

DEC 11 1996

Att 37

## ENGINEERING DATA TRANSMITTAL

Page 1 of 1

1. EDT

617440

2. To: (Receiving Organization) <b>Distribution</b>	3. From: (Originating Organization) <b>Spent Nuclear Fuel Evaluations</b>	4. Related EDT No.: <b>N/A</b>
5. Proj./Prog./Dept./Div.: <b>Spent Nuclear Fuels Project</b>	6. Cog. Engr.: <b>B. J. Makenas</b>	7. Purchase Order No.: <b>N/A</b>
8. Originator Remarks: <b>For Release</b>		9. Equip./Component No.: <b>N/A</b>
		10. System/Bldg./Facility: <b>N/A</b>
11. Receiver Remarks:		12. Major Assm. Dwg. No.: <b>N/A</b>
		13. Permit/Permit Application No.: <b>N/A</b>
		14. Required Response Date: <b>N/A</b>

15. DATA TRANSMITTED					(F)	(G)	(H)	(I)
(A) Item No.	(B) Document/Drawing No.	(C) Sheet No.	(D) Rev. No.	(E) Title or Description of Data Transmitted	Approval Designator	Reason for Transmittal	Originator Disposition	Receiver Disposition
1	WHC-SD-SNF-DQO-012		0	DATA QUALITY OBJECTIVES FOR K WEST BASIN CANISTER SLUDGE SAMPLING	N/A	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	4. Review			1. Approved	4. Reviewed no/comment				
		2. Release	5. Post-Review			2. Approved w/comment	5. Reviewed w/comment				
		3. Information	6. Dist. (Receipt Acknow. Required)			3. Disapproved w/comment	6. Receipt acknowledged				
17. SIGNATURE/DISTRIBUTION (See Approval Designator for required signatures)								(G)	(H)		
Reason	Disp.	(J) Name	(K) Signature	(L) Date	(M) MSIN	(J) Name	(K) Signature	(L) Date	(M) MSIN	Reason	Disp.
1	1	Cog.Eng. B.J. Makenas	<i>B.J. Makenas</i>	12/11/96	HO-40	Distribution Attached				2	
1	1	Cog. Mgr. R.P. Omberg	<i>R.P. Omberg</i>	12/11/96	HO-40						
		QA									
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18. <i>B.J. Makenas</i> B.J. Makenas Signature of EDT Originator		19. <i>12/11/96</i> Date Authorized Representative for Receiving Organization		20. <i>R.P. Omberg</i> R.P. Omberg Cognizant Manager Date <i>12/11/96</i>		21. DOE APPROVAL (if required) Ctrl. No. <input type="checkbox"/> Approved <input type="checkbox"/> Approved w/comments <input type="checkbox"/> Disapproved w/comments					

## DATA QUALITY OBJECTIVES FOR K WEST BASIN CANISTER SLUDGE SAMPLING

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

EDT/ECN: 617440 UC: 2070  
Org Code: 2T650 Charge Code: LB028  
B&R Code: EW 135040 Total Pages: 34 *ew 12-11-96*

Key Words: K Basin, Sludge, N Reactor Fuel

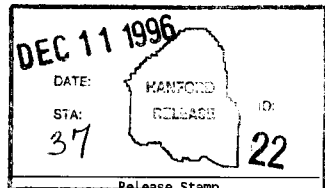
**Abstract:** Data Quality Objectives have been developed for a limited campaign of sampling K Basin canister sludge. Specifically, samples will be taken from the sealed K West Basin fuel canisters. Characterization of the sludge in these canisters will address the needs of fuel retrieval which are to collect and transport sludge which is currently in the canisters. Data will be gathered on physical properties (such as viscosity, particle size, density, etc.) as well as on chemical and radionuclide constituents and radiation levels of sludge. The primary emphasis will be on determining radionuclide concentrations to be deposited on Ion Exchange Modules (IXMs) during canister opening and fuel retrieval. The data will also be useful in determining whether K West Basin sludge meets the waste acceptance criteria for Hanford waste tanks as a backup disposal concept and these data will also supply information on the properties of sludge material which will accompany fuel elements in the Multi-Canister Overpacks (MCOs) as envisioned in the Integrated Process Strategy (IPS).

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*Chris Mullenham*  
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*12-11-96*  
Date



**Approved for Public Release**

**DATA QUALITY OBJECTIVES FOR K WEST BASIN CANISTER SLUDGE SAMPLING**

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Duke Engineering & Services Hanford, Inc.

December 1996

Document Title: DATA QUALITY OBJECTIVES FOR K WEST BASIN  
CANISTER SLUDGE SAMPLING

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## EXECUTIVE SUMMARY

Data Quality Objectives have been developed for a limited campaign of sampling K Basin canister sludge. Specifically, samples will be taken from the sealed K West Basin fuel canisters. Characterization of the sludge in these canisters will address the needs of fuel retrieval which are to collect and transport (to solid waste disposal or TWRS) sludge which is currently in the canisters. Data will be gathered on physical properties (such as viscosity, particle size, density, etc.) as well as on chemical and radionuclide constituents and radiation levels of sludge. The primary emphasis will be on determining radionuclide concentrations to be deposited on Ion Exchange Modules (IXMs) during canister opening and fuel retrieval. This information will also be used in determining whether K Basin sludge meets the waste acceptance criteria for the Hanford waste tanks as a backup disposal concept.

These data will also supply information on the properties of sludge material which will accompany fuel elements in the Multi-Canister Overpacks (MCOs) as envisioned in the Integrated Process Strategy (IPS). Furthermore drying studies will be performed for canister sludge to address residual water issues specific to the IPS.

