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SAMPLING AND ANALYSIS PLAN FOR SLUDGE LOCATED IN FUEL STORAGE CANISTERS OF THE 105-K EAST BASIN

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Abstract: This Sampling and Analysis Plan (SAP) provides direction for the first sampling of sludge from the K East Basin spent fuel canisters. The specially developed sampling equipment used removes representative samples of sludge while maintaining the radioactive sample underwater in the basin pool (equipment is described in WHC-SD-SNF-SDD-004). Included are the basic background logic for sample selection, the overall laboratory analyses required and the laboratory reporting required. These are based on requirements put forth in the data quality objectives (WHC-SD-SNF-DQO-008) established for this sampling and characterization activity.

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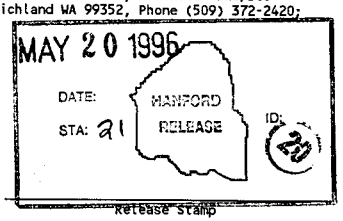

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SAMPLING AND ANALYSIS PLAN FOR SLUDGE LOCATED IN FUEL STORAGE CANISTERS OF THE 105-K EAST BASIN

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May 1996

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ALARA	As Low As Reasonably Achievable
ANOVA	Analysis of Variance
AEA	Alpha Energy Analysis
Basin	105-K East Irradiated Fuel Storage Basin
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CSB	Canister Storage Building
DQO	Data Quality Objective
DSC	Differential Scanning Calorimetry
DST	Double Shell Tank
GEA	Gamma Energy Analysis
IC	Ion Chromatography
ICP	Inductively Coupled Plasma
ISE	Ion Specific Electrode
Main Basin	105-K East Irradiated Fuel Storage Basin Exclusive of Remote Pits
LOI	Letter of Instruction
MCO	Multi-Canister Overpack
MDL	Minimum Detection Limit
NDT	Nondestructive Testing
PCB	Polychlorinated Biphenyls
PNNL	Pacific Northwest National Laboratories
ppm	parts per million
PQL	Practical Quantitation Limit
PTFE	polytetrafluoroethylene
QA	Quality Assurance
QC	Quality Control
RCRA	Resource Conservation Recovery Act of 1976
RPD	Relative Percent Difference; [(result1 - result2)/mean]*100
SAP	Sampling and Analysis Plan
SAR	Safety and Analysis Report
SARP	Safety and Analysis Report for Packaging
SD	Serial Dilution
SFBWP	105-K East Sand Filter Backwash Pit
SNFE	Spent Nuclear Fuel Evaluations Group
SP	Spike Analysis
SVOA	Semivolatilve Organic Analysis
TC	Total Carbon
TCLP	Toxicity Characterization Leaching Procedure
TGA	Thermal Gravimetric Analysis
TIC	Total Inorganic Carbon
TICs	Tentatively Identified Compounds
TIMS	Thermal Ionization Mass Spectroscopy
TLD	Thermoluminescent Dosimeter
TOC	Total Organic Carbon
TWRS	Tank Waste Remediation System
VOA	Volatile Organic Analysis
WHC	Westinghouse Hanford Company
XRD	X-Ray Diffraction

SAMPLING AND ANALYSIS PLAN FOR SLUDGE LOCATED IN FUEL STORAGE CANISTERS OF THE 105-K EAST BASIN

1.0 SAMPLING OBJECTIVES

1.1 PURPOSE

The purpose of this sampling and analysis plan (SAP) is to define the strategy and the methods that will be used to sample and analyze the sludge located in the fuel storage canisters of the 105-K East Basin. The data quality objectives (DQOs) for the sludge sampling campaign are provided in (Makenas et al., 1996a). Canister sludge is a nonhomogeneous mixture of particulate materials likely containing fuel element corrosion products, fuel storage canister corrosion products, environmental materials such as sand and insects, basin rack corrosion products, sloughed-off concrete material, paint flakes from racks, materials accumulated during past basin operations (e.g., ion exchange beads spilled on/in canisters, etc.), and/or fission products.

Specifically this SAP contains the plan for sampling the sludge located in the fuel storage canisters ("canister sludge") and specific requirements for the laboratory analyses to be performed on the samples. This SAP is supplemented by (1) a Canister Choices document, which summarizes the specific canisters to be sampled for sludge in K East Basin and rationale of this selection, (Makenas 1996b), and (2) Letters of Instruction, LOI, which will provide detailed instructions to the laboratories on which samples should receive a particular analysis and how samples should be handled. The DQO (in Sections 2.4.2 and 2.4.3) contains the general rationale for canister selection which was the basis for the Canister Choices document. Additional discussion of this selection is provided in Section 2.2.1 of the present document.

1.2 SAMPLING AND ANALYSIS ACTIVITY OBJECTIVE AND GOALS

The DQO (Makenas et al., 1996a) for this activity summarizes the objectives and logic relating this sampling effort to key Spent Nuclear Fuel (SNF) project activities. The basic decisions requiring this characterization are related to (1) the retrieval, storage, and disposal of K Basin sludge (both canister sludge and floor sludges), and (2) the impact of residual canister sludge accompanying spent fuel elements to the Canister Storage Building (CSB) in Multi-Canister Overpacks (MCO). The specific characterization decisions are:

1. Does the K Basin canister sludge meet established acceptability criteria for storage and treatment in the Hanford Tank Waste Remediation System (TWRS), specifically in one or more double

shelled tanks (DST)? (A secondary consideration here is the potential backup alternative of the sludge being dispositioned as solid waste. In either case the sludge would be stored and then follow the related waste to ultimate disposal in a permanent repository.)

Sampling Goals: Obtain representative samples from the canisters--seven from canisters with fuel elements and two from non-fueled canisters. Deliver samples to a Hanford analytical laboratory. Aliquots then will be submitted for analyses to determine chemical and isotopic compositions of selected constituents. Gross physical properties of sludge will also be obtained from the samples (e.g., settled density, volume, weight, settling time, behavior of selected layers, particle size, etc.). This will provide estimates of the analyte concentrations considered necessary by TWRS (Fowler 1995a, 1995b) to allow their review and acceptance of the sludge for placement in a DST. It will also provide estimates of analyte concentrations important to successful handling of sludge as solid waste (Willis 1993).

2. What actions if any, must be taken during the drying, conditioning, and storage of K Basin spent fuel elements in MCOs as a result of residual canister sludge which accompanies the spent fuel elements?

   Sampling Goals: Obtain drying data from representative samples of canister sludge that are meaningful to pending decisions on proposed drying and conditioning parameters for the fueled MCOs. Quantify any toxic constituents that potentially influence permitting or final disposal of the fuel elements.

3. What is the best choice of equipment to successfully retrieve, handle (e.g., pump, etc.), and process canister sludge?

   Sampling Goals: Obtain fluid and rheological properties which characterize K Basin canister sludge samples to allow accurate development and qualification of designs for sludge handling equipment (i.e., transport, processing, etc.).

4. What are the best simulants to mimic the physical properties of the K Basin canister sludge to allow development and testing of critical equipment designs?

   Sampling Goals: Similar to Item 3.

