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K-Basin Spent Nuclear Fuel Characterization Data Report

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November 1995

Prepared for the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

Pacific Northwest Laboratory
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Richland, Washington 99352

Summary

A "path forward" strategy has been developed for moving the K Basin spent nuclear fuel (SNF) to an interim storage facility (Fulton 1994). This "path forward" includes a process for SNF conditioning (or passivation) recommended in the Independent Technical Assessment (ITA) (Devine 1994). Information needed to support the development of the conditioning process must be obtained from testing activities conducted on fuel samples from the K Basins.

The testing performed to provide initial characterization information on the physical condition and corrosion characteristics of the 105-K West Basin SNF, and to provide SNF characteristics that helped select test specimens for examination and the drying/conditioning response of the SNF material includes the following:

- Visual examinations of two of the three SNF elements shipped to Postirradiation Testing Laboratory (PTL) in the hot cells with the aid of cameras, telescope, and by videotaping through a cell window.
- Weight and density measurements of SNF element SFEC5,4378, sectioned into specimens for metallographic examinations and drying/conditioning testing to ascertain corrosion-induced swelling and material loss.
- Metallographic examinations to identify corrosion products and determine the nature of the oxide layer formed on the specimens for [during?] furnace testing.

The basic conditioning process recommended in the ITA report and the modified process that uses the ITA heating cycles (but involves cold and hot vacuum steps followed by the ITA passivation step) were applied to sectioned specimens from the mid-length of K-Basin SNF element SFEC5,4378.

The results of the visual and optical examinations, the density and the weight measurements, and the furnace testing of cut specimens from the mid-length of element SFEC5,4378 are as follows:

- With the exception of the badly damaged end of the SNF element SFEC5,4378, the remaining Zircaloy™ cladding of the fuel element showed no cladding penetration. About 100° to 120° sector of the fuel in the damaged end has been lost and the surface of the remaining fuel shows large cracks. The identification number on the undamaged end cap is illustrated below.

Summary Figure 1.1. Endcap of SNF Element SFEC5,4378 Showing Identification Numbers

Summary Figure 1.2. Endcap of SNF Element SFEC8,1990 Showing Identification Numbers

- The damaged end of the SNF element SFEC8,1990, had a chip of cladding removed. The surface of the chipped end shows a crack and a deposit of a white corrosion product. The element had only three of the six original shoes remaining. The spot welds of the broken shoes were visible. The identification numbers on both end caps were observed. The undamaged end cap has the numbers 9 and 5 on it; the other end cap with the chip had identification numbers illustrated below.
- The measured density the SNF element SFEC5,4378 was 16.88 g/cc and is not significantly different from the estimated value of 16.78 g/cc. Comparison of the weight of the element in air, 17015.4 g, with the original weight of N Reactor outer element Mark IV(E), 17040 g, indicates only a small amount (i.e., about 24.6 g) if any, of the element was lost due to the cladding breach at one end.
- The scanning electron microscope (SEM) coupled with EDAX (Energy Dispersive Analysis for X-ray) and X-ray diffractometer (XRD) sludge sample examinations filtered from the water that had been in the single fuel element canister (SFEC) 5 and 8, indicated oxides of uranium (e.g., UO_2 and U_4O_9) as the main chemical phases in the sludge; minor constituents consisted of iron oxide(s) and silica. There were no uranium hydride and metallic uranium inclusions in the sludge. The particle sizes ranged from sub-micron to greater than 100 microns.
- Optical microscopy examinations of the transverse and the longitudinal surfaces of the specimens from the mid-length of element SFEC5,4378 element showed inclusions of two different phase structures in the uranium matrix. One of the structures was a uranium-carbide precipitate; the other was probably uranium hydride. There is evidence of lenticular zirconium hydride precipitate in the Zircaloy cladding.
- The results of the furnace testing indicated evolution of moisture and hydrogen from the fuel specimens during the drying cycles. The oxygen pickup by the specimens at 250°C in 2% oxygen and 98% argon mixture during the passivation step translated to a calculated oxide thickness of about 40 microns. For the total amount of oxide formed at this temperature, about 50% of it was lost by spalling. Analysis of the spalled-off oxide by XRD indicated the formation of mainly U_4O_9 chemical phase.
- The results of the "cold vacuum drying" (CVD) followed by "hot vacuum drying" (HVD) showed that a large fraction of the added water came off during the first 150 minutes of the CVD cycle. The amount of moisture left in the specimen after the CVD step and desorbed during the HVD step was insignificant.

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We would like to extend our appreciation to the Postirradiation Test Laboratory staff for their relentless efforts in performing the visual examinations, cutting the specimens for the destructive tests and the installation of the drying/conditioning furnace system. Our thanks go to Ray Bell for his ardent efforts to put together the furnace system and to George Salazar for providing the optical photomicrographs. Thanks to Craig Buchanan, Evan Jenson, Jim Young for providing the scanning electron microscope, X-ray diffractometer and Energy Dispersive Analysis for X-Ray results.

Acronyms and Initialisms

BEI	Back-Scattered Electron Image
BSE	Back-Scattered Electron
CVD	Cold Vacuum Drying
EDAX	Energy Dispersive Analysis for X-ray
GC	Gas Chromatograph
HVD	Hot Vacuum Drying
ICDD	International Centre for Diffraction Data
ITA	Independent Technical Assessment Group
LRB	Laboratory Record Book
MCO	Multi-Canister Overpack
NIST	National Institute for Standard Testing
PNL	Pacific Northwest Laboratory
PTL	Postirradiation Testing Laboratory
ppm	parts per million by weight
XRD	X-Ray Diffractometer
SEM	Scanning Electron Microscope
SFEC	Single Fuel Element Canister
SNF	Spent Nuclear Fuel
SEI	Secondary Electron Image
TI	Test Instruction
WHC	Westinghouse Hanford Company

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1.0 Introduction

The spent nuclear fuel (SNF) project characterization activities will be furnishing technical data on SNF stored at the K Basins in support of a pathway for placement of a "stabilized" form of SNF into an interim storage facility. This report summarizes the results so far of visual inspection of the fuel samples, physical characterization (e.g., weight and immersion density measurements), metallographic examinations, and controlled atmosphere furnace testing of three fuel samples shipped from the KW Basin to the Postirradiation Testing Laboratory (PTL).

Data on sludge material collected by filtering the single fuel element canister (SFEC) water are also discussed in this report.

Visual examinations and physical characterization (e.g., weight and immersion-density measurements) are prerequisite activities in support of sectioning the SNF samples for destructive tests and identifying gross visible failures such as materials loss and/or fuel swelling. The metallographic examinations will identify SNF phase structure (fuel and cladding) and measure oxide thickness and the adherence of the oxide layer to the substrate uranium for "conditioned" samples. The furnace tests, which measure the drying characteristics, dehydriding, and oxygen pick-up by fuel samples, will provide data that will be used to establish the fuel performance parameters when subjected to the pathway conditioning process, that is, the proposed Independent Technical Assessment (DOE 1994) process and/or the modified version that includes the cold vacuum drying.

The applicability of the data collected to the path-forward decision schedule suggests that the data can help to establish process-control parameters. The objective of these examinations is the measurement of the response of these fuel samples to the proposed ITA conditioning process and modifications to that process (e.g., cold vacuum drying). The areas of technical uncertainty are as follows:

- SNF material drying behavior
- Hydride decomposition behavior (if present in the material)
- Formation of a passivated oxide film on exposed uranium metal and/or hydride
- Combustion/pyrophoric behavior of the pre- and post-conditioned spent nuclear fuel.

The limited scope represented by these samples and the limited data obtained from few experimental runs restrict the use of data in this report in support of path-forward decisions. However, the data provided in combination with that available in the literature will help provide a basis for decisions.

The tests used to provide KW Basins SNF conditioning-process response information to support decisions on conditioning-process design and safety analyses are:

Optical microscopy analyses: Metallographic examinations of the SNF material will provide information concerning the type and quantity of corrosion products which have formed on the SNF

during storage in the KW Basin; the effect of the corrosion on the mechanical integrity of the SNF assemblies; the approximate relative amounts of metal, oxide, and hydride to be expected to be loaded into multi-canister overpacks (MCOs) in the basins; and the integrity of the oxide layer formed on the metal surfaces by the conditioning process.

Conditioning (i.e., drying and oxidation kinetics) tests: Conditioning test results on the SNF samples will support decisions concerning the conditioning of the SNF required for long-term dry storage by providing water removal and oxygen uptake data. The tests will provide furnace data concerning the dewatering, drying, and passivation of the as-received K Basin SNF specimens in the conditioning furnace, thereby enabling evaluation of dewatering and/or drying as a step in the conditioning of SNF material. The tests will also provide furnace data concerning oxidation as a step in forming passivated fuel surfaces and/or chemically stable oxides.

Sludge Analyses: Sludge-like material (generated as a result of loading the SNF elements into the K Basins and subsequently transported with the SNF samples) was collected for analyses using the X-ray diffractometer (XRD) and scanning electron microscope (SEM), coupled with EDAX for elemental composition, chemical phase structure, and particle size distribution. The results of these examinations are required to support the choice of sludge-recovery processes and packaging alternatives, and the selection of viable alternatives for SNF drying and conditioning.

2.0 Tests Performed

2.1 Visual Examinations

The spent nuclear fuel (SNF) examinations involved visual inspection with still photography and videotape. Videotaping started when the SNF was unloaded and continued through sectioning of test specimens.

The following activities were performed and recorded during visual examination of the SNF samples:

- Atmospheric conditions (temperature, pressure, and humidity) of F-cell in which the visual inspection was performed were recorded
- The numbering code on the fuel element was identified and recorded
- A two-stage filtration of sludge contained in the SFEC water was done, first through a 106-micron sieve followed by filtering through a 24-micron filter paper
- Full-size photographs of SNF elements SFEC5,4378 and SFEC8,1990 were taken at 0° orientation and at every 90° rotating angle
- Photographs of both ends of the SNF elements SFEC5,4378 and SFEC8,1990 were taken
- SNF elements SFEC5,4378 and SFEC8,1990 were examined for outstanding features, such as corrosion sites, cracks and visible pin holes, swelling, and/or breakage in the fuel element
- Selected magnified photographs of visible features were taken.

2.2 Physical Characterization

Element SFEC5,4378 was examined physically (weight and immersion-density measurements) to estimate the degree of swelling and/or deformation caused by degradation during storage in the KW Basin. The measurements were done in F-cell using a Mettler high-capacity balance installed on top of the cell (all weighings were performed by suspension below the balance using a wire attachment). The readability of the balance was 0.1 g (with reproducibility of 0.1 g). The SNF element was held by a wire attachment to the balance for the dry weight measurement; a six-inch-diameter container located under the balance was filled with de-ionized water for the immersion-density measurement. A 304 stainless-steel replica of the pre-irradiated N Reactor fuel element dimensions was fabricated and calibrated (traceable to NIST standards) by the Westinghouse Hanford Company (WHC) Standards Laboratory as an accurate standard for determining the density of the SNF samples. The water reservoir temperature was monitored using a Type-K thermocouple.

The procedures listed below were used to measure the SNF sample density.

- The SNF was removed from the SFEC and drip-dried before it was weighed in air. The air temperature in the cell was recorded.
- The 304 stainless-steel standard was weighed in air.
- The reservoir tank was filled with de-ionized water and the water temperature was measured.
- The 304 stainless-steel standard was weighed in the de-ionized water.
- The SNF sample was weighed in the de-ionized water.

2.3 Optical Microscopy

2.3.1 Test Specimen Selection and Cutting

Selection of test specimens for optical metallography was based on the visual inspections and physical characterization activities discussed in Sections 2.1 and 2.2.

Using a carbide blade, a fuel element section approximately 2.5-cm thick was cut perpendicular to the longitudinal axis of element SFEC5,4378 at selected locations. Cutting was done in a partially inert enclosure (argon) to reduce the likelihood that the fuel element would react with air. The sectioning followed Test Instruction SNF-CT-003 (see the Appendix) in F-cell air environment with argon-vortex cooling applied at the cutting front.

The one-inch ring cross-section labeled SFEC5,4378-S2 was further sectioned longitudinally using a milling machine with a carbide blade. The cuts were also dried with argon-vortex cooling. Eight 22.5° sector specimens and two 90° sectors were prepared. The specimens were numbered as follows: SFEC5,4378-S2-A, SFEC5,4378-S2-B, SFEC5,4378-S2-C, etc., (SFEC5 for SFEC number; 4378 for K Basin canister number; S2 for sectioned piece and A,B,C, etc., for degree of the sector clockwise [as seen in sectioning SFEC5,4378-S2 diagram in TI SNF-CT-003: A for 22.5°, B for 45°, etc.]). The approximate dimensions and configuration of the specimens are shown on the cutting diagram (see TI SNF-CT-003 in the Appendix).

Figure 2.1. Cut Surface of SNF Element SFEC5,4378. The surface shows the four cutting stages.

Figure 2.2. Schematic of Single Fuel Element Canister Water-Filtering System

2.3.2 Specimen Preparation and Examination

Two of the 22.5°-sector specimens (SFEC5,4378-S2-A and -S2-C) were mounted for metallographic examination, one in cross-section (SFEC5,4378-S2-A) and the other longitudinally (SFEC5,4378-S2-C).

The samples were polished following the procedure in "The Effect of Cooling Rate on the Nucleation and Growth of Beta-Uranium Hydride in Metallic Uranium," (Gardner and Riches 1956). First, the specimens were ground progressively through 180-, 240- and 600-grit silicon-carbide (SiC) abrasive paper.

Next, the specimens were ground by 800- to 1200-grit SiC abrasive paper lubricated with Hyprez OS Type IV (paraffin in kerosene). The specimens were then polished on "gold-polishing" cloth (e.g., Glennel Corp. gold-label polishing cloth), in the following sequence, using the materials listed below.

- 4-8 microns diamond paste suspended in Hyprez OS Type IV lubricant
- 1-2 microns diamond paste suspended in Hyprez OS Type IV lubricant
- 0.05-micron Buehler micropolish II or equivalent, saturated with a 2% solution of chromic acid on a microcloth (or silica slurry on the microcloth) using the rotor- or the Vibra-polisher for 30 seconds to 5 minutes.

The polished surfaces were examined by the metallograph for a series of photomicrographs, as follows:

- Fuel-surface structure with potential magnifications in the range of about 50 to 750X
- Fuel/cladding interface with potential magnifications in the range of about 50 to 750X
- Fuel edge with potential magnifications in the range of about 50 to 750X.

