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## DATA QUALITY OBJECTIVES FOR THE SECOND K WEST FUEL EXAMINATIONS

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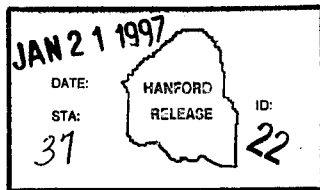
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**Abstract:** The Data Quality Objectives (DQOs) were established for the examinations of the second group of K West fuel samples. The examinations will expand the initial understanding obtained for fuel stored in closed canisters with emphasis on whole element drying and conditioning testing and Thermo-Gravimetric Analysis (TGA) for oxidation behavior, drying, and hydrogen release. The laboratory examinations addressed in this DQO take into account the extent and diversity of the fuel damage as indicated by previous in-basin examinations. A total of fifteen fuel elements are identified for laboratory examinations. Three of these elements are designated for surface coating examinations and two elements are designated for examination of subsurface sludge accumulations and for sectioning of small samples for TGA measurement.

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**DATA QUALITY OBJECTIVES FOR THE SECOND K WEST FUEL EXAMINATIONS**

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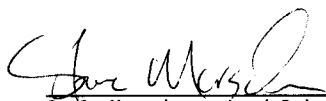
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## EXECUTIVE SUMMARY

The Data Quality Objectives (DQOs) were established for the examinations of the second group of K West fuel samples. The examinations will expand the initial understanding obtained for fuel stored in closed canisters with emphasis on whole element drying and conditioning testing and Thermo-Gravimetric Analysis (TGA) for oxidation behavior, drying, and hydrogen release. The laboratory examinations addressed in this DQO take into account the extent and diversity of the fuel damage as indicated by previous in-basin examinations. Additional in-basin examinations, coincident with fuel selection for shipment, will include detailed visual inspection of elements to further determine the amount of adhering sludge and the extent of fuel degradation.

Laboratory examinations for K West fuel elements will be analogues to the ongoing examinations of the K East fuel samples. A total of fifteen fuel elements are identified for laboratory examinations. Three of these elements are designated for surface coating examinations and two elements are designated for examination of subsurface sludge accumulations and for sectioning of small samples for TGA measurement. Following the subsurface sludge examinations one or two groups of fuel fragments will be collected for drying tests to simulate a Multi-Canister Overpack (MCO) scrap basket. Laboratory testing will focus on drying and conditioning testing of the eleven whole elements and one or two groups of fuel pieces. Samples will be examined visually before and after furnace testing. Small samples will be selected for metallography and TGA based on the results of the furnace tests.

## CONTENTS

1.0	INTRODUCTION . . . . .	5
2.0	DATA QUALITY OBJECTIVE STEP 1: STATEMENT OF PROBLEM . . . . .	7
2.1	REVIEW OF THE PLANNED PROCESS AND ASSUMPTIONS MADE . . . . .	7
2.2	THE PRIMARY PROBLEMS . . . . .	9
2.2.1	Fuel Drying and Conditioning for Integrated Process Strategy . . . . .	9
2.2.2	Reactive Surface Area . . . . .	10
2.2.3	Surface Coatings . . . . .	10
3.0	DATA QUALITY OBJECTIVE STEP 2: IDENTIFY THE DECISIONS . . . . .	13
3.1	QUESTIONS RELATED TO COLD VACUUM DRYING, HOT VACUUM DRYING, AND CONDITIONING . . . . .	13
3.2	QUESTIONS RELATED TO REACTIVE SURFACE AREA . . . . .	14
3.3	QUESTIONS RELATED TO SURFACE COATINGS . . . . .	14
4.0	DATA QUALITY OBJECTIVE STEP 3: IDENTIFY THE INPUTS . . . . .	15
4.1	RELATED INPUTS FROM PREVIOUS DATA QUALITY OBJECTIVES . . . . .	15
4.1.1	Gas and Liquid Sampling . . . . .	16
4.1.2	Sludge Depth Measurements . . . . .	16
4.1.3	Canister Sludge Sampling . . . . .	16
4.1.4	Visual Inspection of Element Surfaces . . . . .	16
4.2	IN-BASIN AND LABORATORY MEASUREMENTS FOR THE CURRENT DATA QUALITY OBJECTIVES . . . . .	17
4.2.1	Visual/Video During In-Basin Fuel Shipping Activities . . . . .	18
4.2.2	Hydrogen Generation Measurement Before Shipment . . . . .	18
4.2.3	Visual/Video During Hot Cell Examinations . . . . .	18
4.2.4	Metallography . . . . .	19
4.2.5	Furnace Testing of Whole Elements and Scrap . . . . .	19
4.2.6	Thermo-Gravimetric Analysis Testing . . . . .	20
4.2.7	Analysis of Element Coatings . . . . .	20
4.2.8	Examination for Subsurface Sludge Deposits . . . . .	20
5.0	DATA QUALITY OBJECTIVE STEP 4: DECISION BOUNDARIES . . . . .	21
5.1	K WEST FUEL AVAILABLE FOR SAMPLE SELECTION . . . . .	21
5.1.1	Surface Coatings . . . . .	22
5.1.2	Damaged Fuel . . . . .	22
5.2	RECOMMENDED SAMPLING . . . . .	23
5.3	THE STATISTICS OF WHOLE ELEMENT FURNACE TESTING . . . . .	24
5.4	LABORATORY TEST CONDITIONS FOR WHOLE ELEMENT DRYING . . . . .	25
6.0	DATA QUALITY OBJECTIVE STEPS 5 AND 6: DECISION RULES AND ACCEPTABLE LIMITS ON DECISION ERROR . . . . .	27
7.0	DATA QUALITY OBJECTIVE STEP 7: OPTIMIZE . . . . .	29
8.0	REFERENCES . . . . .	31

