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Pacific Northwest National Laboratory

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U.S. Department of Energy

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

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Richland, Washington 99352

Summary

An N-Reactor inner element that had been stored underwater in the Hanford 100-Area K-West Basin was subjected to a combination of low- and high-temperature vacuum drying treatments. These studies are part of a series of tests being conducted by Pacific Northwest National Laboratory on the drying behavior of spent nuclear fuel (SNF) elements removed from both the K-West and K-East Basins.

The drying test series was designed to test fuel elements that ranged from intact to severely damaged. The fuel element discussed here was removed from K-West canister 1990 during the first fuel selection campaign conducted in 1995. Visual inspection of this fuel element indicated that it was likely intact, having only a small dent or chip at one end. The element has remained in wet storage in the Postirradiation Testing Laboratory (PTL, 327 Building) since that time.

The drying test was conducted in the Whole Element Furnace Testing System located in G-Cell within the PTL. The furnace testing system is composed of three basic systems: the in-cell furnace equipment, the system gas loop, and the analytical instrument package. Element 1990 was subjected to drying processes proposed under the Integrated Process Strategy, which included a hot drying step. The testing cycles are listed below:

- Cold Vacuum Drying (CVD) at 50°C under vacuum for approximately 8 hr
- Pressure Rise Test under static low-pressure conditions at 50°C for 1 hr
- Gas Evolution Test at 75°C, at near-atmospheric pressure, using an argon background gas for approximately 12 hr
- Hot Vacuum Drying (HVD) at 300°C under vacuum for a total of 120 hr (24 hr for heatup to 300°C at atmospheric pressure, followed by 96 hr at 300°C)
- Water Removal Verification Test at 425°C under vacuum for 6 hr
- System cooldown to ambient conditions.

The removal of free water in the system during the CVD process was not complete. During the Pressure Rise Test and Gas Evolution Test, measurable amounts of water were detected by the moisture probe, representing the remaining free water in the system. The water that was not released during CVD is consistent with evaporation from surface films on the element and on the heated inner surfaces of the furnace.

The Pressure Rise Test in vacuum showed a linear pressure rise with time at constant temperature. The moisture release kinetics are, therefore, independent of concentration of moisture in the system (i.e., a zeroth order reaction). The atmosphere of the system was far removed from saturation, thereby providing

an unlimited sink for water molecule release. The linear release, and the small quantity of moisture evolved, suggests partial evaporation of a surface film of one or more monolayers.

Thermal decomposition of hydrated species was the most significant factor in controlling the removal of water from the system during the HVD. Gas flow and vacuum condition may be secondary factors in the process of drying the SNF element. A temperature above 300°C may be required for complete drying of the hydrated species within a reasonable period of time.

Quality Assurance

This work was conducted under the Quality Assurance Program, Pacific Northwest National Laboratory (PNNL) SNF-70-001, *SNF Quality Assurance Program*, as implemented by the PNNL *SNF Characterization Project Operations Manual*. This QA program has been evaluated and determined to effectively implement the requirements of DOE/RW-0333P, Office of Civilian Radioactive Waste Management *Quality Assurance Requirements and Description (QARD)*. Compliance with the QARD requirements is mandatory for projects which generate data used to support the development of a permanent High-Level Nuclear Waste repository. Further, the U.S. Department of Energy has determined that the testing activities which generated the results documented in this report shall comply with the QARD. Supporting records for the data in this report are located in the permanent PNNL SNF Characterization Project records, *Furnace Testing of N-Reactor Element 1990.*

Contents

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Acronyms

ATS	Applied Test Systems
CVD	Cold Vacuum Drying
DACS	Data Acquisition and Control System
DP	dew point
GC	gas chromatograph
HP	Hewlett Packard
HVD	Hot Vacuum Drying
ID	inside diameter
IPS	Integrated Process Strategy
MS	mass spectrometer
NIST	National Institute of Standards and Technology
OD	outside diameter
PNNL	Pacific Northwest National Laboratory
PTL	Postirradiation Testing Laboratory
QA	Quality Assurance
QARD	Quality Assurance Requirements and Description
SFEC	single fuel element canister
SNF	spent nuclear fuel
UHP	ultra high purity
VP	vapor pressure

1.0 Introduction

The water-filled K-Basins in the Hanford 100-Area have been used to store N-Reactor spent nuclear fuel (SNF) since the 1970s. Because some leaks in the basins have been detected and some of the fuel is breached due to handling damage and corrosion, efforts are underway to remove the fuel elements from wet storage. An Integrated Process Strategy (IPS) has been developed to package, dry, transport, and store these metallic uranium fuels in an interim storage facility on the Hanford Site (WHC 1995). Information required to support the development of the drying processes, and the required safety analyses, is being obtained from characterization tests conducted on fuel elements removed from the K-Basins. A series of whole element drying tests (reported in separate documents, see Section 8.0) have been conducted by Pacific Northwest National Laboratory (PNNL)(a) on several intact and damaged fuel elements recovered from both the K-East and K-West Basins.

This report documents the results of the first of those tests (Run 1), which was conducted on an N-Reactor inner fuel element (1990) that had been stored underwater in the K-West Basin (see Section 2.0). This fuel element was subjected to a combination of low- and high-temperature vacuum drying treatments that were intended to mimic, wherever possible, the fuel treatment strategies of the IPS. The testing was conducted in the Whole Element Furnace Testing System, described in Section 3.0, located in the Postirradiation Testing Laboratory (PTL, 327 Building). The test conditions and methodology are given in Section 4.0, and the experimental results provided in Section 5.0. These results are further discussed in Section 6.0.

(a) Operated by Battelle for the U.S. Department of Energy under Contract DE-AC06-76RLO 1830.

2.0 N-Reactor Inner Element 1990

An N-Reactor fuel assembly consists of an inner element and outer element made from a uranium alloy co-extruded with a Zircaloy-2 cladding. Both elements are annular, right-cylinders. The inner element has a smaller outside diameter and is held in place within the outer element.

The fuel element chosen for Run 1 was an inner element removed from the K-West Basin (canister 1990) in 1995. This element had been in sealed water storage since 1983, and was selected because it had suffered no apparent damage from reactor discharge, handling, or storage. While it had a small chip or dent in one end, a visual inspection did not indicate that the element was breached. A view of the end that contained the small chip or dent is shown in Figure 2.1. This view, taken during the initial visual examination in 1995, does not appear to show signs of a breach in the small damaged area.

The visual inspection revealed that at least one spot-welded "shoe" was broken from the element, but the weld areas appeared intact. The visual inspection did not identify the presence of large areas covered by surface coatings, deep scratches, or other unusual artifacts on the element's exterior surface. The element did appear to have a light gray coating over the surface of one end.

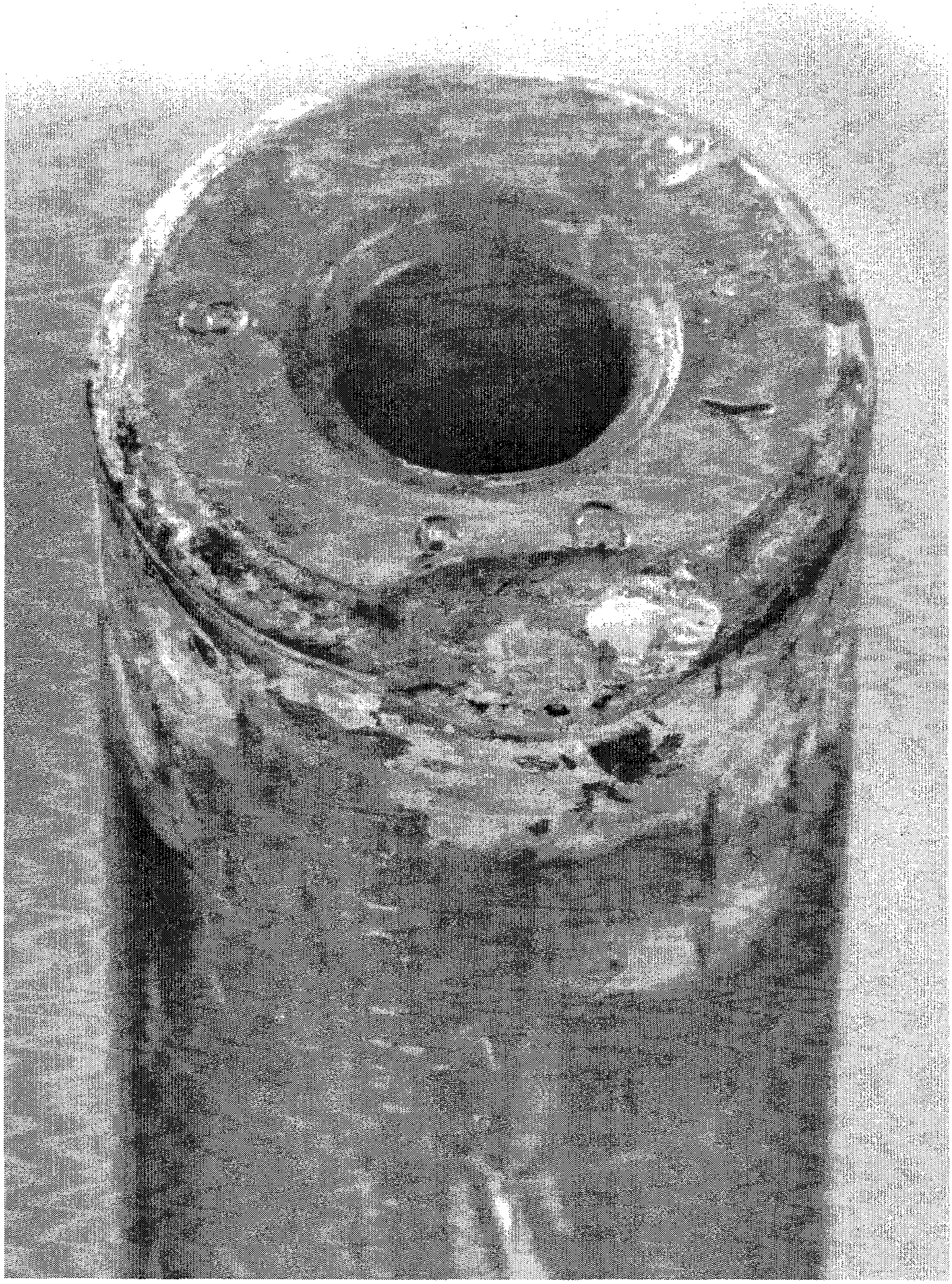

Figure 2.1. End View of N-Reactor Fuel Element 1990

3.0 Whole Element Furnace Testing System

A complete description for the Whole Element Furnace Testing System, including detailed equipment specifications, is provided in Ritter et al. (1998).

3.1 Major Systems Overview

An overview of the furnace testing system is presented in this section. The subsystems pertinent to this test report are as follows:

- Vacuum Pumping System – This system consists of a scroll-type vacuum pump, a condenser with chiller, filters, valves, and piping, which provide the vacuum pressures and flows required for the proposed IPS vacuum processes.

- Process Heating System – This system consists of a resistively heated, clam-shell furnace and a sample chamber (retort) to provide heating to the fuel element and to control process temperatures.

- Gas Supply/Distribution System – This system consists of gas bottles; mass flow controllers; piping; and valves for metering argon, air, or oxygen through the system. A bubbler is also available for adding water vapor to the system if desired.

- Gas Analysis Instrumentation – The gas analysis instrumentation includes a 300-amu quadrupole mass spectrometer (MS) and a gas chromatograph (GC) for monitoring selected elements in the process gas stream.

- Process Instrumentation – The system is equipped with several instruments for measuring process temperatures, pressures, and moisture level.

- Data Acquisition and Control System (DACS) – The DACS consists of an IBM-compatible computer and data acquisition/control unit to monitor/store key system parameters (temperatures, pressures, flows, moisture level), along with controlling the process heating system and a safety argon system.

