) Prepare as many as possible but not more than the required number of samples of each size fraction as specified by the Principal Investigator. Each sample shall weigh ~10 g. Label the samples as directed in the Test Plan. If sample requirements are met, go to Step 10.

Step 3) Place ~30 g of fragments in the Diamonite mortar (plattner-style) with zircaloy discs covering the faces. Strike pestle five times with steel or lead block. Pour fuel into stainless steel sieve screen stack. The screen sizes shall be specified by the Principal Investigator. The number of strikes may be changed by the Principal Investigator if this change is recorded in the notebook.

Step 4) Repeat Step 3 above until sufficient fuel is in screen stack to supply the number of specimens required by the Principal Investigator.

Step 5) Screen all crushed material for 5 minutes.

Step 6) Repeat Steps 1 through 3 until all fuel passes through the largest specified screen. NOTE: Steps 4 and 5 may be interchanged.

Step 7) Prepare the indicated number of samples of each size fraction as specified by the Principal Investigator. Each sample shall weigh ~10 g.

Step 8) Label the samples as directed in the Test Plan.

Step 9) If the sample requirements as specified by the Principal Investigator are not met, repeat Steps 3 through 8 using the largest size fuel fraction.

Step 10) Transfer the samples to the 327 Building "I" Cell.

6.4 Characterization of Pretest Sample

Step 1) Choose a minimum of three fragments with cylindrical curvature that indicates they were adjacent to the cladding and also a minimum of three fragments with all fractured surfaces, as specified by the Principle Investigator.

Step 2) Mount two cylindrical fragments with the cylindrical face perpendicular to the polished plane and two fractured fragments in any orientation. Prepare polished and etched specimens for grain size and porosity determination. The extent of examination, number of photographs, and times for inspection of mount shall be specified by the Principle Investigator.

Step 3) Examine at least one fragment of each type in the SEM to determine the fracture morphology. The Principal Investigator is to be present during this examination.

Step 4) Set aside ~50 mg of fuel from each size fraction in a plastic vial.

Step 5) Conduct x-ray diffraction analysis in accordance with Procedure HTA-3-3 (latest revision) for solids analysis on a sample of fuel specified by the Principal Investigator. Conduct electron diffraction analysis on this sample if specified by Principle Investigator.

Step 6) Conduct SEM and ceramographic examinations of all size fractions of powdered fuels in accordance with Procedure HTA-3-1, to determine surface morphology of grains and particle size distribution.

Step 7) Determine ratio of ^{137}Cs to ^{155}Eu for each size fraction by measuring 0.662 MeV and 1.274 MeV gamma intensity, respectively, using standard gamma-spectroscopy methods for fuel in accordance with Procedure HTA-4-9.

Step 8) Select fragments of fuel to be shipped to 325 Building for burnup analysis as directed by Principal Investigator. Burnup shall be determined in accordance with Procedure HTA-4-25.

6.5 General Procedures and Recordkeeping

- Deviations from the test plan, test matrix, or other programmatic documentation shall be initiated by memo instructions from the Principal Investigator after consultation with the Task Leader.
- Emergency deviations shall be performed by the experiment operator in a manner to preserve the integrity of the experiment as much as possible. A written description of the emergency deviation shall be entered into the Laboratory Notebook, and a copy of the description shall be sent to the Principal Investigator and the Task Leader.
- The following information shall be recorded in the Laboratory Notebook(s): Fuel sample identification, photographs of samples, number of fragments in sample, sample characterization and preparation details and techniques, x-ray diffraction results, SEM results, and ceramography results.
- Equipment calibrations shall be maintained on schedule and shall be recorded in the Laboratory Notebook.

7.0 QUALITY ASSURANCE

Instrument calibrations, experimental procedures, etc. will be carried out in accordance with BQA-1-1, which is the Quality Assurance Program Plan for this procedure.

8.0 REFERENCES

- ANSI/ASME NQA-1, Quality Assurance Program Requirements for Nuclear Facilities, current edition.
- HEDL-WHAN-M-8, Radiation Protection Procedures.
- HEDL-WHAN-M-11, Industrial Safety Manual.
- HEDL-MG-100, Quality Assurance Manual.
- BQA-1-1, "Quality Assurance Program Plan."
- HTA-1-1, "Data and Sample Control for Hydrothermal Testing."