**LIST OF FIGURES**

1. Integrated Process Strategy for K Basin Fuel Removal . . . . .	8
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**LIST OF TABLES**

1. Stakeholders for K West Fuel Examinations . . . . .	6
2. Sequence of In-Basin and Laboratory Activities. . . . .	15
3. In-Basin and Laboratory Examinations to be Performed on K West Fuel as Part of the Current Data Quality Objectives . . . . .	17
4. Fuel Sampling Matrix for Furnace Tests . . . . .	23
5. One-Sided Nonparametric Tolerance Limits . . . . .	25
6. Possible Test Matrix for Fifteen Elements . . . . .	26

## DATA QUALITY OBJECTIVES FOR THE SECOND K WEST FUEL EXAMINATIONS

### 1.0 INTRODUCTION

Spent nuclear fuel (SNF) from N Reactor resides in open canisters in K East Basin and in sealed canisters in the K West Basin. Much of the fuel is in various states of degradation as indicated by recent in-basin examinations. All of the fuel will be moved to interim dry storage over the next few years. Two previous campaigns to retrieve fuel for characterization have occurred, one each in K East and K West Basin (Lawrence 1995a and 1996a). In this document the Data Quality Objectives (DQOs) are established for the examinations of the second group of fuel samples shipped from the K West Basin to the Hanford 327 Building hot cells for examinations. The DQO process was employed to ensure the planned examinations fully support the Integrated Process Strategy (IPS) (WHC 1995) to resolve the safety and environmental concerns associated with the deteriorating fuel in the K Basins (Lawrence 1994).

The topics covered by this DQO include the in-basin examinations concurrent with fuel movement, the selection of fuel for transfer to the hot cells and the detailed hot cell examinations. These examinations complement and expand similar measurements completed for the initial K West fuel samples (Lawrence 1995a) and examinations in progress on the fuel from the open canisters in K East (Lawrence 1996a, 1996b).

These examinations will expand our initial understanding of the expected behavior of the fuel now stored in the K West closed canisters during movement and cleaning. Also ascertained will be the extent of fuel degradation, the fuel's response to the IPS drying and conditioning process, and the degraded fuel's pyrophoric behavior. The scope represented by the samples and data collected will not provide definitive justification for all the decisions that must be made concerning the IPS. These data when combined together with other examination data, along with parallel design efforts and calculations, provide the basis for design decisions.

The applicability of the data collected in these examinations to the IPS is also dependent on the schedule constraints of the Spent Nuclear Fuel Project to begin fuel removal from the K Basins in 1998. Decisions related to fuel handling, cleaning, transportation, and the co-dependent Multi-Canister Overpack (MCO) design may have to be made before sufficient data is obtained from the fuel examinations. In these cases, the data collected may only be confirmatory in nature verifying that there is sufficient latitude incorporated into the corresponding design for the observed fuel behavior or condition.

Stakeholders for the examinations are listed in Table 1. Formal and informal meetings were held with various stakeholders to develop the input and concurrence for this DQO.



Table 1. Stakeholders for K West Fuel Examinations.

Function	Individual Point of Contact
IPS - Fuel Handling - Cold Vacuum Drying - Hot Vacuum Drying/Conditioning - Safety	B.S. Carlisle, W.C. Mills J.J. Irwin C.R. Miska M. Kummerer
K Basin Operations	D. W. Siddoway
RL	J. Shuen
Quality Assurance	D.W. Smith
Characterization - DESH - PNNL	R.P. Omberg* S.C. Marschman

\*Key decision maker.

## 2.0 DATA QUALITY OBJECTIVE STEP 1: STATEMENT OF PROBLEM

### 2.1 REVIEW OF THE PLANNED PROCESS AND ASSUMPTIONS MADE

The IPS has been approved by DOE to mitigate the safety and environmental concerns associated with continued storage of the N Reactor fuel in the K Basins (Hansen 1995).

The IPS process is summarized in Figure 1. In the first step, the fuel in the open and closed canisters in K East and K West respectively, will be moved to a centralized work location within a confinement zone in the basin pool. Then, the fuel will be removed from the canisters, cleaned, and loaded into a tier basket. Some canisters with highly corroded fuel or degraded fuel may have to be mechanically sectioned to remove the elements. Fuel requiring cleaning will be subjected to a flushing-based desludging operation with limited mechanical desludging as necessary. The tier baskets will be loaded and sealed in an MCO; the MCO will be removed from the basin. One basket of fuel rubble is projected to be included in each MCO.

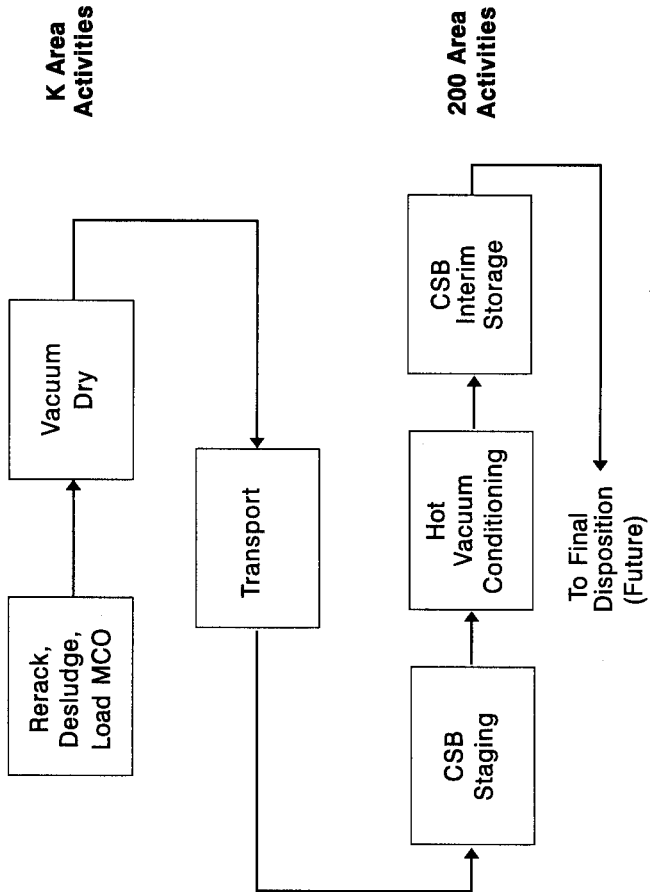
The second step involves a combination of draining and vacuum drying at approximately 50 °C to remove free water from the MCO. Following drying at approximately 3 torr pressure the MCO temperature will be increased to 75 °C and the gases evolved will be monitored. The temperature will be lowered to ambient temperatures of approximately 20 °C prior to shipping. The MCOs will be filled with an inert gas and shipped dry following completion of the vacuum drying process. It is anticipated that the MCO design will require the capability to monitor for hydrogen buildup and provide pressure relief during transportation and staging.