5. Can a non-destructive test method, such as portable gamma scanning, provide an acceptable measure of the fissile content or of marker radionuclides consistent with the fissile accountability needs for K Basin canister sludge handling?

   Sampling Goals: Obtain relationships between fission product radioisotopes (measurable by gamma scan methods translatable to field applications) and plutonium concentration (measured by

analytical laboratory methods) in K Basin canister sludge samples such that, if possible, one or more of the fission products can be identified as a marker for plutonium when quantifying residual transuranics in the sludge.

It should be noted that the current draft of the "Memorandum of Understanding for K Basin Sludge Transfer to Tank Waste Remediation System," between the Spent Nuclear Fuel (SNF) project and TWRS, agrees TWRS will provide complete acceptance criteria to the DST. This task has not been completed. The final criteria for characterization of K Basin canister sludge may increase over the current basis (Fowler 1995a, 1995b) once the final criteria are developed. These additional characterization needs (e.g., repeating some analyses with K Basin sludge mixed with DST tank waste, etc.) will likely be accommodated with the planned contingent sample volume that should be available from this sampling campaign.

2.0 105-K EAST BASIN STATUS AND SAMPLING INFORMATION

2.1 105-K EAST BASIN STATUS

The 105-K East Basin was designed and constructed in 1950 to 1951. The purpose of the basin was to receive and store irradiated fuel from the K East reactor. The basin provided the freshly irradiated fuel with cooling and a 150-day period of time to allow short-lived isotopes to decay. The water in the basin provided the workers with shielding from the nuclear radiation resulting from the isotope decay while they sorted and handled the fuel elements underwater.

The K East reactor stopped the irradiation of fuel in 1971. In 1976, the basin started receiving irradiated fuel from the 105-N Basins. The fuel was stored in the basin in twin barreled canisters open on the top (Baker 1995b). A portion of this fuel is still stored in the basin today, and some fuel elements have breached cladding such that significant corrosion of the exposed uranium fuel material has occurred. These corrosion products are primarily hydrolyzed metal and deteriorated fuel compounds that precipitate as a flocculent sediment in the bottom of the canister barrels and basin.

The basin floor sludge was periodically sampled prior to 1995. Analytical results (Baker 1995b) have shown the material to contain predominantly sands, ferric oxides, aluminum oxides and uranium oxide residuals. The sludge has also been found to contain trace amounts of barium, cadmium, chromium, lead, and samarium. Finally, the sludge is known to contain plutonium based on analysis of materials collected from the 105-K East Sand Filter Backwash Pit (SFBWP) and its transfer channel (Bechtold 1994).

Measurements of floor sludge depths in the 105-K East Main Basin were made in 1994 and have shown that the basin floor is covered with sludge to a depth of 5 to 19 cm (Baker 1995b) and that the Weasel Pit is covered with sludge up to a meter or more in depth. Potential local sources of sludge that could influence the floor sludge characterization were evaluated by the Spent Nuclear Fuel Evaluations (SNFE) group, (Baker 1995c). The local conditions considered were:

- Fuel sludge passing through openings in canisters containing breached fuel elements
- Corrosion of the aluminum canisters
- Sloughing of the unsealed concrete walls that form the basin
- Flaking paint off the fuel storage racks
- Historical activities (e.g., fuel handling) affecting areas near the mouth of the pits (i.e., Weasel Pit, Tech View Pit, Dummy Elevator Pit, and South Load-Out Pit).

Detailed sampling and analyses were recently completed for the floor sludge in the K East Main Basin and Weasel Pit. These results are currently under final review (Silvers 1995; Miller 1996) with a final report scheduled for May 1996. Results thus far have shown agreement with general expectations (Welsh et al., 1995) with the exception of the unexpected identification of polychlorinated biphenyls (PCBs) in some of the sludge samples.

There are no apparent data on depth (i.e., volume) or composition (i.e., characteristics) of the sludge located in the fuel storage canisters in K East Basin except for a limited set of sludge depth measurements made in empty canister barrels and one canister barrel containing five fuel assemblies (Baker 1995b).

To-date the nearest materials to canister sludge that have been chemically analyzed in detail are the recent floor sludge samples from the Weasel Pit and one of the Main Basin Samples (KES-0-09). The Weasel Pit has sludge that was pumped into it from the Segregation-Discharge Chute area. Since this area was used in the past to dump out canister barrels to recover fuel elements, sludge from the canisters was likely included in the accumulated sludge on the floor in this area. This however is likely a very poor basis since there is no way of knowing the canister sludge dilution once mixed with the floor sludges in both the Segregation-Discharge Chute and the Weasel Pit. Similarly, the KES-0-09 sample was taken near breached fuel elements and showed a high concentration of fuel related materials. While this sludge is likely similar to canister sludge the dilution it would have in the components of the floor sludge is unknown.

To better understand the character of the canister sludge prior to the selection of the canister barrels to be sampled for sludge, it is planned that a survey be made of over 50 candidate canister barrels for (1) fuel element condition (accomplished by visual inspection using underwater video cameras) and (2) sludge depth (measured by ultrasound techniques). The survey (Pitner 1996) will be completed prior to the sampling campaign and should provide definitive information to assist in canister selection. Survey data will also assist in selecting related fuel elements that will be removed and shipped from K East Basin for fuel characterization during the same time period.

The primary objectives of the analyses of the canister sludge are verification of (1) its general similarity in dependencies and composition to the floor sludge, and (2) establishment of those areas where its characteristics may differ from floor sludge. Variables considered for the selection of candidate canister barrels to be sampled for sludge are:

- Condition of fuel elements in barrel (e.g., cladding degradation, damaged fuel, etc.)
- Age of fuel in barrel
- Condition (e.g., corroded) and material (e.g., aluminum) of canister barrel
- Type of canister barrel (e.g., open bottom, slotted, etc.)
- Depth of sludge in barrel.

Information on the first four variables are currently available. Information for the fifth variable will not be available until just before sampling when the survey of sludge depths in canisters is completed using the ultrasound methods described previously. Information on all five of these variables will be considered when determining the canisters to be sampled for sludge and this selection documented in the Canister Choices document (Makenas 1996b), also see Section 1.1 of the present document.