Figures 3.1 and 3.2 are photographs of the equipment located inside and outside of the PTL G-Cell. The furnace (including retort) and some of the process piping, instrumentation, and valves are located inside the hot cell. The furnace sits on the cell floor, and the process piping is routed to a rack that hangs on the west cell wall. Process piping and electrical power and instrumentation wires pass through several split plugs on the west side of the cell. The process piping on the outside of the cell is contained within a glove bag, which provides a secondary containment as a precaution in case the process piping lines become contaminated. The vacuum pump, condenser, bubbler, GC, and the remainder of the instrumentation and valves are located inside this glove bag. Instrumentation and electrical power wires are routed through pass-through sleeves on the sides of the glove bag to the instrument rack and computer console. The instrument rack contains the readout/control unit for the pressure sensor, moisture sensor,

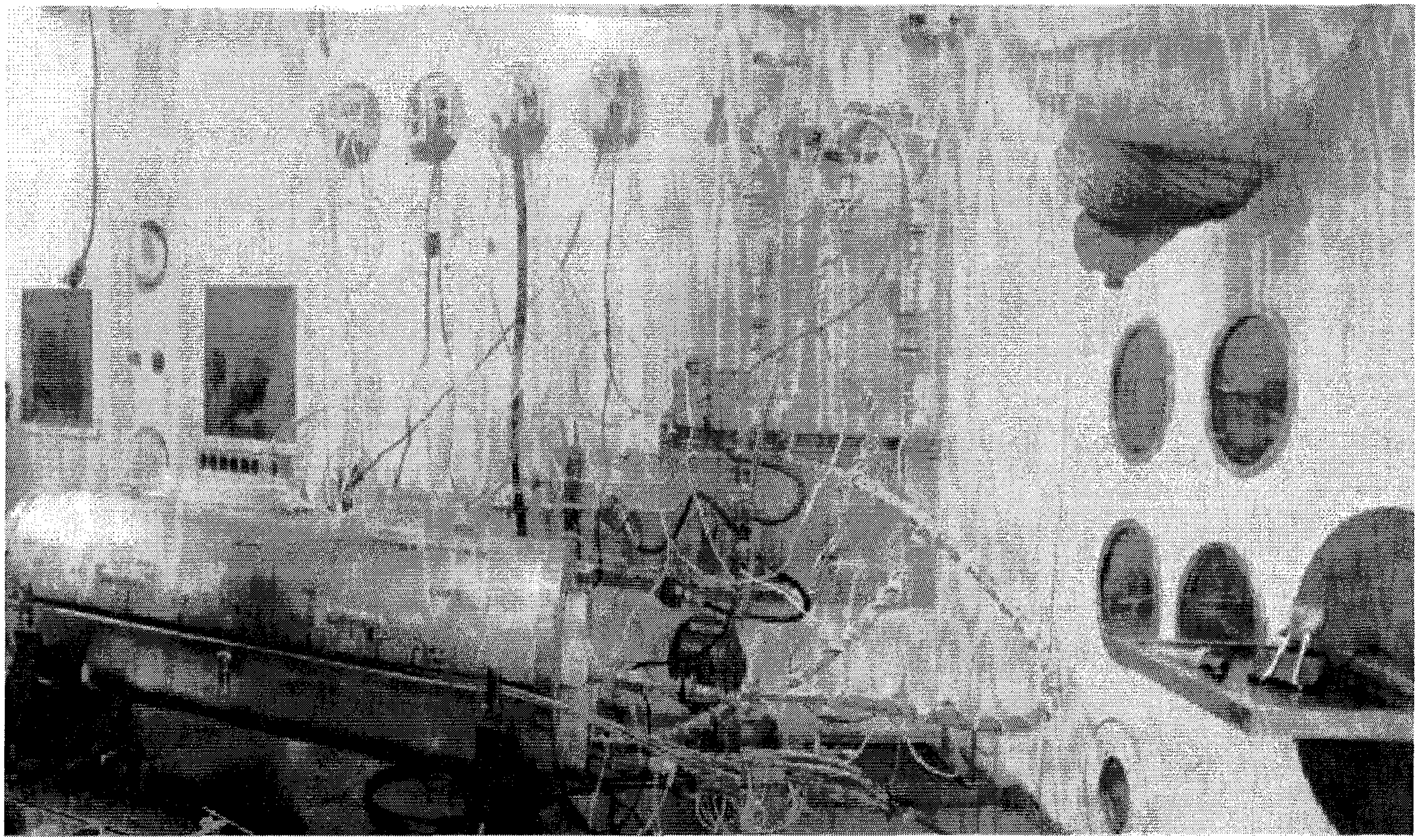

Figure 3.1. Fuel Element Furnace Components (in-cell)

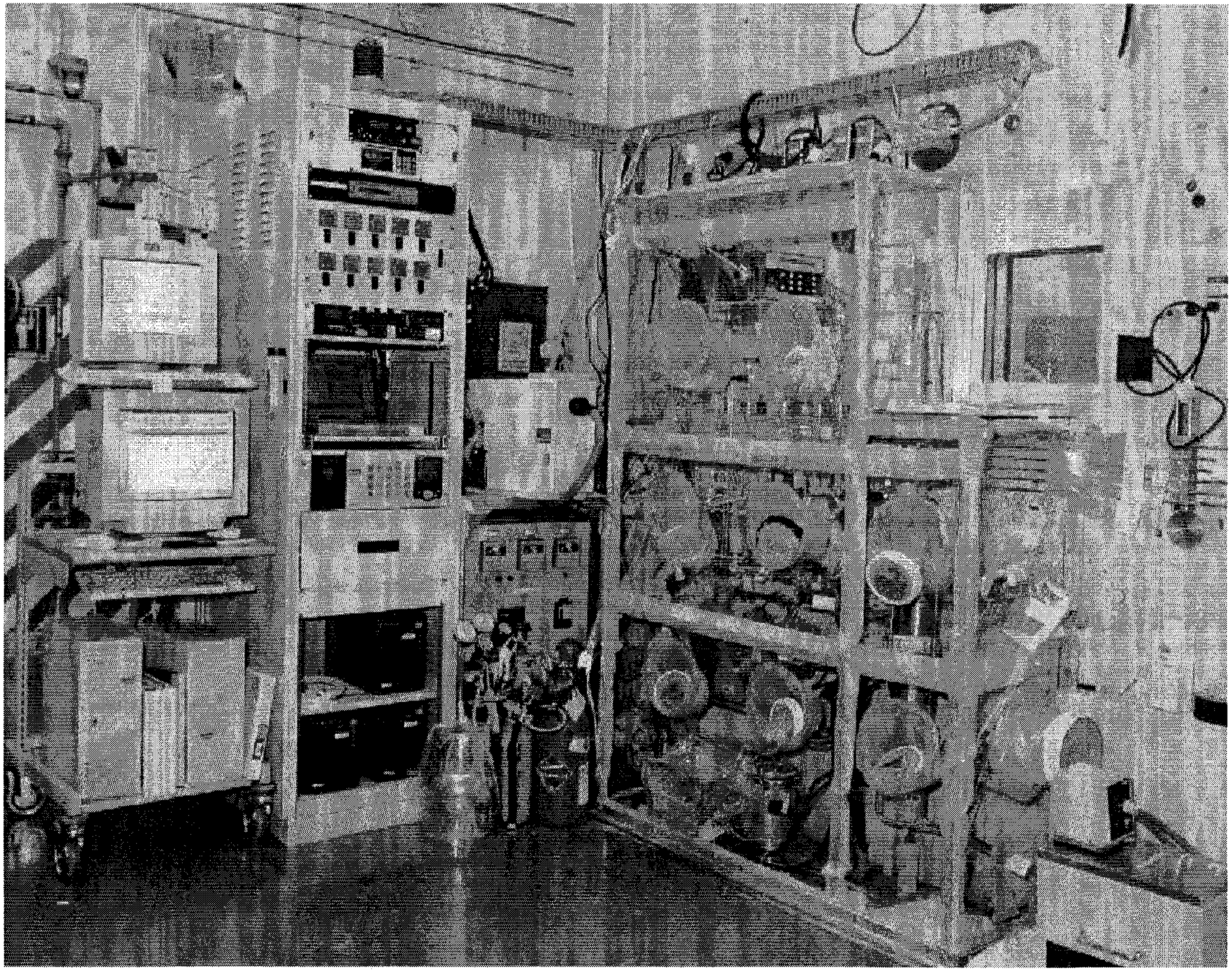

Figure 3.2. Fuel Element Furnace Components (ex-cell)

and flow controllers, along with the heat trace temperature controllers, data acquisition/control unit, GC laptop computer, and uninterruptible power supplies. The computers for the DACS and MS are located next to the instrument rack. The following sections provide more detailed descriptions of the components for these subsystems.

3.2 Vacuum Pumping System

The vacuum pumping system provides the pressures and flows required for the proposed IPS processes. This system connects the furnace retort with all the other components of the test system through various valves, fittings, and piping. The vacuum pumping system consists of the following components:

- scroll pump for evacuating the system to pressures below 1 Torr
- water condenser with refrigerated chiller for gross removal of water
- valves and piping for connecting the various components and controlling the flow direction
- particulate filters to prevent spread of contamination
- heating cords with temperature controllers for preventing condensation in lines.

3.2.1 Varian Scroll Pump

The system vacuum pump is a Varian model 300DS scroll pump. This pump has an ultimate vacuum pressure less than 10^{-2} Torr and a peak pumping speed of 250 l/min (8.8 cfm). These pressures and flows are more than adequate for simulating the conditions of the proposed IPS vacuum processes. For a single fuel element, this amount of flow may be more than desired. Therefore, a metering valve was installed on the pump inlet to throttle the flow to lower levels as required. The desired system pressure is achieved either by using the metering valve or by flowing ultra high purity (UHP) argon into the system through the entire gas loop or via a direct injection of ballast gas at the pump inlet. The use of argon gas helps to prevent the in-leakage of moisture-containing air through small system leaks (which are difficult to eliminate) that would interfere with process monitoring equipment.

3.2.2 Water Condenser

The scroll vacuum pump can be damaged by condensation of liquid water in the scroll mechanism, and, since each fuel element is wet at the start of each test, the possibility of pump damage was considered. A water condenser with corresponding chiller was installed in the system to condense the bulk of the water before it reaches the pump. This condenser can be valved into the system in series with the scroll vacuum pump or can be bypassed if not needed. The condenser cannot trap all the liberated free water, but is efficient at removing the majority of free water in the system. The condenser is only used during the first phase of a Cold Vacuum Drying (CVD) test for a single fuel element. The condenser was custom fabricated specifically for this system. Detailed sketches and specifications for the condenser are given in Ritter et al. (1998).

3.2.3 Piping, Valves, and Filters

The vacuum pumping system connects the system components through various valves, fittings, and piping. A simplified piping schematic for the system is shown in Figure 3.3. This schematic shows the basic flow path of gases through the system that was used for this test, along with the relative locations of the major components, valves, and instruments. Detailed system piping diagrams are provided in Ritter et al. (1998), along with approximate lengths for the piping lines. As seen in Figure 3.3, there are numerous valves in the system that are used to direct the flow to and from the various components. Most of the valves in the system are ball valves and range from 1/4 in. to 1/2 in. nominal size. The system piping is constructed of thin wall tubing (1/4 in. to 1/2 in. OD) and is typically connected using simple Swagelok fittings (tees, elbows, unions, etc.). Ports for gas sampling/analysis and monitoring of system pressure, temperature, and humidity are also provided at key locations in the system piping. Special fittings and pipe-threaded fittings are used in some locations for connecting piping to the process instruments.

Particulate filters are installed in the system on both the inlet and outlet to the retort to help prevent the spread of contamination to the system piping on the outside of the hot cell. These filters are constructed of a microporous fiberglass media in a stainless steel housing. They are 99.9% efficient for particulates that are 0.2 microns and larger in size. Two different size filters, manufactured by Matheson, are used in the system.

3.2.4 System Line Heaters

All of the stainless steel tubing that carries gases into the furnace retort and resultant gases from the retort is heated to about 75°C to ensure condensable water vapor remains in the gas phase. Simple heat "cords" capable of being wrapped upon each other (as required at tees, elbows, and other connections) were found to be a good heating method for this system. The heating cords are controlled by simple proportional controllers, and type-K thermocouples are installed on each heated line to monitor and record temperature using the DACS.