In the third step MCOs will be dry-staged at the Canister Storage Building (CSB) after shipping until the hot vacuum conditioning equipment is available. Cooling of the MCOs would be accomplished by natural convection. They may be monitored and will be vented with a pressure relief system for hydrogen pressure management during staging at the CSB. The MCO temperature is expected to rise to approximately 200 °C during staging in the CSB.

Finally, the fuel will be conditioned. The hot vacuum conditioning process consists of the following five steps:

1. Heat up to 300 °C while purging with pressurized helium for 24 hours.
2. Evacuate and hold for 48 hours at temperature.
3. Cool down to 150 °C with forced air cooling of the MCO exterior.
4. Oxidize the fuel surface by introducing He-O<sub>2</sub> mixtures for 12 hours.
5. Inert with He and cool down to ambient temperatures.

Figure 1. Integrated Process Strategy for K Basin Fuel Removal.



The MCOs will then be sealed and placed in interim storage in the CSB awaiting final disposition.

The following basic assumptions were made concerning the implementation of the IPS. These assumptions were necessary to establish the primary issues to be addressed by the Characterization Program to support the IPS (Lawrence 1995b).

The basic assumptions are:

1. Desludging will not be 100% effective.
2. Independent fuel and sludge laboratory testing will be applicable in processing and storage.
3. Most floor and canister sludge will be transferred to the Double Shell Tanks (DSTs) and/or solid waste for storage.

For the first assumption, a bounding safety limit was established for the quantity of corrosion products remaining in an MCO after different stages in the IPS (WHC 1996b). The nominal and the process design target for corrosion product quantities are 7 kg and 16 kg, respectively.

The testing described in this DQO will consider fuel separately from sludge. However, some tests with commingled material may be conducted if results of the independent tests and new design questions for the IPS call assumption (2) into question.

## **2.2 THE PRIMARY PROBLEMS**

The problems stated below are similar to those raised for K East Basin fuel in a previous DQO. These problems, for this K West DQO document, should be viewed with the one umbrella issue in mind i.e., are there differences in behavior between K East and K West fuel relative to the stated problems?

This sampling campaign in the K West Basin will be the last opportunity to collect fuel samples and examine them in the laboratory before MCO loading begins in mid-1998. The scope of these examinations may not provide the definitive data on the problems identified, however when combined with other ongoing activities they provide primary support for the project decisions that will be necessary to maintain the current schedule.

### **2.2.1 Fuel Drying and Conditioning for Integrated Process Strategy**

Concerns have been raised as to the adequacy of the Cold Vacuum Drying (CVD) and Hot Vacuum Drying (HVD) process temperatures and ramp rates (Rasin 1996; Bergmann 1996). The basis for this concern is lack of experimental data using prototype tests for selecting the process parameters (Rasin 1996). Specifically, there is little data on the evolution of bound and hydrated water and these, if not removed, have the potential to affect the

pressure within the MCO during staging (Rasin 1996). In summary, a better knowledge of hydrate behavior and hydrate content in the sludge that accompanies the fuel in the MCO is necessary for determining the best dehydration conditions such as temperature and time (Rasin 1996). Finally, the geometric configuration of degraded fuel is complex and so this fuel may have water in locations which are not easily accessible for drying. In order to supply data on the efficiency of water removal during prototype testing, fuel with defects which simulate or represent actual degraded fuel conditions should be employed (Rasin 1996).

In addition, ignition testing of fuel samples before and after the IPS conditioning process have not shown any clear effects of the oxidation step on increasing the ignition temperatures (Abrefah 1996). Conditioning (controlled oxidation) may or may not significantly improve the ignition temperature of the stored fuel.

### 2.2.2 Reactive Surface Area

Reactive surface area is one of the fundamental inputs in essentially all design and safety analyses. Its use is necessary in order to calculate the reaction rate with water and other oxidants, the release of heat from these reactions, the formation of corrosion products such as hydrogen, as well as the gettering rate of oxygen. Although very difficult to assess with any degree of precision, at least some estimate of surface area is necessary if any analytical base is to be developed and used for performance prediction. Design and safety analyses are currently based upon the assumption that the principal contributor to reactive SNF surface area is the K East fuel and that the contribution of K West fuel is essentially negligible. This assumption has been called into question by recent in-basin observations (Sellers 1996). It is therefore important to determine if the K West fuel contribution is significant and the extent to which this might impact the current design and safety basis.

If it is not possible to establish an estimate of the surface area, then it is necessary to develop at least some estimate of its global implications. Without either estimate, it will not be possible to predict either pressure within the MCO or the potential for runaway chemical reactions with any certainty. Insofar as global implications are concerned, the net effect of surface area is determined by the interaction of four effects, viz. the magnitude of the area itself, the local morphology of the area, the chemical reactivity of the area, and the general accessibility of the area. The importance of at least some experimental data to reduce uncertainty in this area is indicated in the Technical Baseline Validation (TBVT) report which stated "surface area is a parameter whose value is contentious and whose range requires a path to resolution" (Rasin 1996).

