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SAMPLING AND ANALYSIS PLAN FOR SLUDGE LOCATED ON THE FLOOR AND IN THE PITS OF THE 105-K BASINS

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Abstract: This Sampling and Analysis Plan (SAP) provides direction for the sampling of the sludge found on the floor and in the remote pits of the 105-K Basins to provide: (1) basic data for the sludges that have not been characterized to-date and (2) representative sludge material for process tests to be made by the SNF Project/K Basins sludge treatment process subproject. The sampling equipment developed will remove representative samples of the radioactive sludge from underwater at the K Basins, depositing them in shielded containers for transport to the Hanford Site laboratories. Included in the present document is the basic background logic for selection of the samples to meet the requirements established in the Data Quality Objectives (DQO), HNF-2033, for this sampling activity. The present document also includes the laboratory analyses, methods, procedures, and reporting that will be required to meet the DQO.

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**SAMPLING AND ANALYSIS PLAN FOR SLUDGE LOCATED ON THE
FLOOR AND IN THE PITS OF THE 105-K BASINS**

R. B. Baker, T. L. Welsh, B. J. Makenas, and K. L. Pearce

November 1998

CONTENTS

1.0	SAMPLING OBJECTIVES	5
1.1	PURPOSE	6
1.2	SAMPLING AND ANALYSIS ACTIVITY OBJECTIVE AND GOALS	8
2.0	105-K WEST AND 105-K EAST BASINS STATUS AND SAMPLING INFORMATION	11
2.1	105-K WEST BASIN SLUDGE CHARACTERIZATION STATUS	11
2.2	105-K EAST BASIN SLUDGE CHARACTERIZATION STATUS	12
2.3	SAMPLING INFORMATION	15
2.3.1	Sample Collection and Handling	15
2.3.3	Field Sample Plan Modifications	19
2.3.4	Basis and Locations of Sampling	19
2.3.5	Sample Recovery, Final Compositing, and Preparation at Hot Laboratory	30
3.0	LABORATORY ANALYSIS INSTRUCTIONS	35
3.1	GENERAL	35
3.2	ANALYSES	36
4.0	LABORATORY REPORTING REQUIREMENTS	47
4.1	DATA PACKAGING	47
5.0	FINAL SUMMARY REPORT REQUIREMENTS	49
5.1	STATISTICAL ANALYSIS REQUIREMENTS	49
6.0	SAFETY PLANS	51
6.1	RADIATION/INDUSTRIAL SAFETY	51
6.2	NUCLEAR CRITICALITY SAFETY	51
7.0	GENERAL ALARA PLAN	53
8.0	QUALITY ASSURANCE AND CONTROL PLAN	55
9.0	REFERENCES	57
APPENDIX A	SUMMARY LETTER (DESH-9857199) DOCUMENTING ESTIMATED VOLUMES OF SLUDGE IN K BASINS	61
APPENDIX B	SLUDGE DEPTH ESTIMATES AND SAMPLE LOCATIONS DETAIL FOR K WEST AND K EAST BASINS	77

LIST OF FIGURES

1. Sludge Escaping Out Screened Holes in the Bottom of K East Basin Canister During Typical Move	13
2. Overview of the Sludge Core or Primary Floor Sampling Equipment	16
3. Overview of the Consolidated Floor Sludge Sampling Equipment	17
4. K West Basin Floor and Pit Sample Locations	26
5. K East Basin Floor and Pit Samples	29
6. Flow Chart of Sludge Sample Processing and Analyses	44

LIST OF TABLES

1. General Sampling Scope Proposed by Data Quality Objectives for Current Sampling	12
2. Floor and Pit Sludge Sampling Locations	20
3. Summary of K Basins Floor and Pit Sludge Sampling Projections	21
4. Overall Comparison of Proposed Targets from Data Quality Objectives to Present Sampling Projections for K East and K West Floor and Pit Sludge	22
5. Summary of Sample Primary Uses, Sampler Used, and Compositing	24
6. Summary of Composited Samples	25
7. Proposed Responsible Laboratory and Sample Condition for the Floor and Pit Sludge Samples	33
8. 105-K West and K East Basins Floor and Pit Sludge Analyses	37
9. Analytical Requirements for the 105-K West and East Basins Floor and Pit Sludge Samples	39

