Accession #: D195067309

Document #: SD-SNF-DQO-007

Title/Desc:

DATA QUALITY OBJECTIVES FOR THE REACTION KINETICS STUDIES OF K WEST FUEL SAMPLES

Pages: 26

DEC 14 1995 (21) Sta. 21

ENGINEERING DATA TRANSMITTAL

1. EDT 610187

2. To: (Receiving Organization) Distribution	3. From: (Originating Organization) Nuclear Fuel Evaluations 8M710	4. Related EDT No.:
5. Proj./Prog./Dept./Div.: SNFP	6. Cog. Engr.: L. A. Lawrence	7. Purchase Order No.: N/A
8. Originator Remarks: For approval and information -- Data Quality Objectives for the Reaction Kinetics Studies of K West Fuel Samples		9. Equip./Component No.: N/A
		10. System/Bldg./Facility: K Basins/100 K
11. Receiver Remarks:		12. Major Assm. Dwg. No.: N/A
		13. Permit/Permit Application No.: N/A
		14. Required Response Date:

15. DATA TRANSMITTED

(A) Item No.	(B) Document/Drawing No.	(C) Sheet No.	(D) Rev. No.	(E) Title or Description of Data Transmitted	(F) Approval Designator	(G) Reason for Transmittal	(H) Originator Disposition	(I) Receiver Disposition
1	WHC-SD-SNF-DQO-007		0	DATA QUALITY OBJECTIVES FOR THE REACTION KINETICS STUDIES OF K WEST FUEL SAMPLES	N/A	1,3	1	

16. KEY

Approval Designator (F)	Reason for Transmittal (G)	Disposition (H) & (I)
E, S, Q, D or N/A (see WHC-CM-3-5, Sec.12.7)	1. Approval 2. Release 3. Information 4. Review 5. Post-Review 6. Dist. (Receipt Acknow. Required)	1. Approved 2. Approved w/comment 3. Disapproved w/comment 4. Reviewed no/comment 5. Reviewed w/comment 6. Receipt acknowledged

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1		Cog. Mgr. R. P. Omberg	[signature]	11/30/95	R3-85						
		QA									
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		Env.									

18.	19.	20.	21. DOE APPROVAL (if required)
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DATA QUALITY OBJECTIVES FOR THE REACTION KINETICS STUDIES OF K WEST FUEL SAMPLES

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U.S. Department of Energy Contract DE-AC06-87RL10930

EDT/ECN: 610187 UC: 2070
Org Code: 8M710 Charge Code: LB020
B&R Code: EW 3135040 Total Pages: 22

Key Words: DQO, Fuel Examinations, 105-K West, SNFP

Abstract: The Data Quality Objectives (DQOs) were established for the reaction kinetics studies of the first group of fuel samples shipped from the K West Basin to the Hanford 327 Building hot cells for examinations. A Thermo-Gravimetric Analysis (TGA) system was selected for these measurements and associated hydrogen release and ignition temperature testing. These examinations are an extension of the conditioning testing of sibling samples described in WHC-SD-SNF-DQO-004, Data Quality Objectives for the Initial Fuel Conditioning Examinations.

Karen G. Noland 12/14/95
Release Approval Date Release Stamp

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DATA QUALITY OBJECTIVES FOR THE REACTION KINETICS STUDIES OF K WEST FUEL SAMPLES

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December 1995

Document Title: **DATA QUALITY OBJECTIVES FOR THE REACTION KINETICS STUDIES OF K WEST FUEL SAMPLES**

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EXECUTIVE SUMMARY

The Data Quality Objectives (DQOs) were established for the reaction kinetics studies and associated hydrogen release and ignition temperature testing of the first group of fuel samples shipped from the K West Basin to the Hanford 327 Building hot cells for examinations. A Thermo-Gravimetric Analysis (TGA) system was selected for these measurements. These examinations are an extension of the conditioning testing of sibling samples described in WHC-SD-SNF-DQO-004, Data Quality Objectives for the Initial Fuel Conditioning Examinations.