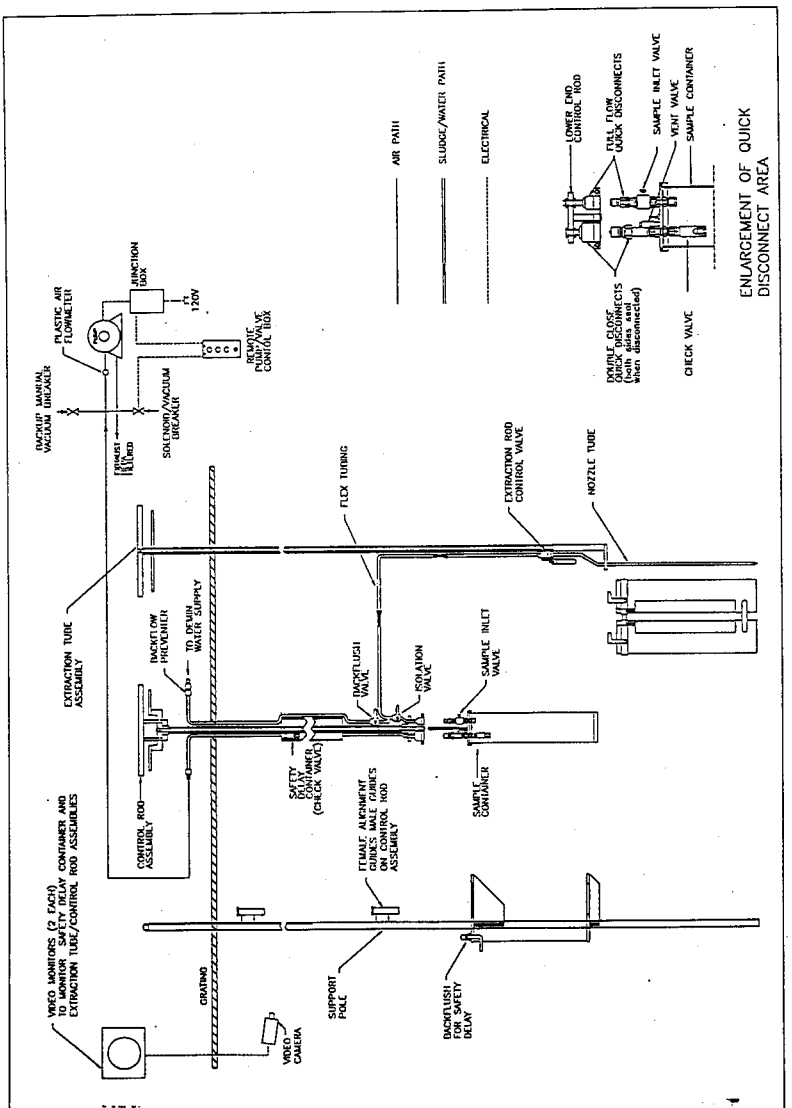
Besides these five variables noted previously it is expected that the same parameters that the basin floor sludge (Welsh et al., 1995) is dependent on will be present for the canister sludge. This follows since the canister barrels in K East Basin are open on the top and will collect a portion of all the materials settling in the basin pool. These floor sludge parameters will be quantified from the characterization analyses and evaluations currently being completed for the K East Basin floor sludge (e.g., Miller 1995). Therefore dependencies of canister sludge on these latter parameters will be in general verified for canister sludge, but analyses will not necessarily be made on every canister sludge sample. In a few instances analyses of the K East Basin floor sludge samples will be assumed to be sufficient and not repeated for the canisters sludge (i.e., cyanide, pH, ammonia, VOAs). Essentially all the types of analyses planned for the canister sludge samples (Tables 1 and 2) were also performed on the recent floor sludge samples.

2.2 SAMPLING INFORMATION

2.2.1 Sample Collection and Handling

The sampling will be conducted such that the operation does not impact the water quality/air emissions at the K East Basin. The sampling apparatus is described in the System Design Description document (Baker 1996). The sampler utilizes specially developed equipment made to sample sludge from the fuel storage canister barrels while keeping the sample at least 10 feet under the surface of the Basin water pool, Figure 1. The sampler is basically composed of a sealed sample container of approximately 10 liters, constructed of stainless steel having "quick disconnect" inlet and outlet ports on the lid. The outlet is attached to a pump that evacuates the air from the sealed sample container. The inlet is attached by flexible tubing to a special extraction tube with nozzle assembly that can be inserted into a fueled canister barrel and controlled by an operator above on the Basin grating. The nozzle is designed with openings to restrict particles larger than 0.25 in. (0.64 cm), leaving them in the canister. The nozzle is controlled by a valve which when opened during sampling allows sludge to be pulled into the sample container under the force of the established vacuum in the sample container. The sludge material is minimally disrupted by this sampling technique. The extraction nozzle will be guided down between the fuel elements and the canister, thus allowing collection of the sludge to the bottom of the barrel in each channel before moving on to the next opening. The sample containers will be cleaned prior to use to minimize possible contamination from fabrication processes (Bridges 1996).

Figure 1. Overview of Canister Sludge Sampling Equipment.



It is planned that each canister sludge sample be about 500 ml, approximately double the size of floor sludge samples taken previously (Baker 1995a). This should provide enough sample for analyses called for in this SAP, plus contingent sample volume to use for future tests including such potential process demonstration tests as K Basin sludge combined with TWRS DST sludge.

The sampling team will follow approved sampling procedures during the collection of the samples. Detailed descriptions of the sampling apparatus and methodology will be provided in the sampling procedure (K Basins OP-43-020E) and Master Work Plan (K Basins MWP-95-005).

Each sludge sample container will be labeled with a unique number. The SNFE group Test Engineer shall verify that the samples are properly identified and the sample location is recorded prior to moving the sample from the 105-K East Basin area. The SNFE group Test Engineer will ensure that the sample custody during transportation will comply with the requirements defined in K Basin procedure MWP-95-005.

After the sludge samples have been collected, they will be moved to the area in the K Basin pool used for hydrogen generation checks and subsequently moved to the Chem-Nuclear cask for transport to the laboratories. The packaging of the samples will comply with applicable WHC procedures.

There is no analytical hold time limit on the samples. It is the intent of this SAP to ship the samples to the analytical laboratories as quickly as possible and be at least within 1 month of when they are collected. If logistics preclude shipment within 1 month from the date of sampling, the SNFE group Project Coordinator will evaluate the situation to confirm applicable storage for the samples. [The samples should be shipped as soon as possible, however logistics may delay transport. Sludge sampling is part of an overall fuel and sludge sampling campaign at K East Basin between April and June 1996. The Chem-Nuclear cask is large (15 tons) and holds only six samples (combination of fuel and sludge sample containers). Sequencing, ALARA concerns, hydrogen monitoring, and waiting for a full cask load could delay shipment of samples to the laboratory up to the 1 month period. There should be no problem with sample integrity during this period as collected samples will be maintained under conditions essentially the same as they were prior to collection (i.e., underwater, same water pressure, same temperature, etc.) and they will be controlled by the Chain of Custody methods.]

In the laboratories the samples will be sealed hermetically to avoid evaporation. Because chemical preservation steps do not make allowance for slurries and would adversely affect the sample (pH change), chemical preservation of samples will not be performed.

It is planned that (1) the analytical laboratories will dispose of the remains of the sludge and water from analyzed samples once the analyses are complete and (2) any significant quantities of sludge not used for analyses will be returned to K Basin if no additional analyses or testing is anticipated.

2.2.2 Sample Record Keeping

A vital part of the sampling activity is the assurance that all of the information and data associated with each sample is accurate and verifiable. Therefore, all pertinent information and data collected during the sampling and sample breakdown will be recorded. The pertinent data to be collected and the corresponding records will be maintained in accordance with the requirements of the sampling procedure (K Basins OP-43-020E), the Master Work Plan (K Basins MWP-95-005), and the QA at the laboratory doing the sample breakdown (i.e., PNNL QA Plan MC-33). A copy of the records for the samples collected at K Basins will be submitted to the SNFE group Project Coordinator the day they are collected. The LOIs for the sampling effort may also provide additional direction on record keeping.