3.3 Process Heating System

The whole element furnace is a 4-ft-long, resistively heated, clam-shell furnace. The furnace, Series 3210 supplied by Applied Test Systems (ATS), has a temperature rating of 900°C and total heating capacity of 13,800 W. The internal dimensions are 5 in. ID by 45 in. long. The furnace has three separate sets of heating elements that allow the heating to be controlled in zones; each zone is 15 in. long and supplies up to 4600 W heating. The zones can be controlled separately to establish a flat temperature profile within the furnace even though heat is lost preferentially out the end with the retort entry flange. A heat reflector consisting of several thin Inconel plates is used to reduce heat loss from the flange end of

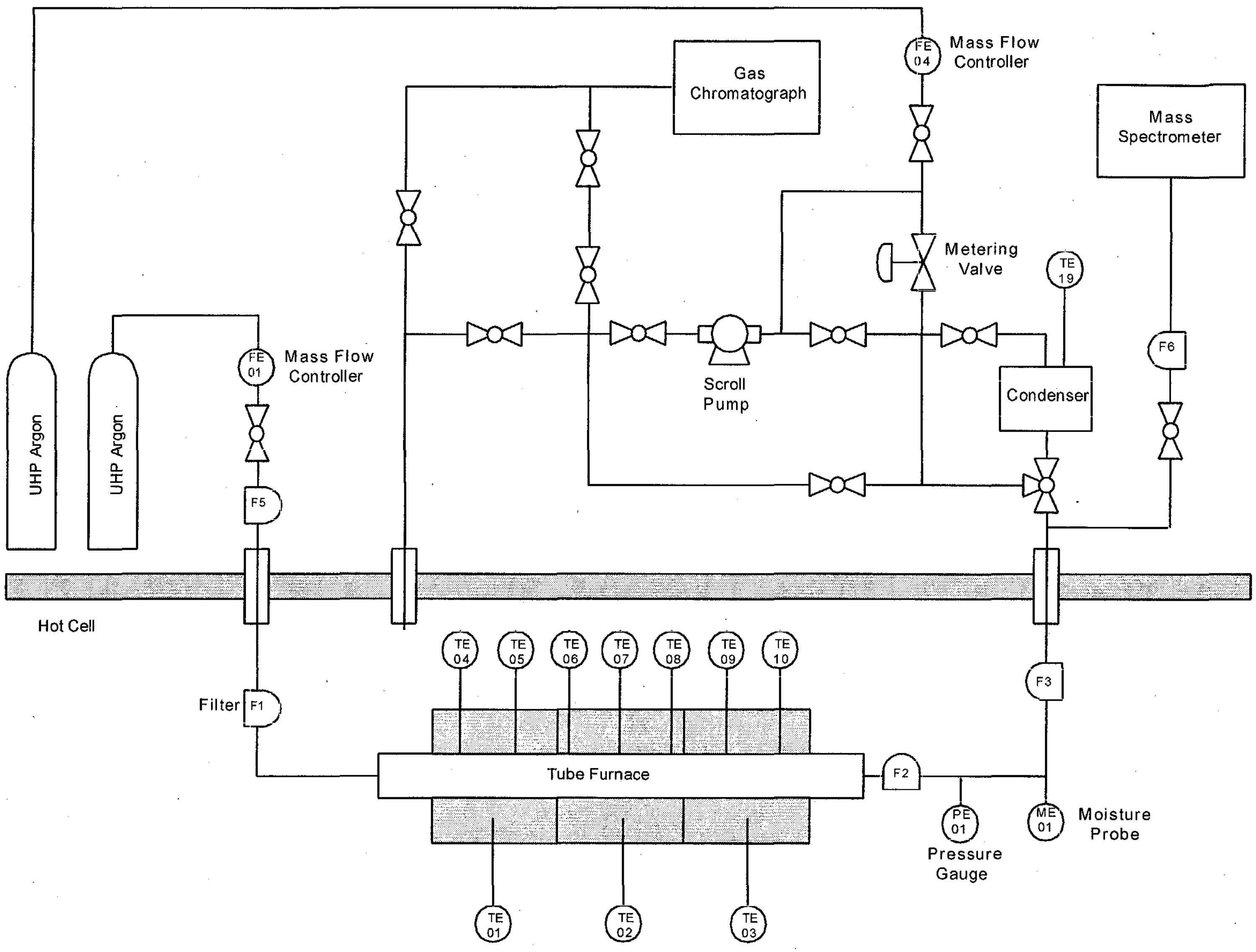

Figure 3.3. Generalized View of the Furnace Test System

the retort. The furnace controller is an ATS Series 3000, which consists of three programmable, self-tuning proportional with integral and derivative controllers. These controllers are also interfaced to the DACS, which can provide limited input to the controllers as required.

The retort, an ATS Series 3910, is an Inconel tube fitted with a gas inlet tube at one end and a gasketed flange at the other. Of all high-temperature materials, Inconel series 600 was selected to reduce the amount of oxidation and water pickup by the retort and associated components. Experience has shown that stainless steel components were easily affected by corrosion, which could then affect test results. The body of the retort is fabricated from schedule 40 Inconel pipe (4.5 in. OD, 4.026 in. ID), and the inside length is about 44.5 in. Seven type-K thermocouples are installed equidistant along one side of the retort and extend into the retort interior approximately 1/8 in. These thermocouples are used to monitor the retort temperature so that if a reaction with the fuel element occurs (which would locally raise the retort temperature), this event can be correlated with the approximate location on the fuel.

An Inconel sample/transfer boat is used to load the fuel element into the furnace. The boat is fabricated from an 11-gauge (0.120-in.-thick) Inconel 601 sheet, which is formed into a flattened u-shape. The boat has a weir and a swivel handle on each end. The weirs are used to keep free water or particulates contained in the boat as required.

3.4 Gas Supply/Distribution System

The gas supply system and vacuum pumping system together are capable of controlling the fuel element environment to vacuum or moderate pressure conditions, and/or exposing the fuel element to a variety of gases or gas mixtures. The gas loop is typically operated as a single-pass system with no capability for recirculation. The gas supply system consists of gas bottles; mass flow controllers; piping; and valves for metering argon, air, or oxygen through the system. A bubbler is also available for adding water vapor to the process gas stream as required, but it was not used in these tests.

The gas supply system contains three Matheson mass flow controllers calibrated for argon, air, and oxygen. All gases are typically specified "ultra high purity" and are additionally filtered for water using molecular sieve columns. Argon is the principal inert gas used, as it is more dense than air; provides reasonable thermal conductivity; and requires simpler handling procedures than lighter gases such as helium. The argon purge gas is introduced into the retort through FE-01, which is a Matheson model 8272-0422 oxygen controller, recalibrated for argon gas at 25°C using a NIST-traceable bubble flow meter. Air and oxygen are not currently used because any oxidative steps have been deleted from the current IPS for the SNF. The manufacturer's specifications for the controllers' flow rate ranges are 0 - 304 sccm argon, 0 - 1000 sccm air, and 0 - 10 sccm oxygen. If higher flow rates are desired, a new mass flow controller with a higher range could be procured and installed in the system.

3.5 Gas Analysis Instrumentation

3.5.1 Balzers Omnistar Mass Spectrometer

The Balzers Omnistar Mass Spectrometer is a compact, computer-controlled, quadrupole MS capable of scanning to 300 amu. The unit is capable of monitoring up to 64 components within a gas stream with a nominal detection limit of less than 1 ppm for most gases other than hydrogen. The MS was used to monitor hydrogen, nitrogen (for air in-leakage), krypton, xenon, and other elements during the test.

The MS was modified as a result of early system testing and calibration to improve the time response to small changes in hydrogen pressure. Prior to testing, the MS was calibrated for hydrogen using mixtures of hydrogen and helium, and hydrogen and argon gas. The residence time of each gas could be measured in the quadrupole chamber, and it was observed that the hydrogen decay time was approximately four times as long as helium. This was not unexpected as turbomolecular pumps have a lower pumping efficiency for very light gases. In standard practice this is acceptable, but, for these tests, where determining hydrogen could be very important, steps were taken to improve the hydrogen decay time. The MS vacuum system was modified by adding a stainless steel flanged tee, a gate valve, and a room-temperature hydrogen getter downstream from the quadrupole. Under vacuum the gate valve can be opened, exposing the getter to the system to help scavenge hydrogen from the system following analysis. This modification reduced the residence time of hydrogen in the system substantially and decreased the background level of hydrogen by about a factor of 2. The getter improved the system response to transient events that may result in the release of hydrogen. The MS was calibrated at ~30 Torr head pressure with a certified gas standard consisting of 1050 ppmv hydrogen in argon.

3.5.2 MTI M200 Gas Chromatograph

The MTI M200 Gas Chromatograph is a high-speed GC that is used to monitor the quantities of hydrogen and other light gases in the furnace testing system gas loop. This instrument is interfaced with a laptop computer to record data. The GC is designed to operate at near-atmospheric pressure; thus it may be configured in two different ways for measurement purposes. At system pressures near atmospheric, the GC is configured to sample directly from the gas loop ahead of the system vacuum pump. When the system is under vacuum, the GC is configured to sample from the exhaust side of the vacuum pump. The gas output from the pump is sufficiently compressed that the GC can sample and analyze this gas. The GC was calibrated with a certified gas standard consisting of 114 ppmv hydrogen in synthetic air. No correction for the difference in the sample pressure and calibration pressure is applied, since both are ~760 Torr (~1 atm).

3.6 Process Instrumentation

The furnace testing system contains several process instruments for monitoring moisture content, pressure, and temperature. The key instruments are as follows:

- Panametrics moisture monitor
- MKS Baratron pressure transducer
- Type-K thermocouples.

3.6.1 Panametrics Moisture Monitor

The Panametrics moisture monitor model MMS35 uses a solid electrochemical probe (model M2L) that measures moisture by measuring the characteristic capacitance of the probe as a function of the moisture in the gas phase. The sensor has a nominal dew point range of -110°C to 20°C. Previous testing indicated that contamination by radioactive elements (e.g., cesium) causes the probe to lose calibration and results in moisture readings that drift with time. To prevent contamination of the probe tip, the probe is installed in the gas loop downstream of two glass particulate filters. Further, the probes are changed following each test and surveyed for radioactive contamination. If no contamination is found, and the data correlate well with the data obtained from the MS, the readings are accepted.

A calibration verification procedure can be performed using calibrated water "leak" tubes. These tubes can be placed inside the furnace and, when heated, will establish a known water vapor pressure in the system. However, this procedure is time intensive; approximately 2 weeks are required to calibrate one probe over the range of moisture likely to be encountered in these tests. This procedure is only used if the moisture monitor results vary widely from the MS data.

Output of the moisture monitor is in dew point (DP) in degrees Celsius. For comparison with other test data, these dew point values were converted to water vapor pressure in Torr using the water and ice vapor pressure data shown in Table 3.1. Interpolation of the data was accomplished using a 6th-order polynomial fit to the log of the vapor pressure (VP) versus temperature data. The resulting conversion expression is as follows:

$$\text{VP (Torr)} = 10^{x}, \qquad (3.1)$$

where

$$\begin{aligned} x = {} & -6.7260\text{E-}12*(\text{DP})^6 - 1.7250\text{E-}09*(\text{DP})^5 - 1.7089\text{E-}07*(\text{DP})^4 \\ & - 7.2618\text{E-}06*(\text{DP})^3 - 2.9668\text{E-}04*(\text{DP})^2 + 3.4414\text{E-}02*(\text{DP}) \\ & + 6.6043\text{E-}01 \end{aligned}$$

3.6.2 MKS Baratron Pressure Transducer

An MKS Baratron model 690 calibrated pressure transducer coupled with an MKS model 270 signal conditioner is used as the primary measurement for the overall system pressure. As shown in Figure 3.3, PE-01 measures the system pressure downstream of the retort outlet, and indicates pressure in the range of 0.1 Torr to 10,000 Torr. The signal conditioner has an analog output that is interfaced to the DACS so that system pressure is continuously recorded.

Table 3.1. Water and Ice Vapor Pressure Data Versus Temperature

Dew Point	**Vapor Pressure (VP)**		
(°C)	**(Pa)[a]**	**(Torr)**	**Log (Torr)**
-80	5.5000E-02	4.136E-04	-3.3834
-75	1.2200E-01	9.174E-04	-3.0374
-70	2.6100E-01	1.963E-03	-2.7071
-65	5.4000E-01	4.061E-03	-2.3914
-60	1.0800E+00	8.122E-03	-2.0904
-55	2.0930E+00	1.574E-02	-1.8030
-50	3.9360E+00	2.960E-02	-1.5287
-45	7.2020E+00	5.416E-02	-1.2663
-40	1.2840E+01	9.656E-02	-1.0152
-35	2.2350E+01	1.681E-01	-0.7745
-30	3.8010E+01	2.858E-01	-0.5439
-25	6.3290E+01	4.759E-01	-0.3224
-20	1.0326E+02	7.765E-01	-0.1099
-15	1.6530E+02	1.243E+00	0.0945
-10	2.5990E+02	1.954E+00	0.2910
-5	4.0176E+02	3.021E+00	0.4802
0	6.1129E+02	4.597E+00	0.6625
10	1.2281E+03	9.235E+00	0.9655

(a) CRC Press. 1997. *Handbook of Chemistry & Physics*, 78th edition.

3.6.3 Thermocouples

Thermocouples provide a simple, reliable method for measuring system temperatures. As shown in Figure 3.3, thermocouples are installed at various locations in the system to provide key temperature measurements. The retort temperatures are of primary importance, and these temperatures are measured by thermocouples TE-04 through TE-10, which are positioned equidistant along the length of the retort. Thermocouples TE-11 through TE-17 are used for controlling the temperature of the heated lines. All thermocouple readings are continuously recorded using the DACS.

3.7 Data Acquisition and Control System

The DACS monitors system parameters, and controls the furnace and the safety argon system. The DACS consists of a Hewlett Packard (HP) 3497A data acquisition/control unit and an IBM-compatible computer. A National Instruments general purpose interface bus card, installed in the IBM-compatible computer, is used to communicate with the HP 3497A. The computer communicates with the furnace temperature controllers over serial port 0 using an RS-232/RS-485 converter. The DACS uses National Instruments LabView for Windows as the control software.