## CONTENTS

1.0	INTRODUCTION . . . . .	5
2.0	DATA QUALITY OBJECTIVES PROCESS . . . . .	7
2.1	STATE THE PROBLEM AND SUMMARIZE PREVIOUS RESULTS . . . . .	7
2.2	IDENTIFY THE DECISIONS . . . . .	11
2.3	INPUTS TO THE DECISIONS . . . . .	12
2.3.1	Measurements of the Amount of Canister Sludge . . . . .	13
2.3.2	Measurements Related to Water Clarity . . . . .	13
2.3.3	Chemistry Measurements in Support of Solid Waste Disposal . . . . .	14
2.3.4	Measurements of Hydrogen Buildup . . . . .	14
2.3.5	Properties to be Measured in Support of Sludge Acceptance by Tank Waste Remediation System . . . . .	14
2.3.6	Properties to be Measured in Support of the Transportation of Sludge to Waste Tanks . . . . .	18
2.3.7	Properties to be Measured in Support of Fuel Storage . . . . .	18
2.4	BOUNDARIES OF THE STUDY . . . . .	18
2.4.1	Equipment and Sampling Procedure . . . . .	19
2.4.2	Sampling Strategy . . . . .	19
2.5	DECISION RULES . . . . .	21
2.5.1	Volume of Sludge . . . . .	21
2.5.2	Water Clarity . . . . .	21
2.5.3	Chemistry of K West Sludge . . . . .	21
2.5.4	Hydrogen Buildup in Ion Exchange Modules . . . . .	21
2.5.5	Acceptance by Hanford Waste Tanks . . . . .	22
2.5.6	Transportation to Tank Waste Remediation System . . . . .	22
2.5.7	Drying Residual Sludge for Multi-Canister Overpack Design Confirmation . . . . .	22
2.6	DECISION ERRORS . . . . .	22
2.6.1	Programmatic Errors . . . . .	23
2.6.2	Errors in Sampling and Analysis . . . . .	23
2.7	OPTIMIZATION . . . . .	23
3.0	REFERENCES . . . . .	25
APPENDIX A	LIST OF STAKEHOLDERS . . . . .	29
APPENDIX B	RECOMMENDED RECIPE FOR SLUDGE THERMO-GRAVIMETRIC ANALYSIS IN SUPPORT OF FUEL STORAGE . . . . .	31

**LIST OF FIGURES**

1. K West Canister Containing Corroded Fuel . . . . .	8
2. K East Canister Sludge As-Settled in the Laboratory . . . . .	9
3. Schematic of Canister Sludge Sampler . . . . .	20

**LIST OF TABLES**

1. List of Physical, Chemical, and Radionuclide Properties to be Measured for K East Basin Canister Sludge . . . . .	15
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## DATA QUALITY OBJECTIVES FOR K WEST BASIN CANISTER SLUDGE SAMPLING

### 1.0 INTRODUCTION

This document defines the data quality objectives (DQOs) for a sludge sampling campaign which encompasses the spent fuel storage canisters in the 105-K West Basin. The two K Basins are water-filled concrete pools which contain 2,100 tons of N Reactor metal fuel elements stored in aluminum or stainless steel canisters. Associated with this Spent Nuclear Fuel (SNF) is an accumulation of particulate layered material which is generally called sludge. Sludge is found on the basin floors, in canisters, and in the basin pits which are used for miscellaneous tasks such as cask handling. In fact, several different types of sludge exist depending on which basin, canister type, or pit location that the particular sludge is found. Each type of sludge is a unique nonhomogeneous mixture possibly containing corroded fuel, debris such as windblown sand or insects, rack and canister corrosion products, and/or fission products (Johnson 1995).

All of the various sludges will need to be transported away from the K Basins and disposed of as envisioned in the Integrated Process Strategy for Fuel and Sludge Disposition documents (Fulton 1994; WHC 1995; Alderman 1995). Different types of sludge however may have markedly different disposal paths. This document will address only the characterization strategy for one type of sludge: that found inside of the K West Basin fuel canisters. It should be considered a supplement to the DQOs for K East floor and canister sludge (Makenas 1995a; Makenas 1996a) in that it seeks to ascertain those characteristics of canister sludge which are unique with respect to those already determined for K East Basin floor and canister sludge.

It is recognized that, due to the accelerated nature of the SNF project, equipment design for fuel and sludge removal is proceeding in parallel with characterization and thus, that the data acquired may lead to changes in existing design assumptions. The DQO methodology followed in this document is that defined in the K Basin Project DQO Strategy Document (Lawrence 1994) with the various standard DQO questions addressed in sequence in the following discussions. This Reference Strategy Document is based on Environmental Protection Agency (EPA) guidance (EPA 1994) but it notes the parts of the process which must be modified for a project, such as SNF, which seeks to determine bounding conditions for design and transportation alternatives. A list of stakeholders in this effort is given in Appendix A.



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## 2.0 DATA QUALITY OBJECTIVES PROCESS

The following seven major sections, 2.1 to 2.7, are the seven steps in this DQO process.

### 2.1 STATE THE PROBLEM AND SUMMARIZE PREVIOUS RESULTS

Video taped examinations of three K West canisters (Makenas 1995b) have documented the presence of sludge in canisters. All of the K West canisters are sealed and many contain broken fuel and corrosion product. This means that fuel corrosion products (uranium oxide and fission products mostly) found in canisters have not generally escaped to the surrounding basin. Figure 1 shows an example of a canister with corroded fuel in K West Basin (circa 1995) and Figure 2 an example of settled sludge recently retrieved from the non-sealed K East Basin canister. The problems to be addressed by the current DQO are:

1. During the complete removal of fuel from K Basins, when the majority of the K West canisters are opened, sludge will be diverted to pass through the water treatment system which includes Ion Exchange Modules (IXMs). Under current plans, the IXMs will remove some fraction of the sludge from the water stream and return the remainder to the basin floor. The first problem is then to determine the amount of sludge in K West canisters. This piece of information is key to estimating the total number of IXMs that will be necessary to replace during processing of all of the K West Basin fuel. The necessity to add prefiltration to the IXM line will also be evaluated based on the estimated total volume of sludge.
2. Since some sludge will pass through the IXMs and back to the basin, physical properties of the sludge will need to be determined to evaluate the effect of the nontrapped sludge on basin operations. Especially important here are properties which affect sludge settling and therefore water clarity.
3. Currently, during routine operations, the amount of material trapped in a given IXM is monitored by comparing the composition of inlet and outlet flow and assuming that the difference is left behind in the IXM. Used IXMs are sent to the solid waste group for burial. IXM replacement is managed to avoid amounts of trapped radionuclides that would cause IXMs to be classified as transuranic (TRU) waste. Metals which would precipitate a hazardous waste designation for IXMs were not found in early baseline analyses and are presumed absent from current modules. Canister sludge may add new chemical and radiochemical components to IXM input or at least alter the ratio of species. Since basin operations must document changes in assumed characteristics, the composition of sludge must be ascertained. Similar arguments apply to any filter disposed as part of the same effort.

Figure 1. K West Canister Containing Corroded Fuel as Seen  
During First K West Fuel Retrieval Campaign.



Figure 2. K East Canister Sludge As-Settled in the Laboratory.