The data obtained from the TGA measurements will be used to benchmark the basic input for the following:

- Oxidation rate data in support of Multi-Canister Overpack (MCO) air ingress analysis, vacuum drying, and passivation.
- Hydrogen generation rate data for determinations of hydrogen influence in reaction kinetics, potential for MCO pressure loading, influence on possible fuel hydride migration, and the potential to mask process parameters.
- MCO pressurization from bound water released from the degraded fuel and accompanying sludge.

The limited scope represented by these samples will not provide definitive data for all the decisions that must be made related to reaction kinetics and hydrogen release. However, the data input when combined with other data from the controlled temperature and atmosphere furnace 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.

CONTENTS

DATA QUALITY OBJECTIVES FOR THE REACTION KINETICS STUDIES OF K WEST FUEL SAMPLES

1.0 INTRODUCTION

The Data Quality Objectives (DQOs) were established for the reaction kinetics studies and drying characteristics of the first group of fuel samples shipped from the K West Basin to the Hanford 327 Building hot cells for examinations. A Thermo-Gravimetric Analysis (TGA) system was selected for these measurements. These TGA examinations are an extension of the conditioning examinations conducted on the initial fuel samples from K West (Lawrence 1995a). The DQO process was employed to ensure the planned examinations fully support the Integrated Process Strategy (IPS) to resolve the safety and environmental concerns associated with the deteriorating fuel in the K Basin (Lawrence 1994).

The examinations covered by this DQO are the TGA measurement of samples to establish the oxidation kinetics, drying behavior, hydrogen release, and ignition temperatures of the irradiated fuel material. This DQO complements and expands the DQO on conditioning for these samples to elaborate on the TGA testing (Lawrence 1995a).

Current conditioning and storage process modeling and design based accident analyses involving air ingress to the Multi-Canister Overpack (MCO) are utilizing oxidation kinetic data published for unirradiated uranium adjusted to account for effects of irradiation. The adjustment factor may unduly penalize the design margins to account for the lack of data on irradiated N Reactor fuel. The TGA measurements will provide the necessary data to bracket expected conditions to compare to the published data. The TGA measurements also provide a complementary tool to provide comparative data for the hydrogen release measured in the conditioning testing (Abrefah 1995) and the ignition temperatures measured in the controlled temperature and atmosphere furnace.

The source term for pressurization of the MCO is strongly dependent on the drying characteristics of the fuel material. The TGA will be used to determine the drying characteristics of the damaged fuel to provide supporting data for process definition to remove the waters of hydration. Similar measurements are in progress for the K East floor and pit sludge to determine their drying characteristics (Makenas 1995).

2.0 DATA QUALITY OBJECTIVE STEP 1: STATEMENT OF PROBLEM

The conditioning process identified for the IPS differs somewhat from the conditioning process proposed by the Independent Technical Assessment (ITA) Team (WHC 1995) which was the original focus of the characterization testing.

The IPS process is summarized in Figure 1. 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 canisters may have to be mechanically sectioned to remove the elements. Fuel requiring desludging 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, and the basin water will be drained from the MCO.

The second step involves a vacuum drying cycle at approximately 50 °C to remove free water from the MCO. 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.

The MCOs would be dry staged at the Container Storage Building (CSB) after shipping until the hot vacuum conditioning equipment is available. Cooling of the MCOs would be accomplished by natural convection and they would be monitored and vented as necessary for hydrogen pressure management during staging at the CSB.

The hot vacuum conditioning process consists of the following six 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.