### 2.2.3 Surface Coatings

Recent in-basin visual examinations of the fuel indicate that several different coatings may be present on the fuel surface. These were not found in the limited examinations performed earlier and so were quite unanticipated.

The coatings tend to present a problem because, on one hand, fuel handling is not currently set up to remove them with certainty, and on the other hand, there is some recent data which indicates that they may contain uranium hydrates. Thus, if the coatings are not removed mechanically during fuel handling or coating water content is not removed thermally during CVD or HVD, the coatings have the potential to increase the pressure within the MCO as well as to supply oxidant supporting an accelerating chemical reaction.

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### 3.0 DATA QUALITY OBJECTIVE STEP 2: IDENTIFY THE DECISIONS

The problems discussed earlier in Section 2.0 lead to a number of questions which in turn may support design decisions, depending upon the data outcome. An attempt is made here to narrow the questions to those answerable by this K West Basin campaign and to frame them in a way that makes comparison to K East Basin elements possible.

#### 3.1 QUESTIONS RELATED TO COLD VACUUM DRYING, HOT VACUUM DRYING, AND CONDITIONING

The following general questions for the K West fuel relate to the successful design of the fuel processing system and to general project decisions.

- How long does it take to dry damaged K West elements under the CVD and HVD conditions?
- How much water remains in the fuel element after drying that may be released to react with the fuel under storage conditions?
- How much fuel degradation will be expected during drying and conditioning?
- How much hydrogen will be released from the fuel elements during the drying process that must be accommodated by the process equipment?

In addition to the general questions listed above, data obtained from these examinations will address these following specific questions. These may not be fully resolved with the data obtained in this sampling campaign.

- What are the locations and morphologies of cracks and are they similar to those in K East? The size of cracks greatly influences the rate at which drying occurs for the underlying material.
- How do the K West damaged elements behave (dripping sludge, widening cracks, etc.)?
- Are the drying and conditioning behavior of the whole elements similar to the whole element testing of K East degraded elements in progress (Lawrence 1996b)? If not, do these differences suggest changes in the fuel processing design (CVD, HVD, and conditioning)?
- Are the K West Basin fuel properties such as oxidation rate, ignition temperatures, and hydride decomposition behavior different from the initial two elements from K West or from the K East elements with examinations in progress (Lawrence 1996b)?
- What is the internal void volume, amount of trapped sludge, and amounts of water trapped on the exterior and interior of the elements to be removed during drying?



### 3.2 QUESTIONS RELATED TO REACTIVE SURFACE AREA

Knowing reactive surface area depends on knowing (1) the number of damaged elements (2) extent of damage in each and (3) the area available for chemical reactions in each. Chemical reactions are generally based on an effective surface area which includes all surfaces available for reaction on a molecular level rather than the geometric surface area which is most easily measured. Confusion has developed in design and safety calculations over what values to use for fuel surface area (Rasin 1996). Examinations of K West fuel will address specifically:

- What is the inventory of damaged fuel?
- Is there any correlation of the appearance of K West fuel damage with the previous gas and liquid sampling of the canisters? A total of 50 canisters (2 barrels per canister) were sampled for liquid (and occasionally gas) composition as part of the in-basin characterization of the K West closed canisters (Makenas 1996a). If such a correlation can be found, it may lead to a way to estimate reactive surface area in the remaining large number of unopened canisters. The current surface area estimate utilizes the release of cesium to the water in K East Basin to calculate an effective reactive surface area (WHC 1996a). It may be possible, based on the results of the canister liquid sampling, to apply the same logic to the individual canisters with a better correlation of observed damage to reactive surface area. This effort would involve the comparison of visual observations made in the hot cell with the measured concentration of fission products liberated during corrosion. Measurements of cesium in the canister water will be conducted under activities in a separate DQO (Section 4.1.1).

### 3.3 QUESTIONS RELATED TO SURFACE COATINGS

From the recent video survey of elements from 24 K West canisters it has been shown that in many (but not all) cases the sludge on cladding surfaces appears to be different in texture and color than that on K East elements. Ultimately, a decision must be made as to whether: (1) the coatings will be removed mechanically during fuel handling, (2) any water which may be in the coatings will be removed thermally during CVD or HVD, or (3) the impact of the water will simply be accepted within the current design and safety basis. To support such decisions, the following questions should be answered:

- To what extent are the coatings adherent and can they be easily removed?
- What is the bound and hydrated water content in the coatings and is it sufficient to suggest that removal of the coating is warranted prior to loading fuel in an MCO?
- Is the adherent K West sludge similar in quantity, adherence, and composition (including hydrates) to corresponding sludge on K East elements?

#### 4.0 DATA QUALITY OBJECTIVE STEP 3: IDENTIFY THE INPUTS

The inputs for the questions raised in Section 3.0 are divided into those which derive from previous DQOs such as, sludge sampling and those associated with the current DQO document.

##### 4.1 RELATED INPUTS FROM PREVIOUS DATA QUALITY OBJECTIVES

The retrieval of fuel and associated laboratory examinations are part of a larger campaign in K West consisting of inter-related activities covered in several documents. These are summarized in Table 2 and in the following subsections.

Table 2. Sequence of In-Basin and Laboratory Activities.