4. Water reaction with uranium metal fuel has potential to generate hydrogen gas as the fuel oxidizes. Buildup of hydrogen in an IXM should be avoided to avoid the formation of flammable gas mixtures. If canister sludge is to be trapped in an IXM, then the potential rate of hydrogen generation by sludge will need to be known so that appropriate mitigation can be applied. The hydrogen generating species (hydride, uranium, metal etc.) should also be identified.
5. A backup option for disposal of K West canister sludge is the shipment of sludge to Double-Shelled tanks of the Hanford Tank Waste Remediation System (TWRS). To obtain agreement to dispose of sludge at TWRS requires the completion of Waste Stream Profile sheets similar to those submitted or in process for K East Basin floor, pit, and canister sludges. Such an option could be activated if sludge is backflushed from K West filters which might precede the IXM entry point. Completion of the profile sheets will require determination of the chemical, radiochemical, and physical properties of the sludge. The information would be used by TWRS personnel to ascertain the acceptability of mixing K West canister sludge with tank waste, the issues involved in sludge handling and the way in which sludge fits within the environmental permitting framework of the waste tanks.
6. K Basin sludge will not utilize the usual TWRS pipelines or receiving station. Physical properties of canister sludge must be known to adequately design a transportation and processing system which must precede deposition of sludge in the waste tanks.
7. The final problem to be addressed by canister sludge sampling is "What is the influence of residual sludge on the performance of a fuel laden Multi-Canister Overpack (MCO) during processing and storage?" N Reactor fuel elements will be placed in MCOs possibly after reracking of elements and/or desludging operations. Reracking will involve removing elements from their current canisters and moving them to a closer packing array in an MCO. Desludging will mean removal of as much sludge as possible from elements before they are placed in an MCO. Desludging methods are not completely defined but may consist of brushing, water-lancing, etc. In any event there will be a specified maximum amount of residual sludge permissible in an MCO. The residual sludge will be subject to the same drying and conditioning cycles as the fuel and will be a source of water. Water that remains after high temperature drying may in turn be subject to hydrolysis which will liberate hydrogen and oxygen and pressurize the MCO to some extent. The amount of water bound to the sludge, the temperatures at which the water is liberated, and the kinetics of the liberation are all of concern to the designers of the MCO and drying cycles. Such data will help to define or confirm the temperatures for hot vacuum drying (HVD), the time at temperature, and the maximum permissible residual amount of sludge that can be left in an MCO after HVD. Similarly knowing the amount of water present in an MCO after cold vacuum drying (CVD) defines the maximum heat contribution from water-uranium reactions during staging between drying cycles.

Early data on K Basin non-canister sludges have been summarized in a report (Baker 1994). More recent characterization data for both K East Weasel Pit and floor sludge are summarized in (Makenas 1996c) and given in more detail in (Miller 1995; Silvers 1995). These samples provide baseline radionuclide, chemical, and physical property assessments but do not include samples from canisters. To date no sampling campaigns specifically aimed at K West canister sludge have been conducted. Some data on K East canister sludge will be forthcoming when the analysis of samples currently in process is completed (Welsh 1996a). Canister sludge is expected to contain significantly higher concentrations of fuel than floor sludge.

In summary, the values for various physical and radiological properties of sludge must be known in order to successfully retrieve and transport sludge. Various chemical properties, in addition to the physical and radiological characteristics, must be known to assure acceptance of sludge by at least one approved waste receiver on the Hanford site. Drying characteristics of sludge must be known to support MCO design and fuel drying activities. Since these data will be used to aid several different design efforts (all of which are proceeding in parallel) the various questions are given equal priority. The characterization data base as it currently exists is inadequate to allow near term sludge disposition activities to go to completion as currently envisioned because the volume and influence of canister sludge are not known.

## 2.2 IDENTIFY THE DECISIONS

The decisions which must be made by the SNF project for sludge retrieval/disposal and for sludge accompanying fuel and which are partially addressed by the current characterization effort are listed below. The decisions are not interdependent and their resolutions should be pursued in parallel.

1. How many IXMs will need to be replaced during the opening of all of the K West canisters and during fuel cleaning, sorting, and MCO loading operations? Will filters need to be mounted upstream of the IXMs to catch a fraction of the canister sludge and thus decrease the number of needed IXMs and the TRU content in each? The scope of the characterization effort will be to specifically answer the question "what is the volume of sludge?"
2. Will current time/motion studies need to be modified to add time for canister sludge to settle between operations during canister handling? Alternatively, will additional filtering be required to remove particles not trapped in the IXM assembly? Characterization will specifically answer the question "what are the physical properties of sludge?"
3. Are assumptions on the minimal hazardous waste (RCRA metals) content of IXMs valid in light of the anticipated canister sludge content of spent IXMs? Are operational/disposal assumptions on the isotopic

content and isotopic ratios of captured material still valid in light of canister sludge radionuclide accumulation in IXMs? Characterization will specifically answer the question "what is the composition of the sludge?"

4. At what rate is hydrogen evolved from canister sludge when the sludge comes in contact with basin water and does this evolution exceed the rate at which an IXM can dissipate the gas and prevent buildup? What hydrogen generating species are present in canister sludge? Characterization will determine these species and give rates of hydrogen evolution in controlled laboratory situations.
5. Does K West canister sludge meet established criteria for storage in the Hanford waste tanks? Information on the radiological, chemical, and physical character of sludge must be transmitted to TWRS Management in order that a decision on the sludge acceptability can be made if this option is exercised. The methodology of the decision on acceptability is laid out in reference (Fowler 1995a). Characterization will furnish the physical and chemical properties of the sludge.
6. What are the correct pore sizes for filters and hydrocyclones? What is the propensity for pipeline plugging? How easily can sludge be resuspended after settling in a shipping container? Are there additional radionuclides present which are not of interest to TWRS and solid waste but which must be known for transport approval? Characterization will provide physical properties and composition data.
7. What actions, if any, must be taken during drying, conditioning, and storage of fuel in MCOs as a result of sludge which accompanies fuel? Central to this question is whether sludge will evolve significant water at elevated temperature and how long, at a given temperature, sludge must be held to induce divestiture of the bound water. Toxic constituents in the sludge may also affect the environmental permitting of MCOs and Canister Storage Building (CSB). Characterization will provide drying data for, and composition of sludge.

## 2.3 INPUTS TO THE DECISIONS

Information to make the above decisions will be acquired through in-situ measurement and sampling of sludge from the K West Basin fuel canisters. With the exception of sludge depth in canisters, values for various properties needed for the above decisions will be obtained through shipment of the samples to laboratories with demonstrated capabilities for the needed analyses. Data values collected for sludge will be compared against the limits and assumptions given in References Willis 1993 (for Solid Waste), Fowler 1995a (for TWRS), and SNF 1996 (for MCO pressurization). Comparison will also be made to as yet unpublished design documentation. New acceptance criteria for sludges are also expected to be published by TWRS in the near future.