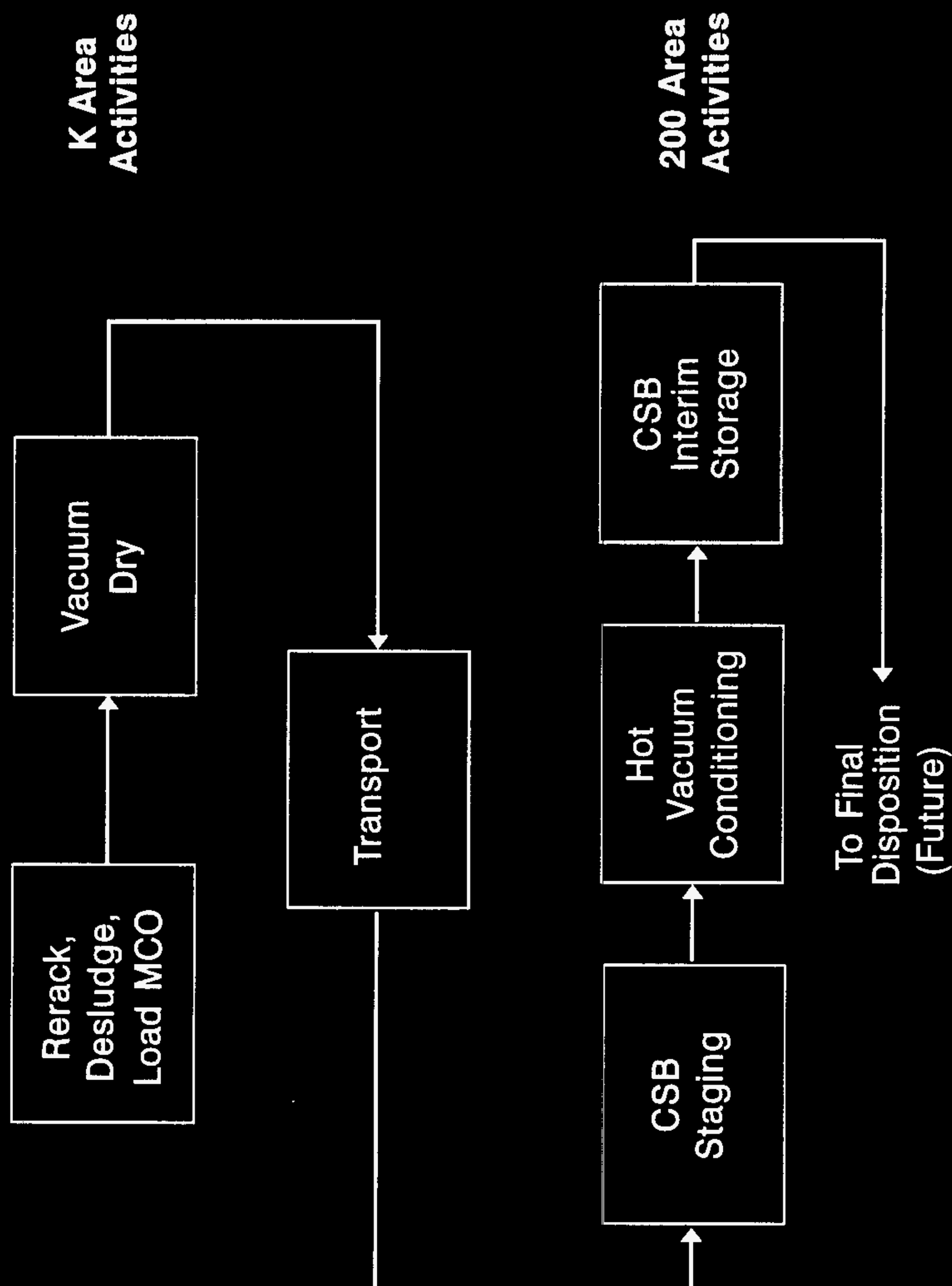
4. Oxidize the fuel surface by introducing He-O_2 mixtures for 24 hours.