LIST OF TERMS

ALARA	As Low As Reasonably Achievable
ANOVA	Analysis of Variance
AEA	Alpha Energy Analysis
Basin	105-K West or East (as used in context) Irradiated Fuel Storage Basin
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CSB	Canister Storage Building
DESH	Duke Engineering & Services Hanford, Inc.
DQO	Data Quality Objective
DSC	Differential Scanning Calorimetry
DST	Double Shell Tank
ECD	Electron Capture Detection
EPA	Environmental Protection Agency
ERDF	Environmental Remediation Disposal Facility
GC	Gas Chromatography
GEA	Gamma Energy Analysis
IC	Ion Chromatography
ICP	Inductively Coupled Plasma
ISE	Ion Specific Electrode
IXM	Ion Exchange Module (at K West Basin)
Main Basin	105-K Irradiated Fuel Storage Basins Exclusive of Remote Pits
MCO	Multi-Canister Overpack
MDL	Minimum Detection Limit
NDT	Nondestructive Testing
NHC	Numatec Hanford Company
PCB	Polychlorinated Biphenyls
PHMC	Project Hanford Management Contract
PNNL	Pacific Northwest National Laboratories
ppb	parts per billion
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; $[(\text{result1} - \text{result2})/ \text{mean}] \times 100$
SAP	Sampling and Analysis Plan
SAR	Safety and Analysis Report
SARP	Safety and Analysis Report for Packaging
SD	Serial Dilution
SFBWP	Sand Filter Backwash Pit
SNFCP	Spent Nuclear Fuel Characterization Project
SOW	Statement of Work
SP	Spike Analysis
SPC	Statistical Process Control
SVOA	Semivolatile Organic Analysis
TC	Total Carbon
TCLP	Toxicity Characterization Leaching Procedure

LIST OF TERMS (Continued)

TGA	Thermal Gravimetric Analysis
TIC	Total Inorganic Carbon
TICs	Tentatively Identified Compounds
TIMS	Thermal Ionization Mass Spectroscopy
TLD	Thermoluminescent Dosimeter
TOC	Total Organic Carbon
TSCA	Toxic Substances Control Act
TWRS	Tank Waste Remediation System
VOA	Volatile Organic Analysis
WHC	Westinghouse Hanford Company
XRD	X-Ray Diffraction

SAMPLING AND ANALYSIS PLAN FOR SLUDGE LOCATED ON THE FLOOR AND IN THE PITS OF THE 105-K BASINS

1.0 SAMPLING OBJECTIVES

Notice

As the present document is being completed the Spent Nuclear Fuel (SNF) project management and SNF Sludge Treatment Process subproject have just finished a review of the cost and benefits related to characterization and process testing data supporting treatment of K Basin sludges. The conclusions of this review are: (1) the benefits (i.e., reduced risks, design basis data, sludge material for process testing, etc.) do not warrant any further sampling or characterization of sludges in the K West Basin and (2) the additional sampling at K East Basin is warranted and should be completed. The project basis for this is the assumption the K East Basin sludge behavior conservatively bounds that of the sludges at the K West Basin. Based on these conclusions no further sampling of sludges will be attempted from the floors or spent fuel storage canisters at K West Basin to support process treatment development and confirmation.

By this notice the reader is being informed that the K West samples will not be taken. In order to maintain required project schedules for the current sludge sampling activities that are imminent, while maintaining consistent basis documentation:

- The present Sampling and Analyses Plan (SAP) retains the references to the original sampling planned for the K West floor sludge.
- The K East Basin floor sludge samples described in the present SAP will be taken now as planned (this will maintain project schedules while being consistent with documentation).
- During the period of time required to take the K East Basin samples, the sampling basis documentation (e.g., data quality objectives, etc.) will be revised reflecting the change of emphasis in sampling (i.e., sampling sludges only at K East Basin).
- Using the revised basis documents the present SAP will be revised as necessary to assure the needs of the Sludge Treatment Process subproject are met for sample material needed for process validation testing.

This latter item may require additional sludge sample material from the K East Basin floor to provide sufficient sludge for validation tests. The equipment for sampling floor sludge at the K Basins will be maintained until the revisions of the basis documents are complete.

1.1 PURPOSE

The data quality objectives (DQOs) for this sludge sampling campaign are provided in Makenas (1998a). 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 (1) located on the floor of the main basin and pits at 105-K West and (2) in selected locations in similar areas at the 105-K East Basin. The objectives for the sampling were rigorously developed by the Spent Nuclear Fuel (SNF) sludge treatment process subproject over the past year and are documented in the general subproject DQO (Pearce 1998a), and the testing strategy documents (Delegard 1998; Flament 1998a), followed by the specific sampling DQO (Makenas 1998) for this SAP. These objectives were further confirmed by the independent review team for the initial sludge process flow diagram (PFD) for treatment (Flament 1998b; Fink 1998). This team strongly encouraged continued characterization of the sludge feed materials to clarify the nominal and bounding cases involved.