2.2.3 Field Sample Plan Modifications

Any modifications to the sludge sampling plan made in the field must be made/approved by the SNFE group Test Engineer and/or SNFE group Project Coordinator, and documented.

2.2.4 Sampling Locations

A representative sludge sample will be obtained from seven fuel storage canister barrels containing fuel elements and from two barrels that are "empty" or nearly empty with respect to fuel elements ("empty" barrels are observed to contain layers of sludge in K East Basin). The DQO (Sections 2.4.2 and 2.4.3) provides a general basis for the number of samples being taken. This number of samples is intended to compare the statistical mean values of the recent floor sludge sample analyses with the statistical mean values resulting for the canister sludge samples analyses. The number of canister sludge samples required for such a statistical comparison was estimated as five for canisters with nearly full fuel loads (i.e., a full load is seven assemblies) and two samples from empty or near-empty (i.e., one or two unbreached fuel elements) canisters. Fewer samples are required from the near-empty canisters since significantly fewer variables are involved (e.g, little or no fuel element dependencies). Two "research" samples are being taken in addition to these seven "normal" samples. Each research sample will be chosen from a canister that has a nearly full or full fuel load, and has conditions similar to one of those in the set of five "normal" samples from fueled canisters. The "research" samples will establish the influence of re-settled layers of sludge that form. Final selection of the research samples will be made after all samples have been collected and observed through the settling studies, to assure a range of behavior is studied in the two research samples chosen. Further discussion on "research" samples is provided in Sections 2.2.5 and 3.1.

The locations and the rational for canisters selected for sampling (and backup locations) is described in the Canister Choices document (Makenas 1996b). This document considers currently available data and rationale for use of the data from the visual and ultrasound survey to be made at K East Basin shortly before sludge sampling of the canisters begins.

The selection of canisters will address the parameters discussed in Section 2.1, plus other requirements imposed by the sampling equipment (i.e., being located directly under a grating slot, being able to fit the extraction nozzle into the canister barrel between fuel elements, etc.).

As noted in Section 2.1, it is assumed because the K East Basin canisters are open at the top with floor sludge sources settling onto them, that the recent characterization of the K East Basin floor sludge is directly related to the non-fuel element components of canister sludge. Verification of this assumption will be made with the selected canister sludge samples. It is acknowledged that canister barrels with open bottoms and/or slotted sides will allow floor sludge from the immediate area to be drawn in with the canister sludge samples. This is viewed as acceptable as it should be a minimal effect and there is a continuum between the canister sludge and floor sludge in these areas (i.e., free exchange of material in these areas).

2.2.5 Sample Preparation

Procedure(s) for sample preparation, including hot cell operations if necessary, similar to WHC-SD-NR-TP-408, "Procedure for Processing Sludge Samples from K East Basin Floor and Weasel Pit" will be prepared by the analytical laboratory performing the analyses. This test plan is being referenced because it is assumed that the 105-K East Basin canister samples will be similar to those samples. The sample preparation procedure will contain the detailed instructions necessary to safely and properly record observations, decant and subsample (as needed), digest (if necessary), handle, package, and label each sample and its derivatives in the hot cell/hood.

The sample preparation (for ICP, GEA, etc.) involves drying the sample in a polytetrafluoroethylene (PTFE; teflon) beaker on a hot plate and then an acid digestion. Therefore, subsamples for organics analyses and other analyses affected by either drying or acid digestion (TIC, TOC, TC, DSC, IC, XRD, etc.) need to be obtained prior to sample preparation. In some cases, as specified in the LOI, the liquid portion and the wet sludge should be sampled for the various analyses.

The "normal" and "research" samples differ in that the research samples will be separated for analyses of individual layers. A "normal" sample will be homogenized and analyses will be performed on the integrated properties. The "research" samples will be separated into major "layers" and each layer analyzed. This is necessary for meaningful rheology measurements and is of interest for possible impact of settled layers behaving differently than the mixed mass of the sample (e.g., plutonium concentration). Layers will be identified as was done for the recent floor sludge samples--that is a visual identification by color and/or texture of regions of sludge material appearing as stratum after settling studies are completed; in the floor sludge samples two layers were typically found.

Analysis of all the sludge samples by layers was considered in order to determine the possible effects such as plutonium being distributed differently between the heavier and lighter sludge layers. It was however concluded the

limited number of research samples (at least two) would be sufficient. Factors involved in this decision (not analyzing by layers in all samples) included: difficulty in objectively determining layers, settling differences between the actual basin itself and in the analytical laboratory, a desire to maintain the maximum sample quantity to minimize potential for inhomogeneities when splitting, and lack of objective data concerning the distribution of the heavier components. This decision also assumes that if the sludge material in the basin is transferred to tank farms or sent to solid waste disposal, it will be sent initially as a mixture, although some separation into layers may occur in transit and after deposited in the DST.

The analytical laboratory will document and photograph both the appearance of the research samples and the number of layers or strata seen based on appearance (e.g., color, texture, etc.). The samples will be reviewed after the settling studies to choose research samples, if unique layering is noted so that more than two research samples are needed, this option will be considered. For each sludge research sample the laboratory preparation personnel will separate the sludge layers into different sampling containers. After separation, each layer will be processed according to the laboratory sample preparation procedure, which will be defined in the LOI. These "research" samples will be chosen to be similar to previously selected "normal" samples, so that comparisons can be made between the samples handled as one unit and those handled in layers. It is very important that the analytical laboratories measure and record the evolving densities of the canister sludge samples (i.e., as-settled, centrifuged, dried, etc.). These densities will allow both comparison to past work (Miller 1995; Silvers 1995) and the state of sludge as collected.

3.0 LABORATORY ANALYSIS INSTRUCTIONS

3.1 GENERAL

Specific instructions on processing of each canister sludge sample in the analytical laboratories will be provided by letters of instruction (LOI) from the SNFE group Project Coordinators prior to shipment of samples.

Each sludge sample will be analyzed to ascertain properties such as fissile and fission product content for criticality, shielding and accountability.

Each sludge sample will be transferred to the analytical laboratory as one unit. Some of the sludge physical properties (e.g., settling rate) will be obtained prior to any subsampling efforts. Others, such as viscosity and particle size/shape, must be determined from aliquots taken prior to centrifuging. After centrifuging (prior to heating or acid digestion), aliquots of both the liquid and the solids will be obtained from each sample. These aliquots will be used by the laboratory for organic, TIC, TOC, XRD, DSC, TGA, and other analyses sensitive to either drying or acid digestion. An aliquot of the thoroughly stirred dried sludge, of sufficient weight (about 10 g) to minimize sample heterogeneity, will then be processed through an acid digest stage. An aliquot of the acid digested sludge shall be prepared and sent to the laboratory. Any solids which remain after the acid digestion step will be analyzed by X-Ray diffraction.