The DACS is designed to measure critical system parameters during fuel conditioning tests, including temperatures, pressures, flow rates, and moisture level. The measured parameters are converted to engineering units, displayed on the computer screen, and stored to disk at user-defined intervals. The data files are stored in a tab-delimited format to allow importing into a standard spreadsheet or plotting program. A plotting screen also allows for plotting of up to six parameters at a time.

Limited control of the furnace can be performed with the DACS. Each of the three furnace zone temperatures can be remotely set by the DACS. In addition, the DACS allows the operator to start and stop the furnace and select one of four temperature profiles that are pre-programmed in the furnace temperature controllers. Note that these profiles must be programmed manually in the furnace controllers before using the DACS to select them.

4.0 Vacuum Drying Testing of Element 1990

The drying test was performed in accordance with the requirements of *Test Plan for Whole Element Furnace Runs 1, 2, and 3*, SNFCT97:053:R00, and the individual Test Procedure, *Furnace Testing of N-Reactor Fuel Element 1990*, PTL-169, Revision 0. These documents are located in the PNNL permanent project records for this test.

The testing consisted of three parts (discussed in this section):

- removing the fuel element from its shipping canister, performing a visual inspection, loading the element onto the furnace system sample boat, and transferring the element to the PTL G-Cell for loading into the furnace.
- drying the fuel element using a combination of Cold Vacuum Drying (CVD) and Hot Vacuum Drying (HVD) processes.
- unloading the furnace, performing a post-test visual inspection, and returning the fuel element to its shipping canister.

4.1 Fuel Element Transfer and Loading

4.1.1 Pre-Test Visual Inspection

The pre-test visual inspection was conducted using a Panasonic Super-VHS video camera located outside the PTL F-Cell (adjacent to G-Cell), where the sample was unloaded from the shipping canister. The results were recorded using a Panasonic Super-VHS resolution video recorder. This examination was conducted to document the condition of the fuel element prior to the test and to determine if any changes had occurred since 1995 when the fuel element was first brought to the PTL. The results of this inspection are presented in Section 5.1.

4.1.2 Fuel Element Rinsing

Fuel element 1990 had been stored in the PTL water storage pool contained in a single fuel element canister (SFEC) that was filled with deionized water following completion of the 1995 visual examination. Before the start of the fuel element drying, the element was rinsed in F-Cell. This rinsing involved raising and lowering the element several times in the SFEC using one of the cell's manipulators. Following rinsing, the element was transferred to G-Cell for loading into the test retort.

4.2 Fuel Element Drying

The element was subjected to cold and hot vacuum drying. The drying test was conducted in six phases:

1. Cold Vacuum Drying
2. Pressure Rise Test
3. Gas Evolution Test
4. Hot Vacuum Drying (first step)
5. Hot Vacuum Drying (second step)
6. Water Removal Verification.

The conditions used for each of these test phases are summarized in Table 4.1. Each test phase is described below.

Table 4.1. Summary of Test Conditions

Test Segment	Test Conditions for Element 1990
A. Cold Vacuum Drying	
System Configuration	Normal
Test Temperature, °C	50
Atmosphere	Vacuum, Ar background
Pressure, Torr	<5
Gas Flow Rate, cc/min	300
Gas Species Monitored	H_2, H_2O, N_2, O_2, CO_2, Ar, Kr, Xe
Duration, hr	The duration for CVD was at least 2 hr or until free water being removed read either background or below detectable limits.
B. Pressure Rise Test	
System Configuration	Test Chamber Isolated
Test Temperature, °C	50
Atmosphere	Vacuum, Ar background
Initial Pressure, Torr	<5
Gas Species Monitored	H_2, H_2O, N_2, O_2, CO_2, Ar, Kr, Xe
Pressure Rise (acceptable levels, Torr)	<3
Duration, hr	1
C. Gas Evolution Test	
System Configuration	Test Chamber Isolated
Test Temperature, °C	75
Atmosphere	Argon
Initial Pressure, Torr	760 (~atmospheric)
Gas Species Monitored	H_2, H_2O, N_2, O_2, CO_2, Ar, Kr, Xe
Pressure Rise (acceptable levels, Torr)	<120
Duration, hr	12

Table 4.1. (contd)

Test Segment	Test Conditions for Element 1990
D. Hot Vacuum Drying (Step 1, heatup)	
System Configuration	Normal
Test Temperature Range, °C	75 to 300
Temperature Ramp Rate, °C/hr	10
Atmosphere	Argon
Pressure, Torr	760 (~atmospheric)
Gas Flow Rate, cc/min	300
Gas Species Monitored	H_2, H_2O, N_2, O_2, CO_2, Ar, Kr, Xe
Duration, hr	~24
E. Hot Vacuum Drying (Step 2, drying)	
System Configuration	Normal
Target Test Temperature, °C	300
Atmosphere	Vacuum, Ar background
Pressure, Torr	<5
Gas Species Monitored	H_2, H_2O, N_2, O_2, CO_2, Ar, Kr, Xe
Duration, hr	~96
F. Water Removal Verification	
System Configuration	Normal
Target Test Temperature, °C	400°C
Atmosphere	Vacuum, Ar background
Gas Species Monitored	H_2, H_2O, N_2, O_2, CO_2, Ar, Kr, Xe
Duration, hr	~6 hr

4.2.1 Cold Vacuum Drying

While the fuel element was being handled and prepared for the drying test, it was sprinkled with deionized water to keep it damp. The amount of surplus liquid water, though small, could not be ascertained. There were no pools of water in the sample boat; however, droplets did exist. The element was then loaded into the furnace, and the test started.

The furnace temperature was raised to approximately 50°C and allowed to stabilize. The system vacuum pump was then turned on, and the system water condenser valved in. Once the system pressure reached the vapor pressure of free water, the vacuum pump was valved out of the gas loop and the condenser valved in to remove the majority of the free water from the system. During this time, the pressure dropped steadily until the water vapor pressure became lower than the condenser could extract. At this point, the water content in the gas phase began to rise, and the condenser was valved out of the system while the vacuum pump was valved back in to continue evacuating the system.

The vacuum pump is capable of reducing the system pressure to levels lower than desired for CVD. To stabilize the system pressure near the desired value of approximately 5 Torr, dry, UHP argon was metered into the system. CVD was conducted at approximately 6.5 Torr for approximately 8 hr. The flow of UHP argon through the furnace system also minimized in-leakage of cell air into the retort tube.

4.2.2 Pressure Rise Test

The Pressure Rise Test involved isolating the fuel element from the effects of flowing gas, and continued vacuum pumping and measuring any system pressure increases while at CVD pressure and temperature conditions. This test was conducted by valving the vacuum pump out of the gas loop, turning off the argon gas flow, and closing the exhaust valves in the gas loop. The test duration was 1 hr.

4.2.3 Gas Evolution Test

This test was conducted at 75°C in a background of UHP argon gas at nearly 1 atmosphere total system pressure. This test was performed by raising the system pressure and temperature simultaneously to the test conditions. The system pressure was elevated by metering UHP argon gas into the system until the desired pressure was reached. The gas flow was then turned off, and the fuel element isolated for 12 hr at these conditions.

4.2.4 Hot Vacuum Drying, Step 1

The first step of HVD involved heating the fuel element from 75°C to 300°C at a slow ramp rate of 10°C/hr in a flowing argon atmosphere near 1 atmosphere system pressure. UHP argon was allowed to flow through the system at approximately 300 cc/min and exhausted to the hot cell. This portion of the test took about 24 hr.

4.2.5 Hot Vacuum Drying, Step 2

The second part of the HVD test cycle required the system to be evacuated to approximately 5 Torr while the temperature was held at ~300°C. The furnace stabilized at about 325°C, where the test was then conducted. Again, argon gas was metered into the system to establish the vacuum conditions in the test system. The second step of the HVD process was conducted for approximately 96 hr.

4.2.6 Water Removal Verification

Previous studies (Abrefah et al. 1998) have shown that water may be released from the fuel through the decomposition of uranium oxy-hydrates. Thus, to check for the presence of high-temperature hydrates and evaluate the completeness of the CVD and HVD cycles, the element was heated to ~400°C. The furnace stabilized at about 425°C. The test was conducted under vacuum for approximately 6 hr with argon metered into the system as the background gas.

4.3 Post-Test Visual Examination

The removal of the element from the furnace test system was documented using a Philips digital color camera. The entire outer surface of the element was inspected, and the results recorded using a Super-VHS videotape recorder (see Section 5.9).

5.0 Experimental Results

In the following sections, the experimental data collected during the drying test are expanded and plotted for each segment. Summary results from the drying test are plotted in Figure 5.1. This figure shows the system moisture level response to the pressure changes and the retort tube temperature during the test. Time intervals for the various test segments are shown in the upper section of the plot, and also outlined in Table 5.1. The temperatures shown in Figure 5.1 were recorded from one of seven thermocouples (TE-06) on the system located near the center of the retort. Measurements conducted in subsequent fuel element tests (described in separate reports) have established that the total pressure data for this first run had a positive offset of approximately 3 Torr.

5.1 Pre-Test Visual Examination

A still image from the video camera examination shows the chipped or dented end of the fuel element (Figure 5.2). This newly captured image still lacks fine definition, but the pre-test visual examination did suggest that the damaged end may have a small crack exposing a small portion of the uranium metal. However, no swelling or ballooning of the fuel was apparent if indeed the fuel element was breached. No other differences were noted in the apparent surface condition since the 1995 visual examination photograph (Figure 2.1).

5.2 Cold Vacuum Drying

The water release from the CVD portion of the test is shown in Figure 5.3. This plot spans the period when the furnace was loaded with the fuel element through when the system was heated for the Pressure Rise Test and the system pressure raised to atmospheric. The baseline moisture partial pressure in the system prior to heating was 5 Torr. This moisture pressure was below the theoretical saturation temperature for the argon/air gas mixture (residual air from when the fuel element was loaded) at ~21°C. This saturation pressure is approximately 20 Torr, yielding a relative humidity of approximately 29% in the system. The moisture pressure increased from the baseline to ~6 Torr when the system was heated to ~50°C.

The total pressure of the closed system also increased from 748 Torr to 834 Torr when the temperature was increased from 21°C to 50°C. Theoretically, for an ideal gas, the expected pressure rise due to a temperature increase from 21°C to 50°C is 74 Torr, 12 Torr below the measured pressure rise of 86 Torr. The additional increase in pressure was likely due to vaporization and/or desorption of water vapor from the SNF element, the sample boat, and small contributions from the remainder of the furnace system. The moisture trace in Figure 5.3 shows that the desorption of the moisture is delayed; occurring rapidly when the system reaches about 50°C.

Figure 5.4 shows that the moisture pressure reached a maximum of about 12.5 Torr. The temperature plot in Figure 5.4 indicates the retort tube, and most probably the fuel element, had not reached the 50°C setpoint temperature before the system was evacuated. Evacuating the system with the water condenser