The K West and K East Basins floor and pit sludge is generally expected to be a nonhomogeneous mixture of particulate materials containing some fuel element corrosion products, environmental materials such as sand and insects, rack and canister corrosion products, sloughed-off concrete material, paint flakes from racks, and/or fuel element fission products. The K West Basin sludge is expected to be somewhat similar in composition to that found to date in K East (Section 2.2 and Makenas 1996c) but with less fuel element related materials, because the canister barrels have lids and barrels have no slots or holes. K West sludges may also contain loose canister lid seal material (e.g., Grafoil), materials related to sludge like material from other Hanford facilities (e.g., many canisters in K West Basin were sealed in N Basin), ion-exchange-module (IXM)-related materials (differing from K East Basin) that were spilled during basin operations, sand and unique basin material backflushed from the basin sand filter, and basin wall materials including sealer.

Sludge from a number of locations on the K East Basin floor and in the Weasel Pit was sampled in 1995, characterized and documented (Makenas 1996c). A number of the samples in the present campaign are being taken in related locations to provide both supplementary analyses and material required for sludge chemical process treatment testing (Flament 1998a). The original floor sludge sample material taken in 1995 was not envisioned to be used to support a treatment development project. Therefore, the quantity of sample taken at that time was purposely kept close to the quantity needed only for projected characterization analyses at that time (to minimize waste and returns to K Basins). Thus, even with only initial testing for the sludge treatment process completed the available sludge sample material from the 1995 sampling campaign has been depleted in most cases. In addition to the samples used for sludge process development testing, samples are being taken at K East Basin in areas where potentially unique local quantities of sludge have either not been previously sampled or where uncharacterized sludge has been mixed with once-characterized sludge inventories (e.g., Weasel Pit). While these sludges are expected to be similar to those originally sampled in other locations at K East Basin, the components may be different due to different historic activities performed in those new locations (e.g., Dummy Elevator Pit, Sandfilter Backwash Pit, Tech View Pit).

Specifically this SAP contains (1) the plan to acquire the sludge samples from the floors and pits of the K West and K East Basins, and (2) the overall requirements for the analytical laboratories to subsample and analyze these samples.

1. Sample Acquisition Activities: Based on the DQO, which in turn is based on the requirements from the subproject responsible for chemical treatment of the K Basins sludge (Flament 1998a; Pearce 1998a; Delegard 1998), a sampling campaign including 25 locations has been planned and described in this SAP. The sampling will use two sampling methods: (1) the system previously developed (Baker 1995a) using a "single pull" system to vacuum a sludge core sample into containers and (2) a modified sampling system for shallow areas of sludge, that provides for a continuous suction of sample material and water into a container with the excess water exhausted through a fine filter trapping the sludge sample; the system also allows sampling of two or more similar physical locations into one container ("consolidated" samples). Once the samples are pulled by the single pull core sampler into the special sample containers, they will be transported using PAS-1 casks to the Hanford 222-S or 325 Building laboratories. The selected subset of samples taken using the consolidation sampler will use a container that is transported in a Chem-Nuclear cask to the Hanford 325 Building Laboratory (by trans-shipping through the 327 Building Laboratory pool facility).
2. Plan for Detailed Laboratory Analyses of Samples: The second purpose of this SAP is to provide the specific basic requirements for detailed handling and analysis of the sludge samples after completion of initial acquisition activities noted in the previous paragraph. These laboratory requirements form the general scope (i.e., specific laboratory procedures, accuracies, etc.) and criteria (i.e., Quality Assurance, reporting, etc.) for the analyses of the sludge samples. Statements of Work (SOW) will be written to the laboratories to supplement this SAP after the samples are recovered from shipping containers and initial observations are complete. These SOWs will provide specific instructions to the laboratories and confirm exactly which samples are to receive particular analyses and any special handling requirements. The final results of the laboratory analyses and their subsequent evaluation, will provide the final documented data satisfying the needs identified by the DQO (Makenas 1998a).

Basic to this sampling campaign are the specific requirements (or needs) the sludge samples address. The DQO document (Makenas 1998a, Sections 2.4.2 and 2.7) contains this rationale. Additional discussion of these requirements are provided as background in Sections 1.2, 2.1, and 2.2 of this SAP.

The overall status of the past sampling campaigns is provided in Sections 2.1 and 2.2 of this SAP. Additional sampling of sludge in the fuel storage canisters in both basins is planned to follow the floor and pit sludge sampling campaign which is the subject of the present SAP. The sampling of canisters in the future is primarily intended to provide prototypic material for development and design testing activities of the sludge chemical process treatment system or its alternative (e.g., direct disposal of portions of the sludge, etc.).