The sludge obtained may settle into two or more layers after retrieval from the Basin. In general, analyses will be performed on homogenized samples. However, analyses of material from each layer will be conducted separately for a limited number of the samples, and properties will be determined for each layer as well as for homogenized samples. After specific canisters are chosen (Makenas 1996d), a sample analysis plan (Welsh 1996b) will specify how many samples are to be separated into layers. The choice of which samples will be analyzed in layers will be made through a letter of instruction based on sludge depth (i.e., sample size) in a particular canister and uniqueness of a particular canister. This choice may be subsequently modified based on unique color and texture of layers observed during laboratory settling studies.

Although samples will not be taken for RCRA waste designation, characterization of the general base chemistry of sludge hazardous constituents is considered good practice. Information on sludge radionuclide content may also support air permit applications for various processes connected with fuel retrieval and processing although initial applications will be filed before these data are available. Thus, some general chemistry analyses will be performed in the current effort in addition to those analyses specifically addressed in the following more focussed discussions. Target accuracy and precision will be specified in the Sampling Analysis Plan (SAP) (Welsh 1996b) to be published as an SD document. Routine accuracy and precision values available through existing laboratory methods are acceptable for this study. The work will be performed using procedures which follow the RCRA technology basis and which are designed using RCRA procedures as a starting point, but unique handling difficulties, sample size constraints, and time constraints for handling radioactive material are recognized.

### **2.3.1 Measurements of the Amount of Canister Sludge**

Sludge content of selected canisters has been measured initially in-basin by ultrasonic measurement of sludge depth. These measurements are accurate to 0.05 in. but can be obscured by fuel and canister seal debris. Since the canister and associated fuel element dimensions are known, a volume of sludge per canister barrel will be derived. Selected canisters will be reopened to retrieve sludge samples. Care must be exercised not to disturb sludge when the canisters are opened (at a minimum canisters must be flooded to eliminate gas bubbles prior to opening). After sludge samples have been secured, laboratories will measure sludge density (in the as-settled, as-centrifuged, and dry conditions) to yield a calculated mass of sludge per canister.

### **2.3.2 Measurements Related to Water Clarity**

The initial data will be video taped observations of sludge settling and water clarity as canisters are opened and fuel elements (Lawrence 1996) are moved. This will be supplemented by settling rate data obtained in the laboratory. Finally laboratory particle size determinations will be made since the efficiency of particulate removal by IXMs varies with particle size.



### 2.3.3 Chemistry Measurements in Support of Solid Waste Disposal

Hazardous metals and radionuclides of interest for solid waste disposal of canister sludge are incorporated in Table 1. Analysis for these constituents shall be performed using procedures that meet the accuracies specified in the SAP for K West canister sludge (Welsh 1996b). Table 1 is a combination of those analytes necessary for solid waste acceptance (Willis 1993) and those necessary for waste tank acceptance discussed in Section 2.3.4.

### 2.3.4 Measurements of Hydrogen Buildup

Hydrogen generation and buildup were identified as an issue for sludge filled IXMs. Shipping containers used to transport sludge from K Basins to laboratories shall be sampled for hydrogen gas buildup prior to partitioning and analysis of the sludge. Volume of recovered gas shall be reported. If bubbles are observed in the sludge after transfer of sludge from shipping to laboratory containers then likewise gas shall be collected in such a manner as to give gas generation per unit time per milliliter of sludge. Similar measurements in the past have been accurate to  $\pm 50\%$ .

### 2.3.5 Properties to be Measured in Support of Sludge Acceptance by Tank Waste Remediation System

Acceptance of sludge by TWRS is directly dependent on the review by East Tank Farms Plant Engineering of sludge properties to be listed on the waste stream profile sheet shown in references (Fowler 1995b; Mulkey 1995; Makenas 1995a). Such a process is in place to ensure that the waste will not (1) react extensively with existing tank waste, or (2) be of a composition outside of the current range of permitted contents (Fowler 1995a; Hunt 1995). Furthermore, the sludge must remain in a transportable state while residing in the tanks to facilitate final disposition of the tank waste. Those areas where knowledge is lacking (i.e., where fuel canister sludge can have different characteristics than those attributed to floor sludge) lead to the data needs listed in Table 1.

It was assumed for this DQO document that problems associated with using the existing waste receiving equipment at the Hanford tank farms will be circumvented by pumping sludge directly from its transportation vehicle or package to the designated tank. Thus, properties which could lead to plugging of lines from the receiving station to the chosen tank are not considered a critical issue. It is expected that one critical piece of data from the acceptance point of view will be the pyrophoricity of sludge which will be a function of the amount of unreacted (unoxidized) metallic uranium or unreacted uranium hydride versus the amount of more benign uranium oxide. The presence of these phases can be established through X-Ray diffraction. Analysis of floor sludge (Silvers 1995) did not find these pyrophoric materials in K East floor or weasel pit sludge but more fuel and active corrosion exist INSIDE of canisters. Recent studies using K East canister sludge have reported a propensity for K East canister sludge to bubble hydrogen but direct confirmation of hydride or metal content is lacking (Bredt 1996).

Table 1. List of Physical, Chemical, and Radionuclide Properties to be Measured for K East Basin Canister Sludge.