Step	Activity	Reference
1	Complete gas and liquid sampling in 50 canisters.	DQO: Makenas 1996a
2	Video coverage of all activities and fuel movements while opening 24 canisters. Measure sludge depth in canisters with an ultrasonic probe. Remove each element if possible and conduct a full length visual/video inspection.	Letter: Makenas 1996c
3	Remove selected canister sludge and ship to hot cells.	DQO: Makenas 1996b
4	Select K West fuel elements and place in a Single Fuel Element Container (SFEC) for shipment to the hot cells. Measure hydrogen gas generation in SFECs before shipment to hot cells. Ship fuel to hot cells.	This DQO
5	Perform laboratory examinations on fuel elements.	This DQO

#### 4.1.1 Gas and Liquid Sampling

These activities are covered under a separate DQO (Makenas 1996a). These sampling activities support the selection of canisters for fuel sampling and provides an assessment of gas/liquid water chemistry for comparison to results of the fuel and sludge hot cell examinations. These data taken together may lead to an improved estimate of reactive surface area (Section 3.2) for unopened canisters when the gas and liquid data are considered with hot cell visual examinations (Section 4.2.3). Specific characteristics of this DQO applicable to these data evaluations is the cesium concentration in the liquid samples.

#### 4.1.2 Sludge Depth Measurements

Canister sludge depths have been measured using an ultrasonic probe that detects the interface between the basin water and the sludge (Pitner 1996d). Measurement at multiple locations within the canister was necessary to obtain a sludge depth profile in the canister. The measurements provided initial estimates of the sludge available for sampling (Makenas 1996c). Sludge depth provides quantitative data on the extent of fuel reaction after encapsulation to possibly compare with cesium measurements. This is additional information that is outside the scope of this DQO but support the analysis.

#### 4.1.3 Canister Sludge Sampling

Canister sludge has been removed from selected canisters for laboratory examinations. These activities are covered in a separate DQO (Makenas 1996b). Sludge drying characteristics relate directly to the sludge that will accompany the fuel in the MCO and support overall drying behavior.

#### 4.1.4 Visual Inspection of Element Surfaces

Elements have been removed from 24 canisters for high resolution video inspection of the fuel element surfaces. Inspections focused on the condition and extent of any degradation resulting from a cladding breach and the condition and extent of any sludge that may be adhering to the element surfaces. These examinations are similar to the full length examinations of K East fuel (Pitner 1996b, 1996c).

Video tapes of the surface inspections will also be used to finalize the selection of elements for transport to the laboratory for detailed examinations that satisfy this DQO document. Detailed visual examinations will also provide some qualitative assessments of the appearance and extent of surface coatings on the fuel elements (Section 3.3). These examinations will provide experience in the video inspection of elements for this cleaned fuel acceptance. Quantitative data on the amount of sludge adhering to the fuel element surfaces may be obtained during the hot cell examinations of the fuel samples.

#### 4.2 IN-BASIN AND LABORATORY MEASUREMENTS FOR THE CURRENT DATA QUALITY OBJECTIVES

This DQO section establishes the tie between specific examinations and the three main questions listed in Sections 3.1 to 3.3. The laboratory measurements for this second batch of K West fuel will in general complement the examinations conducted on the first group of fuel elements from K West (Lawrence 1995a) including the conditioning testing (Lawrence 1995b), and the corresponding Thermo-Gravimetric Analysis (TGA) measurements (Lawrence 1995c) and complement the corresponding K East fuel examinations (Lawrence 1996a, 1996b). In-basin measurements will include visual and quantitative hydrogen generation information. Laboratory measurements include establishing the initial behavior of the fuel when first exposed to cell air atmosphere, detailed visual examinations to supplement in-basin video, metallography for fuel and corrosion product conditions and structures, furnace testing, and TGA measurements to establish conditioned fuel response to anticipated storage conditions.

The examinations proposed here will provide the data for an assessment of the behavior of representative material to the conditioning process including drying behavior, hydride behavior, hydrogen release, continued fuel degradation, and dry storage behavior. Determination of the adequacy of the proposed full scale IPS drying and conditioning processes or the need for any modifications to the current designs will not be fully answered by data obtained here due to the limited nature of the sampling and the number of samples available for testing. These tests will however provide understanding to be expanded if necessary, in the future, with additional fuel samples and testing on a larger more prototypic scale. The inputs discussed below are summarized in Table 3 along with relations back to decisions in Section 3.0.

Table 3. In-Basin and Laboratory Examinations to be Performed on K West Fuel as Part of the Current Data Quality Objectives.

Examination	Decision Supported	Report Section
Video of in-basin activities	Good practice	
Measure hydrogen generation	Surface area	3.2
Hot cell visual examination	Surface area, surface coating, and drying	3.1, 3.2, 3.3
Metallography	Conditioning	3.1
Furnace testing	Drying and conditioning	3.1
Thermo-gravimetric analysis	Drying and conditioning	3.1
Scraping and analysis of coatings	Surface coatings and drying	3.1, 3.3
Subsurface Deposits	Drying and surface area	3.1, 3.2

#### 4.2.1 Visual/Video During In-Basin Fuel Shipping Activities

The fuel element selection and shipping activities will be recorded on video in a similar manner to the first sampling and shipping campaign for K West fuel samples (Lawrence 1995a) and the K East fuel sampling (Lawrence 1996a). This is considered a good practice activity even though the data generated will not directly support this DQO. The video record of the canister and fuel element movements will provide qualitative data on how much sludge will be released to the basin water and what will be the effects on water clarity and visibility. This will supplement the larger video inspection discussed above as well as water clarity objectives listed in the sludge examination DQO (Makenas 1996b).

#### 4.2.2 Hydrogen Generation Measurement Before Shipment

Single Fuel Element Containers (SFECS) containing the selected elements will be monitored for gas generation as a condition for shipment in the Chem-Nuclear cask (Stevens 1994). Elements in the K West Basin are known to be releasing gases during storage as evidenced by the gas collected from the closed canisters. Actively degrading elements may be selected for laboratory examinations and data on the quantities and composition of the gases given off would provide information on the mechanisms responsible for the continued degradation of fuel under water.