The laboratory will analyze each aliquot in duplicate or as directed in Section 8.0 of the SAP and the LOI. Each subsample taken from that aliquot must go through separate sample preparation, if sample preparation is required. If a sufficient amount of dried sludge is not available for the acid digestion step, then analyses for that sample may be limited to higher priority data. The laboratory should subsequently document the reason for not performing any of the analyses called for in the LOI in the narrative of their data report. An alternative to not performing the analyses is to have K Basin Operations take additional samples for the "other" required analyses. The choice of not performing analyses or requesting additional samples will be at the discretion of the SNFE group Project Coordinator.

A flowchart showing the general analysis scheme for the sludge samples in the laboratory is presented in Figure 2. The analytical tests for the analytical laboratory work are shown in Table 1. The reason or need for each of these analyses is defined in the DQO, (Makenas et al., 1996a). In general the samples designated as "normal" will receive all the analyses in Tables 1 and 2 with the exception of those related to rheology measurements. The "research" samples will be split into samples of the observed layers or strata and these will in general receive all the analyses in Tables 1 and 2 including rheology measurements (also see Section 2.2.5 and 3.1). Note that no analyses for waste designation are called for in the DQO (see Section 2.2 of that document). This follows directly from the assumption made by the SNF Project for K Basin sludge that the waste designation will be made based on "process history." As discussed in the DQO pyrophoricity of the sludge is being assessed from the XRD measurements for metallic Zr, metallic U, and U hydride. Exothermic reactions are being evaluated by differential scanning calorimetry.

Figure 2. General Sample Analysis Strategy.

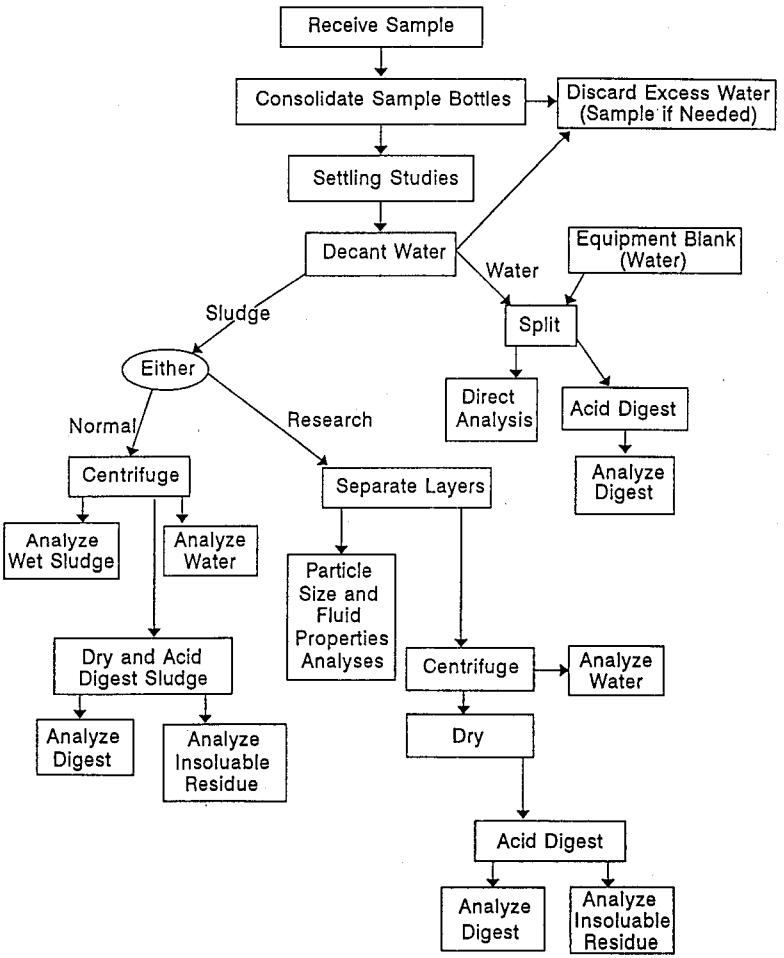


Table 1. 105-K East Canister Sludge Analyses for the Analytical Laboratory.

Requested Analysis	Analysis Technique	Constituents to be Reported
Am-Isotopic Pu-Isotopic	Separation and AEA	^{241}Am, ^{238}Pu, $^{239/240}Pu$, and $^{243/244}Cm$
Sr-90	Separation and beta counting	^{90}Sr
Np-237	Extraction and alpha counting	^{237}Np
Total Gamma	GEA	^{241}Am, ^{134}Cs, ^{137}Cs, ^{60}Co, ^{152}Eu, ^{154}Eu, ^{155}Eu, ^{212}Bi, ^{208}Tl, ^{125}Sb, $^{106}Ru/Rh$ and $^{144}Ce/Pr$ and other gamma emitters in library
Gross Alpha	Gas proportional counting	Detectable alpha emitters
Gross Beta	Gas proportional counting	Detectable beta emitters
U Total	Laser fluorescence or phosphorescence	Total uranium
U Isotopics	TIMS	^{233}U, ^{234}U, ^{235}U, ^{236}U, ^{238}U (also report available data on ^{236}Pu, ^{239}Pu, ^{240}Pu, and ^{241}Pu)
Metals	ICP	Al, Cd, Sm, B, Fe, Ba, Cr, Pb, Ag, Be, Tl, Zn, Cu, Mn, Mg, Ca, Na, K, Se, U, Zr, Bi, P, and Ni (Hf, Sn if in current analyses)
IC Analyses	IC	NO_2^-, NO_3^-, PO_4^{3-}, SO_4^{2-}, F^-, Cl^-, Br^-, oxalate, formate, and acetate
Tc-99	Solvent extraction with liquid sintilation counter	^{99}Tc
Organics	SVOA	Any TICs found in the analysis, polychlorinated biphenyls
Endothermic/Exothermic Reactions	DSC/TGA (use TWRS and Appendix A methods)	DSC/TGA include annotated thermogram
Settling Rate	Settling columns	Volume settled versus time

Table 1. 105-K East Canister Sludge Analyses for the Analytical Laboratory. (Continued)

Requested Analysis	Analysis Technique	Constituents to be Reported
Settled and Centrifuged Densities	Gravimetric/sample preparation procedure	Settled density, volume, weight, and centrifuged density
Residual Analysis	XRD	Qualitative analysis of any undigestible residue
Pyrophoricity	XRD	Qualitative analysis of the sludge prior to heating or digestion. Unreacted metallic uranium and Zr; unoxidized uranium hydride; uranium oxide; hydrates of Al_2O_3, UO_{2x} and FeO_x
Particle Size Distribution	Particle size analyzer	Size, distribution
Particle Shape	Optical microscopy, SEM, or TEM	Shape
Particle Density	Gas pycnometer/ calculation	Density dry/density wet
Viscosity	Viscometer	Viscosity
TIC/TOC/TC	Persulfate oxidation and coulometry	TIC (CO_3^{2-}), TOC, TC
Zeta Potential	Zeta plus instrument	mV
Caustic Demand	Potentiometric titration	Buffering capacity

3.2 ANALYSES

Table 2 shows the analytes, methods, and the laboratory procedures required for the analysis of the samples. The tables also list the requirements for detection levels, precision, and accuracy of data results. Note that if dilution factors change from what is expected based on factors from recent floor sludge sampling (Miller 1995), the detection levels, precision, and accuracy values shown in Table 2 will have to be re-evaluated. One reagent blank will be run if required by the laboratory procedure for each analytical batch. An equipment blank from the sample collection mockup will be run for each set of new sampling equipment (i.e., plan is for one set of equipement for K East Basin and one set for K West Basin). A hot cell blank (if the hot cell is used) will be analyzed for each hot cell batch or as required by laboratory QA. One method control standard will be run with each batch. A matrix spike will be run per matrix, where applicable, as indicated in Table 2.