1.2 SAMPLING AND ANALYSIS ACTIVITY OBJECTIVE AND GOALS

The DQO document (Makenas 1998a) for the present activity summarizes the objectives and logic relating the present sampling effort to key Spent Nuclear Fuel (SNF) project activities. The basic decisions requiring these characterization data are related to (1) "does the previously uncharacterized areas of sludge preclude any of the goals of the sludge treatment process to allow disposition of the resulting streams to the TWRS DST and Environmental Remediation Disposal Facility (ERDF)?," (2) "does the proposed sludge treatment process work on a larger scale?," (3) "does fresh sludge (i.e., unoxidized sludge materials versus the current, circa 1995, floor samples that were stored, and in most cases dried) influence the proposed sludge process?," and (4) "do any of the areas of uncharacterized sludge provide constituents or behavior beyond the bounds previously established for recovery, storage, and handling methods to be used at the K Basins to deliver the sludge to the process treatment facility?." The following enlarge on the DQO specified project decisions being addressed by the present sampling campaign in the context of specific goals:

1. What are the physical properties and composition of the K West Basin and K East Basin floor and pit sludge from previously uncharacterized areas that could impact the envisioned goals of the sludge chemical treatment process?

Sampling Goals: Obtain samples of sludge from those uncharacterized areas that are (1) feasible (physically-able) to sample and (2) contain significant volumes of sludge. The sludge samples will be delivered to the Hanford Analytical Laboratories. Samples from general areas (e.g., single pit, related pit areas, etc.) will be combined where reasonable to form "composite area" samples to maximize the number of basin locations sampled for the amount (i.e., cost) of chemical analyses performed. Data on highly local variations are not available using this approach, but this method is consistent with how the sludge is expected to come to the treatment process. Aliquots of the samples will be subjected to physical property testing, chemical analyses, and analyses for selected isotopic composition at the Hanford Analytical Laboratories. These analyses will provide confirming data for design assumptions being used by the SNF sludge subprojects. [Optionally, in addition, scoping process development tests, specific to the current treatment process, may be performed during these analyses for the sludge process treatment subproject if needs are identified and resources are available.]

2. What are the physical and chemical effects on the envisioned sludge process treatment using "fresh" K Basins sludge samples: (1) with minimal aging (i.e., oxidation of components during drying, etc.) or (2) from basin locations where no historic (i.e., 1995) sludge sample material was available for recently completed validation tests for the sludge process treatment?

Sampling Goals: Obtain representative samples of K West Basin and K East Basin floor and pit sludge (including areas previously sampled and analyzed from K East Basin areas). The "sampling goals" as described in the last portion of the prior paragraph follows similarly for this item.

3. Does the uncharacterized K West Basin and K East Basin floor and pit sludge contain any constituents or combination of constituents that would preclude the use of any processing or treatment alternative that would otherwise be considered as more desirable (i.e., cost effective, timely, ALARA, safer, etc.) than the current chemical treatment process method? It is currently planned to reassess options for treatment to confirm the chemical treatment method for all sludges is the best choice from an engineering and cost standpoint.

Sampling Goals: Obtain representative samples from uncharacterized areas of the basins. Aliquots 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 of importance to TWRS (e.g., Fowler 1995a, 1995b; Alderman 1997; Williams 1996) and also provide estimates of analyte concentrations important to successful handling of sludge as solid waste (Willis 1993). Additional attributes may need to be addressed if other sludge process alternatives (e.g., grinding, direct disposal to solid waste, etc.) are "down-selected" and prove to be sensitive to parameters other than those of the chemical treatment process.

4. Does the currently proposed sludge treatment process work on large scale applications (hot tests using increased volume over the testing recently completed)? While this large scale process testing is beyond scope of the present DQO and SAP, the present sampling scope does include providing the required quantity of fresh "non-canister" sludge material for all subsequent final validation and large scale hot cell testing of the sludge treatment process.

Sampling Goals: The demonstration of the current chemical process treatment on a larger scale requires the quantities of non-canister sludge anticipated for testing by the sludge treatment process subproject (Makenas 1998a; Delegard 1998). These sample quantities would include fresh sludge from both previously (i.e., 1995) sampled areas and areas not sampled to-date. The material would be sampled from representative locations as defined by the DQO. The samples from the same general areas (e.g., within the same pit, same floor area, etc.) would be combined to form samples representative of larger physical areas (as suggested by the sludge process treatment development subproject staff). Aliquots of the resulting sludge sample material would then be analyzed through a set of basic chemical and physical analyses to provide the basis of their use in a process development testing matrix for large scale process demonstration. Such testing would also consider similarities and differences between the historic (i.e., 1995) sludge sample material and the "fresh" sludge (currently being obtained), in order to relate the results of prior and future testing.

