2. To: (Receiving Organization) Distribution		3. From: (Originating Organization) SNF Characterization Project		4. Related EDT No.:	
5. Proj./Prog./Dept./Div.: Spent Nuclear Fuel Project		6. Design Authority/ Design Agent/Cog. Engr.: B. J. Makenas		7. Purchase Order No.: N/A	
8. Originator Remarks:				9. Equip./Component No.: N/A	
				10. System/Bldg./Facility: K Basins/100-K	
11. Receiver Remarks: 11A. Design Baseline Document? [] Yes [X] No				12. Major Assm. Dwg. No.: N/A	
				13. Permit/Permit Application No.: N/A	
				14. Required Response Date: N/A	

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 Design- ator	Reason for Trans- mittal	Origina- tor Dispo- sition	Receiv- er Dispo- sition
1	HNF-SD-SNF-SP-021		0	STRATEGY FOR ANALYSIS OF COATINGS AND SUBSURFACE SLUDGE RECOVERED DURING HOT CELL EXAMINATIONS OF N REACTOR ELEMENTS FROM HANFORD K BASINS		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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STRATEGY FOR ANALYSIS OF COATINGS AND SUBSURFACE SLUDGE RECOVERED DURING HOT CELL EXAMINATIONS OF N REACTOR ELEMENTS FROM HANFORD K BASINS

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U.S. Department of Energy Contract DE-AC06-96RL13200

EDT/ECN: 620793

UC: 2070

Org Code: 2T720

Charge Code: LB027

B&R Code: EW7040000

Total Pages: 43, 18

Key Words: Strategy, Sludge, Coatings, 105-K West, Spent Nuclear Fuel Project, N Reactor

Abstract: Subsurface sludge and/or surface coating material have been collected for four N Reactor fuel elements from K West Basin and one element from K East Basin. It is proposed that examinations of the fairly small volumes of recovered material proceed in order to identify the constituents and their potential impacts on fuel and sludge processing. Lists of potential examination techniques and material allocations are given in this report.

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Release Approval Date

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Document Title:

STRATEGY FOR ANALYSIS OF COATINGS AND SUBSURFACE SLUDGE
RECOVERED DURING HOT CELL EXAMINATIONS OF N REACTOR
ELEMENTS FROM HANFORD K BASINS

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EXECUTIVE SUMMARY

Subsurface sludge and/or surface coating material have been collected for four N Reactor fuel elements from K West Basin and one element from K East Basin. It is proposed that examinations of the fairly small volumes of recovered material proceed in order to identify the constituents and their potential impacts on fuel and sludge processing. Lists of potential examination techniques and material allocations are given in this report.

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**STRATEGY FOR ANALYSIS OF COATINGS AND SUBSURFACE SLUDGE
RECOVERED DURING HOT CELL EXAMINATIONS OF N REACTOR
ELEMENTS FROM HANFORD K BASINS**

1.0 INTRODUCTION

The two most recent campaigns to retrieve and examine N Reactor fuel have resulted in 24 N Reactor outer elements or full assemblies being shipped to hot cells (9 from K East Basin and 15 from K West Basin). Seven of these elements were designated for recovery of coatings from the cladding outer surface. Three of these seven were further designated for peeling of damaged cladding and recovery of fuel sludge found below the cladding surface. The collection and laboratory analysis of coatings from two of these seven elements have been completed and reported previously (Abrefah 1996; Marschman 1997). The retrieval of subsurface sludge and/or coatings from the remaining five elements has recently been accomplished (Pitner 1997a, 1997b, 1997c, 1997d). It is the purpose of this document to specify, in general terms, the particular laboratory analyses to be performed for the coating and subsurface sludge material recovered from these latter five elements. The amounts of material which have been recovered are limited such that priorities must be set based on the needs of the Spent Nuclear Fuel (SNF) Program, the amount of material recovered from specific elements and the amount of material actually required to perform a particular analysis in the laboratory.

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2.0 DRIVERS FOR THE EXAMINATIONS

Examinations of subsurface sludge and cladding coating material are specified in the general examination strategy documents for K West and K East elements (Lawrence 1996; 1997a) as well as in the Data Quality Objectives (DQO) document for the K West elements (Lawrence 1997b). The primary thrust in this vein is the identification of water bearing compounds and the quantification of the difficulty of removing the water during the various SNF processing steps. This comes into play primarily because subsurface sludge and surface coating may accompany fuel into the Multi-Canister Overpacks (MCO) envisioned to hold SNF during interim dry storage. Sludge drying tests to date have been with sludge retrieved from the bottoms of canisters which is in essence a surrogate for subsurface sludge.

