

**PNL TEST PROCEDURE**

**TITLE: SFO-1-2, MEASUREMENT OF SPENT FUEL OXIDATION USING A DRY BATH HEATING SYSTEM**

**1.0 APPLICABILITY**

This procedure provides a method for 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. This procedure applies to the staff members of Pacific Northwest Laboratory who are performing oxidation measurements with a dry bath heating system. These people are responsible for performing oxidation measurements with a dry bath system and for post-test submission of fuel samples for x-ray diffraction, SEM analysis, and ceramographic evaluation.

Specific parameters for individual test runs that are performed according to this test procedure shall be specified in written test instructions. A copy of these instructions shall be attached to the Laboratory Record Book. The Task Leader for Spent Fuel Oxidation shall approve all test instructions.

**2.0 DEFINITIONS**

- **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.
- **Dry Bath System** - The dry bath and its associated atmosphere and temperature control systems.
- **Dry Bath Bank** - Set of dry baths that operate at the same dewpoint.
- **Atmosphere Delivery System** - Apparatus to measure and provide to the dry bath the desired atmosphere.
- **Balance Bubble** - Compartment adjacent to "I" Cell where weighing is conducted.

|                    |  |              |  |                    |  |         |  |
|--------------------|--|--------------|--|--------------------|--|---------|--|
| Concurrence        |  | Date         |  | Approved           |  | Date    |  |
| NA                 |  |              |  | <i>[Signature]</i> |  | 8-15-88 |  |
| Prepared by        |  | Date         |  | QAD Concurrence    |  | Date    |  |
| <i>[Signature]</i> |  | 8/15/88      |  | B. OPIE BARNES     |  | 8/16/88 |  |
| Procedure No.      |  | Revision No. |  | Effective Date     |  | Page of |  |
| SFO-1-2            |  | 2            |  | 08/16/88           |  | 1 of 10 |  |

## PNL TEST PROCEDURE

### 3.0 EQUIPMENT AND MATERIALS

#### 3.1 Equipment

- Balance, hanging weight, four-place or better
- Weight, 10-g, standard NBS traceable
- Crucibles, Ni/Cr, numbered, with Chromel bails
- Dry bath, Techne Model DB-3H, modified, with aluminum heat transfer blocks
- Thermocouples, Type K, initially calibrated
- Lids for dry baths
- Heating tape, flexible, with controllers
- Regulators for air cylinders with low delivery pressure
- Flowmeters, variable area, with control valves
- Water bath, capable of 90°C, equipped with water level control
- Piercing humidity and temperature probe and heated sensing chamber, HUMICAP Model HMP32
- Datalogger or data acquisition system, 30-channel or better
- Humidity probe calibrator, HUMICAP Model HMK-11
- Laboratory Record Books (LRB)
- Cask for transporting fuel specimens between cells in 327 Building and between 327 and 325 Buildings
- Air pump, low-pressure
- Block to hold vial, Petri dish, and crucible while unloading or loading samples
- Crucible protector.

#### 3.2 Materials

- Turkey Point PWR spent fuel samples, from Fuel Assembly B17, prepared according to Procedure SFO-1-1

| Procedure No. | Revision No. | Effective Date | Page | of |
|---------------|--------------|----------------|------|----|
| SFO-1-2       | 2            | 08/16/88       | 2    | 10 |

# PNL TEST PROCEDURE

- BWR and PWR spent fuel samples, as supplied by MCC, prepared according to Procedure SFO-1-1
- Dry air, with dewpoint less than  $-55^{\circ}\text{C}$
- Tap water.

## 4.0 RESPONSIBLE STAFF

### 4.1 Principal Investigator

The Principal Investigator is responsible for overseeing and assisting with the Dry Bath Spent Fuel Oxidation tests and for making and documenting appropriate modifications to the procedure as occasion arises. All 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 is maintained in calibration, for maintaining all records, for reviewing the test data, and for ensuring their quality.

### 4.2 Laboratory Technician

The laboratory technician shall perform the actual test measurements as directed by and occasionally assisted by the Principal Investigator. He shall maintain the continuity of the measurements, make necessary adjustments to the control equipment, and periodically record pertinent run parameters and sample weights.

### 4.3 Hot Cell Technicians

The hot cell technicians shall provide spent fuel specimens when required for test measurements. They shall perform the actual test measurements as directed by the Principal Investigator. They shall appropriately dispose of spent fuel materials that are no longer needed for measurements or analysis. They shall also be qualified radiation workers.