5. Are the current choices of equipment reasonable to retrieve, handle (e.g., pump, etc.), store, and process sludge within K Basins?

Sampling Goals: Obtain fluid and rheological properties which characterize K West Basin and K East Basin sludges not previously sampled to allow accurate development and validation of designs for sludge handling equipment (i.e., transport, processing, filtering, etc.). This goal could also provide required information for developing suitable simulants for equipment development.

6. Can a nondestructive 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 floor and pit sludge handling and receipt? (This was not directly addressed in the DQO but is inferred in Table 2.4 of that document under isotopes of europium).

Sampling Goals: Obtain contingent data for the potential development of relationships between fission product radioisotopes (measurable by gamma scan methods translatable to field applications) and plutonium concentration (measured by analytical laboratory methods) in the 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. The data for europium isotopes can come from the gamma scan analyses being run for other objectives at essentially "no cost" and collected as a future contingent to respond to this issue.

In addition to the large-scale testing of the sludge treatment process, there will be the need for follow-on verification related to the products of the treatment process being directly demonstrated as acceptable to the receivers of the final waste streams. At this time the two receivers would be (1) TWRS double shell tanks (DSTs)-slated to receive the sludge treatment product stream and (2) ERDF-slated for the solid process waste stream.

2.0 105-K WEST AND 105-K EAST BASINS STATUS AND SAMPLING INFORMATION

2.1 105-K WEST BASIN SLUDGE CHARACTERIZATION STATUS

The 105-K West Basin was designed and constructed in 1950 to 1951. The purpose of the basin was to receive and store irradiated fuel from the K West 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 West reactor stopped the irradiation of fuel in 1971. In 1976, the basins started receiving irradiated N Reactor fuel from the 105-N Basin. The fuel was stored in the basin in closed twin barreled canisters (Baker 1995a). 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 may have 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 may be partially released to the basin when lids are removed. In addition some canisters have lid locking bars that have failed (broken), leaving the barrels "closed" but not necessarily "sealed."

Analyses of sludge in the K West Basin has occurred on (1) sludge sampled from nine selected fuel canisters, (2) small samples of sludge from the Sandfilter Backwash Pit (i.e., North Loadout Pit), (3) subsurface sludge analyzed from under cladding, and (4) trace amounts of sludge that accompanied fuel elements removed from canisters in 1995. Item 1 relates to characterization samples that received extensive analyses (Makenas 1998b). Item 2 relates to periodic cursory operations sampling made on the sludge in the Sandfilter Backwash Pit which contains on the order of 4 m³ of sludge (depths of up to about 1 m). Item 3 relates to a series of samples of sludge that were in the shipping container and clung to the fuel elements examined as part of the K Basins fuel element examinations (Makenas 1996c). Because of its possible impact, since it contains about 7% of the sludge at K Basins, it is noted that the appearance of the uncharacterized sludge in the K West North Loadout Pit (Sandfilter Backwash Pit) appears different than other characterized areas. This is described in Dodd (1995). Observed was a gelatinous behavior associated with the release of large bubbles as the sludge was probed for depth measurements.

As part of the prior K West Basin characterization effort gas and water samples were obtained from a number of the sealed canister barrels. Of concern was the gas composition related to hydrogen, cesium fission products in the water, and the levels of corrosion inhibitor remaining in the canister barrel water. Corrosion inhibitor was injected into the barrels as they were initially closed. The DQO document for the canister sludge characterization effort (Makenas 1998b) provides additional background information.

The DQO for the present sampling campaign identifies the detailed status of those areas in the K West Basin that have significant volumes of sludge and if they have been characterized. The information is summarized in Table 1 which provides the base objectives of the sampling as prescribed by the DQO. The volumes of the samples address two basic requirements: (1) the need from uncharacterized locations for chemical and physical characterization analyses and (2) the need of specific sludge sample material for use in treatment process development and validation testing.

Table 1. General Sampling Scope Proposed by Data Quality Objectives for Current Sampling (Table 2.3 of Makenas 1998).

Area	Number of Locations	Target (dry) grams per Sample	Purpose ¹
K East Floor	4 ²	400	Treatment
K East Weasel Pit	2	500	Treatment
K East North Loadout Pit	2	350	Treatment
K East Dummy Elevator Pit	2	300	Characterization and Treatment
K East Tech View Pit	2	200	Characterization and Treatment
K West Floor	5	100	Characterization and Treatment
K West North Loadout Pit	5 ³	200	Characterization and Treatment
K West Tech View Pit	2 ³	200	Characterization and Treatment
K West Dummy Elevator Pit	1	200	Characterization and Treatment
Total	25	6400	Characterization and Treatment

¹Treatment implies the material will be used for process studies.