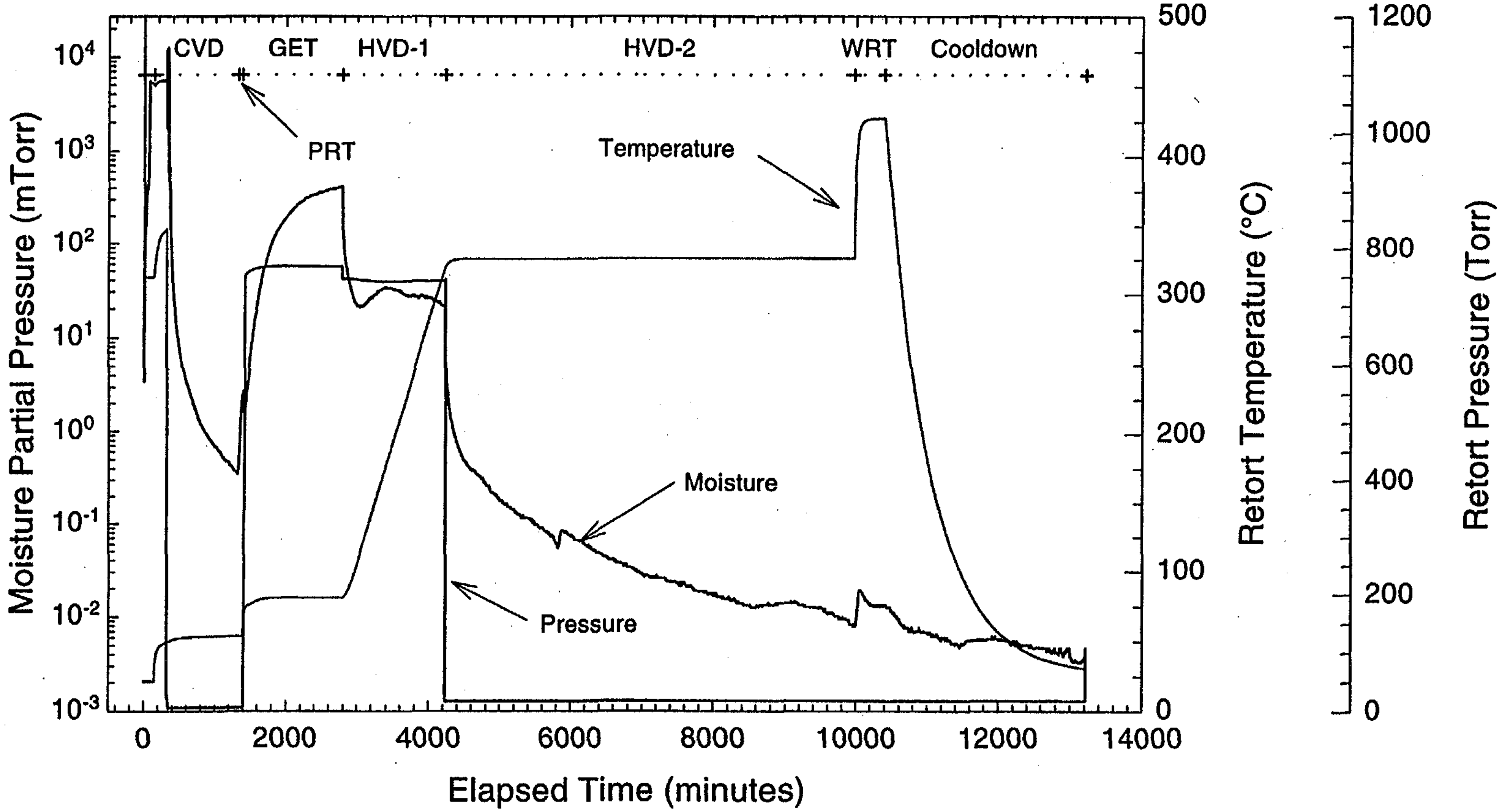

Figure 5.1. Drying of SNF Element 1990, Summary Plot

Table 5.1. Fuel Element 1990 Drying Test Time Line

Activity	Date/Time	Elapsed Time (min)
Start of Test		
Close and seal furnace, start test	9/22/97 12:32	133
Heat furnace to 50°C	9/22/97 12:41	142
Cold Vacuum Drying Test		
Open pump valve and condenser to furnace	9/22/97 15:37	318
Close pump valve	9/22/97 15:39	320
Open pump valve, close condenser valve	9/22/97 15:59	340
Start Ar flow to attain a retort pressure of ~5 Torr	9/22/97 16:12	353
Pressure Rise Test		
Close pump and Ar flow valves (isolate furnace)	9/23/97 8:26	1327
Gas Evolution Test		
Raise furnace temperature to 75°C	9/23/97 9:27	1388
Start Ar flow at ~300 cc/min	9/23/97 9:39	1400
Stop Ar flow when furnace pressure reaches ~1 atm (~760 Torr)	9/23/97 10:09	1430
Hot Vacuum Drying Test (Step 1)		
Raise furnace temperature to 300°C @ 10°C/min		
Start Ar flow at ~300 cc/min (vent to cell)	9/24/97 8:44	2785
Hot Vacuum Drying Test (Step 2)		
Hold furnace temperature and Ar flow rate		
Open pump valve	9/25/97 8:42	4223
Water Removal Verification		
Raise furnace temperature to ~400°C	9/29/97 8:30	9971
Turn off furnace heaters, end test	9/29/97 15:30	10391