In addition to the above, four other objectives can be seen for the examination of the subject material.

1. The need to characterize fuel bearing sludges from a physical standpoint (particle size, density etc.) derives from the water treatment tasks that are associated with cleaning of fuel and canisters during removal of SNF from the Basins. These needs from a canister sludge perspective are discussed in Makenas 1997 but the logic applies equally well to fuel subsurface sludge that may be removed in the fuel washing process.
2. Most of the various kinds of sludges found in the K Basins will, as currently envisioned (Hatch 1997), be subject to a pretreatment process prior to disposal. This process will modify the particle size and chemistry of the sludges. Sludge trapped below the cladding surfaces will, to some extent, find its way into the process stream. It would be advantageous to test the process with samples of the various kinds of actual sludge including that discussed here.
3. The current safety analysis documents in preparation for the Canister Storage Building (CSB) contain various enabling assumptions which must be closed at some point in the review and modification process (Williams 1997). One of these assumptions (007 EA) is the "Subsurface Sludge has the same radionuclide content as the fuel itself." The examinations proposed herein represent a chance to close this assumption. The material collected during the subsurface collection process also represents a source of feed material (larger pieces only) for furnace tests that mimic the processing of an MCO scrap basket.
4. There is no process knowledge basis to indicate that organic compounds such as PCBs are associated with fuel elements. These compounds have to date only been identified in floor sludge and canister sludge. However, the establishment of their absence from element surfaces and interior spaces might be deemed prudent although no statistically valid extension to "all" fuel elements could be developed from our limited sample.

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3.0 AVAILABLE SLUDGE AND COATING MATERIAL

The samples collected from the five elements consists of three types:

1. Coatings: Abrasive pads (accompanied by a small amount of loose particulate) contain coating material from the outer surfaces of cladding from five elements. These pads can be used in some analyses without extracting the coating material from the pad. In other cases loose coating material is available when collection was done with a scraper instead of a pad.
2. Sludge from Cracks: Loose particulate material was collected from element cracks. Often this particulate material is accompanied by large (0.25 to 0.5 in.) pieces of fuel.
3. Sludge from Shipping Containers: Loose particulate material was filtered from the water in the shipping containers (SFECs). This is assumed to be due either to sludge which drained from the element during container loading (in the Basins) or to sludge dislodged or formed during shipment. Filters can be sent directly for analysis of sludge which cannot be extracted.

Table 1 (excerpted from Reference Pitner 1997d) summarizes the elements examined and the amounts of material recovered. Often the majority of the weight of a subsurface sludge sample is made up of a few large chunks and constitutes very little in the way of real sludge (which is by definition less than 0.25 in. in diameter). Also the amount of material recovered from the SFEC is usually significantly greater than that obtained from the element cracks.

Table 1. Samples Recovered During the Examinations.

Element	ID	Description	Gross wt	Tare	Contents
6743U	CS2	Coating sample + Scotchbrite pad	20.20	19.01	1.19
"	SSL4	Fuel particulate from center breach (~1 ml)	23.41	18.99	4.42
"	SSL5	Fuel particulate from bottom breach (<1 ml)	20.61	18.93	1.68
"	R2	Residue from SFEC (~2 ml + filter)	21.31	19.00	2.31
5427E	CS1	Coating sample + Scotchbrite pad	19.74	18.99	0.75
"	FS1	3 fuel pieces from top end (one with cladding)	118.96	18.94	100.02
"	SSL1	Fuel particulate from top end (~3 ml)	34.66	19.13	15.53
"	SSL2	Fuel pieces and particulate collected from hanger (~5 ml)	80.19	18.88	61.31
"	SSL3	Fuel pieces and particulate from lower end (~2 ml)	27.88	19.10	8.78
"	R1	Residue from SFEC w/o filter (~20 ml)	61.74	19.17	42.57
7913U	CS3	Coating sample + Scotchbrite pad	20.00	19.07	0.93
"	SSL7	Fuel particulate from bottom end (~1 ml + fuel piece with cladding)	26.70	18.95	7.75
"	SSL8	Fuel particulate from center breach (~2 ml)	27.24	19.05	8.19
"	R3	Residue from SFEC (~1 ml on filter)	21.23	19.02	2.21
0161M	CS4	Coating sample--mostly flakes (~15 ml)	34.52	26.03	8.49
"	R4	Residue from SFEC (1-2 ml on filter + metallic pieces)	20.44	15.00	5.44
2667U	CS7	Coating (orange) sample (~3 ml)	28.86	26.18	2.68
"	CS8	3 Scotchbrite pads	31.07	26.35	4.72
"	R5	Residue from SFEC (~5 ml + filter)	25.06	15.07	9.99

Weights are in grams.