2.4 Single Fuel Element Canister Sludge Examinations

The sludge that was contained in SFEC 5 and 8 water was filtered in two stages, first through a three-inch-diameter metal sieve with a 106-micron opening size. This was followed by filtering through a filter paper with a 24-micron opening size, using the system shown in Figure 2.2. The collected material was analyzed with Scintag PAD V™ X-ray diffraction (XRD) for crystalline phases present in the sludge. Some samples were also analyzed by scanning electron microscopy (SEM) coupled with EDAX (energy-dispersive analysis for X-ray).

The particles that were retained on the metal sieve (Figures 2.3 and 2.4) were transferred into a petri dish and sent to the XRD and SEM laboratories for examinations. The sieve was then flushed with de-ionized water onto a 24-micron filter paper to collect any leftover sludge material that could not easily be transferred into the petri dish. Last, the filter paper from the second-stage filtering was sampled and analyzed by the XRD and SEM.

Figure 2.3. Three-Inch Stainless-Steel Sieve Showing the Particulates Collected from SFEC 5 Water

Figure 2.4. Three-Inch Stainless-Steel Sieve Showing the Particulates Collected from SFEC 8 Water

2.7

In addition to the filtrate, the sludge samples collected were also analyzed by XRD and SEM. Sludge sample identification is as follows:

Sample 5: Particles from SFEC #5, retained on sieve and transferred to petri dish

Sample 5S: Filter paper from flushing the sieve

Sample 5F: Filter paper from second filtering of SFEC 5 water

Sample 8: Particles from SFEC 8, retained on sieve and transferred to petri dish

Sample 8S: Filter paper from flushing the sieve

Sample 8F: Filter paper from second filtering of SFEC 8 water.

XRD Sample 5 was pulverized using a boron-carbide mortar and pestle, with Collodion™ and amyl acetate added, and examined on a glass slide. The total amount of sample was estimated at <2 mg. Samples 5S, 5F, 8S and 8F were examined as received on the filter paper, mounted on a glass slide with Collodion/amyl acetate. Sample 8 was not examined due to insufficient sample volume. A blank slide of the filter paper was also examined to determine the contribution of the filter paper to the observed diffraction pattern, and to aid in deleting that contribution in the resulting background-subtracted patterns.

The running parameters were 0.02° 2-theta step size (except Sample 5, which had 0.3°); the count time was 25 seconds per step; the range was 5 to 65°. X-ray tube parameters were 45 Kv, 40 mA, anode copper. The slits used were 4, 2, 0.5 and 0.3 mm wide.

The XRD analysis was performed to technical procedure PNL-ALO-268, "Solids Analysis, X-Ray Diffraction." The calibration data were kept in PNL LRB 53224; the daily calibration checks showed the diffractometer was within calibration limits at all times.

2.5 Drying and Conditioning Tests

The hot-cell furnace test activities were performed in the PTL G-cell. A schematic of the furnace system used for the conditioning tests, its inlet gas control capabilities, and its off-gas analyses capabilities are shown in Figure 2.5. Specimen preparation and testing followed requirements in Test Instructions (TI) SNF-CT-005 (for basic ITA conditioning process) and SNF-CT-007 (for cold vacuum drying plus passivation process) (see Attachment in the Appendix).

Two specimens were used in the first two runs of the "conditioning test": one specimen for post-conditioning metallography and the second for post-conditioning archives. Both specimens were individually weighed before and after the test.

Figure 2.5. Schematic of the Furnace System

Specimens SFEC5,4378-S2-H, J, B, I, G, and D were selected from the stored, cut batch. The specimens were stored in a container filled with high-purity argon. The selected specimens were transferred from their temporary storage in E-cell to G-cell, the location of the furnace apparatus. The TIs describe the furnace emplacement configuration of the test specimens.

The basic heating cycle that was used for the conditioning cycle (basically the cycle proposed in the ITA study) consisted of the following:

- A fuel drying step in which the SNF is dewatered (free water removal) either at 373 K under dry argon for approximately 10 hours or at 323 K in vacuum (cold vacuum drying) (10 hours), then dried at 573 K under dry argon (or vacuum) for approximately 24 hours (to remove water of hydration and partially decompose any uranium hydride present).
- A fuel passivation step, in which the fuel is exposed to a 98% argon-2% oxygen atmosphere at temperatures ranging from 423 K to 523 K for about 10 hours, to create an adherent, passive oxide film on any exposed uranium-metal surfaces. In the testing runs for this report, this step was conducted at near atmospheric pressure.

The off-gas from the conditioning tests was monitored for composition (e.g., water, hydrogen, oxygen) and the total water release was measured.

One of the two specimens used in the testing was fitted with a Type-K thermocouple to measure the specimen's temperature. The furnace was purged at ambient temperature with high-purity argon and dried to a moisture level of about 1 ppm, after which the time-temperature-atmosphere conditioning schedule instructed in the TIs was imposed. The moisture trap shown in Figure 2.5 was configured to have only one of the two parallel traps valved-in during the drying part of the conditioning cycle. At the end of the drying step, this trap was valved-off and the other trap valved-in. The first trap was weighed for integral analysis of the water removed from the specimens during the drying operation. Likewise, at the end of the conditioning part of the cycle the second trap was valved-off, removed for analysis, and replaced.

A continuous measurement of water emission from the specimens during the conditioning cycle was provided by a moisture monitor placed in the off-gas train of the furnace, between the furnace and the moisture trap. This instrument measured the water in the off-gas by measuring the dew-point of the gas down to -110°C , corresponding to a detection limit of 1-10 ppm.

Upon completion of the conditioning cycle, the two specimens were removed from the furnace for visual examination and then stored in high-purity argon for later analysis.

3.0 Data and Discussions

3.1 Visual Examinations Data

The videotape records are listed in Table 3.1. Two elements (SFEC5,4378 and SFEC8,1990) are shown in Figures 3.1 through 3.5. Figure 3.1 shows the top half-length of element SFEC5,4378 with the cladding damage at the end of the element. One of the clips that was welded to the outside element of N-Reactor fuel assembly is shown in this photograph. Figure 3.2 shows the full-length view of element SFEC5,4378. With the exception of the damaged end, the rest of the surface cladding of element SFEC5,4378 showed no cladding penetration. There were crud deposits underneath the clips and a few handling scratches on the cladding surface. Figure 3.4 shows a closer view inside the breached cladding of element SFEC5,4378 which indicated a loss of part of the fuel material at the breached cladding location.

Figures 3.4 and 3.5 show the inner N-Reactor fuel element SFEC8,1990. The dented or chipped end of the element is shown in Figure 3.4. A closer view of the damage on this element and part of the element identification code is shown in Figure 3.5.

3.2 Weight and Density Measurements

Weight and immersion density measurements of element SFEC5,4378 are listed in Tables 3.2 and 3.3. Each measurement was performed three times; the average values were used to calculate the sample density.

3.2.1 Discussion

Given a density for metallic uranium of about 18.9 g/cc and a Zircalloy™ density of 6.36 g/cc, the density of SNF element SFEC5,4378 element can be estimated using the following equation:

$$\rho_{SNF} = \frac{W_U + W_{Zr}}{W_U/\rho_U + W_{Zr}/\rho_{Zr}} \quad (3.1)$$

where ρ_{SNF} is the density of the SNF fuel element;
 W_U is the weight of the uranium component of the SNF fuel element;
 W_{Zr} is the weight of the Zircalloy cladding;
 ρ_U is the density of uranium; and
 ρ_{Zr} is the density of the Zircalloy.

SFEC5,4378 is an N-Reactor Mark IV(E) outer fuel element. The weights of uranium and Zircalloy™ cladding for an N-Reactor Mark IV(E) element are 15.95 kg and 1.09 kg, respectively (Abrefah 1994). Using this data and the equation above yields an N-Reactor Mark IV(E) element density of 16.78 g/cc. Comparison of the measured density, 16.88 g/cc, with the estimated value

of corrosion products; that is, either the oxides of uranium and/or uranium hydride, which could have changed the density, should not be present in significant quantities.

Comparison of the weight of the element in air (column 4 of Table 3.3), 17.015 kg, with the original weight of the N-Reactor Mark IV(E) outer element, 17.04 kg, indicates only a small amount (i.e., about 24.6 g) if any, of the element has been lost due to the cladding breach at one end (Figure 3.3).

Table 3.1. Videotaping Records of SNF Characterization Activities

Videotape Number	Recorded Date	Visual Examinations Description
1	3-30-95	Cask off-loading. Cask submersion into PTL basin.
2	3-30-95	Cask lid removal. SFEC transfer to A-cell and back to PTL basin for storage.
3	4-5-95 to 4-6-95	SFEC 5 opening in F-cell. Observation of the contents of SFEC 5 from the top. Emptying the SFEC's water into the immersion tank. View of both ends and the damaged areas of SNF element SFEC5,4378. Return of the SNF element into the SFEC and refilling it with de-ionized water.
4	4-11-95 to 4-12-95	Closer look at the damaged end of SNF element. Horizontal view of SNF element laying in a cradle.
5	4-12-95	Views of SNF element SFEC5,4378 on a vertical hanger at different orientations. Coverage of the filtering of SFEC 5 water. Viewing the inside of the 106-micron sieve after the filtration. Viewing the particulates in the planchet/petri dish.
6	4-18-95 to 4-19-95	Lid removal of SFEC 8 and first view of SNF element SFEC8,1990. Pouring the water in SFEC 8 into the immersion density tank through the 106-micron sieve. Monitoring the empty SFEC with the fuel element. Viewing the particulates collected on the 106-micron sieve. End view of the SNF element while in the SFEC, mounted on the milling machine table.
7	4-19-95	Removing SNF element SFEC8,1990 from the SFEC. Viewing the element in the horizontal cradle. Viewing the damaged end of the SNF element. Return of the SNF element into the SFEC and filling the SFEC with water. End view and side view of the apparent circumferential crack region of the element.
8	4-20-95	Coverage of the filtering of SFEC 8 water through the 24-micron filter paper. Vertical viewing of the SNF element SFEC8,1990 at 0, 90, 180 and 270° orientations.
9	5-4-95 to 5-9-95	SNF element SFEC5,4378 orientation and cross-cut markings. Set-up for sectioning and the first attempted cut with a diamond-impregnated blade. First cross-cut at about 14 inches from the bottom of the element, using a carbide blade.
10	5-10-95 to 5-12-95	Completion of the first cut for specimen SFEC5,4378-S2 at ~14 inches from the bottom of the element. Beginning of the second cut of specimen SFEC5,4378-S2 at ~13 inches from the bottom of the element. Completion of sectioning for specimen SFEC5,4378-S2. Notching of "tic" marks on the top ends of specimens SFEC5,4378-S2-A to J. Cut through 0° and 180° of specimen SFEC5,4378-S2.
11	5-15-95 to 5-24-95	Cutting the eight 22.5°-sector specimens and two 90°-sector specimens.

Figure 3.1. At About the Top 12-Inch Length of SNF Element SFEC5,4378 at 90°Orientation

Figure 3.2. Full Length Photograph of SNF Element SFEC5,4378 at 270° Orientation

Figure 3.3. Angle View of the Damaged End of SNF Element SFEC5,4378

Figure 3.4. Axial View of the Top 11-Inch of SNF Element SFEC8,1990

Figure 3.5. View of Damaged End of Element SFEC8,1990 Tilted at About 40°

Table 3.2. Standardization Measurements

SFEC Number	Element ID Number	Measurement Number	Weight of Standard in Air (g)	Weight of Holder Immersed (g)	Weight of Standard Immersed (g)	Weight of Standard in Water (g)	Temperature of the Water (°C)	Calc. Density of Water (g/cc)	Volume of standard (cm³)
5	SFEC5,4378	1	7866.3	0.0 ± 0.2	6862.4	6862.4	26.92		988.7
		2	7865.8	0.0 ± 0.2	6862.5	6862.5	-----		988.7
		3	7866.0	0.0 ± 0.2	6862.4	6862.4	26.98		988.7
Average Values			7866.0	0.0 ± 0.2	6862.4	6862.4	26.95	1.015	

Theoretical Density of Water, $\rho_{wt} = 0.99654$ g/cc

Table 3.3. SNF Element Measurements

SFEC Number	Element ID Number	Measurement number	Weight of Element in Air (g)	Temperature of Water (°C)	Weight of Element Immersed (g)	Weight of Element in Water	Density of Element
5	SFEC5,4378	1	17015.8	26.31	15991.9	15991.9	
		2	17014.8	-----	15992.0	15992.0	
		3	17015.4	26.28	15992.0	15992.0	
Average Values			17015.4	26.30	15992.0	15992.0	16.876

Units of Measurement: Weights (Kg); Temperature (°C); Volume (m³); and Density (Kg/m³). Note 1 g/cc = 10³ Kg/m³ and 1 cc = 10⁻⁶ m³

Hot Cell's Atmosphere: Temperature 37.4°C; Humidity 16.2%; Pressure Difference -0.63 in H₂O; Atm. Pressure 29.234 in Hg

3.3 Single Fuel Element Canister Sludge Examinations

3.3.1 X-Ray Diffractometer Data on the SFEC Sludge

Plots of all diffraction data obtained are shown in Figures 3.6 through 3.29. Figures 3.6 through 3.10 show plots of the raw data; no data manipulation has been applied. Figures 3.11 through 3.20 show plots with the background subtracted and the $K\alpha$ peak contribution removed. Stick figure patterns of the ICDD (International Centre for Diffraction Data) patterns are included in these figures to show the known patterns for identified phases of U_4O_9 (labeled $UO_{2.5}$ in plots) and UO_2 spectra. (Figures 3.10 through 3.15 have stick patterns under the main plots, while Figures 3.16 through 3.20 have stick figure patterns overlapping the main plots.) Expanded plots around the 56° 2-theta peaks are also included for samples 5, 5F and 8S. The 56° peaks show the maximum differences between U_4O_9 and UO_2 patterns and thus help to determine if UO_2 is present. A simulated fit of U_4O_9 and UO_2 is given for each sample. The simulated fit is the relative smooth line. These plots are shown in Figures 3.21 through 3.26.

Figure 3.27 shows the diffractogram of Sample 5 as first prepared and after resuspension. Figure 3.28 shows the blank of filter paper with Collodion. The comparison of Sample 5 (for background) with a diffractogram of cellulose acetate (millipore filter) and silver filter containing metaschoepite is shown in Figure 3.29.

Discussion of the XRD Data

Diffraction patterns for Samples 5, 5F and 8S (Figures 3.6, 3.7, 3.9, 3.10, 3.12, and 3.14) showed a strong characteristic peak pattern of U_4O_9 , uraninite (ICDD card 20-1344) and UO_2 (uraninite, ICDD card 5-550); the pattern of U_4O_9 was a better fit than that of UO_2 . Samples 5S and 8F (Figures 3.8, 3.10, 3.13, and 3.15) showed very weak crystalline-peak patterns, but the U_4O_9/UO_2 major peak is present in both samples. The U_4O_9 and UO_2 patterns are nearly identical, but the U_4O_9 pattern is at a slightly longer 2-theta position (smaller lattice parameter). The width of the lines was much larger than usually observed (about 1° 2-theta at 28° , compared to a few tenths for most materials). The increased width made the determination of whether or not UO_2 was present difficult.