5. Inert with He and cool down to ambient temperatures over 8 to 16 hours.

6. Monitor for H_2 production rate during an initial storage period.

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

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



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

The basic assumptions are:

1. Fuel will be sorted by visual examination into the following three classifications during reracking.

   a. Visibly undamaged fuel with possible incipient failures.

   b. Intact failed fuel with some dimensional distortion and minimal fuel loss.

   c. Grossly failed fuel, fuel chips, and rubble.

2. Desludging will not be 100% effective.

3. Independent fuel and sludge laboratory testing will be representative of commingling for processing and storage.

4. Basin sludge will be transferred to the Double Shell Tanks (DSTs) for storage.

The first assumption recognizes the opportunity reracking provides for fuel sorting and possible reductions in the number of MCOs or volumes of fuel that needs to be conditioned. The RL approval of the IPS also identifies fuel segregation as a basis for the level of conditioning each MCO will undergo (Hansen 1995).

The IPS recognizes that desludging will not be 100% effective, and has selected a target value of 25 grams of residual sludge or less per assembly as a performance specification (e.g., upper bound) (WHC 1995).

The bulk of the testing will consider fuel and sludge separately however, some tests with commingled material may be conducted based on results of the independent tests and specific design questions to be answered for the IPS.

The following areas of technical uncertainty were established for the conditioning testing in the furnace and they are directly applicable to the complementary TGA measurements (Lawrence 1995a):

- 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 conditions 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. The characteristics of the oxide layer formed on the uranium during the passivation portion of the conditioning process are needed to support process definition.

Evaluations of the initial conditioning data reported (Abrefah 1995) identified reaction rate data and hydrogen generation rate data as valuable information for initial design model calibrations (Wiborg 1995).

Stakeholders for the TGA measurements are listed in Table 1. Formal and informal meetings were held with various stakeholders to develop the input and concurrence was reached for this DQO covering reaction kinetics studies.

Table 1. Stakeholders for Thermo-Gravimetric Analysis Measurements.

Function	Individual Point of Contact
IPS - Cold Vacuum Drying - Hot Vacuum Drying/Conditioning - Safety	J.J. Irwin C.R. Miska J.C. Wiborg
Environmental Input Statement	T.A. Thornton
Advanced Technology Program Office	P.A. Scott
RL	J. Shuen
Quality Assurance	D.W. Smith
Characterization - WHC - PNL	 R.P. Omberg 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 and what are the effects of changes in the time, temperature, gas pressure, and gas composition on the final state of the conditioned fuel? Fuel was selected for the initial examinations to bound the fuel conditions expected to be encountered in the closed canisters in K West (Lawrence 1995a).

Specific project decisions will be focused on establishing the conditioning process definition and safety analyses and 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. The adequacy of the proposed IPS conditioning process or any modifications to the recommended conditioning process will not be fully resolved by these examinations due to the limited nature of the sampling and the number of samples available for testing. These tests will however provide initial understanding of the fuel condition and effects of the IPS conditioning process. In addition these initial data will provide the basis for more extensive testing with additional samples from the second shipping campaign.

4.0 DATA QUALITY OBJECTIVE STEP 3: IDENTIFY THE INPUTS

The data obtained from these measurements will be used to benchmark the basic input for the following calculations.

A. Chemical Reaction Rate Data to:

1. Establish MCO air ingress oxidation models including the subset referred to as ignition theory.
2. Establish vacuum drying and oxidation during drying models.
3. Establish the passivation phase of the conditioning model.

B. Hydrogen Generation Rate Data to:

1. Determine whether hydrogen significantly influences reaction kinetics, and whether it must be removed or it can be accommodated in the drying and conditioning processes.
2. Determine whether hydrogen from the fuel is a potential source for MCO pressurization during staging and long term storage.
3. Determine whether hydrogen will migrate from the interior to the cooler exterior uranium surfaces thus forming exposed uranium hydrides and decreasing the value of conditioning.
4. Determine whether hydrogen will mask any of the process parameters.

C. Drying Characteristics to:

1. Establish the amounts of free and bound water removed during drying cycles.
2. Model MCO pressurization from water remaining in the fuel and sludge following drying.