Property	Task Supported	Applicable Report Section	Notes and Limitations
<b>1. Physical Properties</b>			
Particle size	Transportation, water clarity, tanks, and volume of sludge	2.3.1, 2.3.2, 2.3.5, 2.3.6	
Particle shape	Transportation, water clarity, tanks, and volume of sludge	2.3.2, 2.3.1, 2.3.5, 2.3.6	
Particle density dry and wet	Tanks, transportation, and water clarity	2.3.2, 2.3.5, 2.3.6	Percent solids for tank transfer will depend on amount of water added or subtracted during retrieval/processing.
Viscosity	Transportation and tanks	2.3.5, 2.3.6	
Settling rate	Transportation, tanks, and water clarity	2.3.2, 2.3.5, 2.3.6	
Endothermic/exothermic reactions	Tanks	2.3.5	DSC (calorimetry). Net exothermic minus endothermic reactions.
Pyrophoricity	Tanks	2.3.5	Satisfied by a check for metallic Zr, metallic U, and U hydride through X-Ray diffraction.
Percent water and kinetics of water liberation	Tanks, MCO storage and conditioning	2.3.5, 2.3.7	TGA including annotated thermogram. Run per standard THRS procedure and per Appendix B.
Zeta potential	Transportation	2.3.6	Aids in simulant design for equipment.
Fe and Al hydrates	MCO storage and conditioning	2.3.7	X-Ray diffraction
<b>2. Radionuclides</b>			
$^{235}\text{U}$ , $^{238}\text{U}$ , $^{236}\text{U}$ , and $^{238}\text{U}$	Tanks, air permits, and solid waste	2.3, 2.3.3, 2.3.5	
$^{239}\text{Pu}$ , ( $^{239}\text{Pu}$ , $^{240}\text{Pu}$ , and $^{241}\text{Pu}$ )	Tanks, air permits, transportation, and solid waste	2.3, 2.3.3, 2.3.5, 2.3.6	$^{239}\text{Pu}$ for transportation PDC <sup>(2)</sup>
$^{241}\text{Am}$ and $^{241}\text{Cm}$	Tanks, air permits, transportation, and solid waste	2.3, 2.3.3, 2.3.5, 2.3.6	$^{241}\text{Cm}$ for transportation PDC <sup>(2)</sup>
$^{237}\text{Np}$	Tanks, air permits, and solid waste	2.3, 2.3.3, 2.3.5	
$^{137}\text{Cs}$ and $^{134}\text{Cs}$	Tanks, air permits, and solid waste	2.3, 2.3.3, 2.3.5	
$^{152}\text{Eu}$ , $^{154}\text{Eu}$ , and $^{155}\text{Eu}$	Tanks and solid waste	2.3.3, 2.3.5	Eu is another measure of Pu content for sludge accountability.
$^{90}\text{Sr}$	Tanks and solid waste	2.3.3, 2.3.5	

Table 1. List of Physical, Chemical, and Radionuclide Properties to be Measured for K East Basin Canister Sludge. (Continued)

Property	Task Supported	Applicable Report Section	Notes and Limitations
$^{210}\text{Bi}$ , $^{208}\text{Tl}$ , $^{135}\text{Sb}$ , $^{106}\text{Ru/Rh}$ , $^{14}\text{Pr/Ce}$ , $^{93}\text{Nb}$ , $^{125}\text{Ra}$ , and $^{99}\text{Tc}$	Transportation	2.3.6	For transportation POC <sup>(2)</sup>
<b>3. Radiological Properties</b>			
Total $\gamma/\beta$	All, except water clarity	2.3.1--2.3.7	Radiation levels may be used in radiolysis calculations.
Total $\alpha$	All, except water clarity	2.3.1--2.3.7	
<b>4. Chemical Constituents</b>			
Carbonate	Tanks	2.3.5	Measurement of total inorganic carbon is adequate.
Chloride	Tanks	2.3.5	
Total carbon	Tanks	2.3.5	
TOC	General chemistry, MCO pressurization, and tanks	2.3.2, 2.3.5, 2.3.7	
Fluoride	Tanks	2.3.5	
Hydroxide	Tanks	2.3.5	By caustic demand.
Nitrate	Tanks and solid waste	2.3.3, 2.3.5	
Nitrite	Tanks	2.3.5	
Phosphate	Tanks and solid waste	2.3.3, 2.3.5	
Sulphate	Tanks and solid waste	2.3.3, 2.3.5	
Oxalate, formate, and acetate	Tanks	2.3.5	
Polychlorinated biphenyls	Tanks, air permitting	2.3, 2.3.5	Found in floor sludge Reference (Miller 1995). List other tentatively identified semivolatile organics compounds. Special attention should be paid to the number of solvent contacts as specified in the SAP.
U	Tanks and general information	2.3, 2.3.5	
Na	Tanks and solid waste	2.3.3, 2.3.5	
Fe	Tanks	2.3.5	
Al	Tanks	2.3.5	
Zn	General chemistry of hazardous materials, tanks, and solid waste	2.3, 2.3.3, 2.3.5	

Table 1. List of Physical, Chemical, and Radionuclide Properties to be Measured for K East Basin Canister Sludge. (Continued)

Property	Task Supported	Applicable Report Section	Notes and Limitations
Pb	General chemistry of hazardous materials, tanks, and solid waste	2.3, 2.3.3, 2.3.5	
Ag	General chemistry of hazardous materials, tanks, and solid waste	2.3, 2.3.3, 2.3.5	
Tl	General chemistry of hazardous materials, tanks, and solid waste	2.3, 2.3.3, 2.3.5	
Se	General chemistry of hazardous materials, tanks, and solid waste	2.3, 2.3.3, 2.3.5	
pH	General chemistry of hazardous materials, tanks, and solid waste	2.3, 2.3.3, 2.3.5	
B	General chemistry of hazardous materials, tanks, and solid waste	2.3, 2.3.3, 2.3.5	
Ba	General chemistry of hazardous materials, tanks, and solid waste	2.3, 2.3.3, 2.3.5	
K	Tanks and solid waste	2.3.3, 2.3.5	
Be	General chemistry of hazardous materials, tanks, and solid waste	2.3, 2.3.3, 2.3.5	Fuel brazing material
Ca	General chemistry of hazardous materials, tanks, and solid waste	2.3, 2.3.3, 2.3.5	Found in floor sludge previously
Cr	General chemistry of hazardous materials, tanks, and solid waste	2.3, 2.3.3, 2.3.5	
Cu	General chemistry of hazardous materials, tanks, and solid waste	2.3, 2.3.3, 2.3.5	
Cd	General chemistry of hazardous materials, tanks, and solid waste	2.3, 2.3.3, 2.3.5	
Zr	General chemistry of hazardous materials, tanks, and solid waste	2.3, 2.3.3, 2.3.5	Cladding material
Sand	General chemistry of hazardous materials, and tanks	2.3, 2.3.5	XRD of undissolved residuals
Hf <sup>(1)</sup>	General chemistry	2.3	Clad impurity
Mg	General chemistry	2.3	
Mn	General chemistry	2.3	
Sn <sup>(1)</sup>	General chemistry	2.3	Clad alloy element

(1) Analyze only if routine analysis is available. Extraordinary measures such as purchase of new equipment not required.

(2) These analytes were not addressed in the K East floor sludge campaign.

(3) HPC = package design criteria.

### **2.3.6 Properties to be Measured in Support of the Transportation of Sludge to Waste Tanks**

Various physical properties will need to be known to design simulants to test sludge processing equipment. Specifically the properties to be measured are particle size, particle shape, dry density, and zeta potential.