### 4.4 Task Leader

The Task Leader shall be responsible for checking that testing is carried out according to the approved Dry Bath Spent Fuel Oxidation test matrix, for making changes to the test matrix with the concurrence of the sponsor, approving test instructions that supplement this test procedure, and for integrating test results into the overall oxidation program findings. The Task Leader and the Principal Investigator may be the same person.

|                          |                   |                            |           |          |
|--------------------------|-------------------|----------------------------|-----------|----------|
| Procedure No.<br>SFO-1-2 | Revision No.<br>2 | Effective Date<br>08/16/88 | Page<br>3 | of<br>10 |
|--------------------------|-------------------|----------------------------|-----------|----------|

# PNL TEST PROCEDURE

## 5.0 PROCEDURE

### 5.1 Operation by Dry Bath Systems

#### 5.1.1 Connection and Setup of System

Dry Baths 2, 3 and 4 can operate with atmospheres that have dewpoints up to +80°C. Dry Baths 1 and 5 through 9 can operate with atmospheres that have dewpoints up to room temperature. Dry Baths 1, 5 and 9 form one bank. Dry Baths 6, 7 and 8 form a second bank. Dry Baths 2, 3 and 4 form a third bank.

The Laboratory Technician shall perform all the following steps of this section (5.1.1).

- Step 1) Connect the specified atmosphere delivery system (i.e., moist air or dry gas) to the desired dry bath bank at the 0.25-in. tube connection below the flowmeters.

#### Dry Gas Delivery System

- Step 2) With delivery valve closed, open cylinder, set cylinder delivery pressure to ~10 psig, and open flow valves to obtain gas flow of 25 (15) cc/min.
- Step 3) Record delivery pressure and flow in the LRB. NOTE: If cylinder pressure is below 100 psig, close cylinder, close delivery valve, replace cylinder, and record the new cylinder identification.
- Step 4) Repeat Steps 2 and 3 if cylinder has been changed.
- Step 5) Close cylinder until it is required for testing.

#### Moist Air Delivery System

- Step 6) Fill water supply bottle with tap water.
- Step 7) Turn on wet bath liquid level control and allow wet bath to fill to predetermined level with distilled water. Liquid level control should be set to allow no more than 1/4-in. water level drop prior to refilling.
- Step 8) Close flowmeter valves.
- Step 9) Turn on heating tapes for external lines, cell port, and inside cell. Set voltage (according to chart provided) to obtain a temperature on the appropriate thermocouple that is ~10°C higher than the desired dewpoint.

|                          |                   |                            |           |          |
|--------------------------|-------------------|----------------------------|-----------|----------|
| Procedure No.<br>SFO-1-2 | Revision No.<br>2 | Effective Date<br>08/16/88 | Page<br>4 | of<br>10 |
|--------------------------|-------------------|----------------------------|-----------|----------|

## PNL TEST PROCEDURE

- Step 10) Turn on humidistat.
- Step 11) Turn on air pump.
- Step 12) Set temperature of water bath to provide desired dewpoint.
- Step 13) Open flow valves to get 25 ( $\pm 15$ ) cc/min.
- Step 14) Adjust wet bath temperature until humidistat shows correct dewpoint.
- Step 15) Turn off air pump and shut flowmeters. **NOTE:** After the setup is complete, do not open the flow valves unless the dry baths are at temperature.

### 5.1.2 Operation of Dry Bath Systems

The Hot Cell Technician shall perform all the steps of this section except where noted.

- Step 1) Weigh empty numbered crucibles to  $\pm 0.1$  mg. **NOTE:** Weight should be taken a minimum of five times with a zero point between each measurement and a 10-g standard before and after each set of weighings. The Laboratory Technician shall record all weights and zero points in the LRB.

**NOTE:** At this time, the numbered sample cans should be in the cell in the storage rack, and the numbered crucibles should also be in the cell.