The UO_2 peak position fell within the observed pattern, but not in the center. The U_4O_9 peak fell almost in the center of the observed wide peak. If UO_2 were present, it would distort the observed peak by an amount dependent on the amount present. Since no distortion was apparent, the amount of UO_2 must be small.

Plots of the 56° peak area with superimposed least-squares fit for U_4O_9 and UO_2 (Figures 3.21 through 3.26) show the differences in apparent fit between the two phases. The U_4O_9 was consistently a better fit than UO_2 , and very little if any distortion was apparent. A crystallite-size parameter of 8 nanometers was required to produce the best fit to peak height and width.

ID: #5 SFEC, Particles, Collodion/Glass (from Petri Dish)
File: 950516A.RD Scan: 5- 65/ .03/ 25/#2001, Anode:CU Zero= 0

Figure 3.6. Sample 5 SFEC, Raw Data. Particle with Collodion™ on glass slide.

Figure 3.7. Sample 5F SFEC, Raw Data. Filter paper from canister water, dark.

Figure 3.8. Sample 5S SFEC, Raw Data. Filter paper from sieve wash, light.

ID: #8S SFEC, Filter Paper from Wash Sieve, Dark
File: 950606A.RD Scan: 5- 65/ .02/ 25/#3001, Anode:CU

Zero= 0

Figure 3.9. Sample 8S SFEC, Raw Data. Filter paper from sieve wash, dark.

ID: #8F SFEC, Filter Paper from Cannister Water, Light
File: 950605A.RD Scan: 5- 65/ .02/ 25/#3001, Anode:CU

Zero= 0

Figure 3.10. Sample 8F SFEC, Raw Data. Filter paper from canister water, light.

ID: #5 SFEC, Particles, Collodion/Glass (from Petri Dish)
File: 950516A.DIF Scan: 5- 65/ .03/ 25/#2001, Anode:CU

Zero= 0

Figure 3.11. Sample 5 SFEC. Particles with Collodion, on Glass Slide.
Background subtracted, identified phases shown as stick figures.

Figure 3.12. Sample 5F SFEC. Filter paper from canister water, dark. Background subtracted, identified phases shown as stick figures.

Figure 3.13. Sample 5S SFEC. Filter paper from sieve wash, light. Background subtracted, identified phases shown as stick figures.

ID: #8S SFEC, Filter Paper from Wash Sieve, Dark

File: 950606A.DIF

Scan: 5- 65/ .02/ 25/#3001, Anode:CU

Zero= 0

Figure 3.14. Sample 8S SFEC. Filter paper from sieve wash, dark. Background subtracted, identified phases shown as stick figures.

ID: #8F SFEC, Filter Paper from Cannister Water, Light
 File: 950605A.DIF Scan: 5- 65/ .02/ 25/#3001, Anode:CU
 Zero= 0

Figure 3.15. Sample 8F SFEC. Filter paper from canister water, light. Background subtracted, identified phases shown as stick figures.

ID: #5 SFEC, Particles, Collodion/Glass (from Petri Dish)
 File: 950516A.DIF Scan: 5- 65/ .03/ 25/#2001, Anode:CU

Zero= 0

3.21

1> 20-1344: Uraninite, syn - U O2.25

2> 05-0550: Uraninite, syn - U O2

3> 43-0364: Metaschoepite, syn - U O3 I2 H2 O

Figure 3.16. Sample 5 SFEC. Particles with Collodion, on glass slide. Background subtracted, identified phases shown as overlapped lines.

ID: #5F SFEC, Filter Paper from Cannister, Dark

File: 950612A.DIF

Scan: 5- 65/ .02/ 25/#3001, Anode:CU

Zero= 0

1> 20-1344: Uraninite, syn - U O_{2.25}

2> 05-0550: Uraninite, syn - U O₂

3> 43-0364: Metaschoepite, syn - U O₃ 1/2 H₂ O

Figure 3.17. Sample 5F SFEC. Filter paper from canister water, dark. Background subtracted, identified phases shown as overlapped lines.

ID: #5S SFEC, Filter Paper from Wash Sieve, Light

File: 950608A.DIF

Scan: 5- 65/ .02/ 25/#3001, Anode:CU

Zero= 0

1> 20-1344: Uraninite, syn - U O₂·2.5

3> 43-0364: Metaschoepite, syn - U O₃·2 H₂O

2> 05-0550: Uraninite, syn - U O₂

Figure 3.18. Sample 5S SFEC. Filter paper from sieve wash, light. Background subtracted, identified phases shown as overlapped lines.

ID: #8S SFEC, Filter Paper from Wash Sieve, Dark

File: 950606A.DIF

Scan: 5- 65/ .02/ 25/#3001, Anode:CU

Zero= 0

Figure 3.19. Sample 8S SFEC. Filter paper from sieve wash, dark. Background subtracted, identified phases shown as overlapped lines.

ID: #8F SFEC, Filter Paper from Cannister Water, Light

File: 950605A.DIF

Scan: 5- 65/ .02/ 25/#3001, Anode:CU

Zero= 0

Figure 3.20. Sample 8F SFEC. Filter paper from canister water, light. Background subtracted, identified phases shown as overlapped lines.

3.26

Figure 3.21. Sample 5 SFEC. Particles with Collodion, on glass slide. Simulated fit of U_4O_9 to 56° region of diffractogram.

ID: #5 SFEC, Particles, Collodion/Glass (from Petri Dish)
File: 950516A.DIF Scan: 5- 65/ .03/ 25/#2001, Anode:CU Zero= 0

Figure 3.22. Sample 5 SFEC. Particles with Collodion, on glass slide. Simulated fit of UO_2 to 56° region of diffractogram.

Figure 3.23. Sample 5F SFEC. Filter paper from canister water, light. Simulated fit of U_4O_9 to 56° region of diffractogram.

Figure 3.24. Sample 5F SFEC. Filter paper from canister water, light. Simulated fit of UO₂ to 56° region of diffractogram.

ID: #8S SFEC, Filter Paper from Wash Sieve, Dark
File: 950606A.DIF Scan: 5- 65/ .02/ 25/#3001, Anode:CU Zero= 0

1> 20-1344: Uraninite, syn - U O2.25

Figure 3.25. Sample 8S SFEC. Filter paper from washing sieve, dark. Simulated fit of U₄O₉ to 56° region of diffractogram.

Figure 3.26. Sample 8S SFEC. Filter paper from washing sieve, dark. Simulated fit of UO_2 to 56° region of diffractogram.

ID: #5 SFEC, Particles, Collodion/Glass (from Petri Dish)
File: 950516A.DIF Scan: 5- 65/ .03/ 25/#2001, Anode:CU

Zero= 0

Figure 3.27. Sample 5 SFEC. Particles with Collodion, on glass slide. Comparison of original prep and resuspended preparations.

Figure 3.28. Blank SFEC. Filter paper with Collodion.

Figure 3.29. Comparison of Sample 8S Background with Cellulose Acetate and Silver Filter Backgrounds

Metaschoepite, $\text{UO}_3 \cdot 2\text{H}_2\text{O}$, ICDD pattern 43-364, was found in the 8S sample (Figures 3.9 and 3.14). The 8F sample was too noisy to detect this material. Sample 5 (Figures 3.6 and 3.11) had a detectable peak corresponding to the major peak of metaschoepite, but the other peaks were lost in the noise. This small peak could also have been an artifact, since several peaks were observed in this sample. Sample 5F, (Figures 3.7 and 3.12) showed a definite peak corresponding to the major metaschoepite peak. Sample 5S (Figures 3.8 and 3.13) showed a possible peak in the correct position, but with a very noisy signal.

Some of the sharp, low-intensity lines were believed to be due to crystallites that had not been adequately pulverized and non-randomly oriented. Such particles can lead to peaks which are not representative of the material. Sample 5 was resuspended and dried, then rerun to see if some of these peaks changed. A significant change occurred with the 62° peak, which was completely eliminated. Other peaks, such as the 38 to 40° , 20 , 16 to 18° changed considerably, indicating that they were most likely due to non-randomly oriented particles.

3.3.2 Scanning Electron Microscopy Data on SFEC Sludge

The photomicrographs and X-ray spectra of the particulate samples examined by the SEM coupled with EDAX are shown in Figures 3.30 through 3.59.

Figures 3.30 through 3.47 cover the examination results of sludge samples filtered from SFEC 5 water. The photomicrographs (Figures 3.30, 3.33, 3.34, 3.39, 3.42, 3.44 and 3.46) show the morphology of the sludge particles. Figures 3.30, 3.33, and 3.34 are photomicrographs of sludge sample 5; Figures 3.39 and 3.42 are photomicrographs of sludge sample 5F; and Figures 3.44 and 3.46 are photomicrographs of sample 5S. The photomicrographs indicate very friable particulates that range in size between < 1 micron to > 100 micron.

The elemental composition from the EDAX analyses indicates most of the SFEC sludge particulates are oxides of uranium with minor component of iron oxides, silica, calcium oxide, and alumina. Other elements shown in the spectra are copper (Figures 3.40, 3.41, 3.45, and 3.46); carbon (all the EDAX spectra); magnesium (Figure 3.41); sodium (Figures 3.35, 3.37, and 3.47); and sulphur (Figure 3.47).

The SEM examinations results of sludge samples from SFEC 8 water are shown in Figures 3.48 through 3.59. A photomicrograph of sludge sample 8 is shown in Figure 3.48; the copper wire in the photomicrograph was most probably lying on the sieve from an in-cell activity.

Figures 3.52 and 3.57 show photomicrographs of sludge samples 8S and 8F, respectively. Most of the sludge particles from the SFEC 8 are very small, ranging from < 1 micron to about 100 microns. With the exception of the copper wire, the particulates show elemental composition similar to the sludge material collected in the SFEC 5; that is, oxides of uranium are present as a major chemical phase with minor inclusions of iron oxide, silica, and alumina.

The sludge particles from the two SFECs were sparsely distributed on the 24-micron filter. As shown in the photomicrographs, a large fraction of the sludge material was collected on the sieve by the first filtering process. The hole seen (Figures 3.30a, 3.30b, 3.34c, and 3.34d) might have been a sighting of carbide precipitate which spalled off when the uranium was oxidized.

Discussion of the SEM Data

The sparse distribution of SFEC sludge material on the filter paper indicated that only a small quantity of the K Basin sludge material ended up in the SFEC during SNF sample-loading. The friability of the sludge particles suggests other forms of oxides of uranium other than UO_2 . This is also supported by the broadening and intensity of the oxygen peak in the EDAX spectra.

Uranium carbide precipitates formed in the uranium matrix have geometric shapes similar to the hole shape shown in Figures 3.30a, 3.30b, 3.34c and 3.34d. Most probably, that site in the particle had a carbide inclusion which came off after oxidation of the particle, and left a void space in the particle.

3.4 Metallographic Examinations Results

The photomicrographs of the as-polished surfaces of specimens SFEC5,4378-S2-A in the transverse plane and SFEC5,4378-S2-C in the longitudinal plane are shown in Figures 3.60 through 3.72. The as-polished surface of the transverse plane (specimen SFEC5,4378-S2-A) photomicrographs are shown in Figures 3.60 through 3.63; the as-polished longitudinal plane photomicrographs are shown in Figures 3.64 through 3.72. All of the photomicrographs illustrate the inclusions in the uranium matrix. There are two distinct types of inclusions: one depicts regular geometric shapes as bright areas on the photomicrographs, and the other type are dark spots away from and around the bright inclusions.

The boundary between the Zircalloy cladding and uranium fuel for both planes are shown in Figures 3.62, 3.65, 3.66, 3.67, and 3.69. These photomicrographs show no separation of the cladding from the fuel material and no visible material degradation in the region around the boundary. The bubble-like features on the cladding surface (Figures 3.65, 3.66, and 3.67) were from the polishing and should not be interpreted in any way.

3.4.1 Discussion

The two distinct phase structures illustrated in the photomicrographs of the as-polished uranium surface are uranium-carbide precipitates and probably uranium-hydride inclusions. The carbides are the regularly-structured bright-phase inclusions on the microstructure. Around the carbide precipitates, grain boundaries and micro-cracks are most likely the uranium-hydride inclusions. Some of the dark structures could be voids created by oxidized phases and/or fission gases. The grain boundaries and the micro-cracks are not visible on the as-polished surfaces. Zirconium hydride was also present in the cladding as lenticular inclusions in the Zircalloy microstructure.

Figure 3.30. Particulates from SFEC 5 Water Collected on the 106-Micron Sieve. (a) 130X SEI; (b) 100X SEI showing the marked area for higher magnification and EDAX analyses; (c) SEI of the area marked \$3 on (b) at 1000X; (d) BEI of (c).

Figure 3.31. EDAX Spectrum of Area Marked as \$2 in Figure 35b Showing the Elemental Composition. The abscissa is a log scale in keV.

X-RAY
Live: 300 s
Real: 454 s

Percent 100 s
Dead 0 s
Remaining: 0 s

Figure 3.32. EDAX Spectrum of Area Marked as \$3 in Figure 35b Showing the Elemental Composition. The abscissa is a log scale in keV.

Figure 3.33. Photomicrographs of Area Marked as \$3 in Figure 35b Showing the Size of the Small Particles at 5000X

Figure 3.34. Photomicrographs of Particles from the SFEC 5 Water Showing the Marked Areas and Inclusions that were Analyzed by EDAX. The magnifications for (a), (b), (c) and (d) are at 1000X.

Figure 3.35. EDAX Spectrum of Area Marked as 10 in Figure 39b Showing the Elemental Composition. The abscissa is a log scale in keV.

N-REACT
 Live: 100s Preset: 100s Remaining: 0s
 Real: 148s 32% Dead

< .2 5.296 keV 10.4 >
 FS= 16K OS= 128 ch 274= 177 cts
 MEM1: J0002 N-REACTOR FUEL #5 \$ 11

Figure 3.36. EDAX Spectrum of Area Marked as 11 in Figure 39b Showing the Elemental Composition. The abscissa is a log scale in keV.

X-RAY
 Live: 100s Preset: 100s Remaining: 0s
 Real: 155s 35% Dead

Figure 3.37. EDAX Spectrum of Area Marked as 12 in Figure 39b Showing the Elemental Composition. The abscissa is a log scale in keV.

Figure 3.38. EDAX Spectrum of Area Marked as 13 in Figure 39b Showing the Elemental Composition. The abscissa is a log scale in keV.