Characterization implies the samples address a here-to-for uncharacterized area of sludge.

²Includes one sample with intentionally large concentration of resin beads.

³Samples spread between main pit and transfer channel.

2.2 105-K EAST BASIN SLUDGE CHARACTERIZATION STATUS

K East Basin has a general operating history similar to that noted for K West Basin. The differences between the basins are: (1) K East Basin was the first of the two put into operation for N fuel storage, (2) the fuel storage canisters have no lids, (3) and the fuel is in a much more degraded condition. The canister barrels in some instances also have slotted sides and open bottoms that can leak much of the canister sludge as they are moved in the basin during normal handling, Figure 1. The basin itself is uncoated concrete, as opposed to K West Basin where the basin was drained and coated to

Figure 1. Sludge Escaping Out Screened Holes in the Bottom of K East Basin Canister During Typical Move.

seal the concrete prior to the current storage mission. As noted in the DQO and in Appendix A, the K East Basin contains significantly more sludge on its floor and in the pits than K West Basin (about 50 m³ K East versus 7 m³ K West).

The K East Basin main basin floor and Weasel Pit sludges were recently characterized, based on 20 samples taken in 1995 (Makenas 1996c). In addition nine (9) samples of sludge from K East Basin fuel storage canisters were subsequently taken in 1997 (Makenas 1996b). The nine canister samples were taken to compare the mean property measurement values from the canister sludge to those of the floor/pit sludge. The K East Basin floor sludge was also periodically sampled prior to 1995 (Baker 1995b). The analytical results have indicated the floor sludge 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. Some sludge samples from the floor, Weasel Pit, and canisters showed the presence of polychlorinated biphenyls (PCBs). Samples from the canisters also showed sludge samples generated gas both in some of the shipping containers and during the settling studies. These samples contained very high concentrations of fuel (e.g., uranium concentrations in dry canister sludge was high as 65 wt%). The gas formed in the K East canister sludge was found to be hydrogen with traces of fission gases and tritium. This leads to the conclusion that unreacted uranium metal fuel could be present, however no such metal fuel was detected in the samples analyzed by X-Ray diffraction (XRD). This lack of confirmation may result from there only being a comparatively few particles of uranium or that the particles are quickly coated with an oxide layer masking the uranium metal from the XRD analysis.

The sludge in the 105-K East Basin Sand Filter Backwash Pit (SFBWP) and its transfer channel have been sampled periodically by operations to monitor fissile inventory buildup and a special sampling effort was completed in 1994 related to a safety issue (Bechtold 1994; Baker 1995b).

The most recent overall set of measurements of floor sludge depths in the 105-K East Basin main basin were completed in 1994 and have shown that the basin floor is covered with sludge to a depth of 5 cm to 19 cm (Baker 1995a) and that the Weasel Pit is covered with sludge up to a meter or more in depth (Makenas 1996c), see Appendix B, Figure B1. Potential local sources of sludge that could influence the floor sludge characterization were evaluated by the Spent Nuclear Fuel Characterization Project organization (SNFCP) at that time (Baker 1995b). The local sources (besides the environmental sand, insects, etc.) 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, IXM maintenance, etc.) affecting areas near the mouth of the pits (i.e., Weasel Pit, Tech View Pit, Dummy Elevator Pit, North Loadout Pit, and South Loadout Pit).

As noted earlier, detailed sampling and analyses for the floor sludge in the K East Basin main basin and Weasel Pit (Makenas 1996c) agreed with expectations, with the exception of the unexpected identification of PCBs in some of the sludge samples and the quantity of IXM beads found in certain locations.

The DQO for the present sampling campaign identifies the detailed status of those basin areas that have significant volumes of sludge and have not been characterized in the original detailed sampling campaign in 1995. The primary reason these were not sampled at that time was because of physical debris obstructed sampling; in some cases these locations can now be sampled. Several other areas sampled in 1995 or 1994 now either have significant new sludge added to the area (i.e., Weasel Pit) or did not have a full complement of characterization analyses run on them (i.e., Sandfilter Backwash Pit). Finally, because almost all the sludge samples from the prior floor and pit characterization campaign have been depleted during sludge treatment process testing, more material is required for the validation tests from the general basin floor area and Weasel Pit. The information summarized in Table 1 provides the base objectives of the sampling as prescribed by the DQO. As with the K West Basin sampling, the volumes of the samples in K East Basin address the two primary objectives: (1) the need of sludge from areas yet uncharacterized so chemical and physical characterization analyses can be made and (2) the need of the sludge treatment process development subproject related to development and validation testing.