4.0 ANALYSES TO BE PERFORMED

The analyses proposed for the material collected in this campaign are listed in Table 2. The details presented indicate whether an individual analysis applies to coating, sludge from cracks, or SFEC material and how much material is necessary to perform the analysis.

Table 3 prioritizes the analyses for each type of sample. Thus, where the amount of sludge or coating is small, the material will be allocated first to the highest priority item. Tables 2 and 3 were prepared with the assumption that, since SFEC sludge is the most plentiful, the material archived for sludge process demonstration and that dedicated to physical properties will come from SFEC sludge. Larger pieces of material will be archived for sludge process tests or Thermo-Gravimetric Analysis (TGA).

Examinations will be performed in 325 Building Laboratory. Compound identification (by X-ray Diffraction) will confirm the presence of such species as hydrates and oxides of uranium in coatings and sludges. Previous work with one K East fuel element indicated that the grayish coating on the cladding surface is a mixture of UO_2 hydrates (Marschman 1997). However, the red and translucent coatings investigated here are expected to be quite different from previously investigated material. Chemistry and isotopic determinations through Inductively Coupled Plasma (ICP) analysis, Mass Spectrometry (MS), and Gamma Energy Analysis (GEA) will give elemental identification of constituents which are noncrystalline or present in minute quantities. TGA will give information that is complementary to the drying information previously generated for canister sludge. However, given the limited amount of available material for the present campaign, the TGA sample sizes used here may only be 10% of that used previously. Analysis for organic compounds will be by MS but initial detection of PCBs may be by Electron Capture (EC). It may also be possible to measure coating thickness in the case there translucent material is removed in sheet-like pieces from the cladding surface. This may be done via micrometer measurements or more likely by Scanning Electron Microscopy (SEM). The advantage of the latter method is that simultaneous energy dispersive X-ray measurements can be made that will give elemental composition information which complements ICP results.

Table 2. Analyses Proposed for Coatings and Subsurface Sludge.

Determination	Coating/Sludge/SFEC	Technique
Structure	C, S	XRD
Chemistry and isotopes	C, S	ICP/GEA/MS
Water evolution	C, S	TGA
Physical properties: Particle size, density	SFEC	Sieving, laser scattering, macro-settling
PCBs/organics	C, S	EC and MS
Sludge pretreatment/ disolution	SFEC	Archive sludge <1/4 in.
Scrap basket oxidation	C/S/SFEC	Archive pieces >1/4 in.
Coating thickness	C	Micrometer and/or SEM

Table 3. Prioritization* of Examinations.

Examination	Amount Needed (gm)	Coating	Coating Pads	Crack Sludge	SFEC Sludge	SFEC Filter	>1/4 in. Fuel Pieces
XRD	0.1		1	1		1	
ICP GEA MS	0.5 to 1	1	2	3		2	
Sieving, laser scattering, settling	10				1		
TGA (drying)	0.05 to 1	2		2			
SEM	0.1	3					
EC and MS (organics)	0.5 to 1	4				3	
Sludge process demonstration archive	--				2		
Furnace scrap test archive	--						1

*1 = Highest priority.

4 = Lowest priority.

5.0 GOVERNING DOCUMENTS AND SCHEDULES

The procedures, accuracies, and precisions for this work will be those specified in the last Sample Analysis Plan (SAP) for K West Canister Sludge (Welsh 1997). Work will be initiated through letters of instruction (LOIs) to Pacific Northwest National Laboratory. Quality Assurance requirements will be specified in the LOIs and these will be the same as those for fuel element examinations (Lawrence 1997c). Some subsurface sludge is expected to accompany the fuel in an MCO and it therefore falls under the Office of Civilian Radioactive Waste Management Quality Assurance Requirements (DOE/RW-033P, Quality Assurance Requirements and Description).

The subject work will be scheduled such that it does not interfere with the ongoing analysis of K West canister sludge which is governed by a near term performance agreement. Thus, it is expected that the proposed analyses will overlap fiscal years 1997 and 1998.

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