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V.L. Birkland
V.L. Birkland

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7. Abstract <p>The Data Quality Objectives (DQOs) were established for the response of the first group of fuel samples to the proposed Path Forward conditioning process. Controlled temperature and atmosphere furnace testing will establish performance parameters using the conditioning process proposed by the Independent Technical Assessment (ITA) Team as the baseline.</p> <p>The major question to be addressed by these examinations is what is the response of these fuel samples to the proposed ITA conditioning process. The following four areas of technical uncertainty have been established for the process; fuel and corrosion product drying behavior, sludge drying behavior, hydride decomposition behavior, and passivation.</p> <p>The limited scope represented by these samples and conditioning examinations will not provide definitive data for all the decisions that must be made related to the conditioning process definition. However, the data input when combined with other data along with parallel design and analysis provide the basis for the decisions. Decisions may by necessity be made before sufficient data is obtained from these examinations. In these cases, the data collected will be confirmatory.</p> <p>The testing will focus on the proposed ITA process at one atmosphere pressure for these initial samples. The order of testing will be to first select a sample away from the damaged area of one of the elements to provide baseline behavior for uncorroded fuel followed by samples selected from the corroded surfaces. Modifications to the ITA proposed conditioning process may be considered for a second series of tests to be conducted with samples from these three elements..</p>		
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DATA QUALITY OBJECTIVES FOR THE INITIAL FUEL CONDITIONING EXAMINATIONS

L. A. Lawrence
Westinghouse Hanford Company

May 1995

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Approved by:

RP Omberg 5-1-95

R. P. Omberg, Manager
Spent Nuclear Fuels
Characterization Program

5/1/95
Date

Approved by:

D.W. Smith

D. W. Smith, Manager
SNFP Quality Assurance

5/5/95
Date

Approved by:

S. C. Marschman

S. C. Marschman, Manager
Fuel and Dry Storage
PNL Spent Nuclear Fuels Program

5-5-95
Date

Approved by:

P. J. Turner

P. J. Turner, Manager
Hanford Technical Support Project
Pacific Northwest Laboratory

5-8-95
Date

Approved by:

Jian-Shun Shuen

J. Shuen, Program Engineer
Spent Nuclear Fuels Project Office
Characterization Program
U.S. Department of Energy
Richland Operations Office

5-8-95
Date

EXECUTIVE SUMMARY

The Data Quality Objectives (DQOs) were established for the response of the first group of fuel samples shipped from the K West Basin to the Hanford 327 Building hot cells for examinations to the proposed Path Forward conditioning process. Controlled temperature and atmosphere furnace testing will establish performance parameters using the conditioning process proposed by the Independent Technical Assessment (ITA) Team as the baseline.

The major question to be addressed by these examinations is what is the response of these fuel samples selected for the first shipment to the proposed ITA conditioning process. The areas of technical uncertainty for the process can be placed in the following four general categories:

1. Fuel and Corrosion Product Drying Behavior
2. Sludge Drying Behavior
3. Hydride Decomposition Behavior
4. Passivation

The limited scope represented by these samples and conditioning examinations will not provide definitive data for all the decisions that must be made related to the conditioning process definition. However, the data input when combined with other data along with parallel design and analysis provide the basis for the decisions. Decisions may by necessity be made before sufficient data is obtained from these examinations. In these cases, the data collected will be confirmatory.

The testing will focus on the proposed ITA process at one atmosphere pressure for these initial samples. The order of testing will be to first select a sample away from the damaged area of one of the elements to provide baseline behavior for uncorroded fuel followed by samples selected from the corroded surfaces. Modifications to the ITA proposed conditioning process may be considered for a second series of tests to be conducted with samples from these three elements.

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DATA QUALITY OBJECTIVES FOR THE INITIAL FUEL CONDITIONING EXAMINATIONS

1.0 INTRODUCTION

The Data Quality Objectives (DQOs) were established for the conditioning examinations of the first group of fuel samples shipped from the K West Basin to the Hanford 327 Building hot cells for examination. These examinations of the conditioning behavior of the fuel samples are an extension of the initial K West fuel examinations (Lawrence 1995). The DQO process was employed to ensure the planned examinations fully support the Path Forward to solve the safety and environmental concerns associated with the deteriorating fuel in the K Basins (Lawrence 1994).