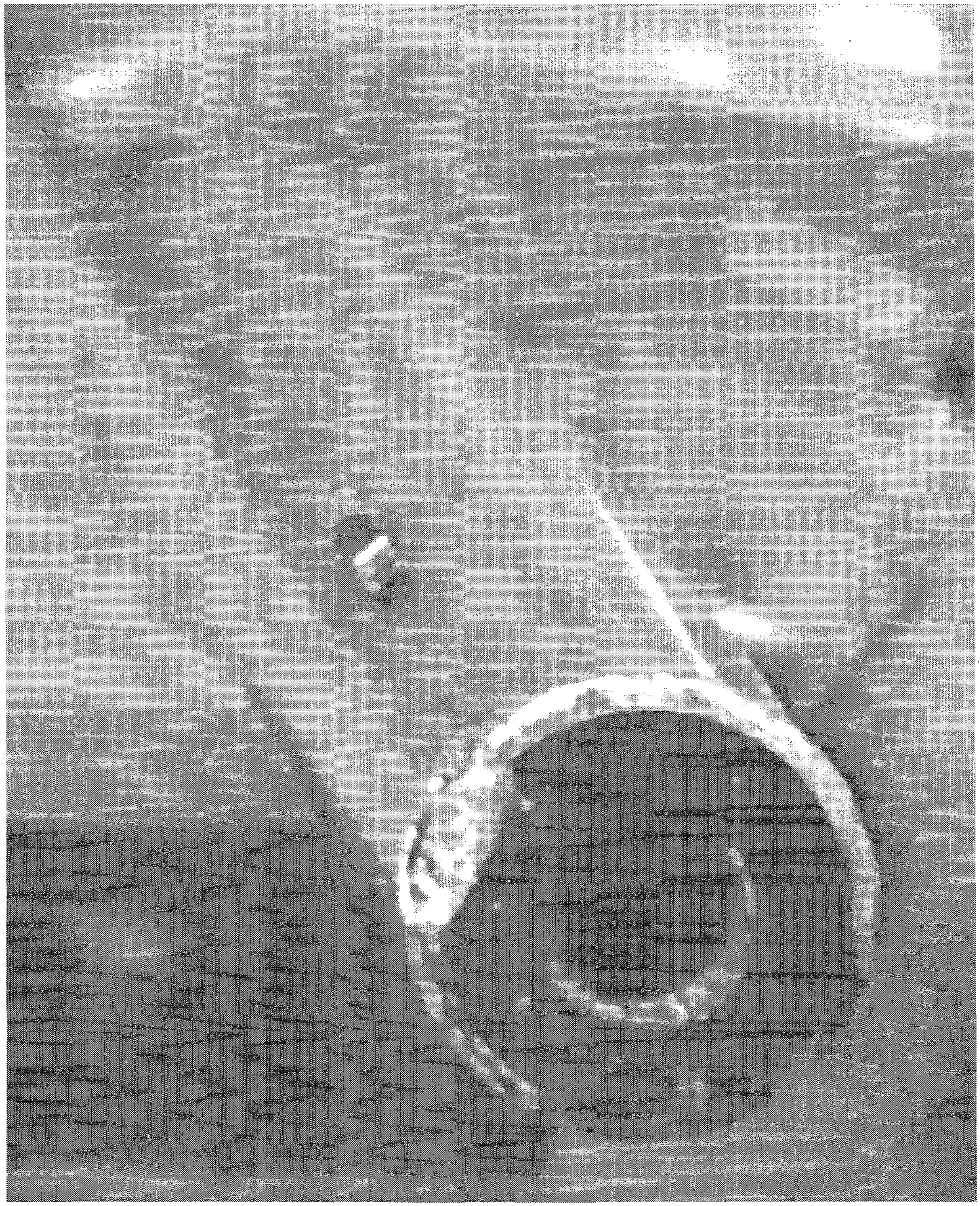

Figure 5.2. End View of Element 1990 Prior to Drying Test

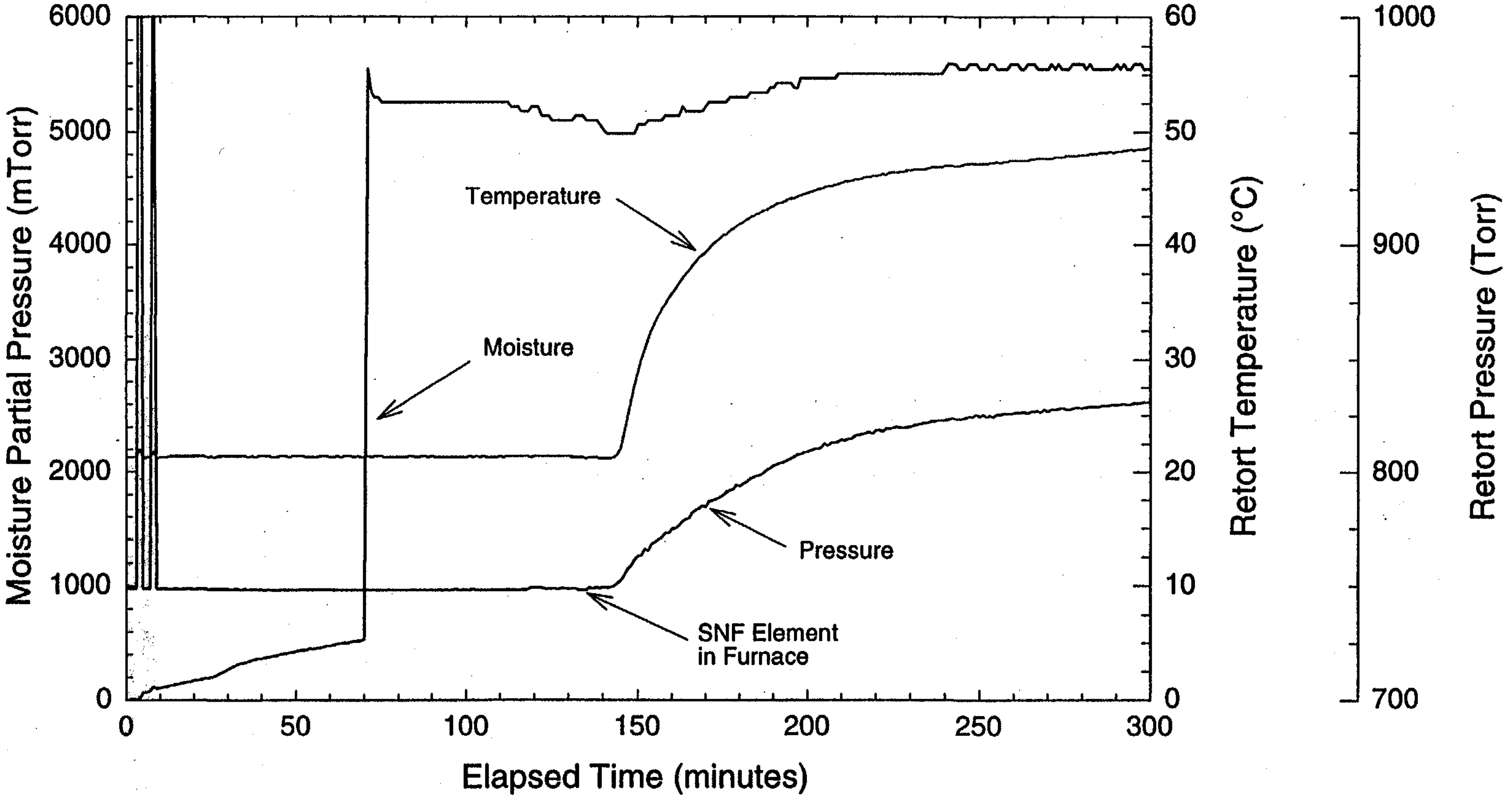

Figure 5.3. Drying of SNF Element 1990, Prior to CVD

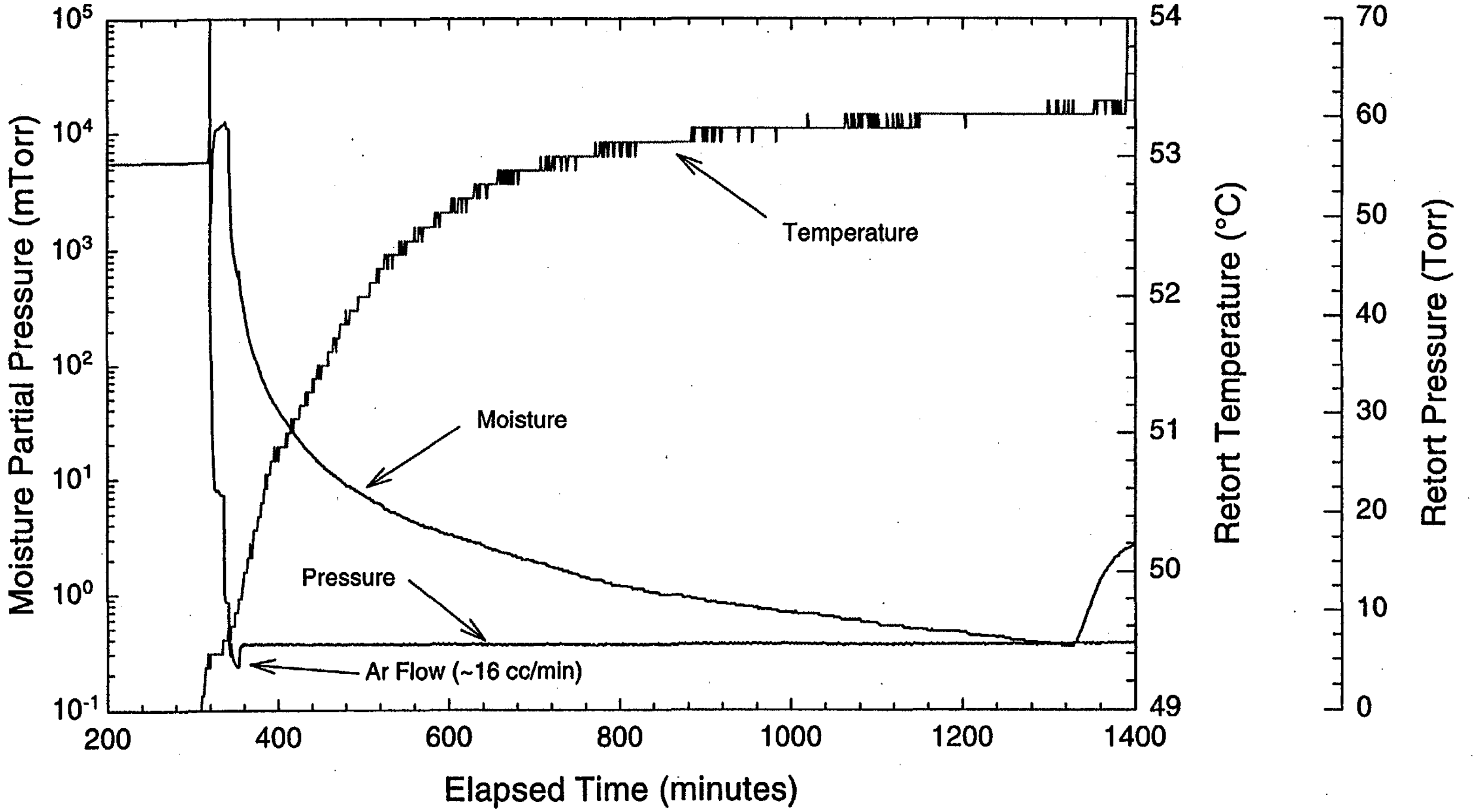

Figure 5.4. Drying of SNF Element 1990, CVD in Vacuum

and scroll pump resulted in a decrease in the total pressure as well as in the partial pressure of moisture. The decrease in the total pressure was more rapid than the decrease in the moisture pressure. The slight system pressure increase shown as a step in Figure 5.4 was due to the introduction of argon gas into the system to provide a controlled background atmosphere.

The continuing decrease in the moisture partial pressure at the end of the CVD indicates that the total CVD time of ~17 hr was insufficient to remove all the *free* (i.e., not chemically bound) water from the system. System moisture pressure was 0.35 mTorr at the end of CVD. An extrapolation of the moisture data would predict that reaching a pressure of ~0.1 mTorr would require an additional 25 hr of CVD.

5.3 Pressure Rise Test

The Pressure Rise Test results are shown in Figure 5.5. Noise on the total pressure data is due to undersampling of the Baratron pressure gauge output and resolution limitations with the 0 to 10,000 Torr Baratron sensor.

As a result of this noise, no meaningful indication of total pressure rise could be obtained. However, the partial pressure of the moisture in the system increased linearly with time, as indicated by the regression line shown in Figure 5.5. The rate of increase was ~0.04 mTorr/min. Assuming ideal gas behavior of the water vapor, the rate of desorption of the water will be constant, given by

$$\frac{dn}{dt} = \frac{kV}{RT} \tag{5.1}$$

where n is the number of moles of gas, V is the volume of the system (~9100 cm^3), R is the gas constant (82.06 $\text{cm}^3\cdot\text{atm/g-mol}\cdot\text{K}$), T is the temperature (326.3 K), and k is a constant and equal to the slope of the regression line (i.e., dP/dt). The total amount of water released to the system during the Pressure Rise Test is given by the integral of the above equation. For a period of 1 hr, the total amount of water released was ~20 micrograms. The linearity of this release, and the small quantity of moisture evolved, are consistent with the partial evaporation of a surface film. Since the slope of the line did not change for the duration of the test, this suggests that the surface moisture was not reduced to less than a monolayer.

5.4 Gas Evolution Test

Figure 5.6 shows the increase in moisture partial pressure during the Gas Evolution Test at a furnace temperature of approximately 81°C. The release of moisture occurs over three distinct regions.

The first region showed negligible rise in the moisture pressure, which may be due to the slow response of the SNF element to the retort temperature ramp. This first region was followed by a rapid linear increase in the water vapor pressure, likely from delayed heating of the element. The third region of pressure rise is also linear with a slightly smaller slope than the preceding region. This latter region likely corresponds to the point at which the fuel element temperature reached a constant temperature. The

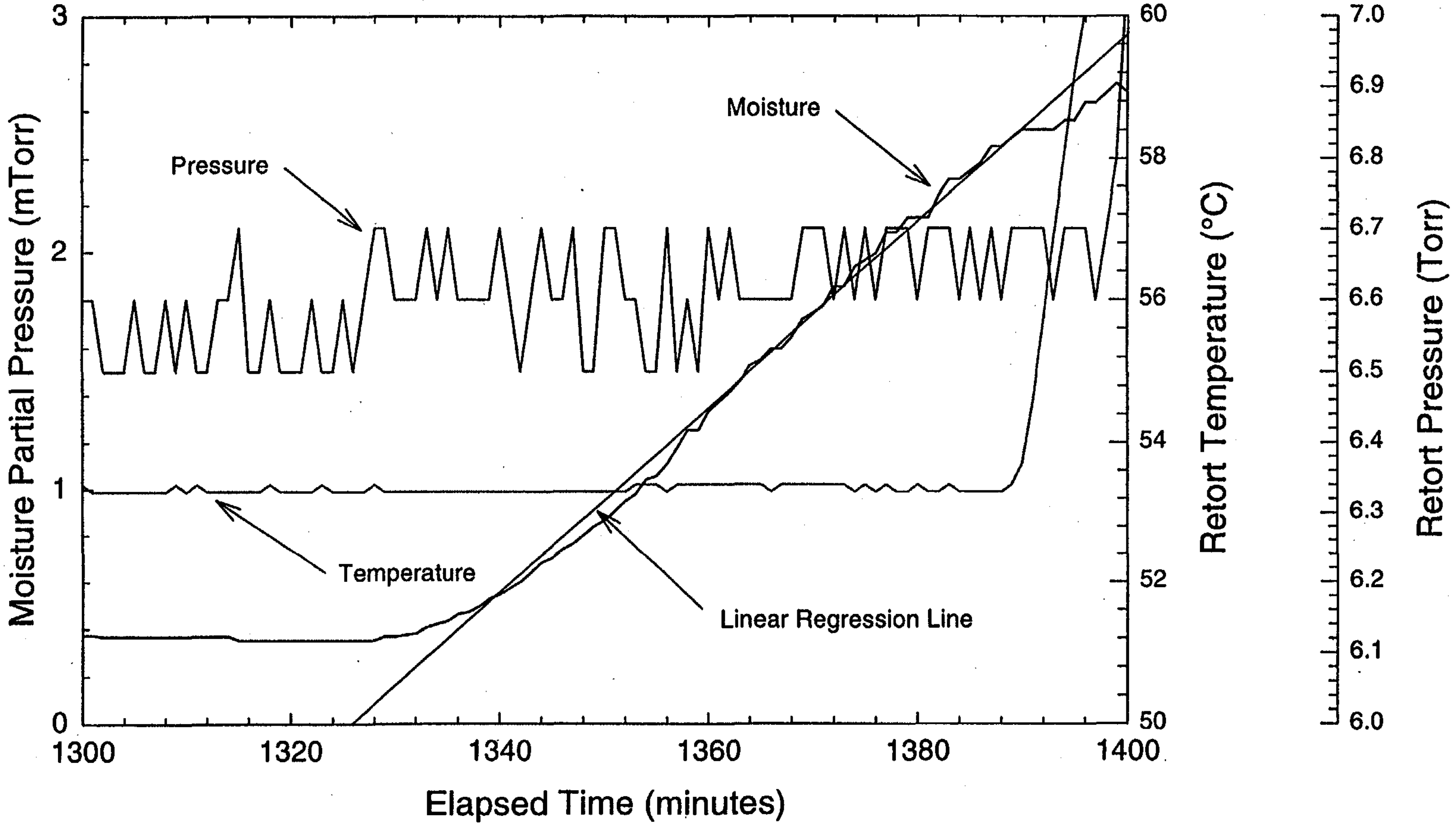

Figure 5.5. Drying of SNF Element 1990, Pressure Rise Test

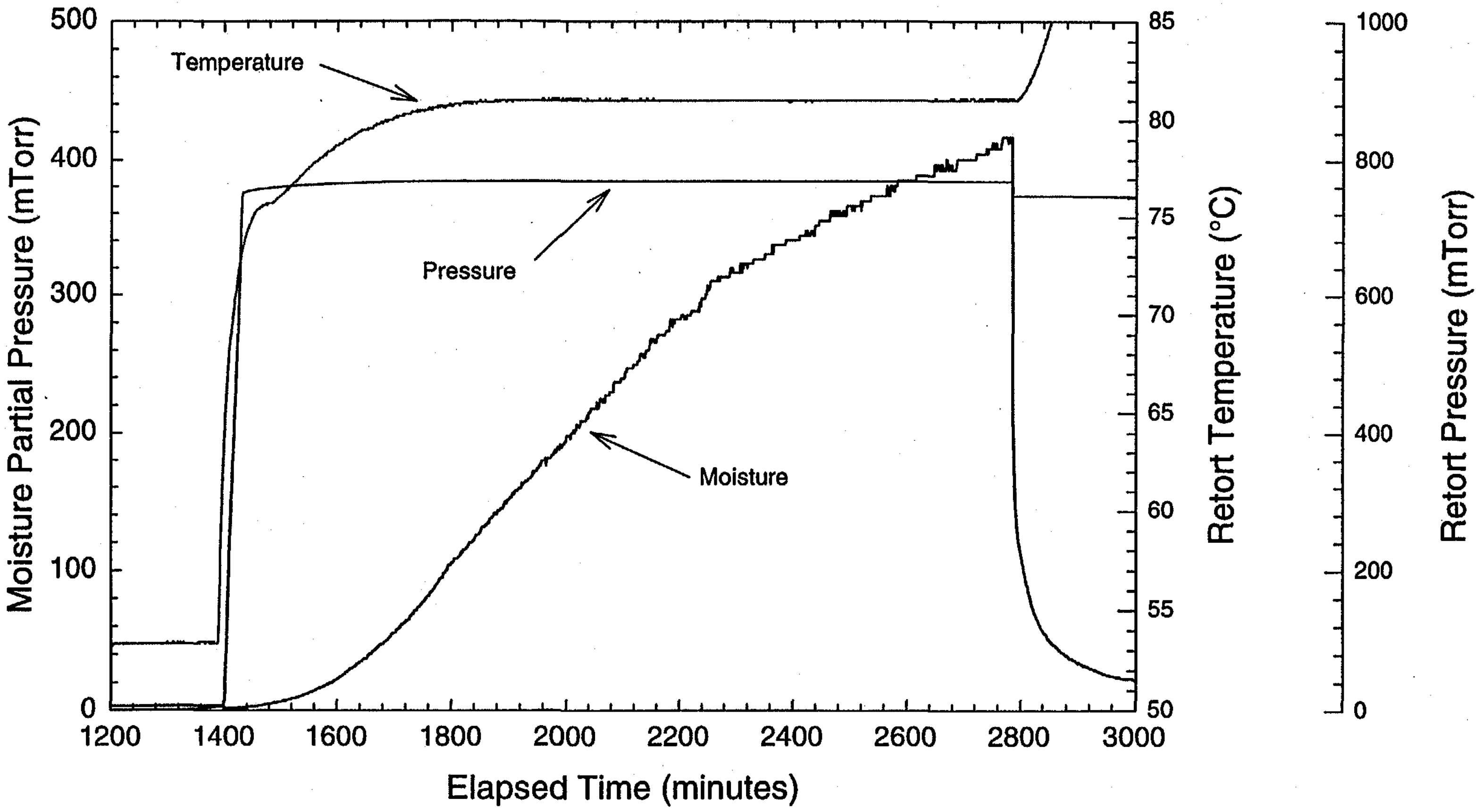

Figure 5.6. Drying of SNF Element 1990, Gas Evolution Test

rate of pressure rise in this equilibrium region was 0.21 mTorr/min. This rate is higher than that determined at 50°C, suggesting a temperature-activated desorption process for the water release.