Additional analyses of the liquid fraction of the sludge samples, beyond those noted in Figure 2, are potential options to support (1) calculation of maximum theoretical leachate concentration for RCRA heavy metals, and/or (2) to test bench scale water/sludge processing concepts. These will be addressed in the LOI to the laboratories if they are to be performed. It is acknowledged that due to the potential dose rate of these samples such analyses could require significant additional preparation and costs at the laboratories.

Quality assurance (QA) and quality control (QC) information will conform to those required for a "full" data package (as discussed in the following paragraph). The QA/QC standards must conform to Table 2 and/or the applicable analytical procedures. The control standards, spike recoveries, precision between duplicates, hot cell blanks, and equipment blanks need to be within the specified requirements as noted in Table 2. If the precision between duplicates or the spike recoveries exceed the appropriate criteria there are two options; (1) make one rerun to see if acceptable results are obtained (if they are not report QC failure to SNFE group) or (2) report QC failure and suggested actions immediately to SNFE group Program Coordinator for evaluation.

The data package will give the duplicate results, all the QC data (laboratory method control standard results, spike recovery results, hot cell blank results, the equipment blank results), deviations from any of the requirements, and a narrative.

Practical Quantitation Level (PQL) as used in this document is the detection level which is expected to be achievable by the laboratory to analyze a listed constituent, yet is low enough to detect whether the constituent is present in concentrations significant to resolving data quality objectives.

Precision and accuracy requirements for laboratory analytical results specified in this document are based on an assessment of achievable laboratory capabilities, given the complex nature of the samples to be analyzed, their radioactive nature; and the consequent handling, dilution, and analysis methods prescribed. The precision and accuracy statements calculated from the

recent K East Basin floor sludge samples were also considered in determining the requirements presented in Table 2. The DQOs for this sampling effort (Makenas et al. 1996a) consolidate and incorporate the various analyses performed to assure that the overall process will produce the analytical results leading to a realization of the sampling objectives.

Table 2. Analytical Requirements for the 105-K East Basin Canister Sludge Samples (222-S and 325 Laboratories).

Process	Constituents	Procedure	Required PQL[1]	Precision[2]	Accuracy[3]
Separation, AEA					
	^{238}Pu	LA-943-128 PNL-ALO-455/469	8.0 µCi/g	±25%	--
	$^{239/240}Pu$	LA-943-128 PNL-ALO-455/469	3.0 µCi/g	±25%	±25% Sp
	^{241}Am $^{243/244}Cm$	LA-953-103 PNL-ALO-469	3.0 µCi/g 6.0 µCi/g	±25%	±25% Sp Am only
Fluorimetric	U total	LA-925-009 PNL-ALO-445	10 µg/g	±20%	SPC
Thermal Ionization Mass Spectroscopy (TIMS)	U Isotopics ^{233}U, ^{234}U, ^{235}U, ^{236}U, ^{238}U (^{236}Pu, ^{239}Pu, ^{240}Pu, and ^{241}Pu)[7]	PNL-ALO-455	25 µg/g	±5%	SPC
GEA		LA-548-121 PNL-ALO-450/451			
	^{134}Cs		0.4 µCi/g	±25%	--
	^{137}Cs		0.003 µCi/g	±25%	SPC
	^{60}Co		0.002 µCi/g	±25%	SPC
	^{241}Am		3.0 µCi/g	±25%	--
	^{152}Eu		0.3 µCi/g	±25%	--
	^{154}Eu		0.3 µCi/g	±25%	--
	^{155}Eu		0.4 µCi/g	±25%	--
	^{212}Bi		2.0 µCi/g	±25%	--
	^{208}Tl		6.0 µCi/g	±25%	--
	^{125}Sb		2.0 µCi/g	±25%	--

[1] Required Practical Quantitation Limit (PQL) has been established based on expected concentrations and use of the data, taking into account different capabilities of the laboratories for the equipment and analysis methods in use.

[2] Measured sample precision determined by duplicate analyses. The Relative Percent Difference (RPD) between the duplicate analyses is within the specified bounds; e.g., -25% < RPD < 25%. RPD = [(result1 - result2)/mean]*100.

[3] All spike recoveries should be within ±25%. All method standard recoveries should be within statistical process control (SPC). A serial dilution (SD) will be used to evaluate the method accuracy for some metal analytes.

[4] The sample preparation procedure will provide the calculation used to determine the sludge settled density.

[5] Analyze for PCB if PCBs are detected as TICs in SVOA. Precision and accuracy will be provided in LOI.

[6] If available from routine analysis, extraordinary measures (purchase of new equipment) not required.

[7] Report if data are generated as part of other requested analyses.

Sp--At least one spike analysis required.

Table 2. Analytical Requirements for the 105-K East Basin Canister Sludge Samples (222-S and 325 Laboratories). (Continued)

Process	Constituents	Procedure	Required PQL[1]	Precision[2]	Accuracy[3]
	$^{106}Ru/Rh$		7.0 µCi/g	±25%	--
	$^{144}Ce/Pr$		5.0 µCi/g	±25%	--
	^{94}Nb		0.2 µCi/g	±25%	--
	^{226}Ra		9.0 µCi/g	±25%	--
Gross Alpha	Total α	LA-508-101 PNL-ALO-461	3.0 µCi/g	±25%	±25% Sp
Gross Beta	Total ß	LA-508-101 PNL-ALO-463	0.10 µCi/g	±25%	±25% Sp
ICP	Metals	LA-505-151/161 PNL-ALO-211			
	Al		100 µg/g	±25%	±10% SD
	Cd		15 µg/g	±25%	±25% Sp
	Sm		200 µg/g	±25%	±25% Sp
	B		100 µg/g	±25%	±25% Sp
	Fe		100 µg/g	±25%	±10% SD
	Ba		100 µg/g	±25%	±25% Sp
	Cr		20 µg/g	±25%	±25% Sp
	Pb		200 µg/g	±25%	±25% Sp
	Ag		20 µg/g	±25%	±25% Sp
	Be		10 µg/g	±25%	±25% Sp
	Tl		400 µg/g	±25%	±25% Sp
	Zn		20 µg/g	±25%	±25% Sp
	Cu		20 µg/g	±25%	±25% Sp

[1] Required Practical Quantitation Limit (PQL) has been established based on expected concentrations and use of the data, taking into account different capabilities of the laboratories for the equipment and analysis methods in use.