A TGA system was selected for these measurements. The system shown schematically in Figure 2 was placed in a shielded glovebox for the measurements. The system includes a TGA and Differential Scanning Calorimeter (DSC) coupled with a Quadripole Mass Spectrometer (QMS). The system consists of a sample crucible located in a controlled temperature and atmosphere furnace. The samples are connected to an analytical balance for real time sample weight changes. A gas system is provided to control sample atmosphere and to sweep any off gas species to the QMS for analysis. A skimmer orifice is included in the gas stream to concentrate the sample off gas to improve the QMS measurements. The system is interfaced with a computer for both control and data collection.

The interaction of the TGA/DSC with the QMS is summarized in Figure 3. The QMS provides mass discrimination during the measured sample mass changes (TGA). The DSC provides enthalpy change data for sample specific heat

Figure 2. Schematic of Thermo-Gravimetric Analysis/Differential Scanning Calorimetry/Quadripole Mass Spectrometry System.

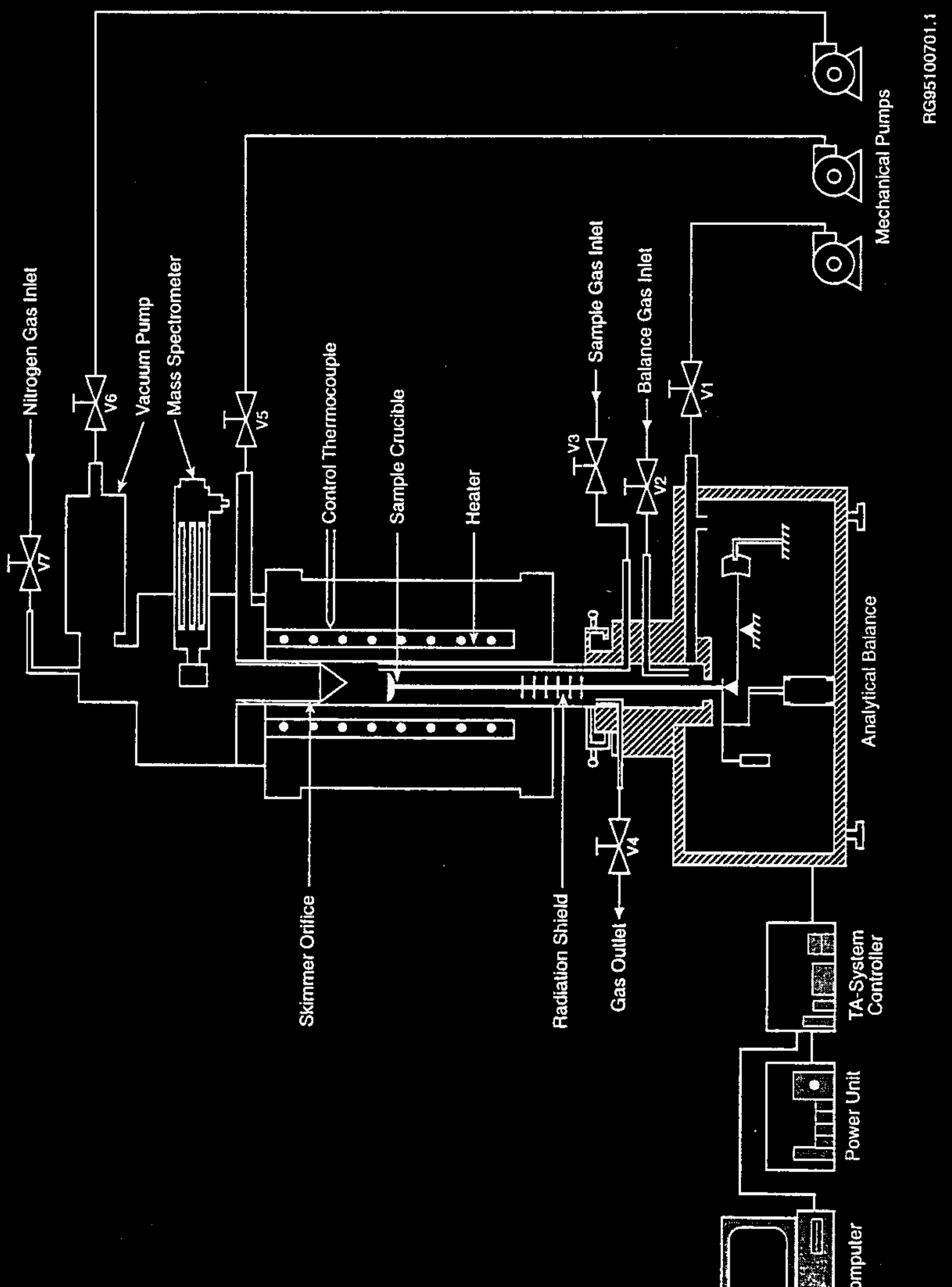
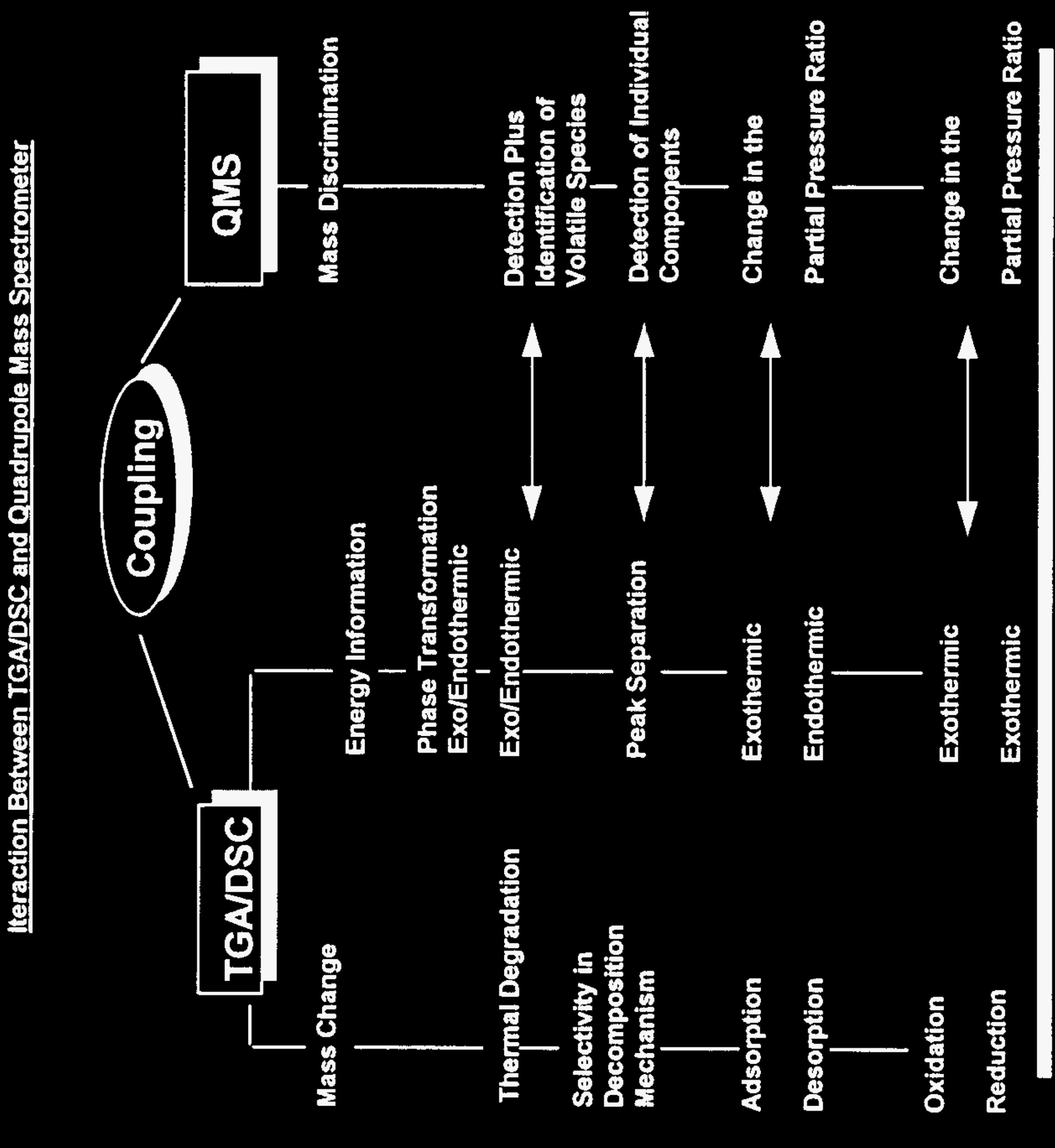