The examinations covered in this DQO are the controlled temperature and atmosphere furnace testing to establish the fuel performance parameters when subjected to the conditioning process proposed by the Independent Technical Assessment (ITA 1994) and complementary Thermo Gravimetric Analysis (TGA) of selected samples. The TGA testing will provide kinetic data on small sample water loss for drying and oxygen uptake during the passivation process. The ITA proposed process will be the basis for the testing however specific parameters of time, temperature, and atmosphere will be evaluated for the different fuel states represented by the samples from the first fuel shipment from K West (Makenas 1995).

Combustibility and pyrophoricity have also been identified as information needs for this material following prolonged underwater storage and degradation (Abrefah 1994). Controlled atmosphere and temperature testing of materials from the fuel elements could provide data for these needs to support safety evaluations and process definition. Pre- and post-conditioning ignition tests will provide a measure of the effects of the passivation on fuel behavior.

The applicability of the data collected in this examination to the Path Forward decision schedule suggests that the data collected will establish process control parameters. The Path Forward from the K Basins (DOE 1995) calls for early conditioning of the SNF placed in the Multiple Canister Overpacks (MCOs). The accelerated program calls for proceeding as directly as possible from the point of packaging to drying and conditioning. WHC was directed to begin fuel removal from the K Basins by December 1, 1997 and begin conditioning by June 1998. Specification of the conditioning process is scheduled for October 1995 after limited data on material response has been obtained from the testing activities outlined in this DQO. Process validation is scheduled for the end of FY 1996 providing an opportunity to obtain bounding behavior for the conditioning process defined.

2.0 DATA QUALITY OBJECTIVE STEP 1: STATEMENT OF PROBLEM

The conditioning process proposed by the ITA team summarized in Figure 1 contains three major steps (ITA 1994). First the water is removed and the material dried. This is expected to require temperatures up to 300 °C for up to 35 hours to remove adsorbed, and absorbed water as well as water of hydration. The second step, termed passivation, involves controlled additions of oxygen to the system at higher temperatures for approximately 10 hours. The MCO is then backfilled with an inert cover gas and sealed for long term interim storage.

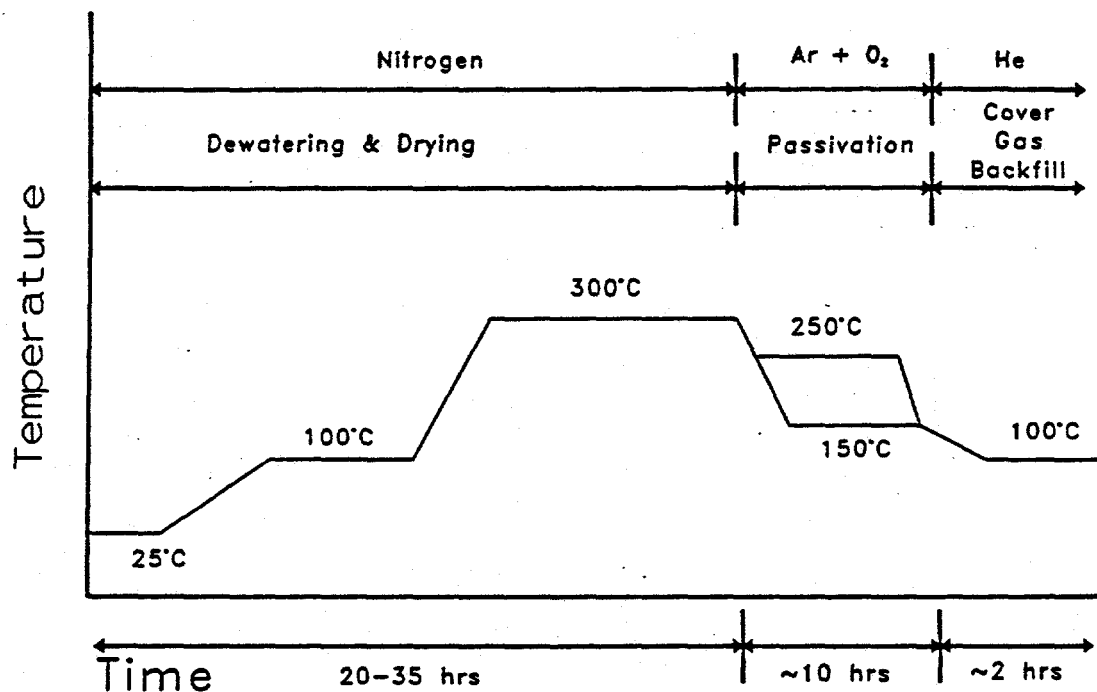
The drying step will remove water that remains after the MCO is drained following transport to the conditioning facility. This is accomplished in two stages. The first stage at a temperature of 100 °C is intended to remove free water from the fuel elements and from any fuel corrosion products and accompanying sludge in the MCO. The higher temperature portion of the drying step at 300 °C or higher will be in vacuum to remove the adsorbed and absorbed water and the water of hydration associated with the corrosion products and sludge. Complete removal of the hydride is not expected; the main objective is to adequately dry the material. The acceptable level of dryness for the material in the MCO has not been established.