Figure 3.39. Photomicrograph of Sludge Particulates from SFEC 5 Water Collected on the 24-Micron Filter Paper. (a) 500X SEI; (b) 500X BSE; (c) 5000X; and (d) 5000X BSE showing the areas marked for analyses.

Figure 3.40. EDAX Spectrum of Area Marked \$3 on Figure 44d Showing the Elemental Composition. The abscissa is a log scale in keV.

Figure 3.41. EDAX Spectrum of Area Marked \$4 on Figure 44d Showing the Elemental Composition. The abscissa is a log scale in keV.

Figure 3.42. Photomicrograph of Sludge Particulates from SFEC 5 Water Collected on the 24-Micron Filter Paper at 5000X Magnification. (a) SEI; and (b) BSE showing the area marked for analysis.

X-RAY

Live: 200s Preset: 100s Remaining: 0s
Real: 298s 33% Dead

5.136 keV 10.3 >
FS= 16M LOG ch 256= 672 cts
MEM: 00013 3F \$ 1

Figure 3.43. EDAX Spectrum of Area Marked \$1 on Figure 47b Showing the Elemental Composition. The abscissa is a log scale in keV.

Figure 3.44. Photomicrograph of Sludge Particulates from SFEC 5 Water Flushed from the Sieve and Collected on 24-Micron Filter Paper. (a) 500X SEI; (b) 500X BSE; (c) 5000X SEI; and (d) 5000X BSE. The entire particle was analyzed.

Figure 3.45. EDAX Spectrum of the Entire Particle Area on Figure 49d Showing the Elemental Composition. The abscissa is a log scale in keV.

Figure 3.46. Photomicrograph of Sludge Particulates from SFEC 5 Water Flushed from the Sieve and Collected on 24-Micron Filter Paper at 3000X magnification. (a) SEI; and (b) BSE showing the area marked \$2 for analysis.

X-RAY	100 s	Preset	100 s	Remaining	0 s
Live	126 s				
Real		21% Dead

<.0	5.176	keV	10.3	>
FS=	2M	LOG	ch	268=
MEM1:	20014	53		
			114	cts

Figure 3.47. EDAX Spectrum of Area Marked \$2 on Figure 51b Showing the Elemental Composition. The abscissa is a log scale in keV.

Figure 3.48. Photomicrograph of the Particulates from SFEC 8 Water Collected on the 106-Micron Sieve. (a) 15X SEI; (b) 200X SEI showing the marked area for higher magnification and EDAX analysis; (c) BEI of the area marked \$2 on (b) at 500X.

Figure 3.49. EDAX Spectrum of Area Marked \$1 on Figure 53a Showing the Elemental Composition of the Wire and Particles Collected on it Within That Area. The abscissa is a log scale in keV.

Figure 3.50. EDAX spectrum of Area Marked \$2 on Figure 53b Showing the Elemental Composition. The abscissa is a log scale in keV.

X-RAY

Live: 100s Preset: 100s Remaining: 0s
Real: 139s 28% Dead

< .0 5.136 keV 10.3 >
FS= 16K OS= 256 ch 266= 314 cts
MEM1: J0002 #8 \$ 3

Figure 3.51. EDAX Spectrum of Area Marked \$3 on Figure 53c Showing the Elemental Composition. The abscissa is a log scale in keV.

Figure 3.52. Photomicrograph of Sludge Particulates from SFEC 8 Water Flushed from the Sieve and Collected on 24-Micron Filter Paper at 500X Magnification. (a) SEI; and (b) BSE showing the areas marked \$1, \$2, \$3 and \$4 for analyses.

Figure 3.53. EDAX Spectrum of Area Marked \$1 on Figure 3.52b Showing the Elemental Composition. The abscissa is a log scale in keV.

Figure 3.54. EDAX Spectrum of Area Marked \$2 on Figure 3.52b Showing the Elemental Composition. The abscissa is a log scale in keV.

Figure 3.55. EDAX Spectrum of Area Marked \$3 on Figure 3.52b Showing the Elemental Composition. The abscissa is a log scale in keV.

X-RAY
 Live: 100s Preset: 100s Remaining: 0s
 Count: 125s 20% Dead

Figure 3.56. EDAX spectrum of Area Marked \$4 on Figure 3.52b Showing the Elemental Composition. The abscissa is a log scale in keV.

Figure 3.57. Photomicrograph of Sludge Particulates from SFEC 8 Water Collected on the 24-Micron Filter Paper. (a) SEI at 500X; (b) BSE at 500X; and (c) BSE at 3000X showing areas marked for analyses.

Figure 3.58. EDAX Spectrum of Area Marked \$1 on Figure 3.57c Showing the Elemental Composition. The abscissa is a log scale in keV.

Figure 3.59. EDAX Spectrum of Area Marked \$2 on Figure 3.57c Showing the Elemental Composition. The abscissa is a log scale in keV.

Figure 3.60. Photomicrograph, 500X, of the As-Polished Specimen SFEC5,4378-S2-A in the Transverse Plane. The photo illustrates the different phase structures in the uranium matrix.

Figure 3.61. Photomicrograph, 500X, of the As-Polished Specimen SFEC5,4378-S2-A in the Transverse Plane, Illustrating the Different Phase Structures in the Uranium Matrix. Around the inclusion are shadowy small precipitates.

Figure 3.62. Photomicrograph, 500X, of the As-Polished Specimen SFEC5,4378-S2-A in the Transverse Plane. The photo shows the boundary of the uranium and the Zircalloy cladding and the different phase structures in the uranium matrix.

Figure 3.63. Photomicrograph, 50X, of the As-Polished Specimen SFEC5,4378-S2-A in the Transverse Plane Illustrating More of the Different Phase Structures in the Uranium Matrix

Figure 3.64. Photomicrograph, 50X, of the Mid-Region of As-Polished Specimen SFEC5,4378-S2-C in the Longitudinal Plane (Parallel to the Axis of the SNF Element). The photo illustrates the different phase structures in the uranium matrix. The bright, almost regular geometric precipitates are most likely uranium carbide.

Figure 3.65. Photomicrograph, 50X, of As-Polished Specimen SFEC5,4378-S2-C in the Longitudinal Plane (Parallel to the Axis of the SNF Element). The photo illustrates the different phase structures in the uranium matrix and the fuel/cladding boundary.

Figure 3.66. Photomicrograph, 250X, of the Cladding of As-Polished Specimen SFEC5,4378-S2-C in the Longitudinal Plane (Parallel to the Axis of the SNF Element). The photo illustrates the lenticular zirconium-hydride phase in the region close to the outside surface (top) of the cladding.

Figure 3.67. Photomicrograph, 50X, of As-Polished Specimen SFEC5,4378-S2-C in the Longitudinal Plane (Parallel to the Axis of the SNF Element) Illustrating the Fuel (Top) and Cladding (Bottom) Boundary

Figure 3.68. Photomicrograph, 150X, of the Mid-Region of As-Polished Specimen SFEC5,4378-S2-C in the Longitudinal Plane (Parallel to the Axis of the SNF Element). The photo illustrates the inclusions in the uranium matrix.

Figure 3.69. Photomicrograph, 250X, Illustrating the Fuel/Cladding Boundary of As-Polished Specimen SFEC5,4378-S2-C

Figure 3.70. Photomicrograph, 250X, Illustrating the Mid-Region of the As-Polished Specimen SFEC5,4378-S2-C

Figure 3.71. Photomicrograph, 250X, Illustrating the Inclusions in the Uranium Matrix of As-Polished Specimen SFEC5,4378-S2-C After Heat-Tinting. The shadowy, small dark precipitates around the uranium carbide precipitates (bright) are probably hydrides.

Figure 3.72. Photomicrograph, 250X, Illustrating the Inclusions in the Uranium Matrix of As-Polished Specimen SFEC5,4378-S2-C After Heat-Tinting. The small, dark precipitates and the dark regions around the uranium carbide precipitates (bright) are probably hydrides.

The distribution of the zirconium hydrides (close to the surface which was in contact with water coolant in the N-Reactor) suggested that they were formed from Zircalloy oxidation during reactor operation.

3.5 Drying and Conditioning Results

3.5.1 Data

The dimensional measurements of the specimens SFEC5,4378-S2-H and -J were done by taking photographs of each cut surface (Figures 3.73 through 3.80).

The boundary between the Zircalloy cladding and the oxidized uranium surface is shown in the figures. Each division of the scales shown is 0.5 mm. Figures 3.75 and 3.79 show the notch on the cladding indicating the top end of the specimens.

The dimensions of all specimens used in the furnace testing, using TI SNF-CT-005, are in Tables 3.4 through 3.6. Columns 2 and 3 of Tables 3.4 and 3.5 show the before- and after-test weights for specimens SFEC5,4378-S2-H, -J, -B, and -I. Table 3.6 shows the before- and after-weights of specimen SFEC5,4378-S2-D used in the cold vacuum-drying test. The weight results show that specimens SFEC5,4378-S2-H, J, B, and I lost about 181.6 mg, 180.2 mg, 169.6 mg, and 181.5 mg, of the oxide layer, respectively. On the other hand, specimen SFEC5,4378-S2-D gained about 6.1 mg of oxygen.

The specimen temperature measured by a Type-K thermocouple, the hydrogen in the off-gas stream measured by gas chromatography (GC), the moisture concentration in the off-gas stream detected by the moisture monitor, and the oxygen concentration upstream after the specimens were measured by GC are plotted in Figures 3.81 through 3.88.

Figure 3.81 shows the two drying cycles for specimens SFEC5,4378-S2-H and -J. There was an increase in the moisture content for the first drying cycle during the transient heat-up of the specimens, and the hydrogen concentration was below the GC detection limit. During the second drying cycle, both hydrogen and moisture concentration peaked during the transient heat-up of the specimens. The moisture eventually decreased to the lowest detection limit of the moisture monitor, but the hydrogen leveled off at about 9 ppm in the off-gas stream. The integral values of the hydrogen at the end of the second cycle was 0.1 mg; the total moisture was 0.3 mg.

Figure 3.82 shows the results of the passivation cycle for specimens SFEC5,4378-S2-H and -J. The figure shows depletion of oxygen in the gas stream, measured by GC. The total oxygen pick-up by the specimens with small contributions from the rest of the furnace materials is about 70 mg. Hydrogen and moisture concentration of the off-gas, detected by GC and moisture monitor, are shown in the figure. Both the moisture level and the hydrogen concentration peaked during the transient heat-up of the specimens, similar to the observations during the drying cycle (Figure 3.81).

Table 3.4: Specimen Measurements (SFEC5,4378-S2-H and -J)

Measurement Number	Specimen Identification Code SFEC5,4378-S2-H								
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm)						
			Length		Thickness	Inner Chord		Outer Chord	
			Z _a	Z _b		X _{1a}	X _{1b}	X _{2a}	X _{2b}
1	28.7545	28.5726	24.1	24.1	9.7	5.7	6.4	8.8	8.7
2	28.7543	28.5727	24.1	24.2	9.5	5.7	6.4	8.8	8.8

Measurement Number	Specimen Identification Code SFEC5,4378-S2-J								
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm)						
			Length		Thickness	Inner Chord		Outer Chord	
			Z _a	Z _b		X _{1a}	X _{1b}	X _{2a}	X _{2b}
1	29.6718	29.4918	24.7	24.8	9.3	5.7	6.3	8.7	9.3
2	29.6719	29.4917	24.7	24.8	9.3	5.7	6.3	8.7	9.2

Table 3.5. Specimen Measurements (SFEC5,4378-S2-B and -I)

Measurement Number	Specimen Identification Code SFEC5,4378-S2-B								
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm)						
			Length		Thickness	Inner Chord		Outer Chord	
			Z _a	Z _b		X _{1a}	X _{1b}	X _{2a}	X _{2b}
1	31.9330	31.7634	25.1	25.1	9.5	6.7	5.6	10.6	9.9
2	31.9330	31.7634	25.1	25.1	9.1	6.2	5.5	10.6	9.9

Measurement Number	Specimen Identification Code SFEC5,4378-S2-I								
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm)						
			Length		Thickness	Inner Chord		Outer Chord	
			Z _a	Z _b		X _{1a}	X _{1b}	X _{2a}	X _{2b}
1	37.4591	37.2777	24.5	24.5	9.5	7.6	7.75	11.2	11.3
2	37.4593	37.2778	24.5	24.5	9.6	7.7	7.7	11.1	11.0

Table 3.6. Specimen Measurements (SFEC5,4378-S2-D and -G)

Measurement Number	Specimen Identification Code SFEC5,4378-S2-D								
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm)						
			Length		Thickness	Inner Chord		Outer Chord	
			Z _a	Z _b		X _{1a}	X _{1b}	X _{2a}	X _{2b}
1	28.8632	28.8694	25.2	25.0	9.6	4.7	6.4	8.1	9.0
2	28.8633	28.8694	25.1	25.1	9.2	4.9	6.3	8.2	8.9

Measurement Number	Specimen Identification Code SFEC5,4378-S2-G								
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm)						
			Length		Thickness	Inner Chord		Outer Chord	
			Z _a	Z _b		X _{1a}	X _{1b}	X _{2a}	X _{2b}
1			24.0	24.0	9.4	7.5	7.6	10.5	10.7
2			24.1	24.1	9.8	7.6	7.5	10.6	10.6

Figure 3.73. Longitudinal Plane of Specimen SFEC5,4378-S2-H. Each division of the attached scale is 0.5 mm. The PTL log is Q-1258.

Figure 3.74. Other Longitudinal Plane of Specimen SFEC5,4378-S2-H. Each division of the attached scale is 0.5 mm. The PTL log is Q-1259.

Figure 3.75. Top End of Specimen SFBCS,4378-S2-H. Each division of the attached scale is 0.5 mm. The PTL log is Q-1260.

Figure 3.76. Bottom End of Specimen SFEC5,4378-S2-H. Each division of the attached scale is 0.5 mm. The PTL log is Q-1261.

Figure 3.77. Longitudinal Plane of Specimen SFEC5,4378-S2-J. Each division of the attached scale is 0.5 mm. The PTL log is Q-1262.

Figure 3.78. Other Longitudinal Plane of Specimen SFEC5,4378-S2-J. Each division of the attached scale is 0.5 mm. The PTL log is Q-1263.

Figure 3.79. Top End of Specimen SFEC5,4378-S2-J. Each division of the attached scale is 0.5 mm. The PTL log is Q-1264.

Figure 3.80. Bottom End of Specimen SFEC5,4378-S2-J. Each division of the attached scale is 0.5 mm. The PTL log is Q-1265.

The hydrogen concentration in the off-gas stream during this passivation cycle was lower than the amount observed during the drying cycle and it was also lower than the moisture concentration in the off-gas stream. When the oxygen supply in the gas stream was turned off at the end of the cycle (Figure 3.82), the hydrogen concentration increased. The total amount of hydrogen observed during this cycle was 0.01 mg and the moisture concentration was 0.3 mg.