2.3 SAMPLING INFORMATION

2.3.1 Sample Collection and Handling

The sampling at K Basins will be conducted such that the operation does not impact the water quality/air emissions. The sampling apparatus is described in Baker (1998b and 1995c). Two similar systems will be used to acquire samples. The first, the "single pull" sludge core sampler, is the same equipment used for the 1995 K East Basin floor sampling campaign, Figure 2. It utilizes a metal tube (or pipe) to isolate representative core samples of the sludge material on the floor of the main basin and in remote pits. This core is vacuumed into a set of special 4 liter sample bottles on the basin grating above. 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-43-031E [KE] and OP-43-032W [KW]) and Project Work Plan (K Basin PWP-98-001).

The second sampling system, Figure 3, the "consolidated sampler," is similar to the first except it is designed to collect sludge samples from areas where the layer of sludge is relatively shallow and does not provide enough depth for reasonable core sampling (e.g., depths less than about 0.4 in.). This sampler uses a similar nozzle system, but collects a sample into a 10 liter, underwater container (similar to that used for canister sludge sampling, Makenas 1998b). The system does a continuous draw of

Figure 2. Overview of the Sludge Core or Primary Floor Sampling Equipment.

Figure 3. Overview of the Consolidated Floor Sludge Sampling Equipment.

LAYOUT/SCHEMATIC CONSOLIDATED SAMPLER

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material through a fine (e.g., 5 micron) filter, exhausting the filtered water back into the basin pool. Thus, the sampler is expected to capture most particles greater than 5 microns and a portion of those 5 microns or less. The prior floor and canister sludge samplers pull directly into containers, retaining all water and material, that is no filters are used and no water or material is exhausted back to basin (i.e., "single pull"). This was done to assure minimal change in the physical properties of the sludge sample pulled. For the subject shallow depth sludge samples it was judged much more important to obtain significant volumes of material for analysis and testing than the concern for the effects of the filter on physical properties and minor loss of very fine particles.

Each sludge sample container will be labeled or marked with a unique sample number. The SNFCP organization Test Engineer shall verify to ensure the samples are properly identified and the sample location recorded prior to moving the sample from the K Basins area. The SNFCP organization Test Engineer will ensure that the sample custody during transportation will be tracked at all times by "chain-of-custody" protocol, resulting in documented traceability.

After the sludge core or "single pull" samples have been collected into the sample bottles, they will be loaded in the PAS-1 Cask containers per K Basin procedure OP-43-016 "Load and Ship K Basin Sludge Samples in PAS-1 Cask." For the consolidated sampler containers, similar logic will be used based on procedures currently being developed. The containers in this case will be moved from K Basins to the 327 Building facility using the Chem Nuclear cask (instead of the PAS-1 casks), and from there to the 325 Building Laboratory using a Hanford 300 Area inter-building cask.

It is the intent of this SAP to ship the samples to the analytical laboratory within a week of when they are collected. If logistics preclude shipment within one week from the date of sampling, the SNFCP organization Project Coordinator will evaluate the situation to provide applicable storage for the samples. The consolidated sampler containers may be stored at the K Basins in the pool for a longer period of time (i.e., 1 month or more). In the basin pools these containers remain in an environment (i.e., temperature, chemistry, etc.) similar to the sludge under general basin conditions, so the stored samples should maintain their integrity.

Once the sludge samples arrive at the laboratories they will be stored and handled to avoid evaporation as much as possible. 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 should be noted that since hydrogen gas generation has occurred in the canister sludge samples, safety precautions are required in sealing any small containers in laboratories and hot cells.

As in the past campaigns, it is planned that the analytical laboratories will dispose of the remains of the sludge and water samples after the analyses and sludge treatment process development tests are completed. If it is found beneficial, and the operations groups at K Basins concur, decanted water may be returned to the K Basins from the laboratories.

2.3.2 Sample Record Keeping

A vital part of the sampling activity is the assurance that all of the information and data associated with each sample are accurate and verifiable. Therefore, all pertinent information and data collected during the sampling sample breakdown, analyses and reporting 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-43-031E and OP-43-032W), and the QA at the Laboratory doing the sample breakdown, analysis and reporting (see Section 8.0). Chain of custody (COC) forms will be used at each step of collection and transfer by field and laboratory personnel (i.e., required in K Basin Project Work Plan, etc). The SOWs for the sampling effort may also provide additional direction on record keeping.