- Step 2) Place crucible (designated by Principal Investigator) in the transfer block.
- Step 3) Take designated sample can from storage rack and place it in the clamp in the transfer block. Tighten the clamp by moving the lever.
- Step 4) Remove tape from sample can and remove lid.
- Step 5) Place funnel designated for sample type in the crucible.
- Step 6) Unlock clamp and pick up the can of fuel.
- Step 7) Pour fuel into the funnel and into the crucible.
- Step 8) Remove funnel.
- Step 9) Lift crucible by the bail and place in the transfer holder between the "I" Cell and the bubble. Transfer crucible to bubble. Close door between "I" Cell and bubble.

| Procedure No. | Revision No. | Effective Date | Page | of |
|---------------|--------------|----------------|------|----|
| SFO-1-2       | 2            | 08/16/88       | 5    | 10 |

## PNL TEST PROCEDURE

- Step 10) Weigh the filled crucible according to the protocol given in Step 1 of this section. The Laboratory Technician shall record data as in Step 1.
- Step 11) Place crucible in transfer unit. Open door between "I" Cell and bubble. Move transfer unit into "I" Cell.
- Step 12) Place crucible in designated dry bath position. The Laboratory Technician shall record sample number and dry bath location in the LRB.
- Step 13) Place crucible protector in place.
- Step 14) Repeat Steps 2 through 9 for all samples in the order given by the Principal Investigator. **NOTE:** The order of loading the dry bath can be changed by the Principal Investigator if recorded in the LRB.
- Step 15) Cover each dry bath and close clamps, if available, after the loading of each dry bath is complete.

The Laboratory Technician shall perform Steps 16-21 of this section.

- Step 16) Turn on dry baths and set to the temperature specified in the test instructions (according to calibration chart). Adjust controller to the specified temperature.
- Step 17) Record temperature on either datalogger or data acquisition system at maximum 10-minute intervals from the time the temperature is within 30°C of desired point until stabilization occurs. **NOTE:** Thereafter record at minimum one-hour intervals.
- Step 18) Turn on Dry Gas Delivery System to dry bath, if required (see Section 5.2.1), and record cylinder pressure and gas flow, OR
- Step 19) Turn on Moist Air Delivery System (see Section 5.1.1, Steps 6 - 15), if used. Ascertain that dry baths are at temperature before continuing in order to prevent condensation on the samples. Record date, time, and water bath temperature.
- Step 20) Turn on air pump and set flowmeter for 25 ( $\pm$ 15) cc/min. Record flow.
- Step 21) Manually check temperatures, humidity, and flow cylinder pressure at least once each working day.
- Step 22) The Hot Cell Technician shall remove empty cans from the hot cell.

|                          |                   |                            |           |          |
|--------------------------|-------------------|----------------------------|-----------|----------|
| Procedure No.<br>SFO-1-2 | Revision No.<br>2 | Effective Date<br>08/16/88 | Page<br>6 | of<br>10 |
|--------------------------|-------------------|----------------------------|-----------|----------|

## PNL TEST PROCEDURE

### 5.1.3 Interim Examination

- Step 1) The Laboratory Technician shall shut the flow valve and wait 30 minutes.
- Step 2) The Laboratory Technician shall shut off controller (using on/off switch) to desired dry bath and cool to room temperature. During cooldown, record temperatures at maximum 10-minute intervals until temperature drops at least 30°C. Record time in the LRB when temperature dropped 30°C.

The Hot Cell Technician shall perform the remaining steps of this section (5.1.3) except where noted.

- Step 3) Remove lid from dry bath. The Laboratory Technician shall record dry bath number in the LRB.
- Step 4) Remove crucible protector from crucible to be examined.
- Step 5) Remove crucible from dry bath and record identification. Place each crucible in transfer block and transfer to weighing bubble. NOTE: Close door between "I" Cell and weighing bubble once the transfer block is in the bubble. Only one crucible is to be out of the dry baths at any one time.
- Step 6) Record balance zero, place 10-g standard on balance, and weigh. Weigh standard three times, removing weight from balance each time. Record zero after third standard weight. The Laboratory Technician shall record zero readings in the LRB. NOTE: This step is to be conducted before and after weighing all the crucibles from any one dry bath. If the differences between the average of the initial and of the final standard weights is more than 0.1 mg after correction for zero drifts, the balance should be recalibrated and the crucibles reweighed.
- Step 7) The Laboratory Technician shall record the balance zero point in the LRB. Then hang the crucible on the balance. Record the crucible's weight. Remove the crucible and record zero point. Repeat this step at least six times.
- Step 8) Place crucible in transfer block. Open door between "I" Cell and bubble. Transfer the crucible to "I" Cell.

| Procedure No. | Revision No. | Effective Date | Page | of |
|---------------|--------------|----------------|------|----|
| SFO-1-2       | 2            | 08/16/88       | 7    | 10 |

## PNL TEST PROCEDURE

- Step 9) Reidentify crucible. Place crucible in dry bath. Replace crucible protector.
- Step 10) Repeat Steps 4 through 9 until all designated crucibles from bath are weighed.
- Step 11) After all designated crucibles are weighed, replace lid and clamp, if required.
- Step 12) The Laboratory Technician shall turn on dry bath controller (using on/off switch). Record the temperature as it stabilizes (Section 5.1.2, Step 17). Set flow to 25 ( $\pm$ 15) cc/min.