Figure 3.83 shows the drying cycles for specimens SFEC5,4378-S2-B and -I. The characteristics of this test run were the same as that for specimens SFEC5,4378-S2-H and -J (Figure 3.81). The total amount of hydrogen measured was below the detection limit for the first cycle and was 0.1 mg after the second cycle. The integral moisture in the off- gas stream for the whole drying process is 0.1 mg.

The plots in Figure 3.84 also show characteristics similar to Figure 3.82. The total oxygen pick-up is 73 mg. Integration of the areas under the moisture and the hydrogen curves yielded total hydrogen of 0.008 mg and 0.2 mg of moisture.

A black, powdery residue was found in the specimen holder after the second furnace testing. The residue, which weighed 0.6081 g, was collected and sampled for XRD analysis. The results of the analysis is shown in Figures 3.85 through 3.88. The presence of this residue could account for the weight loss by the specimens in the first two runs (about 712.9 mg) caused by spalling off the oxide layer during the passivation cycle. The XRD spectra identified the residue as mainly U_4O_9 phase (Figures 3.87 and 3.88).

Figures 3.89 and 3.90 show the results of the conditioning test when no specimen was loaded into the furnace (i.e., a blank run). The drying cycles indicated that only a small amount of moisture desorbed from other surfaces inside the furnace system; there was no detectable hydrogen measured. The oxygen pick-up by the rest of the furnace material, Figure 3.90, is also insignificant.

The results of the cold-vacuum drying (CVD) followed by a hot-vacuum drying (HVD) are plotted in Figure 3.91. The moisture concentration plot shows that a large fraction of the added water came off during the first 150 minutes of the run. The amount that was left on the specimen that desorbed during HVD was insignificant. However, not all of the water added to the specimen was measured in the experiment. The total moisture detected by the moisture monitor was about 0.15 g, compared to the 5 g of water that was added to the specimen in the specimen-holder. This difference was ascribed to large flow rate; attempts will be made in subsequent runs to fix it.

3.5.2 Discussion

The results of the furnace testing (Runs 1 through 3) indicate evolution of moisture and hydrogen from the fuel specimens. The probable sources of the observed moisture are (a) absorbed water in the oxide layer formed in the specimen during specimen-cutting operations; and (b) adsorbed water on the specimens. The hydrogen, however, could be from the following sources:

- Dissolved hydrogen in the cladding;
- Uranium hydride decomposition and/or reaction with moisture and/or oxygen;
- Zirconium hydride decomposition and or reaction with oxygen and/or water; and/or

- Uranium reaction with moisture in the specimen.

The low limit of solubility of hydrogen in uranium cannot account for the level of hydrogen measured but can contribute a small fraction to the amount of hydrogen measured. The depletion of the moisture source (Figures 3.81 and 3.83) even though hydrogen is being released, and the moisture response to the oxygen addition (Figures 3.82 and 3.84) seems to diminish contribution from uranium reaction as the source.

The consistency of probable oxide spalling off (i.e., weight loss by the specimens) from the four specimens used in Runs 1 and 2 shows that the 10-hour passivation time probably is too long for oxidation at 250°C, if the objective is to form a passivated layer. The XRD analyses of the residue show that the U_4O_9 phase is formed at this temperature instead of the anticipated UO_{2+x} . The formation of the higher oxide phase might be the cause of the oxide spalling.

For a uranium specimen with an exposed area of 6.68 cm² (specimen SFEC5,4378-S2-H) using the following assumptions:

- Only the uranium surface is oxidized;
- Oxidation front is uniform; and
- Product of the oxidation is UO_2 .

The calculated oxide thickness for the passivation is 40 microns (uranium dioxide density of 10.96 was used in the calculations); the total weight of the oxide layer is 295 mg. The calculations show that a loss of about 60% by weight of the oxide layer could account for the observed weight loss in the four specimens (Tables 3.4 and 3.5).

Figure 3.81. Plots of Specimen Temperature, Moisture Content, and Hydrogen Concentration in the Off-Gas Stream for the Drying Cycles of Furnace Test Run 1

Figure 3.82. Plots of Specimen Temperature, Oxygen Concentration, Moisture Content, and Hydrogen Concentration in the Off-Gas Stream for the Passivation Cycle of Furnace Test Run 1

Figure 3.83. Plots of Specimen Temperature, Moisture Content, and Hydrogen Concentration in the Off-Gas Stream for the Drying Cycles of Furnace Test Run 2

Figure 3.84. Plots of Specimen Temperature, Oxygen Concentration, Moisture Content, and Hydrogen Concentration in the Off-Gas Stream for the Passivation Cycle of Furnace Test Run 2

ID: SFEC-5, 4378-S2-C1, RESIDUE FROM BOAT/FURNACE. Thin glass.
File: 950720A.DIF Scan: 5- 65/ .02/ 1/#3001, Anode:CU

Zero= 0'

Figure 3.85. Sample SFEC5,4378-S2-C1. Residue from the furnace system specimen holder. Particles with Collodion, on glass slide. Background subtracted, identified phases shown as overlapped lines.

Figure 3.86. Sample SFEC5,4378-S2-C1. Residue from the furnace system specimen holder. Particles with Collodion, on glass slide. Background subtracted, identified phases shown as overlapped lines. Simulated fit of U₄O₉ to the peaks.

Figure 3.87. Sample SFEC5,4378-S2-C1. Residue from the furnace system specimen holder. Particles with Collodion, on glass slide. Background subtracted, identified phases shown as stick figures.

Figure 3.88. Sample SFEC5,4378-S2-C1. Residue from the furnace system specimen holder. Particles with Collodion, on glass slide. Simulated fit of U₄O₉ to the 56° region of diffractogram.

Figure 3.89. Plots of Specimen-Holder Temperature, Moisture Content, and Hydrogen Concentration in the Off-Gas Stream for the Drying Cycles of Furnace Test Run 3. Run 3 had no SNF specimen.

Figure 3.90. A Plot of the Oxygen Concentration for the Passivation Cycle of Furnace Test Run 3. Run 3 had no SNF specimen.

Figure 3.91. Plots of the Specimen Temperature, Moisture Concentration and the Furnace System Pressure for the Cold and Hot Vacuum Drying Cycles of Furnace Test Run 4

Figure 3.92. Plots of Specimen Temperature, Oxygen Concentration, and Moisture Content in the Off-Gas Stream for the Passivation Cycle After the Cold and Hot Vacuum Drying of Furnace Test Run 4

Appendix

Test Instructions

Test Instruction	Rev. No.: 0	Page 1 of 7
Title: Sectioning of SNF Sample in SFEC #5 for Metallographic and Furnace Testing Specimens		
Work Location: 327 (Building/Room or "General")	TI No.: SNF-CT-003	
Author: John Abrefah	Effective Date: 05/01/95 Supersedes Date:	
Signatures:		
Author <u> </u>	(Signature)	<u>05-01-95</u> (Date)
Technical Reviewer <u> </u>	(Signature)	<u>5-2-95</u> (Date)
Approval:		
Project Manager, HTSP <u> </u>	(Paul J. Turner)	<u>5-2-95</u> (Date)
Concurrences:		
PTL Manager <u> </u>	(James M. Seay)	<u>5/2/95</u> (Date)
HSNFP Charact. Manager <u> </u>	(Ronald P. Omberg)	<u>5/2/95</u> (Date)

Test Instruction	Rev. No.: 0	Page 2 of 7
Title: Sectioning of SNF Sample in SFEC #5 for Metallographic and Furnace Testing Specimens		

Purpose/Scope The purpose of this test instruction is to direct the sectioning of the Spent Nuclear Fuel (SNF) samples shipped from 105-K basin to the Postirradiation Testing Laboratory (PTL).

Applicability This test instruction is applicable to SNF element SFEC5, 4378 (outer element with split open end cap).

Responsible Staff All PTL Operators and other persons authorized in writing by PTL management will perform routine operations under guidance of the PNL cognizant scientist and the cognizance of PTL management via this test instruction and the appropriate PTL examination plan.

1.0 Sectioning of the SNF Element SFEC5,4378

Element SFEC5,4378 will be sectioned using the milling machine in F-cell. Stainless steel specimen containers will be used to store the cut specimens under high purity argon.

Note: The SNF sample contained in the SFEC could be friable (i.e., easily crushed or damaged) due to irradiation and degradation in storage. Extreme care should be taken in handling to avoid breaking the fuel sample.

- 1.1 Provide video coverage of the activities as directed by cognizant scientist.
- 1.2 Prior to removing the SNF sample for the following activities, measure and record the cell's atmospheric conditions.
- 1.3 Remove the SNF element from the SFEC and mount it on the milling machine for the first cut.
- 1.4 Mark the 0° and 90° (clockwise) orientations along the entire length of the SNF element. The 0° should be at the same orientation as was used in the visual examinations.
- 1.5 Measure 14 inches (35.56 cm) from the undamaged end of the SNF element and mark that length with a visible pen or paint on the element (see the sectioning diagram in attachment 1, the first cut mark).
- 1.6 Measure 13 inches (33 cm) from the bottom (see the diagram in attachment 1, the second cut mark). Mark the measured length with a visible pen or paint.
- 1.7 Place the slotted template over the ring piece (SFEC5-2) of the fuel sample, aligning it with the 0° orientation mark on the ring sample.

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Title: Sectioning of SNF Sample in SFEC #5 for Metallographic and Furnace Testing Specimens		

CAUTION: There is the potential for the SNF sample to react with the cell's atmosphere. For observable energetic reaction in air, **DO NOT** immerse the element in the SFEC water but purge the element with Argon to preclude further sample degradation. Inform the cognizant scientist. If the reaction stops, re-insert the element in the SFEC. If not, continue the purging with the argon to control the burning of the SNF sample.

- 1.8 Mark 22.5°, 45°, 67.5°, 90° and 180° clockwise on the outside cladding of the ring with a visible pen or paint.
- 1.9 Place the template on the other half of the ring section and mark 22.5°, 45°, 67.5°, 90° counterclockwise. (See attachment 2).
- 1.10 Attach the adhesive K-type thermocouples (TCs) to both sides of the cutting location to measure temperature increases during the cutting.
- 1.11 Place the Argon trough box over the element.
- 1.12 Cut the SNF element at the marked location (first cut in the sectioning diagram) using the milling machine with vortex spray cooling. Start the cutting at a minimum speed and increase the speed as necessary to optimize the cutting time with minimal rise in temperature, at the direction of the cognizant scientist.
- 1.13 Let an operation assistant measure and record the temperature of the SNF sample at the beginning of the cutting and every 15 minutes during the cutting.
- 1.14 Cut the 1 inch (2.54 cm) ring section (labelled SFEC5-2 in the diagram) from the piece with the undamaged end cap.
- 1.15 Insert sections labelled SFEC5-1 (on top with the cut surface down) and SFEC5-3 (at the bottom with the cut surface up) into the SFEC # 5.
- 1.16 Add de-ionized water to the SFEC until full and place the lid on. Store the SFEC in a vertical position.

2.0 Sectioning of the Ring Piece of SNF Element for Test Specimens

The ring section (labelled SFEC5-2 in attachment 2) of the SNF sample will be further sectioned along the longitudinal direction. The sectioning will provide a maximum of eight 22.5° sector specimens and two 90° sections.

- 2.1 Cut the ring section into two halves through the 0° and 180° marks.

Test Instruction

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Title: Sectioning of SNF Sample in SFEC #5 for Metallographic and Furnace Testing Specimens

- 2.2 Label the container with identification numbers such as SFEC5-4378-S2-A, SFEC5-4378-S2-B, SFEC5-4378-S2-C, etc, (i.e., SFEC5 for SFEC #, 4378 for K basin canister #, S2 for sectioned piece and A,B,C, etc, for degree of the sector clockwise per sectioning SFEC5-2 diagram in attachment 2, i.e., A for 22.5°, B for 45°, etc.). Add the sectioned date to the labelling.
- 2.3 Mount one half-piece of the ring and section the four marked 22.5° sectors (shown in attachment 3) with the milling machine .
- 2.4 Place each specimen in the corresponding glass specimen container.
- 2.5 Repeat 2.2 - 2.4 for the other half-ring.
- 2.6 Place the glass containers in the stainless steel sample storage container. Evacuate the container with mechanical pump and backfill it with the high purity argon.

Attachment 2

SIDE VIEW OF SFEC5-2

ESTIMATED
WEIGHT = 650g

OUTSIDE ZR. CLADDING

SECTIONING OF SFEC5-2

Test Instruction	Rev. No.: 0	Page 7 of 7
Title: Sectioning of SNF Sample in SFEC #5 for Metallographic and Furnace Testing Specimens		

Data Sheet # 1

Technician: _____ **Date:** _____ **Cognizant Scientist** _____ **Date:** _____

SNF Temperature

SFEC #	Time (min)	Temperature (°C)	
		Thermocouple 1	Thermocouple 2

Hot Cell's Atmosphere: Temperature _____ **; Humidity** _____ **;**

Pressure Difference _____ **; Atm. Pressure** _____

Test Instruction	Rev. No.: 0	Page 1 of 8
Title: Metallographic Examinations of SNF Element SFEC5,4378 Specimens		
Work Location: 327 (Building/Room or "General")	TI No.: SNF-CT-004	
Author: John Abrefah	Effective Date: 5/16/95 Supersedes Date:	
Signatures:		
Author		5-16-95
	(Signature)	(Date)
Technical Reviewer		5-16-95
	(Signature)	(Date)
Approval:		
Project Manager, HTSP		5/16/95
	(Denis K. Kreid)	(Date)
Concurrences:		
PTL Manager		5/18/95
	(James M. Seay)	(Date)
HSNFP Charact. Manager		5/18/95
	5/18	(Ronald P. Omberg) (Date)

Test Instruction	Rev. No.: 0	Page 2 of 8
Title: Metallographic Examinations of SNF Element SFEC5,4378 Specimens		

- Purpose/Scope** The purpose of this test instruction is to direct the metallographic examinations of polished and etched Spent Nuclear Fuel (SNF) specimens. The specimens were sectioned from SNF element SFEC5,4378, which was shipped from 105-KW basin to the Postirradiation Testing Laboratory (PTL).
- Applicability** This test instruction is applicable to two pre-conditioned sectioned SNF specimens from the middle portion along the SFEC5,4378 element.
- Responsible Staff** All PTL Operators and other persons authorized in writing by PTL management will perform routine operations under guidance of the PNL cognizant scientist and the cognizance of PTL management via this test instruction and the appropriate PTL examination plan.

1.0 Specimen Mounting, Polishing and/or Etching for Examinations

Two 22.5°-sector SNF specimens, sectioned from the element SFEC5,4378 (per TI: SNF-CT-003) will be selected for mounting, polishing and/or etching along the transverse and longitudinal-section planes.