[2] Measured sample precision determined by duplicate analyses. The Relative Percent Difference (RPD) between the duplicate analyses is within the specified bounds; e.g., -25% < RPD < 25%. RPD = [(result1 - result2)/mean]*100.

[3] All spike recoveries should be within ±25%. All method standard recoveries should be within statistical process control (SPC). A serial dilution (SD) will be used to evaluate the method accuracy for some metal analytes.

[4] The sample preparation procedure will provide the calculation used to determine the sludge settled density.

[5] Analyze for PCB if PCBs are detected as TICs in SVOA. Precision and accuracy will be provided in LOI.

[6] If available from routine analysis, extraordinary measures (purchase of new equipment) not required.

[7] Report if data are generated as part of other requested analyses.

Sp--At least one spike analysis required.

Table 2. Analytical Requirements for the 105-K East Basin Canister Sludge Samples (222-S and 325 Laboratories). (Continued)

Process	Constituents	Procedure	Required PQL[1]	Precision[2]	Accuracy[3]
	K		1000 µg/g	±25%	±25% Sp
	Se		200 µg/g	±25%	±25% Sp
	Mn		20 µg/g	±25%	±25% Sp
	Mg		200 µg/g	±25%	±25% Sp
	Ca		200 µg/g	±25%	±25% Sp
	Na		100 µg/g	±25%	±25% Sp
	Zr		20 µg/g	±25%	±25% Sp
	Ni		40 µg/g	±25%	±25% Sp
	P		400 µg/g	±25%	±25% Sp
	Bi		200 µg/g	±25%	±25% Sp
[6]	Hf		TBD	±25%	±25% Sp
[6]	Sn		TBD	±25%	±25% Sp
	U		1000 µg/g	±25%	±25% Sp
IC	Anions--F^-, Cl^-, NO_2^-, NO_3^-, PO_4^{3-}, SO_4^{2-}, Br^-, oxalate, formate, and acetate	LA-533-105 PNL-ALO-212	20 µg/ml	±25%	±25% Sp
Solvent Extraction with Liquid Scintillation Counter	^{99}Tc	LA-438-101	0.01 µg/g	±25%	SPC
Exothermic/ Endothermic	DSC	LA-514-113 PNL-ALO-508	--	±25%	SPC

[1] Required Practical Quantitation Limit (PQL) has been established based on expected concentrations and use of the data, taking into account different capabilities of the laboratories for the equipment and analysis methods in use.

[2] Measured sample precision determined by duplicate analyses. The Relative Percent Difference (RPD) between the duplicate analyses is within the specified bounds; e.g., -25% < RPD < 25%. RPD = [(result1 - result2)/mean]*100.

[3] All spike recoveries should be within ±25%. All method standard recoveries should be within statistical process control (SPC). A serial dilution (SD) will be used to evaluate the method accuracy for some metal analytes.

[4] The sample preparation procedure will provide the calculation used to determine the sludge settled density.

[5] Analyze for PCB if PCBs are detected as TICs in SVOA. Precision and accuracy will be provided in LOI.

[6] If available from routine analysis, extraordinary measures (purchase of new equipment) not required.

[7] Report if data are generated as part of other requested analyses.

Sp--At least one spike analysis required.

Table 2. Analytical Requirements for the 105-K East Basin Canister Sludge Samples (222-S and 325 Laboratories). (Continued)

Process	Constituents	Procedure	Required PQL[1]	Precision[2]	Accuracy[3]
Drained Solids (TWRS)	Percent water (TGA)	LA-560-112 PNL-ALO-508	--	±25%	SPC
Drained Solids (MCOs)	Percent water (TGA)	Appendix A	--	--	--
Hot Persulfate/ Coulometry	TIC (CO_3^{2-}), and TOC/TIC/TC	LA-342-100 PNL-ALO-381	40 µg/g	±25%	±25% Sp
Separation and Beta Counting	^{90}Sr	LA-220-101 PNL-ALO-433	0.2 µCi/g	±25%	±25% Sp
Extraction and Alpha Counting	^{237}Np	LA-933-141 PNL-ALO-415/422	5 µCi/g	±25%	±25% Sp
Gravimetric	Settled and centrifuged density/volume	Sample preparation procedure[4]	--	--	--
Caustic Demand	Buffering capacity	LA-211-104	0.04 mole/kg	±25%	SPC
XRD--Prior to Sample Heating and Acid Digestion	U (metallic), Fe hydrates, Al hydrates, U hydrides, U oxides, and Zr metal	PNL-ALO-268	--	--	--
XRD--Digestate Residue	Any identifiable compounds (analytes)	PNL-ALO-268	--	--	--
Settling Rate	Volume settled versus time	Sample preparation procedure	--	--	--
Particle Size Analyzer	Particle size and distribution	LT-519-101 PNL-ALO-530	--	--	--
Particle Shape	Shape	TBD	--	--	--

[1] Required Practical Quantitation Limit (PQL) has been established based on expected concentrations and use of the data, taking into account different capabilities of the laboratories for the equipment and analysis methods in use.

[2] Measured sample precision determined by duplicate analyses. The Relative Percent Difference (RPD) between the duplicate analyses is within the specified bounds; e.g., -25% < RPD < 25%. RPD = [(result1 - result2)/mean]*100.

[3] All spike recoveries should be within ±25%. All method standard recoveries should be within statistical process control (SPC). A serial dilution (SD) will be used to evaluate the method accuracy for some metal analytes.

[4] The sample preparation procedure will provide the calculation used to determine the sludge settled density.

[5] Analyze for PCB if PCBs are detected as TICs in SVOA. Precision and accuracy will be provided in LOI.

[6] If available from routine analysis, extraordinary measures (purchase of new equipment) not required.

[7] Report if data are generated as part of other requested analyses.

Sp--At least one spike analysis required.

Table 2. Analytical Requirements for the 105-K East Basin Canister Sludge Samples (222-S and 325 Laboratories). (Continued)

Process	Constituents	Procedure	Required PQL[1]	Precision[2]	Accuracy[3]
Viscometer	Viscosity	LT-519-115 PNL-ALO-502	--	--	--
Organics	SVOA--all TICs	LA-523-131/406 PNL-ALO-120/ 344/345	Report TIC >10 ppm	--	--
	Polychlorinated biphenyls (PCB)[5]	PNL-ALO-345 LA-523-434	1 ppm	--[5]	--[5]
Zeta Potential	Zeta potential	TWRS-95-5.6a-2, Rev. 0	TBD	TBD	TBD

[1] Required Practical Quantitation Limit (PQL) has been established based on expected concentrations and use of the data, taking into account different capabilities of the laboratories for the equipment and analysis methods in use.

[2] Measured sample precision determined by duplicate analyses. The Relative Percent Difference (RPD) between the duplicate analyses is within the specified bounds; e.g., -25% < RPD < 25%. RPD = [(result1 - result2)/mean]*100.

[3] All spike recoveries should be within ±25%. All method standard recoveries should be within statistical process control (SPC). A serial dilution (SD) will be used to evaluate the method accuracy for some metal analytes.