Some release of radioactive species from the material is expected during the high temperature conditioning. The expected releases are considered to be low due to the low exposure levels of the fuel material and expected low diffusion rates at the selected temperatures. However, this is unknown and must be bounded with experimental data.

The second step termed passivation is intended to oxidize uranium hydrides, reactive metal sites on the surface, and any uranium fines present after the hydride removal. The MCO temperature at which the oxygen is admitted is very important because it affects the degree to which the oxygen reacts with exposed uranium. If oxygen were to be admitted at a temperature at which oxidation of bulk uranium is significant, much of the oxygen would be consumed by exposed uranium metal rather than by oxidation of uranium hydride and other reactive sites. The oxidation of exposed solid unirradiated uranium as a function of oxygen partial pressure and temperature is well known, however significant differences compared to the irradiated and corroded fuel make a direct application of the laboratory data questionable. For the N Reactor fuel, the ITA Team proposes that oxygen be admitted at a MCO temperature in the range of 150 °C to 250 °C.

The ITA Team proposed that the oxygen volume fraction in the inert (argon) gas be 2% for the initial step in the passivation process. This concentration was selected based on information on the French process and that planned by the United Kingdom to passivate MAGNOX type fuel. The oxygen concentration was set to restrict the available oxygen supply and potential heat release from uranium hydride and metal oxidation, in order to avoid reaching a temperature at which significant uranium oxidation would occur. Therefore, significant uncertainty also exists in the oxygen volume fraction selected for the K Basin fuel materials.

Figure 1. Independent Technical Assessment Proposed Conditioning Process for K Basin Fuel Material.



To ensure adequate penetration and replenishment of oxygen, during passivation the French employ a pressure pulsing technique and changes in the oxygen concentration. The oxygen volume fraction is increased from the initial 2% value up to 20%. Again there is essentially no data on the response of the K Basin materials to these temperatures and gas environments.

The ITA Team identified the following areas where information was necessary to confirm the proposed process selected (ITA 1994).

- Fuel corrosion product drying behavior (water loss versus time and temperature).
- Sludge drying behavior (water loss versus time and temperature).
- Aluminum oxide (hydrates) drying behavior (water loss versus time and temperature).
- Experimental and theoretical data concerning optimum oxygen additions for MCO design and loading.
- Uranium corrosion rates in K East and K West Basins and estimate of the area of uranium exposed to corrosion.
- Uranium hydride content of the fuel corrosion product and sludge for fuel in open and closed canisters.

These areas of technical uncertainty can be grouped into three general categories for the conditioning testing in the hot cells. They are:

- Fuel and Corrosion Product Drying Behavior: What are the dry out characteristics of the K Basin fuel material (fuel and corrosion products) as a function of time, temperature, pressure, and gas composition?
- Sludge Drying Behavior: What are the dry out characteristics of sludge which will accompany the fuel in the MCO as a function of time, temperature, pressure, and gas composition?
- Hydride Decomposition Behavior: What is the degree to which the uranium hydride present dissociates as a function of the in-MCO condition of time, temperature, pressure, and gas composition?