#### 4.2.3 Visual/Video During Hot Cell Examinations

All the in-cell activities will be closely monitored for changes in sample appearance and recorded on video where appropriate. These examinations will provide data sequential to visual examinations performed in the basin during canister and fuel movement for shipment.

These examinations provide the first opportunity to observe the behavior of the material in air after substantial underwater storage in closed canisters. This is analogous to what may happen in the case of a "dry" shipment. An understanding of the behavior of the material after exposure to air is necessary to evaluate postulated shipping accident scenarios where the MCOs are damaged and the elements are accidentally exposed to air during handling or transport. The examinations of elements from the initial K West shipment showed no degradation or reactions when the fuel originally stored in closed canisters was first exposed to the cell atmosphere. Reactions, such as pyrophoric tendencies, would directly impact the IPS as noted in Section 3.1.

These detailed visual examinations and photography supplement and expand the in-basin video inspections and supplement the earlier data for K West fuel elements. Some loose cladding may be removed to allow improved photography of underlying fuel. The cracking patterns and amounts of associated corroded fuel and sludge shall be documented as input to questions covered in Sections 3.1 and 3.2 for process and surface area determinations. These examinations also characterize the various surface coatings on the elements selected for laboratory examinations (Section 3.3).

#### 4.2.4 Metallography

Selected fuel samples will be removed from the fuel elements for metallographic examinations. These examinations will be focused on establishing the characteristics of the corrosion occurring in the breached and failed elements. Examinations will establish the fuel condition, extent of oxidation, morphology of the oxide layer, and possibly hydride formation. Sample preparation techniques utilized for the initial K West fuel examinations have been successful in identifying the presence of hydrides in the fuel. The presence and distribution of hydrides and the characteristics of oxide layers are specific inputs to the questions raised for the IPS process design in Section 3.1.

#### 4.2.5 Furnace Testing of Whole Elements and Scrap

Controlled temperature and atmosphere furnace testing will be conducted on whole elements and possibly on selected small samples to determine fuel and corrosion product drying behavior and corresponding hydride decomposition and hydrogen release from the exposed fuel. A gas chromatograph on this furnace measures gases emitted by the sample at high temperature (such as water emitted during drying) and measures atmosphere components such as oxygen scavenged by the samples from the circulating input gas. One or two collections of fuel rubble similar to the expected contents of an MCO scrap basket will also be processed in the furnace. Fuel ignition tests of small samples may also be conducted in the controlled temperature and atmosphere furnace. The questions to be answered using these measurements are those listed in Section 3.1 for CVD, HVD, and conditioning are generally the same as those established in the DQO for the furnace testing of samples from the first K West campaign (Lawrence 1995b).

Furnace testing will be expanded from initial small samples (up to 30 g each) for the first K West fuel studies up to complete unsectioned inner or outer elements for the current campaign. Fuel response to the drying and conditioning process is expected to be strongly dependent upon the fuel surface exposed. Furnace testing of the K East fuel samples focused on whole elements (Lawrence 1996a, 1996b). The current tests will include whole elements from K West. In addition to the whole elements one or two collections of fuel pieces, similar to the expected contents of an MCO scrap basket, will be dried and conditioned in the hot cell furnace using the IPS process. Surface areas for the expected contents of an MCO scrap basket are expected to represent a significant portion of the total surface area in an MCO for continued fuel corrosion and gas generation prior to hot vacuum drying and conditioning.

Whole element samples will be visually examined after drying and conditioning to evaluate the effects of the conditioning process on the character of the oxide layers formed.

#### 4.2.6 Thermo-Gravimetric Analysis Testing

Chemical reaction rates, hydrogen release, and fuel drying characteristics will be established with small samples in a TGA system. The TGA gives a continuous measurement of weight change as temperature is raised and thus tracks the emission of volatiles and absorption of gases as a function of temperatures. Samples up to approximately 20 g in weight will be tested in the system. The data inputs and the questions to be answered with these measurements are given in Section 3.1 and are generally the same as those established in the DQO for TGA testing of the first group of samples from K West (Lawrence 1995c). Testing may be expanded to consider parametric effects as well as some fuel variables not considered during the first K West campaign.

#### 4.2.7 Analysis of Element Coatings

Coatings will be scraped from the cladding of selected fuel elements in the laboratory. Collected coatings will be weighed to give a weight of coating per area scraped. X-ray diffraction analysis will be performed to identify the compounds present through crystal structure determination. Precursor information may also be obtained by brushing selected elements in the K West Basin to give qualitative adherence characteristics. Samples may be tested in the TGA for drying behavior based on results of the X-ray diffraction analysis.

#### 4.2.8 Examination for Subsurface Sludge Deposits

Subsurface deposits of sludge have been postulated based on the visible appearance of the fuel elements. These may prove difficult to dry during the IPS. Selected K West elements will be examined, using the techniques developed for the K East fuel elements, to look for internal void volumes and amounts of trapped sludge (Lawrence 1996b). These examinations relate to questions raised concerning the drying behavior during CVD and HVD (Section 3.1), as well as the possible reactive surfaces available for oxidation (Section 3.2).

## 5.0 DATA QUALITY OBJECTIVE STEP 4: DECISION BOUNDARIES

The fuel shipped to the laboratory for examinations will be restricted to K West damaged fuel representative of the most degraded fuel that will be handled, dried, and conditioned as whole elements in the IPS and to representative examples of the different fuel surface deposits observed in the visual examinations of the K West fuel elements.