[4] The sample preparation procedure will provide the calculation used to determine the sludge settled density.

[5] Analyze for PCB if PCBs are detected as TICs in SVOA. Precision and accuracy will be provided in LOI.

[6] If available from routine analysis, extraordinary measures (purchase of new equipment) not required.

[7] Report if data are generated as part of other requested analyses.

Sp--At least one spike analysis required.

4.0 LABORATORY REPORTING REQUIREMENTS

The laboratories will be required to submit a final report within a period defined in the LOI from SNFE group Project Coordinator to the laboratories. Opportunities for parallel operations will be developed and schedules will be prepared for laboratory work which will minimize the time actually required. Samples will be processed through the laboratory with the priority required to meet the completion date given in the LOI. The laboratory will be required to submit interim preliminary data reports to the SNFE group Project Coordinators as requested. The final reports submitted to the WHC SNFE group Project Coordinators for technical review will conform to the requirements for a "full" data package (e.g., similar to Silvers 1995; Miller 1996). The final report shall document any deviations from the requirements of this document, provide a summary of the analytical results (duplicates, control standard recoveries, and spike recoveries), and give a narrative. Additional information on the data report content is given in Sections 3.1 and 3.2.

Data related to dose rates measured on samples to be shipped between the 325 Building and 222-S analytical laboratories, should be transmitted from the shipping laboratory to the receiving laboratory at least 5 working days prior to the actual shipment.

4.1 STATISTICAL ANALYSIS REQUIREMENTS

A statistical analysis of the data will be performed by the Process Chemistry and Statistics group. For each analyte, analysis of variance (ANOVA) techniques will be used to fit a statistical model to the data. This statistical model can be used to:

- Estimate the variability between samples (heterogeneity of the canisters sampled)
- Estimate the analytical variability (precision)
- Compute a 95% confidence interval on the mean concentration for each analyte
- Compare canister sludge data to floor sludge data.

The report providing the results of the statistical analysis of the data may be separate from the full data package to expedite data communication.

4.2 DATA PACKAGING

Analytical analysis results will be reviewed and processed on a priority basis by the analytical laboratory and other supporting organizations to meet the schedules provided in the LOI.

5.0 REPORTING REQUIREMENTS

The completed data packages from the analytical laboratory will be sent to the SNFE group Project Coordinator who will coordinate data review and final data report preparation. The Project Coordinator will ensure the data and reports become a part of the permanent facility records per WHC-CM-3-5, Document Control and Records Management Manual. Content of the data report is further discussed in Section 3.2.

6.0 SAFETY PLANS

6.1 RADIATION/INDUSTRIAL SAFETY

An industrial safety assessment will be performed prior to the start of these activities. The requirements of HSRCM-1, Hanford Site Radiological Control Manual, and WHC-CM-4-3, Industrial Safety Manual, shall be adhered to during the performance of sampling activities.

6.2 NUCLEAR CRITICALITY SAFETY

A Criticality Safety Assessment will be performed in accordance with WHC-CM-4-29, Nuclear Criticality Safety, prior to the start of these activities. The requirements determined by the assessment shall be adhered to during the performance of the sampling activities.

7.0 ALARA PLAN

The sampling procedure for the activities discussed in Section 2.2.1 will take into consideration exposure reduction techniques which will minimize the radiation exposure to the sampling team as required by WHC-CM-4-11, ALARA Program Manual.

ALARA considerations will be an integrated part of the design effort for the sampling and sampling equipment to minimize personnel exposure. The following exposure reduction techniques or their equivalent will be used during the sampling process (also see Baker 1996c):

a. Approved procedures will be used which take into account the anticipated radiation exposure rate levels.

b. Personnel will be trained on the procedures prior to using them.

c. Personnel training will include cold facility training through the procedures.

d. Shielding and/or remote operation of the equipment will be used where possible.

e. Time required to collect samples will be minimized through thorough analysis of each sampling activity.

f. A contingency procedure will be provided for use when sampling problems arise.

g. As required Health Physics staff will conduct a pre-job meeting with the sampling team, and the SNFE group Test Engineer will conduct a daily briefing with the sampling team on the planned activities for that day.

h. Hand exposure during sample transfers will be monitored using finger ring TLDs, if required.

i. Members of the sampling team, including observers, will be limited to only those absolutely necessary to properly perform the sampling activities.

j. Health Physics personnel will establish dose rate "standby areas" for the sampling team members.

As a result of the above ALARA actions, estimates of the overall total whole-body exposure to the sampling team personnel and Radiation Technician will be projected and closely monitored.

8.0 QUALITY ASSURANCE AND CONTROL PLAN

All sampling, sample handling, sample packaging/shipping, and analytical process activities will be performed in accordance with the requirements of this plan, along with the approved sampling and analysis procedures. The controls identified in this plan, along with those contained in the program plan, have been established to assure the applicable quality assurance requirements of WHC-CM-4-2, Quality Assurance Manual, are satisfied during the performance of this activity. In addition, the analytical laboratory will perform the analyses to its internal quality assurance program plans. The 222-S Laboratory will follow WHC-SD-CP-QAPP-016, Quality Assurance Program Plan for Laboratory Analysis and Process Testing. The 325 Laboratory will follow MCS-033, Quality Assurance Plan for Activities Conducted by the Analytical Chemistry Laboratory.

Method specific quality control and quality assurance such as calibrations and blanks are found in the analytical procedures noted in Table 2. Sample quality control (duplicates, spikes, and standards) are identified in Table 2, in Section 3.1 or in the following paragraph. If no criteria are provided in the current document, the performing laboratory shall perform to its quality assurance plan(s).

Sample groups will be assigned categories in LOI that reflect the number of duplicates and spike analyses that should be run. The categories are:

Category

1 Laboratories will select four of the samples (from the seven normal samples) for duplicate analyses and one sample for spike analyses. The balance of the samples will be handled as normal laboratory batches with QA as per the first paragraph of this section.

2 For the two Research Samples the laboratories will run duplicates on layer analyses, no spike analyses are required.

As noted previously, duplicates are repeats of all analyses after completion of settling analyses. Selection of samples within categories should optimize the use of samples with larger volumes for duplicates, allowing the maximum number of requested analyses to be performed for each category.

Care should be taken with water samples processed in conjunction with the sludge analyses (e.g., equipment blanks, etc.), processing them on a best effort basis with criteria similar to that called out in Table 2, the applicable laboratory procedures, or as called for in the final LOI.

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A P P E N D I X A

GENERAL RECIPE FOR SLUDGE THERMAL-GRAVIMETRIC ANALYSIS IN SUPPORT OF K BASIN SPENT FUEL CONDITIONING AND STORAGE IN MULTI-CANISTER OVERPACKS

APPENDIX A

GENERAL RECIPE FOR SLUDGE THERMAL-GRAVIMETRIC ANALYSIS IN SUPPORT OF K BASIN SPENT FUEL CONDITIONING AND STORAGE IN MULTI-CANISTER OVERPACKS

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 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 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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