In addition the pyrophoric properties of the material after conditioning are needed to support process design and safety considerations and the characteristics of the oxide layer found on the uranium during the passivation portion of the conditioning process are needed to support process definition.

Stakeholders for the initial conditioning examinations are listed in Table 1. They are essentially the same as those for the initial K West fuel examinations (Lawrence 1995).

Table 1. Stakeholders for Initial Fuel Conditioning Examinations.

Function	Individual Point of Contact
1. Path Forward	MCOs--L.H. Goldmann Transportation--A.T. Kee Stabilization--C.R. Miska
2. Basin Operations	B.S. Carlisle
3. Regulatory Integration	V.C. Hoeffer
4. Technical Oversight	J.C. DeVine
5. K Basin Engineering	J.P. Schmidt
6. Quality Assurance	D.W. Smith
7. RL	J. Shuen
8. Characterization	WHC--R.P. Omberg PNL--S.C. Marschman

3.0 DATA QUALITY OBJECTIVE STEP 2: IDENTIFY THE DECISIONS

The major questions to be answered by these examinations are what is the response of these selected fuel samples to the conditioning process proposed by the ITA and what are the effects of changes in the time, temperature, gas pressure, and gas composition on the final state of the conditional fuel? Fuel was selected for the initial examinations to bound the breach conditions expected to be encountered in the closed canisters in K West.

Specific project decisions will be focused on establishing the conditioning process definition for facility design and construction. These examinations will provide the initial data for the behavior of representative material to the general parameters of the conditioning process. Specific questions to the adequacy of the proposed ITA conditioning process or any modifications to the recommended conditioning process will not be fully answered by these examinations due to the limited nature of the sampling and the number of samples available for testing. These tests will however be the initial data and provide the basis for more extensive testing with additional samples from the second shipping campaign from the K West Basin.

4.0 DATA QUALITY OBJECTIVE STEP 3: IDENTIFY THE INPUTS

A controlled temperature and atmosphere furnace was selected for the hot cell examinations of the fuel conditioning process. The design requirements for the furnace are summarized in Table 2. The furnace has the capability of accommodating both small, i.e., approximately 1 cm³ samples and large ring sections of an outer element up to 8 in. long.

The furnace designed by Pacific Northwest Laboratory (PNL) based on these design requirements is shown schematically in Figure 2. The majority of equipment will be placed outside the hot cell for access and control. Furnace design should be optimized as much as possible to provide data on both the moisture remaining in the sample as well as the quantity of oxygen uptake for the sample during the passivation part of the conditioning process. The proposed furnace will be capable of ignition testing as well as conditioning testing the fuel materials.

The reference ITA conditioning process will be the furnace temperature cycles and atmospheres for the initial sample testing at one atmosphere pressure for the passivation portion of the conditioning process (Figure 1). The order of testing will be to first select a sample away from the damaged area of one of the elements to provide baseline behavior for uncorroded fuel followed by samples selected from the corroded surfaces. The controlled temperature and pressure testing of the conditioning process will be conducted first followed by any complimentary TGA measurements provided the equipment preparations and installations are compatible with the examination schedules. Ignition testing with the furnace will be conducted last.

A total of four nearly identical samples will be, if possible, selected from a given fuel element sample for the following testing to support conditioning.

- One sample will be examined metallographically to establish the pre-conditioning characteristics of the sample. Examinations will be focused on the extent of hydride, fuel oxidation, and corrosion products present.
- Two samples will be designated for concurrent conditioning in the furnace, at given times and temperatures. Initial testing will consider the proposed ITA process parameters (ITA 1994).
- Following testing one sample will be examined metallographically to establish the post conditioning characteristics of the sample.
- The remaining sample after conditioning will be stored under controlled conditions for complementary thermogravimetric (TGA) testing.
- One sample be removed as a spare sample for possible pre-conditioning TGA testing.

Table 2. Design Requirements for Furnace for N Reactor Fuel Samples.

1. Sample

1.1 Geometry and Dimensions

Both small and large samples will be tested in the furnace.