### **2.3.7 Properties to be Measured in Support of Fuel Storage**

Drying curves will be key to making decisions related to the limits on residual sludge and residual water in an MCO loaded with fuel elements. Thermo-Gravimetric Analysis (TGA) will provide a quantitative measure of temperatures at which bound water is liberated from sludge. To accomplish this, the entire plot of weight loss versus temperature must be reported (not simply total percent water in a sample). Various constituents of sludge such as canister corrosion products and fuel compounds will liberate their water at different temperatures and at different rates. The TGA done as part of Section 2.3.5 probably will not suffice. TGA done for TWRS issues is done according to an established recipe prescribed by tank safety documents. The goal for TWRS is primarily to determine the total water inventory and as such the recipe may not allow sufficient time at a given temperature to resolve water evolution peaks attributable to different chemical species. Appendix B gives a more desirable recipe for TGA to address MCO issues. It is recognized that this may mean that TGA may have to be performed twice on similar subsamples to satisfy the needs of Sections 2.3.5 and 2.3.7. The recipe in Appendix B for K West canister sludge will be changed if the evaluations of K East canister sludge TGA data indicate that significant water liberation occurs in sludge at other discreet temperatures. The letter of instruction for this activity shall provide specific pressures and material quantities for TGA to address water transport effects.

## **2.4 BOUNDARIES OF THE STUDY**

This Sampling Campaign will be done within the manpower availability of K Basin operations staff and within SNF budget constraints. Sampling will be done exclusively from canisters in K West Basin and data is, in general, not expected to indicate the same results as K East Basin canister sludge. Operations personnel will run the sampling equipment and perform those tasks (such as cask loading) needed to accomplish shipment of samples to laboratories. Operations will also need to support the training and readiness review processes for this activity. The work will be performed utilizing practices which conform to ALARA for radiation dosage to workers and performed within the limits of the K Basin Safety Analysis Report.

Modifications to K West Basin South loadout pit are planned which might preclude loading of sludge into casks at this location. Sludge sampling must occur prior to these modifications. By performance agreement with DOE, the completed data package must be issued by September 30, 1997. Sampling shall occur early enough to allow for shipping of sludge to hot cells, settling studies, and at least 110 days for chemical analysis prior to this milestone.

### 2.4.1 Equipment and Sampling Procedure

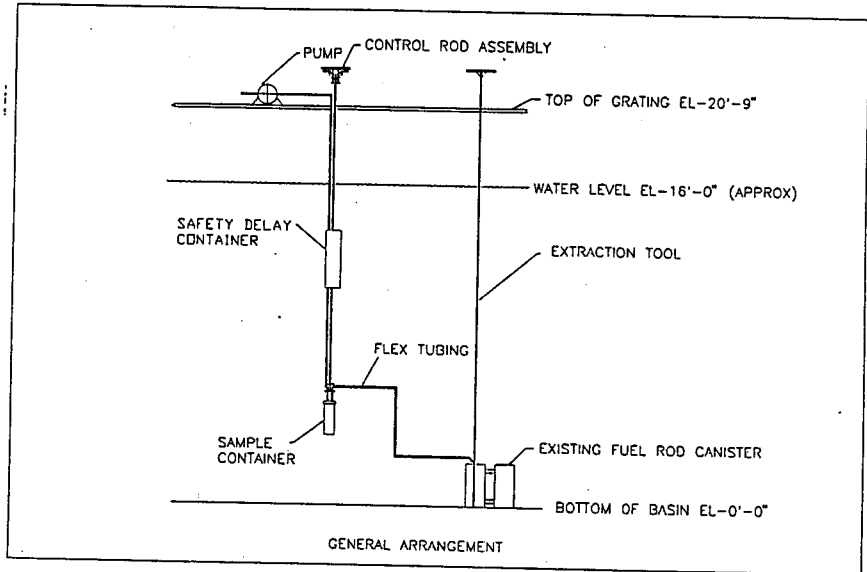
The samples of sludge are to be retrieved using an apparatus whose design criteria are outlined in (Baker 1996). Samples of sludge are to be pumped from the canisters to a point in the water pool below the grating for collection in containers. The basic concept is shown schematically in Figure 3. This apparatus differs from the version of the mechanism that was used to acquire floor sludge samples in that the earlier collection containers remained above the pool and grating. This change was necessitated by expected radiation levels that are higher for canister sludge than for floor sludge. Acceptance testing for the equipment is outlined in reference (Bridges 1996) and essentially consists of demonstrations in the 305 Building pool utilizing a rudimentary sludge simulant. Usage of this equipment to retrieve canister sludge in K East Basin proved quite successful.

Operation of the envisioned sampling equipment in K West Basin will require that a vacuum extraction tube and nozzle be inserted into the sludge between fuel elements prior to sampling and that sludge be vacuumed nearly completely [at least 70% recovery for particles less than 0.64 cm (0.25 in.) in diameter for a volume less than 500 ml] from within a canister barrel containing seven fuel assemblies (as confirmed by acceptance testing done outside of K Basins). Consideration was given to increasing the amount of sludge collected by utilizing a series of filters and thus collecting more sludge and less water. The larger amounts of sludge for large scale drying studies were judged to be uneeded since large amounts of sludge will not be dried in an MCO. Furthermore, possible damage to the sludge during filter packing (and resulting artifact data) was judged to be unacceptable.

### 2.4.2 Sampling Strategy

Samples will be taken from nine canister barrels. During sampling of K East floor sludge a selection of 20 samples was determined (Welsh 1995) to satisfy TWRS required confidence levels given the expected variations from area-to-area in the Basin. Subsequently, for K East canisters, nine samples were determined (Welsh 1996a) to be a sufficient number to allow determination of whether the mean canister sludge differed significantly from floor sludge. Since K West canister-to-canister variations are expected to be less than in K East (less debris in sealed canisters) the same logic should apply. Nine samples will therefore produce enough data to allow legitimate comparison of the mean of previous K East floor sludge and K East canister sludge property measurements with K West canister sludge property measurements. These may or may not be the same canisters as needed to fulfill the objectives for fuel sampling (Lawrence 1996) and specific canister numbers will be specified in the canister choices document (Makenas 1996d). Specific canisters will be chosen through review of canister gas and liquid radionuclide concentrations for 50 canisters (100 barrels) (Makenas 1996e) and a review of video tapes of the K West fuel element and canister survey encompassing 20 canisters (40 barrels). This latter survey includes the ultrasonic measurements of sludge depth. These surveys should indicate candidates which represent greatest sludge depth and radionuclide concentration. Samples will be taken from both aluminum and stainless steel (SS) canisters and samples will include

Figure 3. Schematic of Canister Sludge Sampler.



sludge from canisters with highly damaged fuel if available based on the video survey. Such canisters are assumed to have the potential for sludge with the largest uranium concentrations. The primary consideration will be greatest depth of sludge in a chosen canister.