The configuration used for the Gas Evolution Test did not allow for meaningful observation of moisture release from decomposition of hydrated species. Typically, such measurements are conducted using a temperature ramp under metered gas flow conditions. The present test conditions were static at near-atmospheric pressure and resulted in an accumulation of water in the retort, thus masking any discernible release events from hydrate decomposition.

5.5 Hot Vacuum Drying

The HVD segment of the test shown in Figure 5.7 includes the ramp from 75°C to 300°C in flowing argon gas (~324 cc/min) at atmospheric pressure. The moisture pressure decreased from 350 mTorr to approximately 25 mTorr when the gas flow was established in the system. This decrease in pressure was followed by a slight increase when the system temperature exceeded 100°C. This slight increase may be an indication of a different source of moisture, perhaps from release from a chemisorbed site (i.e., hydrated species) at higher temperatures. Because of a high background signal from free water released during this segment of the test, however, the onset of thermal decomposition of the hydrate did not yield a well-defined peak.

Figure 5.8 shows the continuation of the HVD test segment. Clearly, removing moisture from the system by the vacuum pump is more efficient than only flowing gas through the system. The moisture pressure decreased sharply from approximately 25 mTorr to 4 mTorr when the system was again evacuated. This was followed by a slow decrease for the remaining time of the HVD test segment. It should be noted that the HVD was conducted at a temperature near 325°C rather than at the planned temperature of 300°C. The higher temperature resulted from the system never having been operated using a uranium fuel element in the retort. The furnace controllers were set to lower setpoints for subsequent furnace testing.

The data from the HVD suggest slow release kinetics of the moisture indicative of decomposition from chemically bound water. The slow decrease of the moisture level suggests that the water removal process is kinetically controlled by thermal decomposition of the hydrated species.

5.6 Water Removal Verification

This segment of the test was conducted to determine if there was any moisture remaining in the system following the HVD. Figure 5.9 shows the moisture response to the final temperature ramp to the target temperature of 400°C (test performed at 425°C).

The moisture pressure increased slightly with the increase in temperature, indicating that a very small amount of water remained in the system. This result points out the difficulty in achieving what can be defined as "complete drying" of the system. It is unlikely that the state of dryness of a fuel element can

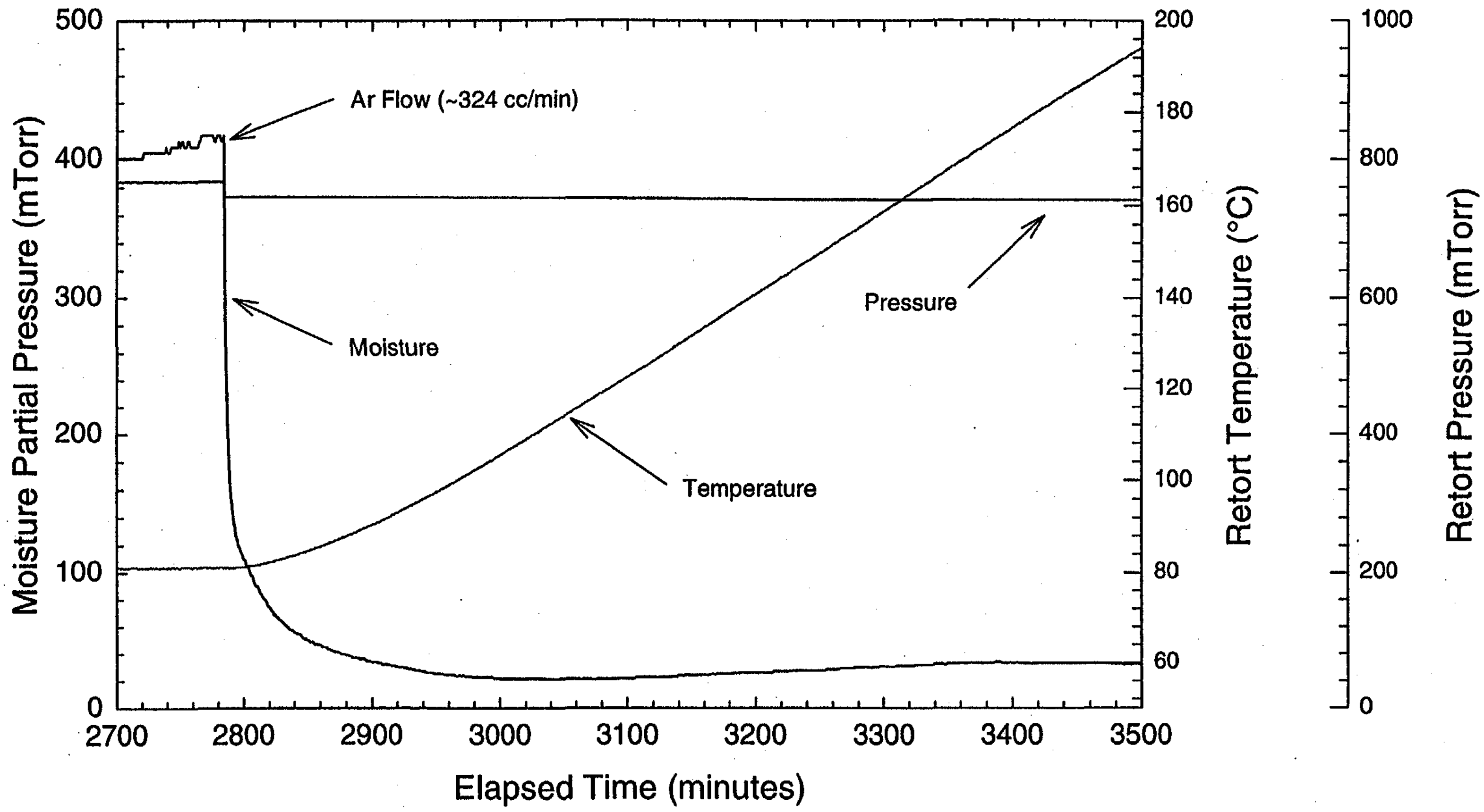

Figure 5.7. Drying of SNF Element 1990, Hot Vacuum Drying – Step 1

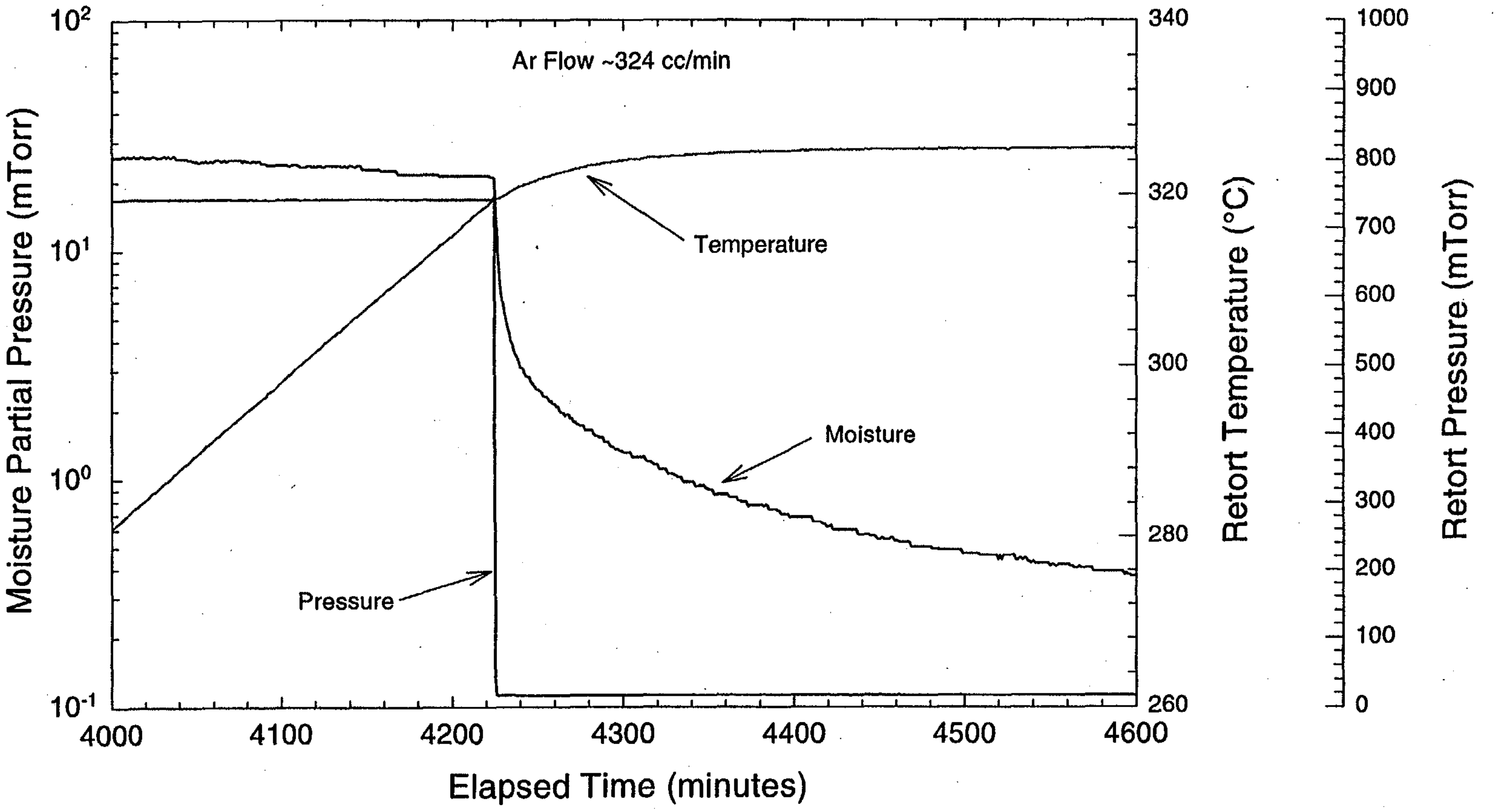

Figure 5.8. Drying of SNF Element 1990, Hot Vacuum Drying – Step 2

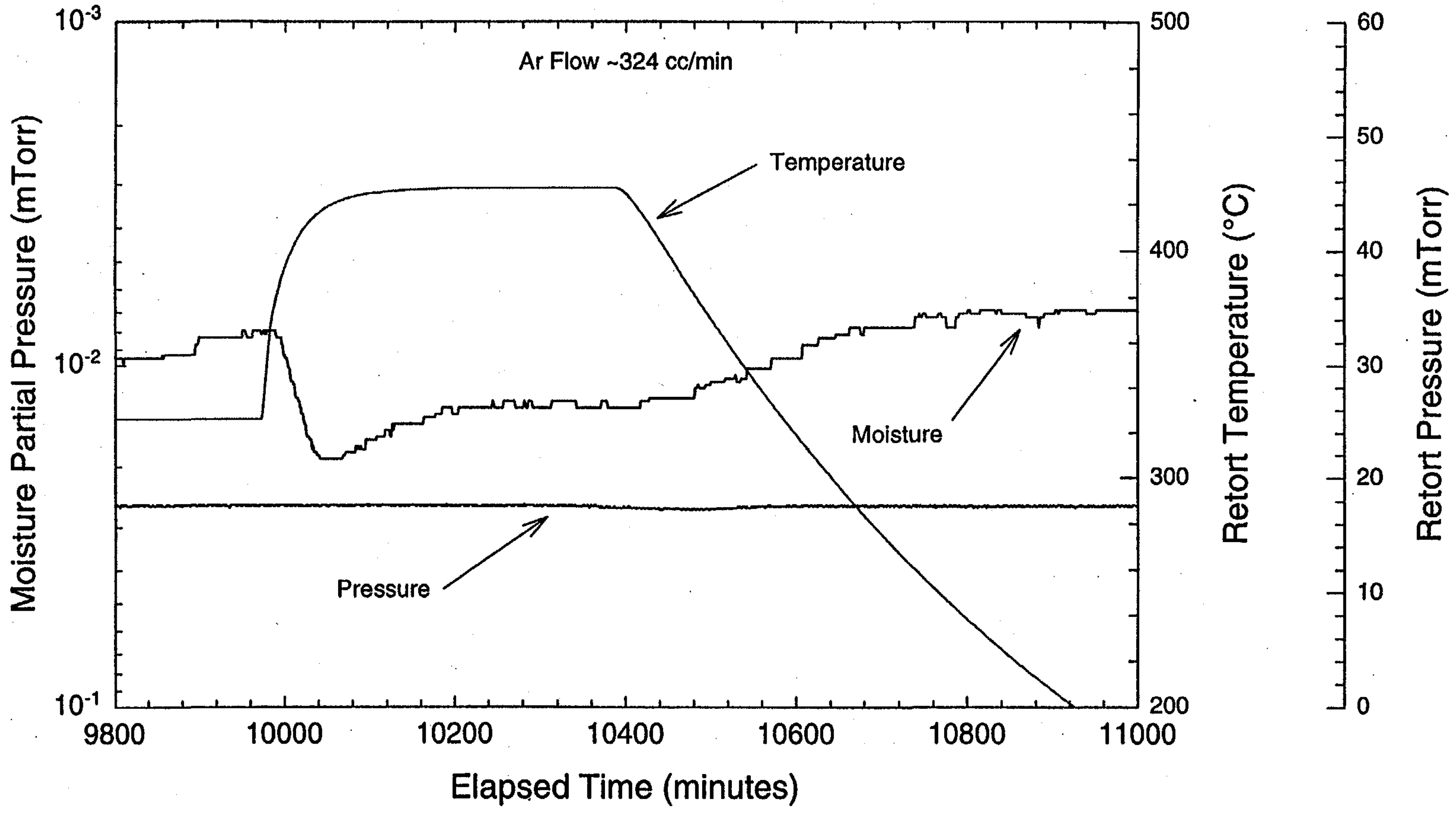

Figure 5.9. Drying of SNF Element 1990, Water Removal Verification

be determined by comparison with the baseline water content of the empty furnace system. It will only be possible to determine the state of dryness of a fuel element in relative terms by determining the lowest level of moisture achieved during a particular test.