Small samples will be approximately 1 cm³ fuel samples with or without the cladding attached. Samples may be placed in canister sludge to represent drying conditions.

Large samples may be ring sections of an outer fuel element. The length of the fuel element segment will be up to 8 in. long.

2. Furnace Temperature

2.1 Heating Range

Room temperature to 700 °C.

2.2 Measurement Accuracy

+/- 5 °C

3. Sample Temperature

3.1 Temperature Range

Room temperature to 1500 °C/2000 °C for ignition testing only

3.2 Measurement accuracy

Room temperature to 700 °C +/-5 °C

Above 700 °C +/-50 °C

4. Atmosphere

4.1 Gas Composition

Air saturated with water to inert (nitrogen, argon)

4.2 Gas Pressure in active furnace zone

Vacuum (1 torr) to atmosphere

Pressure test pulsing capability up to 8 atmospheres

4.3 Measurement Accuracy

Gas Composition: Bottle analysis or mass flow control fractions to +/-5%

Pressure: +/-1 torr or +/-1% whichever is larger

Table 2. Design Requirements for Furnace for N Reactor Fuel Samples. Cont.

5. Temperature Cycle

5.1 Temperature Rise/Cool Down Time

Ramp rates up to 5 to 15 °C/minute

Cool down times will be established for the specific furnace configuration fabricated.

5.2 Time at Temperature

Complete temperature cycle of up to 2 days.

5.3 Measurement Accuracy

Same as 3.2

6. Sample Handling

Capability to protect samples before and after testing will be based on initial sample handling and examination.