Note: The specimens should be mounted such that the top surface (indicated in step 1.2 for the transverse mount and 1.5 for the longitudinal mount) will be ground and polished for metallographic examinations. The bottom plane should be protected from damage by a tape or sticky foil so that if the surface polishing technique does not show a hydride phase, the other surface will be prepared for examination.

- 1.1 Identify the two specimens selected for metallographic examinations as SFEC5,4378-S2-A, B, C or D.
- 1.2a Mount one specimen such that the transverse planes are available for grinding, polishing and etching (see the illustration in Attachment 2). Use Fiberlay Clear Cast resin for the mount.
- 1.2b Mark the top orientation of the specimen on the mounting ring. This direction should be the same direction as the top of SNF element SFEC5,4378.
- 1.3a Mount the second specimen such that the longitudinal surfaces are available for grinding, polishing and etching (see Attachment 2). Use Fiberlay Clear Cast resin for this mount.
- 1.3b Mark the radial orientation of the specimen on the mounting ring. This direction should be the specimen's degree orientation (e.g., for specimen SFEC5,4378-S2-A per sectioning diagram, the markings will be 0° and 22.5°).

Test Instruction	Rev. No.: 0	Page 3 of 8
Title: Metallographic Examinations of SNF Element SFEC5.4378 Specimens		

- 1.4 Progressively, grind the top of first specimen and the lower degree of the second specimen on the 180 followed by the 240 grit silicon carbide (SiC) abrasive paper.
- 1.5 Take the specimens through 400 and 600 SiC abrasive paper followed by 3/0 emery paper, using a lubricant of paraffin in kerosene (50 g paraffin per liter of kerosene) or the Hyprez OS Diamond liquid.
- 1.6 Polish on "Gold Polishing" cloth, using, in steps the following:
 1. 4 - 8 microns diamond paste, at approximately 40 to 60% Ratiotrol.
 2. 1 - 2 microns diamond paste, at approximately 40 to 60% Ratiotrol.
 3. Linde B alumina saturated with a 2% solution of chromic acid on a microcloth using the Rotor or the Vibra for 30 seconds to 5 minutes (or use silica slurry on the microcloth).
- 1.7 Examine the polished surface structure (see Attachment 3 for illustration of uranium hydride agglomerate) in the following sequence:
 1. Fuel Surface Structure: 36X micro; 36 - 75X mosaic per request of the cognizant scientist; and 36 - 700X photomicrographs as selected by the cognizant scientist.
 2. Fuel/Cladding Interface: 36X micro and 36-700X selected photomicrographs by the cognizant scientist.
 3. Fuel edge: 36X micro; and 36 - 700X photomicrographs as selected by the cognizant scientist.

Get the cognizant scientist approval to proceed to the following steps.

- 1.8 Oxidize the polished surfaces under a halogen lamp (about 1 to 2 inches) for 15 to 30 minutes to get the 'halo' effect (see Attachment 3 for the illustration of the 'halo' effect).
- 1.9 Examine the oxidized specimens in the following sequence:
 1. Fuel Surface Structure: 36X micro; 36 - 75X mosaic per request of the cognizant scientist; and 36 - 700X photomicrographs as selected by the cognizant scientist.
 2. Fuel/Cladding Interface: 36X micro and 36-700X selected photomicrographs by the cognizant scientist.

If the above steps do not positively indicate the presence of a hydride phase, then proceed to the following steps using the bottom planes of the specimens. If a hydride phase is revealed, do not proceed with the following steps:

- 1.10 Progressively grind the bottom plane of the first specimen and the higher degree of the second specimen on the 180 followed by the 240 grit SiC abrasive paper.
- 1.11 Take the specimens through 400 and 600 SiC abrasive paper followed by 3/0 emery paper, using a lubricant of paraffin in kerosene (50 g paraffin per liter of kerosene) or the Hyprez OS Diamond liquid.
- 1.12 Polish on Tex-Met Paper, using saturated solution of Linde A alumina in oxalic acid.
- 1.13 Electropolish for one minute in a solution of 1 part of ethylene glycol, 1 part phosphoric acid and 1 to 2 parts ethyl alcohol at a current density of 10 to 30 mA/cm² and 18 - 20 VDC.
- 1.14 Electroetch for approximately one minute in a solution of 10 g citric acid, 2.5 ml nitric acid and 490 ml de-ionized water at a current density of 15 mA/cm² using a 1 to 1.5 volt dry cell.
- 1.15 Examine the etched surface of each specimen (see Attachment 4 for the illustrations of small uranium hydride inclusions in uranium matrix) in the following sequence:
 - 1. Fuel Surface Structure: 36X micro; 36 - 75X mosaic per request of the cognizant scientist; and 36 - 700X selected photomicrographs by the cognizant scientist.
 - 2. Fuel/Cladding Interface: 36X micro and 36-700X photomicrographs as selected by the cognizant scientist.
 - 3. Fuel edge: 36X micro; and 36 - 700X photomicrographs as selected by the cognizant scientist.

Note: Additionally, if the polishing and etching steps in steps 1.17 and 1.18 fail to work, then the cognizant scientist will select one of the etching solutions in Attachment 1 for cathodic or electropolishing.

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Title: Metallographic Examinations of SNF Element SFEC5,4378 Specimens		

Attachment 1

Electropolishing Solutions For Uranium and Uranium Alloys

1. For cathodic etching: 1 part ortho-phosphoric acid ($\text{ortho-H}_3\text{PO}_4$), 1 part of water. Comments: 30 V open circuit, stainless steel cathode.
2. Electropolish Solution: 1 part of a solution consisting of 118 g chromic acid (CrO_3) dissolved in 100 mL water, 3 to 4 parts glacial acetic acid. Comment: 40 V open circuit.

Attachment 2

TRANSVERSE PLANE MOUNTING LONGITUDINAL PLANE MOUNTING

Attachment 3

Photomicrographs, 500X top, 100X bottom, illustrating the large uranium hydride agglomerates found in one of the Jominy specimens. Note normal size hydride to left of agglomerate in top and bottom photos, also the characteristic "halos" in the bottom photo. Samples in as polished condition; bottom photo taken after heat tinting for 15 minutes.

Attachment 4

D Group

D Group specimen, slow cooled, recrystallized beta quenched, 0.12 ppm hydrogen. Slight indication of a grain boundary precipitate.

B Group

B Group specimen, slow cooled, recrystallized beta quenched, 0.26 ppm hydrogen. Note large hydride inclusions at grain boundaries. (Arrows)

AM Group specimen, in as machined condition from beta quenched rod, 3.3 ppm hydrogen. Note grain boundary precipitate of a larger size than that present in either H or A Group.

Photomicrographs (100X) of Specimens after Inclusion Etch, Appendix I, A. Bright Field Illumination.

Test Instruction

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Title: Furnace Testing of SNF Element SFEC5,4378 Specimens

Work Location: 327
(Building/Room or "General")

TI No.: SNF-CT-005

Author: John Abrefah

Effective Date: 6/23/95
Supersedes Date:

Signatures:

Author

(Signature)

6/28/95

(Date)

Technical Reviewer

(Signature)

6-29-95

(Date)

Approval:

Project Manager, HTSP

7/5/95

(Denis K. Kreid)

(Date)

Concurrences:

PTL Manager

(James M. Seay)

7/7/95

(Date)

HSNFP Charact. Manager

(Ronald P. Omberg)

7/5/95

(Date)

Test Instruction	Rev. No.: 0	Page 2 of 9
Title: Furnace Testing of SNF Element SFEC5,4378 Specimens		

Purpose/Scope The purpose of this test instruction is to direct the drying and conditioning testing of Spent Nuclear Fuel (SNF) specimens in a controlled temperature and atmosphere furnace. The specimens were sectioned from SNF element SFEC5,4378, which was shipped from 105-KW basin to the Postirradiation Testing Laboratory (PTL).

Applicability This test instruction is applicable to SNF specimens taken from the mid-length of the element SFEC5,4378.

Responsible Staff All PTL Operators and other persons authorized in writing by PTL management will perform routine operations under guidance of the PNL cognizant scientist and the cognizance of PTL management via this test instruction and the appropriate PTL examination plan.

Prerequisite The assigned operator(s) should read and follow the steps in the procedure PTL-151: *Operation of Conditioning Furnace System*.

1.0 Equipment Readiness, Specimen Preparation and Loading Into the Furnace

Two 22.5°-sector SNF specimens will be selected for loading into the resistively heated furnace shown schematically in Attachment 1.

- 1.1 Perform the *Equipment Readiness/Start Up* steps of the procedure PTL-151: *Operation of Conditioning Furnace System*. Record completion of each step with the operator's initials, time and date in the PTL log book.
- 1.2 Configure the valves of Attachment 1 for purging the furnace system by closing valves 2, 3, 4, 10 and 12 and opening valves 1, 5, 6, 7, 8, 9 and 11.

Note: For pumping the system, CLOSE valves 2, 3, 4, and 11, and OPEN valves 1, 5, 6, 7, 8, 9, 10 and 12.

- 1.3 Dry the furnace system before loading the test specimens using the method in step 1.3.1 below. If the method in step 1.3.1 is ineffective then try the step 1.3.2 after discussions with the cognizant scientist otherwise disregard step 1.3.2.
 - 1.3.1 Dry the furnace system by flowing high purity gas (e.g. 99.999% Ar) through it for about 24 hours. Heat the lines connected to the system and the furnace tube to about 100°C during this drying period. Computer #1 will record the moisture concentration in the system measured by the moisture monitor. Verify that the data is being acquired by the computer and when a constant concentration is reached, stop the drying process.

Test Instruction	Rev. No.: 0	Page 3 of 9
Title: Furnace Testing of SNF Element SFEC5,4378 Specimens		

Hold Point: Consult with the cognizant scientist before trying the alternate drying method in step 1.3.2 below and/or proceeding to the remaining steps of this test instruction.

- 1.3.2 Dry the furnace system under vacuum condition for about 24 hours. Heat the lines connected to the system and the furnace tube to about 100°C during this drying period. Computer #1 will record the moisture concentration in the system measured by the moisture monitor. Verify that the data is being acquired by the computer and when a constant concentration is reached, stop the drying process.
- 1.4 Identify and record the specimens identification codes in blocks 9 & 28 on the data sheet of Attachment 2.
- 1.5 Use a calibrated ruler (record the calibration information in blocks 7 & 8 on the data sheet of Attachment 2) with an accuracy of ± 1 mm to measure and record in blocks 12 through 27 and 31 through 46 on the data sheet of Attachment 2, each specimen dimensions shown in Attachment 3. Repeat each dimensional measurement. If there are any sharp 'lips' that interfere with the measurements, sand them off with silicon carbide abrasive paper. Do not use a lubricant.
- 1.6 Weigh each specimen twice on a QA category one balance (record the balance calibration information in blocks 1 through 3 on the data sheet of Attachment 2) with 0.1 mg sensitivity and record their weights in blocks 10 & 19, and 29 & 38 on the data sheet of Attachment 2.

Note: If the subsequent steps are performed immediately following step 1.3 then make sure the furnace system has cooled down to about ambient hotcell temperature per agreement with the cognizant scientist before loading the specimens. Minimize the time the furnace tube and gas lines will be open to air in the cell.

- 1.7 Mount the two specimens in the sample holder such that they will be in the center of the uniform heated zone when loaded into the furnace. Ensure that one of the specimens is in contact with the specimen thermocouple.
- 1.8 Load the sample holder into the furnace tube and assemble the rest of the system (i.e., the gas supply components and the effluent gas analytical components).

Test Instruction	Rev. No.: 0	Page 4 of 9
Title: Furnace Testing of SNF Element SFEC5,4378 Specimens		

- 1.9 Prior to the start of the test, check for leakage in the system by either pumping it down to about one Torr pressure or using other leak detection techniques (e.g., a portable leak detector). Record completion of this step with the operator's initials, date and time in the PTL log book.

2.0 Drying Test

The specimens will be initially tested to determine the water content by driving off absorbed, adsorbed and water of hydration through heating in inert atmosphere. The moisture content of the off-gas will be detected by the moisture monitor, and the cumulative water in the specimens will be trapped in the moisture trap. Before and after weights of the trap will be a measure of the water that came off the specimens.

- 2.1 Prior to beginning the drying test of the specimens, determine the weights of the moisture traps on a QA category one balance (record the balance calibration information in blocks 4 through 6 on the data sheet of Attachment 2) with 10 mg sensitivity. Repeat each measurement and record their weights in blocks 47 & 51 and 49 & 53 on the data sheet of Attachment 2.
- 2.2 Purge the system with high purity (e.g. 99.999%) argon gas for a period of at least 30 minutes. Use an argon flow rate of 100 ± 2 cm³/min.
- 2.3 Verify that all the analytical instruments (e.g., GC, moisture monitor, pressure gauge, mass flow controllers and the computers) are responding properly by looking at the readings on both computers. Notify the cognizant scientist of any anomalous readings.
- 2.4 Turn on and maintain the heating tapes downstream from the furnace at a fixed temperature within the range of 50°C to 100°C throughout the drying cycles. The heating tape upstream from the furnace will be turned off or on at the direction of cognizant scientist.
- 2.5 Configure valves 6 to 9 of Attachment 1 (i.e., either valves 6 and 8 opened with valves 7 and 9 closed or valves 6 and 8 closed with 7 and 9 opened) so that gas flows through only one of the moisture traps.
- 2.6 Turn on the furnace and program it (refer to the Omega Temperature Controller/Programmer manual):
 - 2.6.1 to heat the furnace at a constant rate, within the range of 10 to 15°C/min, to $100 \pm 2^\circ\text{C}$
 - 2.6.2 hold the $100 \pm 2^\circ\text{C}$ for 10 hours
 - 2.6.3 resume the constant heating rate (i.e., at the same rate as in step 2.6.1) to $300 \pm 2^\circ\text{C}$

Test Instruction	Rev. No.: 0	Page 5 of 9
Title: Furnace Testing of SNF Element SFEC5,4378 Specimens		

- 2.6.4 hold the $300 \pm 2^\circ\text{C}$ for 24 more hours
- 2.6.5 then allow the furnace to cool to ambient temperature.
- 2.7 Record in block 55 on the data sheet of Attachment 2 the time that the furnace heat-up is initiated, and verify that the computers are monitoring and recording the:
 - 2.7.1 Water vapor concentration with the moisture monitor
 - 2.7.2 Hydrogen and oxygen concentrations with the GC
 - 2.7.3 Furnace and specimen temperatures
- 2.8 After the furnace and specimen have reached ambient temperature, remove the in-line moisture trap (e.g., trap # 1 or 2) and weigh it twice on a QA category one balance with 10 mg sensitivity. Record the weights in blocks 48 & 52 or 50 & 54 on the data sheet of Attachment 2.