## **2.5 DECISION RULES**

### **2.5.1 Volume of Sludge**

If the amount of sludge found to be present in sampled K West canisters results in a projected unacceptably number of IXM replacements (as called out in design documents to be published later), then filtration will be added to the K West water treatment scheme. SNF design engineers will use these data in conjunction with other design inputs to arrive at a decision.

### **2.5.2 Water Clarity**

If unfiltered canister sludge presents a clarity problem and if particle size determinations show sludge particles are mostly outside of the efficient range of an IXM, then prefiltration or postfiltration is indicated. Alternatively operations may be managed to allow settling time. The data acquired here will be used in conjunction with video tapes of sludge behavior from previous canister openings and with processing throughput requirements to decide if action is warranted.

### **2.5.3 Chemistry of K West Sludge**

Solid waste burial is the reference case disposal path for K West canister sludge. If the amounts of radionuclides and hazardous metals in the sludge sampled from the nine canisters imply that IXMs will exceed amounts defined in Willis 1993 for low level waste then the IXMs will be classified as TRU or regulated waste and another disposal path must be found. Alternatively K Basin operations will change out IXMs with an increased frequency which precludes exceeding TRU/hazardous waste concentration limits. Sludge may also be caught on filters and sent to TWRS.

### **2.5.4 Hydrogen Buildup in Ion Exchange Modules**

If the rate of hydrogen generation from sludge exceeds the capability of IXMs to vent gas then design changes (such as additional relief holes) will be made to IXMs to allow additional release of gas from areas prone to gas accumulation. The data collected by this sampling program will be compared, by SNF design engineers, to existing IXM venting capabilities and to safety requirements for hydrogen accumulation prior to any design changes.



### **2.5.5 Acceptance by Hanford Waste Tanks**

It is currently envisioned that K West canister sludge will not follow the same disposal path as K East floor sludge (i.e., to the Hanford waste tanks). However, this option is being carried as a contingency. If the properties of K West canister sludge, as defined by this characterization effort, are within the limits imposed by reference (Fowler 1995a) and by TWRS sludge acceptance criteria (to be published) and if the sludge is without pyrophoric materials such as metallic uranium and uranium hydride, then the sludge should be acceptable for storage in the Hanford Waste Tanks. If the fissile or fission product concentrations are higher than those detected previously in floor sludge (Miller 1995 and Makenas 1996c), which is likely true, then an addendum to TWRS Waste Profile sheets for K Basin sludge will be necessary to reflect these higher concentrations if this option is exercised.

### **2.5.6 Transportation to Tank Waste Remediation System**

The data acquired on physical properties will be used primarily to formulate sludge simulants which will in turn be used to design or accept sludge handling equipment. The data acquired here does not, of itself, lead to a final decision on equipment related to transportation. Other inputs such as criticality specifications, throughput requirements, et al., will also be used for the final decisions.

### **2.5.7 Drying Residual Sludge for Multi-Canister Overpack Design Confirmation**

If canister sludge contains significantly more bound water than can be released at temperatures and times specified for the current MCO with related process designs, then either the drying process, the MCO configuration (including the venting/sealing scenario), or the allowable residual sludge volume will need to change. Acquired data will be compared to the most recent version of Reference SNF 1996 and other applicable process design documentation. These data will be used in concert with reaction kinetic calculations, thermal models, and radiolysis data to arrive at the pertinent decisions.

## **2.6 DECISION ERRORS**

Two types of errors can be identified which are related to the canister sludge sampling effort: those that influence the programmatic success of the Fuel and Sludge Disposition Path (i.e., prevent the successful removal of fuel and sludge from the basin to a disposal site in a timely cost-effective manner) and those that are associated with individual sampling and analytical methodologies.

### 2.6.1 Programmatic Errors

If radiochemical and physical properties of K West canister sludge are not further defined (or are defined incorrectly), then the design of sludge processing equipment (IXMs, filters, etc.) will proceed with only the limited data available (that from K East canister sludge). This increases the risk that the amount of one or more K West canister sludge constituents will not be enveloped by the equipment capabilities and operational/regulatory scenario or that the equipment will need to be over designed for all conceivable eventualities with resulting cost increases. If decisions on the acceptability of sludge for burial as solid waste or storage in waste tanks are not made with data that includes high fuel content canister sludge, the risk increases that a decision to deposit sludge as solid waste or in tanks will be countermanded by subsequent analyses of the retrieved and transported sludge (with the resultant waste of resources, time, and effort). Finally, if the drying characteristics of canister sludge are not ascertained, the assumptions made in the design of MCO; with respect to removal of water at specific temperatures will remain unsubstantiated. This may result in an unnecessarily robust or complicated design to contain radiolytic hydrogen from residual water bound to sludge or in a challenge to MCO integrity from pressurization.

### 2.6.2 Errors in Sampling and Analysis

Reference (Fowler 1995c, Revision 1) specifies that criticality, flammability, and energetics properties necessary for waste tank acceptability decisions need to be known to the 90 percent confidence level. Corrosion related data (nitrites, nitrates, etc.) must be known to 80 percent confidence. No other guidance is available from the other users of the data nor has an acceptable programmatic risk been quantified. Since regulatory (RCRA) sampling is not a goal of this current effort, laboratory good-practice standards are judged to be acceptable (Fowler 1995d). An effort is underway by Welsh, et al., to quantify laboratory accuracies for individual analytes which will lead to the required confidence levels and provide valid comparisons to previous floor sludge data. This study will be included in the Sampling Analysis Plan (Welsh 1996b).

## 2.7 OPTIMIZATION

To maximize the amount of sludge collected, canisters with the largest observed amounts of sludge will be sampled (specific canisters to be documented in Makenas 1996d). Visual survey and ultrasonic measurements used in concert with DQOs to make canister choices will be documented in Pitner 1997. This will provide primarily a bounding case since more sludge probably implies more fuel corrosion and thus more radionuclides per canister. If the samples from the K West Basin canisters have an unexpectedly large variation in properties, analysis may dictate the necessity for future

additional sampling since the variation would indicate less certainty that a bounding case had indeed been found. An estimate of the average amount of sludge and radionuclide content in K West Basin canisters could be obtained by comparison of sludge data to the ongoing analyses of liquid from canisters. If a good correlation is obtained between amount and/or composition of sludge versus radionuclide concentration in liquid then the much larger data base of liquid samples (120) could be used to estimate the average sludge characteristics in K West canisters.

If the current sludge characterization effort is successful in adequately bounding the properties of canister sludge in K West Basin then future characterization efforts may concentrate on sludge from remote pits in K East or K West Basins as well as on K West floor sludge. Fuel element samples will also be shipped to hot cells (Lawrence 1996) shortly after sludge. Since some sludge will adhere to fuel elements, residual sludge found in fuel shipping containers or in elements during examinations will be an additional source of material for sludge characterization analysis.