Figure 3. Interaction Between Thermo-Gravimetric Analysis/Differential Scanning Calorimetry, and Quadripole Mass Spectrometry.



determinations. The QMS also provides identification of volatile species as well as changes in partial pressures supporting TGA/DSC measurements of thermal degradation, absorption, desorption, oxidation, and reduction.

The technical specifications for the TGA/DSC/QMS system are summarized in Table 2. The system has the capability of handling fuel samples up to 20 gm which is at least 100 times greater than most TGA systems.

An initial test matrix of 28 samples will be measured for oxidation kinetics, (Table 3) and hydrogen release, and ignition temperatures (Table 4). The hydrogen release and ignition testing is complementary to similar measurements using the controlled temperature and atmosphere furnace in the hot cell (Lawrence 1995a). Detailed test matrices for the drying studies will be developed following an evaluation of the data from these initial 28 samples.

The test matrix for oxidation kinetics (Table 3) includes samples from the first two elements to be examined, including samples from the damaged and undamaged areas of the elements which have and have not been conditioned using the vacuum conditioning process. The overall comparisons and impacts of the measurements are also summarized in Table 3. Two tests under each set of conditions are planned to provide some assurance that the results from a given test will be reproducible.

Samples are obtained from the first three elements removed from the K West Basin for examination (Lawrence 1995a). Small samples are sectioned from the elements mid-point and from the degraded end. Samples will be sectioned further to remove the cladding material and conform to the size and weight limitations of the TGA System (Table 2).

The test matrix for hydrogen release and ignition temperatures (Table 4) is smaller due to the complementary nature of this testing with the hotcell furnace testing (Table 4). These tests include samples from all three elements shipped to the hot cells again with samples from the damaged and undamaged portions. The intent of these measurements and the comparisons to be made are also summarized in Table 4.

Table 2. Technical Specifications for Thermo-Gravimetric Analysis/Differential Scanning Calorimetry, and Quadripole Mass Spectrometry Systems.

Thermo-Gravimetric Analysis (TGA) Unit

1. Sample

   Small samples with weight range from 5 mg to 20 g. Samples will have known geometric surface areas and dimensions. Balance accuracy of 0.1%.

2. Furnace Temperature

   Heating range: room temperature to 2000 °C.

   Measurement accuracy ±1.0 °C.

3. Sample Temperature

   Temperature range: room temperature to 2000 °C.

   Temperature measurement accuracy is ±1.0 °C below 1000 °C and ±5.0 °C above 1000 °C.

4. Furnace Atmosphere

   Gas composition: air saturated with water to inert conditions.

   Gas pressure in furnace zone: vacuum (0.1 Torr) to one atmosphere.

   System can operate in static and/or dynamic environments with gas flow up to 500 cc/min.

5. Temperature Cycle

   Multi-step and isothermal capabilities.

   Heating and cooling rates variable from 0.1 to 100 °C/minute in 0.1 °C/minute steps.