3.0 Conditioning Testing

The SNF passivation step in the furnace testing will involve exposing the test specimens to a gas mixture of 98% argon and 2% oxygen by volume at a flow rate of $100 \pm 2 \text{ cm}^3/\text{min}$.

- 3.1 Configure valves 6 to 9 (i.e., either valves 6 and 8 opened with valves 7 and 9 closed or valves 6 and 8 closed with 7 and 9 opened) so that gas flows through the moisture traps that was not used in the drying cycle.
- 3.2 Adjust the heating tape (downstream from the furnace) temperature to a constant value in the range of 50 to 100°C or the value requested by the cognizant scientist.
- 3.3 Set the argon and oxygen flow controllers to give the flows indicated above (i.e., 98% argon, 2% oxygen per GC measurement, at a total flow rate of $100 \pm 2 \text{ cm}^3/\text{min}$).
- 3.4 Switch the gas flow to the Gas Chromatograph (GC) through the GC bypass line (i.e., opening valve 4 and closing valves 1, 2, 3, and 11 of Attachment 1). Record in block 56 on the data sheet of Attachment 2 the switch over time. Program computer #2 to monitor and record the oxygen concentration in the gas stream every 2 minutes for a period of at least 15 minutes after equilibration to establish the baseline.

Test Instruction	Rev. No.: 0	Page 6 of 9
Title: Furnace Testing of SNF Element SFEC5,4378 Specimens		

- 3.5 Switch the gas flow to the furnace by opening valve 1, 5, and 11, and by closing valves 2, 3, 4 and 10. Record in block 57 on the data sheet of Attachment 2 the switch over time. Program computer #2 to monitor the oxygen concentration in the gas stream measured by the GC every 5 minutes until a constant oxygen concentration is reached. At that constant concentration, monitor the gas stream for at least 30 minutes to establish the baseline concentration.
- 3.6 Turn on the furnace and program it (refer to the Omega Temperature Controller/Programmer manual) to heat at a constant rate (e.g., between 12 to 15°C/min) to 250±2°C and hold at that temperature indefinitely. (Note that furnace cooling will be initiated manually after about 10 hours as described in Step 3.7.) Verify that the computers are monitoring and recording the:
 - 3.6.1 Moisture concentration using the moisture monitor every 5 minutes.
 - 3.6.2 Oxygen and hydrogen concentrations using the gas chromatograph every 5 minutes.
 - 3.6.3 Furnace and specimen temperatures every 5 minutes.
- 3.7 After 10 hours of conditioning of the specimens, turn off the oxygen flow and the furnace, and allow the specimen to cool to ambient temperature in flowing argon. Record in block 58 on the data sheet of Attachment 2 the time oxygen gas is turned off.
- 3.8 Open the furnace and carefully remove the specimens for weighing using a QA category one balance with 0.1 mg sensitivity. Weigh each specimen twice and record their weights in blocks 11 & 20 and 30 and 39 on the data sheet of Attachment 2. Record other observations in the PTL log book.
- 3.9 Perform the test shut down steps in the procedure PTL-151. Record completion of each step with the operator's initials, date and time in the PTL log book.
- 3.10 At the direction of the cognizant scientist, remove and store the two 0.2 µm filters for possible radionuclide analyses at a later date. Record the storage location and date in the PTL log book.

Test Instruction

Rev. No.: 0

Page 7 of 9

Title: Furnace Testing of SNF Element SFEC5,4378 Specimens

Attachment 1

Schematic Of The Furnace System

Test Instruction Data Sheet	Title: Furnace Testing of SNF Element SFEC5,4378	Rev. No.: 0	Page 8 of 9
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Operator: _____ Date: _____ Cognizant Scientist: _____ Date: _____

EQUIPMENT DESCRIPTION

1 - Sample Balance Description	2 - Sample Balance Calibration Number	3 - Calibration Expiration Date
4 - Moisture Trap Balance Description	5 - Moisture Trap Balance Calibration Number	6 - Calibration Expiration Date
	7 - Ruler Calibration Number	8 - Ruler Calibration Date

SPECIMEN MEASUREMENTS

9 - Specimen Identification Code									
Measurement Number	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm)						
			Length		Thickness	Inner Chord		Outer Chord	
			Z _a	Z _b		X _{1a}	X _{1b}	X _{2a}	X _{2b}
1	10	11	12	13	14	15	16	17	18
2	19	20	21	22	23	24	25	26	27

Measurement Number	28 - Specimen Identification Code								
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm)		Thickness	Inner Chord		Outer Chord	
			Length			X _{1a}	X _{1b}	X _{2a}	X _{2b}
			Z _a	Z _b					
1	29	30	31	32	33	34	35	36	37
2	38	39	40	41	42	43	44	45	46

Measurement Number	Moisture Trap #1		Moisture Trap #2	
	Weight Before Test (g)	Weight After Test (g)	Weight Before Test (g)	Weight After Test (g)
1	47	48	49	50
2	51	52	53	54

SYSTEM STATUS

55 - Time/Date	56 - Time/Date	57 - Time/Date	58 - Time/Date
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Specimen Dimensions

Test Instruction	Rev. No.: 0	Page 1 of 10
Title: Vacuum Drying and Conditioning Testing of Initially Wet SNF Element SFEC5,4378 Specimens		
Work Location: 327 (Building/Room or "General")	TI No.: SNF-CT-007	
Author: John Abrefah	Effective Date: 7/5/95 Supersedes Date:	
Signatures:		
Author <u> </u>	<u>7/5/95</u>	(Date)
Technical Reviewer <u> </u>	<u>7-5-95</u>	(Date)
Approval:		
Project Manager, HTSP <u> </u>	<u>7/6/95</u>	(Date)
Concurrences:		
PTL Manager <u> </u>	<u>7/6/95</u>	(Date)
HSNFP Charact. Manager <u> </u>	<u>7/7/95</u>	(Date)

Test Instruction	Rev. No.: 0	Page 2 of 10
Title: Vacuum Drying and Conditioning Testing of Initially Wet SNF Element SFEC5,4378 Specimens		

Purpose/Scope The purpose of this test instruction is to direct the drying and conditioning testing of Spent Nuclear Fuel (SNF) specimens in a controlled temperature and atmosphere furnace. The specimens were sectioned from SNF element SFEC5,4378, which was shipped from 105-KW basin to the Postirradiation Testing Laboratory (PTL).

Applicability This test instruction is applicable to SNF specimens taken from the mid-length of the element SFEC5,4378.

Responsible Staff All PTL Operators and other persons authorized in writing by PTL management will perform routine operations under guidance of the PNL cognizant scientist and the cognizance of PTL management via this test instruction and the appropriate PTL examination plan.

Prerequisite The assigned operator(s) should read and follow the steps in the procedure PTL-151: *Operation of Conditioning Furnace System*.

1.0 Equipment Readiness, Specimen Preparation and Loading Into the Furnace

Two 22.5°-sector SNF specimens will be selected for loading into the resistively heated furnace shown schematically in Attachment 1.

- 1.1 Perform the *Equipment Readiness/Start Up* steps of the procedure PTL-151: *Operation of Conditioning Furnace System*. Record completion of each step with the operator's initials, time and date in the PTL log book.
- 1.2 Configure the valves of Attachment 1 for purging the furnace system by closing valves 2, 3, 4, 10 and 12 and opening valves 1, 5, 6, 7, 8, 9 and 11.

Before proceeding further in the procedure PTL-151 perform the following steps:

- 1.3 Dry the furnace system before loading the test specimens using the method in step 1.3.1 below. If the method in step 1.3.1 is ineffective then try the step 1.3.2 after discussions with the cognizant scientist otherwise disregard step 1.3.2.
 - 1.3.1 Dry the furnace system by flowing high purity gas (e.g. 99.999% Ar) through it for about 24 hours. Heat the lines connected to the system and the furnace tube to about 100°C during this drying period. Computer #1 will record the moisture concentration in the system measured by the moisture monitor. Verify that the data is being recorded by the computer and when a constant concentration is reached, stop the drying process.

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Hold Point: Consult with the cognizant scientist before trying the alternate drying method in step 1.3.2 below and/or proceeding to the remaining steps of this test instruction.

- 1.3.2 Dry the furnace system under vacuum condition for about 24 hours. Heat the lines connected to the system and the furnace tube to about 100°C during this drying period. Computer #1 will record the moisture concentration in the system measured by the moisture monitor. Verify that the data is being recorded by the computer and when a constant concentration is reached, stop the drying process.
 - 1.4 Identify and record the specimens identification codes in blocks 9 & 28 on the data sheet of Attachment 2.
 - 1.5 Use a calibrated ruler (record the calibration information in blocks 7 & 8 on the data sheet of Attachment 2) with an accuracy of ± 1 mm to measure and record in blocks 12 through 27 and 31 through 46 on the data sheet of Attachment 2 the specimens dimensions shown in Attachment 3. Repeat each dimensional measurement. If there are any sharp 'lips' that interfere with the measurements, sand them off with silicon carbide abrasive paper. Do not use a lubricant.
 - 1.6 Weigh each specimen twice on a QA category one balance (record the balance calibration information in blocks 1, 2 & 3 on the data sheet of Attachment 2) with 0.1 mg sensitivity. Record the specimens weights in blocks 10 & 19, and 29 & 38 on the data sheet of Attachment 2.
- Note:** If the subsequent steps are performed immediately following step 1.13 then make sure the furnace system has cooled to the ambient hotcell temperature before loading the specimens. Minimize the time the furnace tube and gas lines will be open to air in the cell.
- 1.7 Mount the two specimens in the sample holder such that they will be in the center of the uniform heated zone when loaded into the furnace. Ensure that one of the specimens is in contact with the specimen thermocouple.
 - 1.8 Fill the sample holder with 5 ± 0.2 ml of de-ionized water.
 - 1.9 Load the sample holder into the furnace tube with extreme care to avoid loss of water and assemble the rest of the system (i.e., the gas supply components and the effluent gas analytical components).

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- 1.10 Prior to the start of the test, check for leakage in the system by either pumping it down to about one Torr pressure or using other leak detection techniques (e.g., a portable leak detector). Record completion of this step with the operator's initials, date and time in the PTL log book.

2.0 Vacuum Drying Test

The specimens will be initially tested to determine the water content by driving off free and bound water first at low temperature vacuum followed by relatively high temperature vacuum. The moisture content of the off-gas will be detected by the moisture monitor, and the cumulative water will be trapped in the moisture traps. Before and after weights of the traps will be a measure of the water that came off.

- 2.1 Prior to beginning the drying test of the specimens, determine the weights of the moisture traps on a QA category one balance (record the balance calibration information in blocks 4, 5 & 6 on the data sheet of Attachment 2) with 10 mg sensitivity. Repeat each measurement and record their weights in blocks 47 & 51 and 49 & 53 on the data sheet of Attachment 2.
- 2.2 Pump the system down and control the system pressure to within the range of 50 to 100 Torr after closing valves 1, 2 and 11 and opening valves 5, 6, 7, 8, 9 and 10.
- 2.3 Configure valves 6 to 9 of Attachment 1 so that the pumping is through the two moisture traps (i.e., valves 6 to 9 opened).
- 2.4 Verify that all the analytical instruments (e.g., moisture monitor and pressure gauge) are responding properly by looking at the readings on the computer #1. Notify the cognizant scientist of any anomalous readings.
- 2.5 Turn on and maintain the heating tapes downstream from the furnace at a fixed temperature within the range of 50°C to 100°C throughout the drying cycles. The heating tape upstream from the furnace will be turned off or on at the direction of the cognizant scientist.
- 2.6 Close valves 7 and 9 of Attachment 1 so that only moisture trap #1 is used in the low temperature vacuum drying cycle.

Note: Due to the swapping of gas traps after the first low temperature vacuum drying cycle, the test must be scheduled such that the first cycle ends during regular working hours, otherwise arrange for that step to be performed during off hours.

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- 2.7 Turn on the furnace and program it (refer to the Omega Temperature Controller/Programmer manual):
 - 2.7.1 to heat at a constant rate (e.g., between 10 and 15°C/min) to 50±2°C and hold the 50±2°C for 10 hours.
 - 2.7.2 resume the constant heating rate after swapping the gas traps in Step 2.9 (i.e., at the same rate as in Step 2.7.1) to 300±2°C and hold the 300±2°C for 24 more hours
 - 2.7.3 then allow the furnace to cool to ambient temperature.
- 2.8 Record in block 62 on the data sheet of Attachment 2 the time that the furnace heat-up is initiated, and verify that computer #1 is monitoring and recording the:
 - 2.8.1 Water vapor concentration with the moisture monitor
 - 2.8.2 Pressure with the pressure gauge
 - 2.8.3 Furnace and specimen temperatures
- 2.9 After the cold vacuum dry cycle, close valves 6 to 8 and open valves 7 and 9 (Attachment 1) so that the moisture trap #2 is connected to the system.
- 2.10 Remove the moisture trap #1 during the second heating up rate to set the testing temperature at 300±2°C. Weigh the trap on a QA category one balance with 10 mg sensitivity. Repeat the weight measurement and record the weights in blocks 48 & 52 on the data sheet of Attachment 2.
- 2.11 After the second drying cycle, and with the furnace and specimen at ambient temperature, remove moisture trap #2 and weigh it twice on a QA category one balance with 10 mg sensitivity. Record the weights in blocks 50 & 54 on the data sheet of Attachment 2.

3.0 Conditioning Testing

The SNF passivation step in the furnace testing will involve exposing the test specimens to a gas mixture of 98% argon and 2% oxygen by volume at a flow rate of 100±2 cm³/min.

- 3.1 Configure valves 6 to 9 (i.e. either valves 6 and 8 opened with valves 7 and 9 closed or valves 6 and 8 closed with valves 7 and 9 opened) so that gas flows through only one of the moisture traps.
- 3.2 Adjust the heating tape (downstream from the furnace) temperature to a constant value in the range of 50 to 100°C or the value specified by the cognizant scientist.