Processing activities for K Basin fuel and sludge are constrained by the overall project schedule to begin loading the MCOs and removing fuel from the basins in 1998 (Hudson 1996). This accelerated schedule dictates only one additional sampling campaign in K West with a moderate amount of time, i.e., approximately 1 year, for examinations before the process strategy is finalized with the loading and movement of the first fuel elements. In addition, the current fuel shipping pathway to the laboratory will be unavailable after FY 1997. Equipment to be installed in the K West Basin South loadout pit will eliminate that facility for future shipments with the Chem-Nuclear cask. If additional samples are needed in the future for process definition and safety evaluation a new pathway will have to be developed with major schedule and cost impacts to the SNF Project schedule.

Current budget and operational support limitations dictate a sampling of nine canister sludge samples (Gerber 1996). The current planning budget supports a total of four cask shipments from the K West Basin to the laboratories for both the sludge and fuel samples. The cask can accommodate a maximum of six fuel and sludge containers for a total of 24 samples for the four shipments. Therefore, the maximum number of fuel samples that can be removed for examination within the current budget and schedule constraints is 15.

The analysis of the K West Basin fuel had been reduced from nine to three fuel elements as a cost savings based on the assumption that there was less damaged fuel in K West than K East. As a result of increased fuel damage discovered during the visual inspection of the element surfaces (Section 4.1.4) the K West fuel laboratory characterization has been increased to 15 fuel elements (Hudson 1996). The current Multi-Year Program Plan (MYPP) and approved change requests SNF-96-071 and SNF-96-085 authorize shipment of three fuel elements to the hot cells for examinations (Gerber 1996). The shipment of samples to the hot cells will be limited to three elements unless the MYPP is revised or a change request is approved. An urgent change request may be necessary to meet the target dates for sampling and shipment to the hot cells.

### 5.1 K WEST FUEL AVAILABLE FOR SAMPLE SELECTION

The visual inspections of the K West fuel surfaces (Section 4.1.4) identified the fuel attributes that need to be considered in developing the sampling strategy for the K West Basin. The fuel elements observed in the 24 canisters that were examined can be divided into two categories for selection of samples for the laboratory examinations, i.e., fuel exhibiting coatings or damaged fuel.



### 5.1.1 Surface Coatings

Five different coatings were observed during the visual examinations. These five coatings are summarized as follows:

1. White crystals
2. An orange coating on the fuel (iron or uranium oxides and hydrates?)
3. A thin translucent coating
4. A thick white, brittle coating
5. A thin coating similar to the ones found in the K East Basin.

The thin translucent coating and white brittle coating most likely will not survive the fuel handling and cleaning process and are generally not considered to be part of the sampling process for the fuel. The identification of this material may however, be necessary for the removal and disposal of this material following the fuel recovery and cleaning process.

### 5.1.2 Damaged Fuel

The damaged fuel in the K West Basin can be grouped into the following four general categories for possible sampling based on recent in-basin visual examinations.

1. Cracked Fuel -- Damaged fuel elements were observed with extensive cracks on the outer surfaces with and without significant outer damage.
2. Broken End Caps -- Damaged fuel with displaced and missing top and/or bottom end caps was found during the visual examinations and is similar to damage seen in the K East Basin. However, in the case of K West there appears to be less sludge trapped within the damaged fuel compared to K East fuel with similar broken or missing end caps. This is evidenced by the lack of sludge dripping from cracks.
3. Broken Fuel -- Fuel elements were examined that broke into large pieces somewhat similar to the K East fuel. These broken ends of the elements do not show the fuel loss generally seen with end cap failures.
4. Severely Damaged Fuel -- K West fuel elements with a combination of missing end caps, extensive fuel loss, axial cracking, deformation, and fractured large pieces have been seen. This is, in general, what was observed in K East for the severely damaged fuel.

Differences in the extent and nature of the fuel damage may dictate the drying behavior under CVD and HVD conditions. It may be easier to remove the fuel and bound water from severely damaged fuel which has good communication with the process environment than from less damaged fuel with trapped water and sludge.

## 5.2 RECOMMENDED SAMPLING

An attribute driven sampling plan for the damaged fuel similar to the strategy selected for the K East fuel examination would support the following sampling matrix for whole element drying studies (Table 4).

Table 4. Fuel Sampling Matrix for Furnace Tests.

Attribute	Recommended Number of Samples
Cracked fuel	3
Broken end caps	4 (2 top/2 bottom)
Broken fuel element (approximately half an element)	1
Severely damaged fuel	3
Total	11

For whole element furnace testing a total of three elements each will be selected for the cracked and severely damaged fuel since either may be the bounding elements. Three samples provide for more than simple duplication and are considered to be a minimum number to support a greater understanding of the influences of fuel damage on drying. Three elements, each with top and bottom failures, are currently being tested from the K East fuel for this reason (Lawrence 1996b). Two K West elements each will be selected for the top and bottom end cap failures respectively since three of each are already being tested for K East fuel. Two elements provide duplication to address the question of whether K West fuel drying behavior is bounded by K East fuel behavior (for similar fuel damage) or whether K West fuel represents a different population from K East (Section 2.2).

Samples are also required in order to characterize the element coatings (Section 4.2.7), to provide elements for examination for subsurface sludge deposits (Section 4.2.8), and to facilitate TGA measurements (Section 4.2.6). Three elements will be selected to characterize the three coatings (Numbers 1, 2, and 5 in Section 5.1.1). These elements would also provide the small samples for TGA testing and metallography in a similar manner to the current K East examination (Lawrence 1996b).

Examinations of subsurface sludge deposits will require at least two damaged elements which most likely will not be the elements selected for surface coatings or small samples. Only one element was available for the K East fuel for this examination step which is considered insufficient to provide anything beyond a general qualitative understanding of whether or not subsurface sludge deposits are to be expected in the damaged fuel.

In summary, this sampling strategy, based on the attributes of the fuel seen in K West, corresponds to a minimum of 16 elements. However, the program is constrained to 15 elements. Therefore, one of the sampling categories must be reduced to stay within the program boundaries. The reduction in the matrix for individual attributes is best made after the fuel samples are retrieved from the Basin and their actual condition can be used to adjust the testing matrix in a manner similar to what was done with the K East sampling campaign.