Quadrupole Mass Spectrometer (QMS)

1. Mass Range

   The off gas content and composition within the mass range 1 to 300 amu with a mass resolution of 1 amu can be detected.

2. Temperature of the Off Gas

   System can handle gas temperature up to 2000 °C.

   Vacuum pumping to achieve 10^{-6} Torr in the QMS chamber.

Table 3. Test Matrix for Oxidation Kinetics (K) Testing.

Run	Element/ Sample	Damaged Fuel	Conditioned Sample	Intent			
TGA-1	4378	No	No	K For Undamaged Fuel in 4378	Impact of Conditioning on K for Undamaged Fuel in 4378	Impact of Damage on K for Conditioned and Unconditioned Fuel in 4378	Impacts of Fuel from 4378 and 4366 on K
TGA-2	4378	No	No				
TGA-3	4378	No	Yes	K For Conditioned Undamaged Fuel in 4378			
TGA-4	4378	No	Yes				
TGA-5	4378	Yes	No	K For Damaged Fuel in 4378	Impact of Conditioning on K for Damaged Fuel in 4378		
TGA-6	4378	Yes	No				
TGA-7	4378	Yes	Yes	K For Conditioned Damaged Fuel in 4378			
TGA-8	4378	Yes	Yes				
TGA-9	4366	Yes	No	K For Damaged Fuel in 4366	Impact of Conditioning on K for Damaged Fuel in 4366	Impact of Damage on K for Conditioned and Unconditioned Fuel in 4366	
TGA-10	4366	Yes	No				
TGA-11	4366	Yes	Yes	K For Conditioned Damaged Fuel in 4366			
TGA-12	4366	Yes	Yes				
TGA-13	4366	No	No	K For Undamaged Fuel in 4366	Impact of Conditioning on K for Undamaged Fuel in 4366		
TGA-14	4366	No	No				
TGA-15	4366	No	Yes	K For Conditioned Undamaged Fuel in 4366			
TGA-16	4366	No	Yes				

Table 4. Test Matrix for Hydrogen Release (H) and Ignition Temperature (T_I) Testing.

Run	Test Objective	Element/ Sample	Damaged Fuel	Intent		
TGA-17	H	4378	No	Impact of Damage on H for Fuel in 4378		Impact of Fuel From 4378 4366 and 1990 On H For Undamaged Fuel
TGA-18	H	4378	Yes			
TGA-19	H	4366	No	H For Undamaged Fuel in 4366	Impact of Damage on H for Fuel From 4366	
TGA-20	H	4366	No			
TGA-21	H	4366	Yes	H For Damaged Fuel in 4366		
TGA-22	H	4366	Yes			
TGA-23	H	1990	No	H For Fuel from 1990		
TGA-24	H	1990	No			
TGA-25	T_I	4378	No	Effect of Damage on T_I for Fuel in 4378	Impact of Fuel From 4378 and 4366 on T_I	
TGA-26	T_I	4378	Yes			
TGA-27	T_I	4366	No	Effect of Damage on T_I for Fuel in 4366		
TGA-28	T_I	4366	Yes			

5.0 DATA QUALITY OBJECTIVE STEP 4: DECISION BOUNDARIES

The TGA examinations in this DQO will be restricted to the fuel samples from the initial shipment from the K West Basin. Test parameters will in general be similar to the IPS 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. The test parameters for the TGA will explore conditions reported in the literature to provide a comparison of measured values for the irradiated fuel with literature values. The data developed will be used in establishing a decision rule for conditioning when the product criteria is determined.

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 examinations in a similar manner to the initial examination of the K West fuels (Lawrence 1995a, 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 IPS proposed process and corresponding limits on the decision error.

7.0 DATA QUALITY OBJECTIVE STEP 7: OPTIMIZE

The examinations will be conducted 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 as well as the TGA testing. Information gained from the materials responses during the first series of tests 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 conditions.

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