7. Sample Off Gas

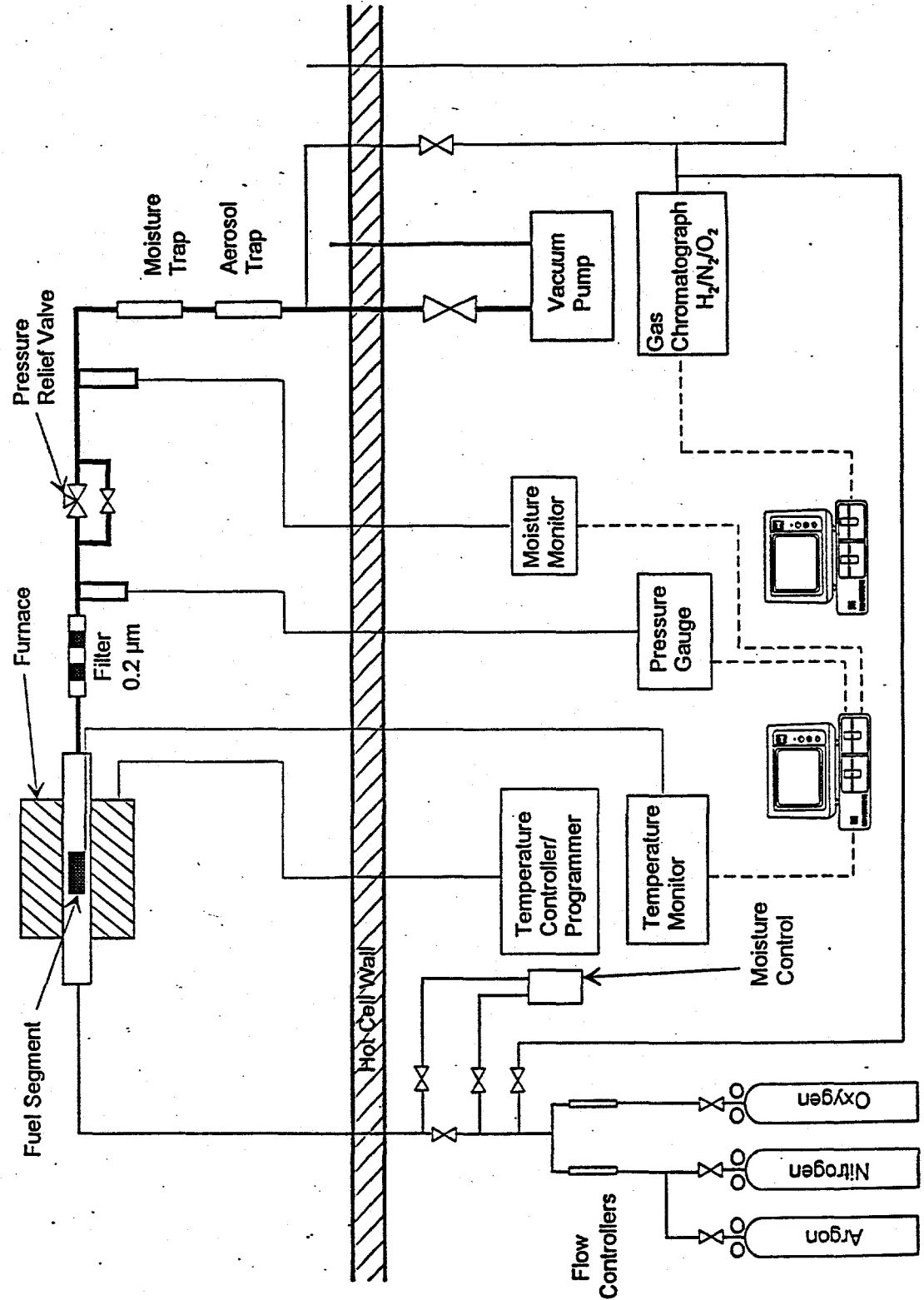
7.1 Content

The quantity of gas released from the samples will be measured to +/-5%

7.2 Composition

The composition of the gas released from the samples will be determined.

Figure 2. Schematic Diagram of Spent Fuel Drying, Passivation, and Ignition Apparatus.



The initial conditioning tests will include fuel only. No attempts will be made with the initial testing sequence to include sludge samples concurrent with the fuel. It is recognized that sludge will most likely accompany the fuel samples to the conditioning facility, however, for these initial tests with limited samples the decision was made to focus on the intact fuel element behavior.

Modifications to the ITA proposed conditioning process will be considered for the second series of tests to be conducted. Parameters of interest from the second series of tests include time and temperature, and gas composition. The second series of tests will also be at one atmosphere pressure. Pressure testing of the passivation portion of the conditioning process will be conducted after the materials response to one atmosphere is established and sufficient experience has been obtained with the system and the in-cell testing.

5.0 DATA QUALITY OBJECTIVE STEP 4: DECISION BOUNDARIES

The conditioning examinations in this DQO will be restricted to the fuel samples from the initial shipment from the K West Basin. Test parameters and conditioning cycles will be limited to the proposed ITA process and any possible adjustments of time, temperature, gas composition and pressure that might be required to meet defined acceptance standards for the conditioned fuel material. Acceptance standards for the conditioned material have not been established at this point in development of the Path Forward for the material. Planned post-test examinations of conditioned samples will provide data on extent of oxide buildup and provide qualitative information on the bonding of the oxide layer to the metal matrix that can be utilized to develop acceptance criteria. In addition, post-conditioning ignition testing of selected samples are expected to quantify any change in combustion temperatures for the material.

6.0 DATA QUALITY OBJECTIVE STEPS 5 AND 6: DECISION RULES AND ACCEPTABLE LIMITS ON DECISION ERROR

A limited scope DQO is being used for these initial conditioning examinations in a similar manner to the initial examination of the K West fuels (Lawrence 1995, Lawrence 1994). The development of a decision rule and acceptable limits on decision error are beyond the scope of these DQO activities. The lack of an acceptance standard for the conditioned fuel makes it difficult to develop a decision rule on the applicability of the process or any alterations in the ITA proposed process and corresponding limits on the decision error.

7.0 DATA QUALITY OBJECTIVE STEP 7: OPTIMIZE

The examinations will be optimized within the confines of the three elements shipped to the hot cells from the K West Basin. Detailed examination plans for the three elements will include both samples for initial metallography to define the initial fuel state and sister samples for conditioning testing. Information gained from the materials responses during the first series of tests with the proposed ITA process at atmospheric pressure will be used to develop the specific testing parameters for the follow on tests with the material. Limitations in the quantities of test samples available from this initial fuel shipment may make it difficult to explore more than a limited range of conditioning process parameters beyond the proposed process.

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