### 5.3 THE STATISTICS OF WHOLE ELEMENT FURNACE TESTING

The larger number of samples for K West also provides an opportunity to consider statistics in the recommended sampling. The amount of damaged fuel in the K West closed canisters appears to be similar to the amount for the K East fuel established with the video surveys. One sided nonparametric tolerance limits were selected for consideration of the drying and conditioning behavior of the damaged elements (Welsh 1996).

Nonparametric tolerance limits are based on rank or order statistics, i.e., the data are sorted from low to high. For a group of damaged elements a one-sided nonparametric tolerance limit will establish the confidence that a given portion of the population lies below the largest observed behavior. In the case of the K West damaged fuel, the value selected is the response to the IPS drying and conditioning process. Specific questions, such as how long does it take to remove the free water during cold vacuum drying or correspondingly the bound water during hot vacuum drying, will be the ordered observed values. There may or may not be a correlation of extent of damage with drying or conditioning behavior. Water may be removed more easily from fuel elements with extensive damage and with a lot of exposed fuel compared to elements with corrosion products trapped under cladding with only limited access to the process atmospheres. The results of the order statistics for the one-sided nonparametric tolerance limits are summarized in Table 5.

A sample size of 11 gives reasonable confidence (i.e., on the order of 90%) that a large percentage (i.e., 80%) of the population lies below the largest value observed for element drying and conditioning. The corresponding 10% uncertainty in the data is considered to be acceptable for the application of the results to the decisions outlined in this DQO. Reducing the confidence level increases the percentage of the population that lies below the largest value observed.

Table 5. One-Sided Nonparametric Tolerance Limits.

Sample Size	Confidence Level (%)	Percentage of Population that Lies Below Largest Value Observed (%)
7	50	90
7	75	80
7	85	75
14	50	95
14	75	90
14	95	80
22	90	90
29	95	90
45	90	95
59	95	95

In summary, the second K West fuel laboratory examinations will consist of 15 fuel elements. Eleven elements will be dedicated to whole element furnace testing initially using the reference CVD, HVD, and conditioning times, temperatures, pressures, and atmospheres. This recommended sample provides the opportunity to conduct duplicate tests which is not the case for the current strategy for K East or examine some changes to the drying and conditioning process parameters such as temperature, ramp rates to HVD, and pressure. This sample size also provides the first opportunity to consider statistics in bounding the fuels response to the IPS drying and conditioning processes.

#### 5.4 LABORATORY TEST CONDITIONS FOR WHOLE ELEMENT DRYING

The following test matrix (summarized in Table 6) for the 15 elements is provided for demonstration purposes only on what could be accomplished with the recommended sample size. This option will retain the original number of 15 fuel elements in the sample and adjust the test matrix to address the two test objectives of establishing fuel drying behavior for the reference IPS drying process and evaluate alternate temperatures for cold and hot vacuum drying.

Table 6. Possible Test Matrix for Fifteen Elements.

Test	CVD Temperature		HVD Temperature		Ramp Rate		
	50 °C	75 °C	300 °C	375 °C*	Nominal	Slow	Fast
1-6	X		X		X		
7		X	X		X		
8	X			X			
9	X		X			X	
10	X		X				X
11		X		X	X		

Elements 12 through 15 selected for surface deposits, subsurface examinations and sectioning for small samples.

\*For demonstration only, this temperature would be established if it was necessary to evaluate a higher process temperature.

One of the recommendations from the TBT report for fuel drying was, "characterization and prototype testing data should be available to help set the final process parameters for CVD," (Rasin 1996). The expanded K West test sample size of 11, compared to six for K East, provides an opportunity to evaluate some of the process parameters such as CVD temperature. Similarly the French data suggests that 300 °C may be sufficient for HVD however, the kinetics may be such that increasing the temperature by 50 °C to 100 °C greatly improves the process times. The expanded sample size for drying provides sufficient samples to evaluate effects of temperature. These changes may be dictated by the initial result of the whole element drying tests with K East elements. Long drying times for some of the damaged fuel may suggest an increased process temperature would be appropriate.

This possible test matrix is conditional on the actual appearance of the elements selected and shipped to the hot cells for testing and on the relative importance of establishing bounding drying behavior and establishing the effects of different process temperatures and ramp rates on the fuels drying behavior.

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

A limited scope DQO process is being used for the examination of the K West fuels. Therefore, the development of decision rules and of acceptable limits on the decision errors is beyond the scope of this document. The justification for this is that the in-basin examinations and laboratory measurements provide additional data on the behavior of the fuel material after extended storage in closed canisters and expand the furnace testing to include whole elements and representations of the contents of a scrap basket. These examinations will provide data in areas where none presently exist or where only limited information is available to address questions related to fuel retrieval, cleaning, and drying and conditioning following loading of fuel into the MCOS.

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## 7.0 DATA QUALITY OBJECTIVE STEP 7: OPTIMIZE

The examinations are being optimized by the use of a flexible sampling plan to select the samples for hot cell examinations in a similar manner to the ongoing K East fuel examinations. Information gained during each step of the examination will be utilized to obtain the most relevant information from subsequent examination steps. A detailed sampling plan will be developed supplementing this DQO (Makenas 1996d). This plan identifies the specific canisters and elements to be shipped to the hot cells for examinations.

Approval of this DQO will support the recommended sampling of 15 elements for bounding the drying behavior of K West damaged fuel using the reference IPS temperatures and pressures. A strategy for the examinations of these 15 elements will be developed based on the actual condition of the elements retrieved and shipped to the laboratory for examination in a similar manner to the current K East fuel examinations (Lawrence 1996b). This strategy document will be reviewed and approved by the same program participants who approve this DQO.



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