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- 3.3 Purge the system with pure argon flow of 100 ± 2 cm³/min (i.e., by opening valves 1, 5, 6&8 or 7&9, and 11, but with valves 2,3,4, and 10 closed) for about 45 minutes and monitor the moisture content in the system. If the moisture monitor indicates high concentration (i.e., greater than -50°C dew point) then consult with the cognizant scientist before proceeding to the next step.
- 3.4 Set the argon and oxygen flow controllers to give the flows indicated above (i.e., 98% argon, 2% oxygen at a flow rate of 100 ± 2 cm³/min).
- 3.5 Switch the gas flow to the GC through the bypass line (i.e., opening valve 4 and closing valves 1, 2, 3, and 11 of Attachment 1). Record in block 63 on the data sheet of Attachment 2 the switch over time. Program computer #2 to monitor and record the oxygen concentration in the gas stream every 2 minutes for a period of at least 15 minutes after equilibration to establish the baseline.
- 3.6 Switch the gas flow to the furnace by opening valve 1, 5, and 11, and by closing valves 2, 3, 4 and 10. Record in block 64 on the data sheet of Attachment 2. Program computer #2 to monitor and record the oxygen concentration in the gas stream measured by the GC every 5 minutes until a constant oxygen concentration is established. At that constant concentration, monitor the gas stream for at least 30 minutes to establish the baseline concentration.
- 3.7 Turn on the furnace and program it (refer to the Omega Temperature Controller/Programmer manual) to heat at a constant rate (e.g., between 12 to 15°C/min) to 150 ± 2 °C and hold at that temperature indefinitely. (Note that furnace cooling will be initiated manually after about 10 hours as described in Step 3.8.) Verify that the computers are monitoring and recording the:
 - 3.7.1 Moisture concentration using the moisture monitor for every 5 minutes.
 - 3.7.2 Oxygen and hydrogen concentrations using the gas chromatograph for every 5 minutes.
 - 3.7.3 Furnace and specimen temperatures every 5 minutes.

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- 3.8 After 10 hours of conditioning of the specimens, turn off the oxygen flow and the furnace, and allow the specimen to cool to ambient temperature in flowing argon. Record in block 65 on the data sheet of Attachment 2 the time oxygen gas is turned off.
- 3.9 Open the furnace and carefully remove the specimens for weighing using a QA category one balance with 0.1 mg sensitivity. Weigh each specimen twice and record their weights in blocks 11 & 20 and 30 & 39 on the data sheet of Attachment 2. Record other observations in the PTL log book and on a sheet to be pasted in the Laboratory Record Book (LRB).
- 3.10 Perform the test shut down steps in the procedure PTL-151. Record completion of each step with the operator's initials, date and time in the PTL log book.
- 3.11 At the direction of the cognizant scientist, remove and store the two 0.2 μ m filters for possible radionuclide analyses at a later date. Record the storage location and date in the PTL log book.

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Attachment 1

Schematic Of The Furnace System

Operator: _____ Date: _____ Cognizant Scientist: _____ Date: _____

EQUIPMENT DESCRIPTION

1 - Sample Balance Description	2 - Sample Balance Calibration Number	3 - Calibration Expiration Date
4 - Moisture Trap Balance Description	5 - Moisture Trap Balance Calibration Number	6 - Calibration Expiration Date
	7 - Ruler Calibration Number	8 - Ruler Calibration Date

SAMPLE MEASUREMENTS

Measurement Number	9 - Specimen Identification Code								
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm)						
			Length		Thickness	Inner Chord		Outer Chord	
			Z _a	Z _b		X _{1a}	X _{1b}	X _{2a}	X _{2b}
1	10	11	12	13	14	15	16	17	18
2	19	20	21	22	23	24	25	26	27

Measurement Number	28 - Specimen Identification Code								
	Weight Before Test (grams)	Weight After Test (grams)	Sample Dimensions (mm)						
			Length		Thickness	Inner Chord		Outer Chord	
			Z _a	Z _b		X _{1a}	X _{1b}	X _{2a}	X _{2b}
1	29	30	31	32	33	34	35	36	37
2	38	39	40	41	42	43	44	45	46

Measurement Number	Moisture Trap #1		Moisture Trap #2	
	Weight Before Test (g)	Weight After Test (g)	Weight Before Test (g)	Weight After Test (g)
1	47	48	49	50
2	51	52	53	54

SYSTEM STATUS

55 - Time/Date	56 - Time/Date	57 - Time/Date	58 - Time/Date
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Attachment 3

Specimen Dimensions

Test Procedure

Procedure Worksheet

Org. Code: D7N27 Procedure No.: PTL-151 Rev. No.: R00	Title: OPERATION OF CONDITIONING FURNACE SYSTEM
	Author: WJ Gray
Determine Stakeholders	
Gather Background Information	
Identify Source Requirements	
Conduct Hazards Assessment	Hydrogen from operation Air from hot cell
Plan Hazard Mitigation Methods	Vent to cell exhaust Return air to cell

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<h2 style="margin: 0;">PNL Operating Procedure</h2>	Org. Code: D7N27 Procedure No.: PTL-151 Rev. No.: R00
Title: OPERATION OF CONDITIONING FURNACE SYSTEM	
Work Location: 327/1 (Building/Room or "General")	Page 1 of <u>23</u> 7/26/11/15
Author: WJ Gray	Effective Date: 06/14/95 Supersedes Date: New
Identified Hazards: ☒ Radiological ☐ Hazardous Materials ☐ Physical Hazards ☐ Hazardous Environment ☐ Other:	Was a Procedure Worksheet Completed? <div style="text-align: center;"> ☐ Yes ☒ No </div> Are One-Time Modifications Allowed to this Procedure? <div style="text-align: center;"> ☒ Yes ☐ No </div>
Signatures:	
Author <u>WJ Gray</u> (Signature)	<u>6/14/95</u> (Date)
Technical Reviewer <u>Gene Marshall</u> (Signature)	<u>6-14-95</u> (Date)
Approval:	
Line Manager <u>[Signature]</u> (Signature)	<u>6/14/95</u> (Date)
Concurrences, as appropriate:	
Building Manager <u>WA Harkin</u> (Signature)	<u>6/16/95</u> (Date)
Laboratory Safety <u>[Signature]</u> (Signature)	<u>6/14/95</u> (Date)
Quality Programs <u>[Signature]</u> (Signature)	<u>6/14/95</u> (Date)
Radiological Control <u>Terry W. Milham</u> (Signature)	<u>6/14/95</u> (Date)
(Other) _____ (Signature)	_____ (Date)

PNL Operating Procedure

Org. Code: D7N27
Procedure No.: PTL-151
Rev. No.: R00

Title: OPERATION OF CONDITIONING FURNACE SYSTEM

Purpose/Scope

The purpose of this procedure is to establish a set of instructions for operation of the conditioning furnace system.

Applicability

This procedure applies to the operation of a furnace, which is used for the conditioning of spent nuclear fuel specimens and associated materials, and associated gas handling and data acquisition equipment in G-Cell. A schematic diagram of the apparatus is shown in Exhibit #1. Many of the following instructions refer to items identified in that figure.

Definitions (optional)

TI -- Test Instruction. An approved set of step-by-step instructions for conducting a single test or related group of tests in accordance with the general guidelines of this procedure.

GC -- Gas Chromatograph

Responsible Staff

Qualified PTL operations staff members and other personnel authorized in writing by PTL management may perform routine operations under the cognizance of PTL management and via the use of this procedure.

Hot-Cell Technician -- Operates all equipment inside the hot-cell under the direction of the Test Operator and/or the Cognizant Scientist. May also operate equipment outside the hot-cell under the direction of the Test Operator and/or the Cognizant Scientist.

Test Operator -- Operates equipment outside the hot-cell according to this procedure and applicable TI and/or under the direction of the Cognizant Scientist.

Cognizant Scientist -- Responsible for conducting tests according to this procedure and applicable TI. May make minor modifications to the procedure or TI so long as they do not change the intent of the procedure or TI, and the changes are recorded on the applicable TI data sheet. May operate the equipment outside the hot-cell.

Emergency Response

Notify PNL's single-point-of-contact number at 375-2400. Dial 911 in the event there is no response at 375-2400.

Prerequisites

The PTL operations staff member shall read and comply with PTL-060, G-Cell General Operating Procedure.

The following reference materials are available for more detailed information regarding the equipment used in this procedure:

- M200 Gas Chromatograph Operations Manual
- Moisture Monitor Series 35 User's Manual
- EZChrom Data System User's manual
- AIMAX Plus Software Instruction Manual

- Mass Flow Control Box Instruction Manual
- Omega Temperature Controllers Operations Manual

Precautions and Limitations

Operate cell within normal operating limits.

If specimen temperatures are to exceed 500°C, the system must include an aerosol trap (see Exhibit #1).

Gas supply to furnace shall not exceed 20% oxygen.

Do not set the pressure regulators on the argon, nitrogen, and oxygen gas cylinders above 150 psig.

Work Instructions

1.0 Equipment Readiness/Start Up

- 1.1 Ensure that the valves on the Argon, Nitrogen, and Oxygen gas cylinders are closed.
- 1.2 Ensure that all gas and vacuum valves outside the hot-cell are in the off position.
- 1.3 Establish all gas, vacuum, and electrical connections between gas cylinders, equipment rack, vacuum pump, and hot-cell.
- 1.4 Supply electrical power to the equipment rack.
- 1.5 Turn on MKS Baritron Pressure Gauge (including heater).

Note: It is recommended that this equipment be turned on at least 4 hours before start of test.

- 1.6 Turn on the Temperature Controller/Programmer for the furnace.
- 1.7 Ensure that the power to the furnace is set in the "off" position.
- 1.8 Program furnace over-temperature protection to 50°C above furnace set-point.
- 1.9 Turn on the Moisture Monitor.
- 1.10 Turn on the Flow Controller.
- 1.11 Activate the Gas Chromatograph.
 - 1.11.1 Turn on the GC computer (#2).
 - 1.11.2 Check the time and date and correct if necessary.
 - 1.11.3 From the windows screen, select "EZ Chrom" icon. Click (using the left mouse button) on "OK" when the EZCHROM title screen comes up.

- 1.11.4 Check the calibration status and recalibrate according to Section 4.0 as necessary.
- 1.11.5 Click on "Reports" in the upper left corner of the screen, then click on "External Standard" to display the gas concentrations.
- 1.11.6 Click on "Start" in the lower right corner of the screen.
- 1.11.7 Click the cursor into the box labeled TEST ID and make the TEST ID equal to the date of the test in the form of MMMDDYY (e.g., JUN0991).
- 1.11.8 Set the number of runs and the time between injections according to the test instructions.
- 1.11.9 Make sure the SAVE and PRN boxes have X's in them.
- 1.11.10 Check disk space and make sure that at least 25 M of hard disk space is available.
- 1.11.11 Click on "Start".

Note: The GC will now collect data unattended until the end of the test.

1.12 Turn on Computer #1 and associated I/O Plexer for warmup.

- 1.12.1 Check the time and date and correct if necessary.
- 1.12.2 Ensure that the clocks on computers 1 and 2 are in sync within ± 30 seconds.
- 1.12.3 Ensure that there is at least 500K file space on the hard disk to record data from the Historical Trend task.
- 1.12.4 Run "Start Background" from main menu.
- 1.12.5 Run "Start Foreground" from main menu.
- 1.12.6 Switch to the "Real-Time Trend" task and ensure that viable readings exist for all 6 channels. If not, notify cognizant scientist.

Note: The computer will now collect data unattended until the end of the test.

2.0 Test Procedure

- 2.1 Place Fuel Segment(s) in Furnace Tube according to applicable TI.
- 2.2 Place Furnace Tube in Furnace and reestablish gas-line connections.

- 2.3 Configure the Pressure Control Valve and associated bypass valve (#5) according to applicable TI.
- 2.4 Configure the Moisture Traps and associated valves (6 thru 9) according to applicable TI.
- 2.5 If specimen temperatures are to exceed 500°C, ensure that the optional Aerosol Trap is in place as depicted in Exhibit #1.
 - 2.5.1 Turn on the Temperature Controller for the Aerosol Trap Heater.
 - 2.5.2 Set the temperature to 500°C.
 - 2.5.3 Allow the heater temperature to come to equilibrium.
- 2.6 Adjust the heat tape temperatures according to the applicable TI.
- 2.7 Establish gas flow through the Flow Controller according to applicable TI.
 - 2.7.1 Configure valves 1, 2, 3, 4, 10, and 11 according to applicable TI.
 - 2.7.2 Start the flow of gas and record the time, as read from either of the computers, on the applicable TI data sheet.
- 2.8 Conduct the test according to applicable TI.

Note: The TI will provide details of the various test phases such as furnace temperature, gas composition and flow rate, when to turn on the vacuum pump, duration of the different test phases, etc.

- 2.9 At the end of the test, record the time, as read from either of the computers, on the applicable TI data sheet.

3.0 Test Shutdown

- 3.1 Stop the GC if it is still running.
- 3.2 If more than two days will elapse before the start of the next test, turn the column temperatures on the GC down to 35°C.
- 3.3 Exit to the main menu on computer #1 and run "Stop Background".

Note: Either or both computers can be turned off if desired.
- 3.4 Ensure that the Furnace, Heat Tapes, Aerosol Trap Heater (if used), and Moisture Control Heater and Vacuum Pump are turned off.
- 3.5 Ensure that the valves on the Argon, Nitrogen, and Oxygen gas cylinders are closed.

- 3.6 Ensure that all gas and vacuum valves outside the hot-cell are in the "off" position.

4.0 GC Calibration

Note: Calibration frequency of the M200 Gas Chromatograph is anytime after cold shutdown. The calibration interval can be up to 30 days of continuous operation or warm standby mode.

The following instructions are adapted from the M200 Operations Manual (MTI part # 20050 Revision 1.3) and the EZChrom 200 Chromatograph Data System manual (MTI part # 20049 Revision 2.2) published by Microsensor Technology Inc. (Fremont, CA).

- 4.1 Activate the GC as described in Section 1.11.

- 4.2 Turn on the calibration gas and allow it to flow through the system for about 5 minutes before analyzing a sample with the GC.

- 4.3 Make twenty runs.

Note: Store with a unique filename, e.g., cal6995.

- 4.4 Go to "Data" and "Clear Statistics".

- 4.5 Go to "Method" and "Calibration Setup".

- 4.6 Set the "Calibrate After" entry to "15".

- 4.7 Go to "Method" and "Peak Calibration".

- 4.8 Click on the "Next" box until the calibration information of the desired gas is displayed.

- 4.9 Enter the concentration of the calibration gas in the box marked "Cal. Amount".

- 4.10 Click on the "OK" box.

- 4.11 Go to "Start".

- 4.12 Enter filename from above (e.g., cal6995) in the proper place.

- 4.13 Select "Recall".

Note: GC will average the last five runs and enter results in the "Peak Calibration" section for each gas in each channel.

- 4.14 Check the External Standard Report (see Section 1.11.5) and verify that the calibration gas concentration is displayed on the appropriate row of the report.

Note: The above steps assume that you have a method that properly analyzes each peak. If not, notify the cognizant scientist. The time to

refine the method is just after step 4.3. Remember to store the method under a new name. Save the old method for archives in case you need it later. Also remember to save the method after step 4.13 so the calibration will be held in permanent memory.

Exhibits/Attachments (optional)

Exhibit #1: Schematic Diagram of Apparatus

Exhibit #1

SCHEMATIC DIAGRAM OF APPARATUS

Appendix

4.0 References

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