When specific TWRS acceptance criteria are published for sludge, as proposed in (Fulton 1996), it is possible that additional reactivity, mixing, and caustic fusion data needs for sludge may be indicated. Also, if thermal models being developed for the MCO and CSB require properties such as thermal conductivity and specific heat for sludge, which accompanies fuel to the CSB, such tests will be conducted on archive sludge material.

Thermo-Gravimetric Analysis as well as a hot cell furnace (with mass spectrometer and vacuum capability) to test drying and oxidation characteristics of fuel element sections are currently operational or under construction in 325 and 327 Buildings, respectively (Lawrence 1995a, 1995b). They mimic a process which is expected to be utilized to dry and condition all K Basin fuel for long term storage. These furnaces, run in a mode which determines ignition temperature, could add to the expected sludge data. For example, if X-Ray Diffraction (XRD) examinations reveal that some hydride or metal fragments are present in the sludge or if safety considerations dictate additional confirmation of XRD data, then furnace data (i.e., ignition temperatures) could reveal whether the fragments add a pyrophoric character to the sludge. Furthermore, these furnaces can be run in an alternate mode that could ascertain whether this pyrophoric character can be destroyed through controlled additions of oxygen at high temperature. Synergistic effects can also be explored when sludge samples are run at high temperature in intimate contact with fuel specimens.

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## **A P P E N D I X   A**

### **LIST OF STAKEHOLDERS**



## List of Stakeholders for Sludge Sampling

Path Forward for Sludge	C.J. Alderman and F.W. Moore
K Basin Operations	D.W. Siddoway
Safety Analyses	C. Defigh-Price
Fuel Process Engineering	C.R. Miska, L.H. Goldman, R.G. Cowan, and A.T. Kee
QA	D.W. Smith
Cold Vacuum Drying	J.J. Irwin
TWRS Sludge Disposition Interface	K.L. Pearce
222-S Laboratory	G.L. Miller
325 Building Laboratory	J.M. Tingey and K.L. Silvers
Equipment Design	A.E. Bridges and P.J. MacFarlan
Equipment/Operations Interface	R.B. Baker and D.W. Bergmann
DOE/RL	J. Shuen
Characterization Management	R.P. Omberg*
Sample Analysis Plan	T.L. Welsh and R.B. Baker
Fuel Element Recovery	B.S. Carlisle and W.C. Mills
Canister Storage Building Air Permitting	R.F. Hinz and W.L. Willis
K West Basin Water Treatment	K.H. Bergsman
Environmental Permitting	D.J. Watson

\*Key decision maker.

**A P P E N D I X   B**

**RECOMMENDED RECIPE FOR SLUDGE THERMO-GRAVIMETRIC ANALYSIS  
IN SUPPORT OF FUEL STORAGE**

**(Excerpted From Reference Miska 1995)**

**APPENDIX B****RECOMMENDED RECIPE FOR SLUDGE THERMO-GRAVIMETRIC ANALYSIS  
IN SUPPORT OF FUEL STORAGE**

Because of the major impact that canister sludge (oxidized fuel particulates) could potentially have on the amount of water retained with the fuel after both vacuum drying and hot conditioning, the best available information is critical to ensure appropriate criteria is specified for several Spent Fuel Projects. Specifically this information must support proper definition of fuel cleaning criteria, and both Vacuum Drying and Hot Conditioning process conditions and performance.

Canister sludge simulant samples should be dried at 50 °C until the weight is stable before running a TGA. Vacuum should be applied to the TGA apparatus, and the temperature should be slowly ramped\* from 50 °C to 300 °C. Above 300 °C, the off-gas composition should be determined along with the sample weight loss.

Data from previous floor sludge TGAs show that all of the free water did not come off the sample at 100 °C. A relatively large amount of water was removed above 100 °C. How much of this water was free water or bound water is not easy to determine from the previous data. By holding and drying the sample in vacuum or dry flowing gas temperature at 50 °C, all or most of the free water can be removed before going to higher temperature.

The ramp rate of the temperature above 50 °C should be slow enough to reduce mass transport effects in the sample. If the ramp rate is too fast, as in the case of previous floor sludge samples, the mass transport effects in the sample may be limiting and water may be released over a much broader range of temperatures than with a slower ramp rate. Ramping the sample temperature at a higher rate, with appropriate temperature hold points, i.e., 100 °C and then 300 °C, may be an alternative method of determining free water and bound water release data needed.

In addition, monitoring of the sample off-gas composition above 300 °C should be done to determine if all of the sample weight loss is due to the release of water.

Selection and analysis of canister sludge is needed to provide essential data to Hot Conditioning, and other interrelated SNF projects. This data will provide an estimate of the characteristics of oxidized fuel particulates in the feed to Vacuum Drying and Hot Conditioning systems. The test results will also allow prediction of the fuel particulate characteristics after Vacuum Drying and Hot Conditioning.

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\*Rate, pressures, sample size, and hold points will be supplied in a letter of instruction.

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Name	MSIN	Text With All Attach.	Text Only	Attach./ Appendix Only	EDT/ECN Only
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## Rust Federal Services of Hanford, Inc.

S. M. Joyce	T6-04	X
A. G. King	T6-03	X
J. E. Mercado	G1-11	X
G. L. Miller	T6-06	X

## Duke Engineering & Services Hanford, Inc.

C. J. Alderman	R3-48	X
R. B. Baker (2)	H0-40	X
D. W. Bergmann	R3-86	X
L. D. Bruggeman	H0-40	X
B. S. Carlisle	R3-85	X
S. A. Chastain	H0-40	X
R. G. Cowan	R3-86	X
C. Defigh-Price	X3-79	X
D. R. Duncan	R3-86	X
J. R. Fredrickson	R3-86	X
L. H. Goldmann	R3-86	X
S. L. Hecht	H0-40	X
F. G. Hudson	R3-11	X
J. J. Jernberg	X3-72	X
A. T. Kee	R3-86	X
L. A. Lawrence	H0-40	X
P. G. LeRoy	R3-11	X
B. J. Makenas (3)	H0-40	X
C. R. Miska	R3-86	X
R. P. Omberg	H0-40	X
A. L. Pajunen	R3-86	X
K. L. Pearce (2)	R3-48	X
R. W. Rasmussen	R3-86	X
P. K. Shen	H0-40	X
J. A. Swenson	R3-11	X
C. A. Thompson	R3-85	X
D. J. Trimble	H0-40	X
SNF Project Files	R3-11	X
Central Files	A3-88	X