### 5.1.4 Test Termination

- Step 1) The Hot Cell Technician shall place numbered transfer cans in hot cell on storage rack.
- Step 2) The Laboratory Technician shall shut down the dry bath and weigh samples as directed in Section 5.1.3, Steps 1 through 10.

The Hot Cell Technician shall perform the remaining steps of this section (5.1.4).

- Step 3) Identify crucible when returned to "I" Cell. Then place it in transfer block.
- Step 4) Clamp numbered metal or glass transfer can (same number as crucible) on transfer block.
- Step 5) Remove lid from transfer can.
- Step 6) Dump contents of crucible into the can and cover the can. Presstape tab securely and unclamp transfer can.
- Step 7) Place transfer can and crucible on storage rack.
- Step 8) Repeat Steps 2 through 7 until all the samples specified by the Principal Investigator to be terminated have been placed along with the corresponding crucibles in the storage rack.
- Step 9) Transfer sample cans and crucibles out of the "I" Cell.
- Step 10) Clean crucibles with ethyl alcohol in an ultrasonic bath and then dry them.

| Procedure No. | Revision No. | Effective Date | Page | of |
|---------------|--------------|----------------|------|----|
| SFO-1-2       | 2            | 08/16/88       | 8    | 10 |

## PNL TEST PROCEDURE

- Step 11) Weigh crucibles on the outside of the cell on the same balance used for hanging weights and using the same procedure as in Section 5.1.2, Step 1.
- Step 12) Transfer indicated samples or parts of samples to appropriate cells for post-test examination (as specified by the Principal Investigator).
- Step 13) Retain remainder of samples in transfer cans until instructed (by Principal Investigator) to discard them.

### 5.2 Post-Test Procedures

Following the oxidation measurement, the fuel specimen or portions thereof may be submitted by the Laboratory Technician for ceramographic examination, x-ray diffraction analysis, transmission electron microscopy (TEM), ion microprobe or other analyses

The appropriate analytical laboratory staff shall perform the following analyses if requested:

- The ceramographic sample preparation shall conform to the current revision of Procedure SFO-1-1, "Sample Preparation for Spent Fuel Oxidation Testing Using a Dry Bath Heating System."
- The x-ray diffraction analysis shall conform to the current revision of Procedure HTA-3-3, "Solids Analysis: X-Ray Diffractometry."
- The SEM examination shall conform to the current revision of Procedure HTA-3-1, "Solids Analysis: Scanning Electron Microscopy."
- The TEM examination shall conform to the current revision of procedure HTA-3-2, "Solids Analysis: TEM/STEM."
- The ion microprobe examination is experimental in nature and not subject to formal procedural controls at this time.

### 5.3 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 consulting 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 shall be entered into the LRB and a copy of this description sent to the Principal Investigator and the Task Leader.

| Procedure No. | Revision No. | Effective Date | Page | of |
|---------------|--------------|----------------|------|----|
| SFO-1-2       | 2            | 08/16/88       | 9    | 10 |

## PNL TEST PROCEDURE

- The following information shall be recorded in the LRB(s): fuel sample identification, photographs of samples, number of fragments in sample, initial sample and crucible weight, interim sample weights, flowmeter readings, temperature and humidity measurement from humidity probe, temperature and humidity measurement details and techniques, calculations based on data from measurements, x-ray diffraction results, SEM results, ceramography results, and general observations and conclusions. Written test instructions shall be attached to pages of the LRB.
- Maintain equipment calibrations and record them in the LRB.
- Start up instructions shall be recorded in the LRB.

|                          |                   |                            |            |          |
|--------------------------|-------------------|----------------------------|------------|----------|
| Procedure No.<br>SF0-1-2 | Revision No.<br>2 | Effective Date<br>08/16/88 | Page<br>10 | of<br>10 |
|--------------------------|-------------------|----------------------------|------------|----------|