5.7 Mass Spectrometer Measurements

The Balzers Omnistar MS was to be used to collect hydrogen and other gas release data over the test run. During the test it was learned that the MS was not configured appropriately to monitor the gas stream over all the test segments. For periods of time when the furnace system was at atmospheric pressure, the MS had to be valved off because the inlet pressure to the MS was too high to allow operation of the system (this was corrected for subsequent furnace testing).

Data that were collected during the portions of the test conducted at vacuum (e.g., CVD, HVD step 2), proved to be so low as to be within the noise level of the MS detector. Thus, no meaningful data were obtained for the release of hydrogen or fission gases from this test of a relatively undamaged fuel element.

5.8 Gas Chromatograph Measurements

The GC yielded data for the release of hydrogen in the gas stream during the heating ramp from 75°C to 300°C (HVD step 1). During that period (when hydrogen was detected) argon was flowing through the system at a rate of about 324 cc/min, and the moisture level in the gas stream was within the pressure range of 0.3 to 410 mTorr.

Figure 5.10 shows the hydrogen concentration as a function of time in the gas stream together with plots of temperature and moisture partial pressure, also as a function of time. The lowest temperature at which hydrogen was measured was about 180°C. Hydrogen continued to evolve at an increasing linear rate with increasing temperature. When the maximum ramp temperature (325°C) was reached, the level of hydrogen began to decrease slowly with time. The likely sources of hydrogen are release of hydrogen from the metal in the retort and the fuel element, and hydrogen generated by the decomposition of water with the metal surfaces in the system. Decomposition of metal hydrides is not a likely source of hydrogen in this test, as no significant change in the slope of the hydrogen release curve was observed with increasing temperature. Hydrogen from metal surfaces seems to be the most plausible source.

The data do not indicate that uranium hydride was decomposed. Even though the fuel element appeared to have had a very small breach at one end, the hydrogen release characteristics, i. e., the smooth slope, and relatively small quantity released, indicate that no significant quantity of uranium metal was exposed. A subsequent "dry-run" test, with no fuel element in the retort, yielded hydrogen release data similar to this fuel element test. Both tests showed an approximately linear increase in hydrogen release with increasing temperature.

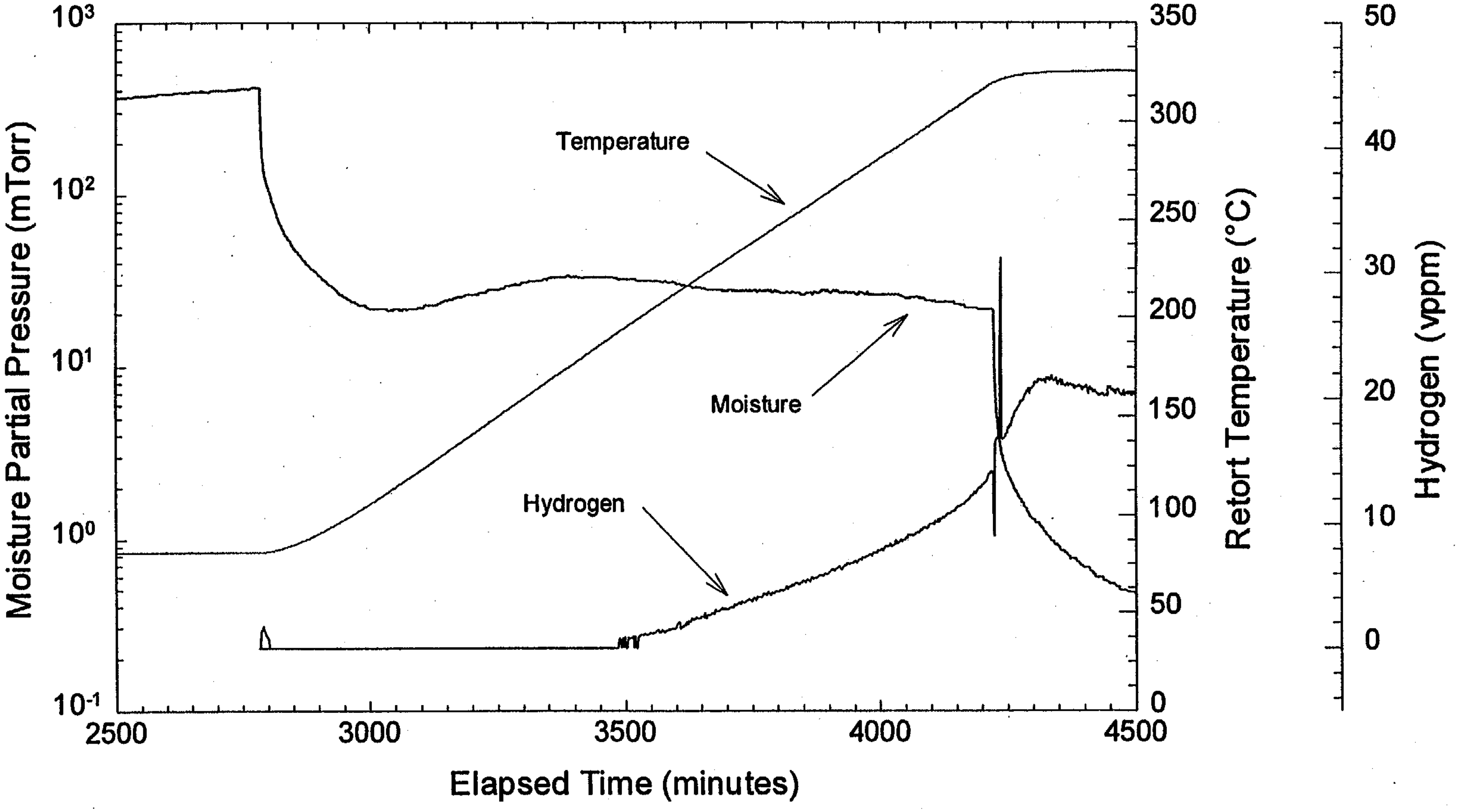

Figure 5.10. Hydrogen Release During HVD

The primary difference was the magnitude of the release. The highest concentration of hydrogen observed in the "dry-run" test was less than half that observed in the fuel element test. This suggests that a portion of the hydrogen release in the fuel test is attributable to the fuel element, probably from the cladding.

5.9 Post-Test Visual Examination

The post-test visual examination was conducted using a small video camera placed inside the hot cell. Figure 5.11 is a video still, captured from the videotape showing the damaged end of the fuel element. This view also suggests that the fuel element may have been breached; the entire width of the end cap is visible, the cladding thickness is visible, and a dark material (presumed to be uranium metal) appears in-between. Again, no major swelling or bulging was observed in this region. The remainder of the fuel element surface showed no unusual surface anomalies, new breaches, fractures, or other defects.

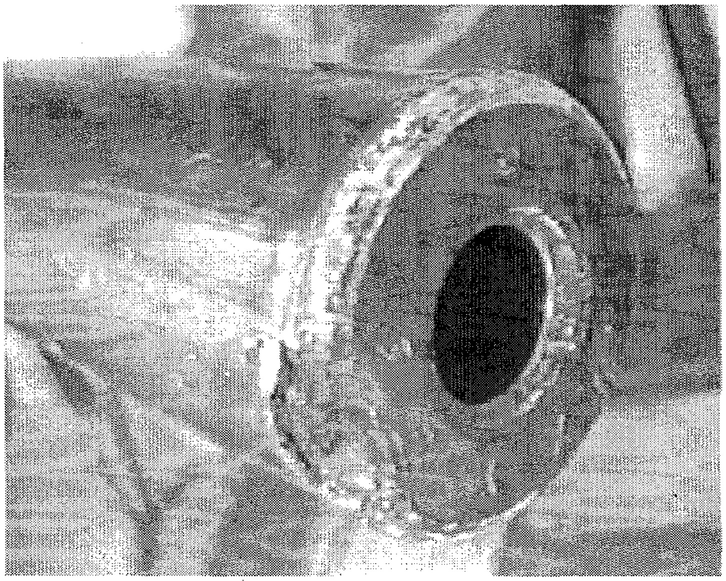

Figure 5.11. End View of Fuel Element 1990 After Removal From Furnace

6.0 Discussion

The increase in pressure during the initial static system conditions was due to temperature effects and vaporization of moisture in the system. However, the total moisture pressure increase detected was only 6.4 Torr, which accounts for approximately half of the excess 12 Torr pressure measured. Other gaseous products could have been contributing to the increase; however, nothing was detected in the gas stream by either the GC or the MS to account for the pressure difference of approximately 5.6 Torr. The most probable explanation for this excess pressure is a slightly higher bulk gas temperature than indicated by the retort thermocouple. This is possible because the gas lines were heated to temperatures higher than 50°C. A gas temperature of ~52°C is sufficient to account for the observed pressure difference.

The furnace system was opened to the cell's atmosphere during the SNF element loading, which increased the moisture level in the retort tube. Thus, the total free water in the system at the beginning of the drying test was from the following sources:

- adsorbed/absorbed water on the fuel element
- water in the sample boat
- water in the gas stream due to the cell's air mixing with gas in the system
- adsorbed/absorbed water in the retort tube.

The removal of free water in the system during the CVD process was not complete, and the test supports this conclusion. An extrapolation of the data indicates that more than 42 hr would be required to remove all the free water to a pressure of ~0.1 mTorr. The remaining free water was removed in other parts of the test at temperatures above 50°C. During the Pressure Rise Test and Gas Evolution Test, measurable amounts of water were detected by the moisture probe, representing the remaining free water in the system. Because of the test configuration used, no discernible release events attributable to hydrate decomposition were observed.

Both the Pressure Rise and the Gas Evolution Tests showed an increase in the moisture pressure due to the release of remaining free water. The release rate was linear and showed a temperature dependence. The moisture release kinetics are, therefore, independent of concentration of moisture in the system (i.e., a zeroth order reaction). Increasing the temperature from 50°C to 75°C increased the moisture release rate from 0.04 mTorr/min to 0.21 mTorr/min. The atmosphere of the system was far removed from saturation, providing an unlimited sink for water molecule release. The linear release, and the small quantity of moisture evolved, suggests partial evaporation of a surface film of one or more monolayers.

Thermal decomposition of the hydrated species was significant in controlling the removal of water from the system during the HVD. Thermal decomposition of hydrated species occurred when the system temperature exceeded 100°C, and the removal of these hydrates was kinetically controlled by the decomposition reactions. Gas flow and vacuum condition may be secondary factors in the process of drying the SNF element. A temperature above 300°C may be required for complete drying of the hydrated species within a reasonable period of time.

Absolute drying of the system (furnace + fuel) was not achieved by the CVD and HVD cycles of the test. The HVD step was necessary to remove the free water remaining after approximately 17 hr of CVD, and most of the water attributed to hydrated species.

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8.0 Supporting Documents and Related Reports

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Reports are written separately for the whole element drying test series as follows:

System Design Description for the Whole Element Furnace Testing System

Spent Fuel Drying System Test Results (First Dry-Run)

Spent Fuel Drying System Test Results (Second Dry-Run)

Spent Fuel Drying System Test Results (Third Dry-Run)

Drying Results of K-Basin Fuel Element 1990 (Run 1)

Drying Results of K-Basin Fuel Element 3128W (Run 2)

Drying Results of K-Basin Fuel Element 0309M (Run 3)

Drying Results of K-Basin Fuel Element 5744U (Run 4)

Drying Results of K-Basin Fuel Element 6603M (Run 5)

Drying Results of K-Basin Fuel Element 1164M (Run 6)

Drying Results of K-Basin Fuel Element 2660M (Run 7)

Drying Results of K-Basin Fuel Element 6513U (Run 8)