2.3.3 Field Sample Plan Modifications

Any modifications to the sludge sampling plan made in the field must be made/approved by the SNFCP organization Test Engineer and/or SNFCP Project Coordinator and documented. Changes affecting quality related information will include QA approval.

2.3.4 Basis and Locations of Sampling

Samples will be taken from locations consistent with the DQO (Makenas 1998a). In addition to the general DQO criteria, the selection process also considered: (1) accessibility of the areas by the sampling crew at the basins, (2) possible disruption of the base sludge layers by recent operation and construction activities, and (3) current safety restrictions at the basins. As in past sampling campaigns, a criterion was used to sample in reasonably accessible locations (i.e., sampling through grating slots--no grating removal, sampling around debris--no major debris removal, and sampling around operation obstructions--not removing current restrictions around floor drain valves, etc.). Using these criteria were judged to have no significant impacts on the quality of the samples with respect to their use. This follows since the sludge within the areas being sampled is expected to be uniformly deposited across these areas due to the expected dispersion of the material through the pool of water.

Tables 2 and 3 define the samples and corresponding intended locations. In two locations, the total number of samples taken was changed slightly from the DQO. Target versus projected sludge volumes are noted in Table 4. One of the two samples to be taken in the K East Basin Tech View Pit was deleted because it was not possible, in the very shallow sludge found present, to get a reasonable quantity of material that would warrant two core samples. Correspondingly, a sample was added to the K East Basin main floor area that will be taken between the barrels of a canister with damaged fuel to provide additional data (only one such sample was taken in 1995 campaign, "KES-0-09"). Such data are of concern to sludge treatment process subproject for evaluating the alternative of recovering the K East Basin floor sludge, grouting it, and sending it directly to solid waste. One of the five samples indicated in the

Table 2. Floor and Pit Sludge Sampling Locations.

Location	Sample Location Identification	Specific Sample Location Information
KW Basin		
Main/East Bay	KW-(1)-C1	Deepest area of this bay (0.1 in. see SD-TI-054).
Main/Central Bay	KW-(2)-C1	Deepest area in this bay (0.1 in. see SD-TI-054). Consolidate during sampling with KW-(1)-C1.
Main/West Bay-North	KW-(3)-C2	Deepest area in NW corner of this bay (0.271 in. see SD-TI-054). Consolidate during sampling with KW-(5)-C2.
Main/West Bay-Center	KW-4	Core sample from deepest position in bay/main basin. Near center of West wall, 0.68 in., see SD-TI-054.
Main/West Bay-South	KW-(5)-C2	Deepest area in SW corner of bay (0.4 in. see SD-TI-054). Consolidate during sampling with KW-(3)-C2.
North Loadout Pit-1	KW-6	Center of Northwest open area beyond restriction in main pit.
North Loadout Pit-2	KW-7	Center of Southwest open area beyond restriction in main pit.
North Loadout Pit Trs-1	KW-8	Center of West half of transfer channel.
North Loadout Pit Trs-2	KW-9	Center of East half of transfer channel.
Dummy Elevator Pit	KW-(10)-C3	Deepest area in pit, consolidate during sampling with KW-(12)-C3.
Tech View Pit South Channel	KE-11	Deepest area in Tech View South trans channel, best effort.
Tech View Pit Main	KE-(12)-C3	Deepest area in main pit (0.11 in., see SD-TI-054). Consolidate during sampling with KW-(10)-C3.
KE Basin		
Main/East Bay	KE-1	Cubical 0168 South side, similar to KES-K-12 location.
Main/East Bay-South	KE-2	Cubical 1065 North side.
Main/West Bay-IXM	KE-3	Same as KES-H-08 location, cubical 6755 only North side, deepest area.
Main/West Bay	KE-4	Cubical 6722 North side, similar to KES-I-15.
North Loadout Pit	KE-5	Middle unrestricted area of main pit.
North Loadout Pit Trs	KE-6	Middle of transfer channel.
Dummy Elevator Pit-1	KE-7	Best effort middle of East half of pit.
Dummy Elevator Pit-2	KE-8	Best effort middle of West half of pit.
Weasel Pit-East	KE-9	Midway between where KES-R-18 and KES-S-19 were taken.
Weasel Pit-West	KE-10	Midway between where KES-P-16 and KES-Q-17 were taken.
Tech View Pit North Channel	KE-11	North channel midway between doors, along South wall, deepest sludge.
Main Basin East-IXM-2	KE-12	Second IXM main floor sample, cubical 6756 to deepest area, similar to KES-H-08.
Between Barrels	KE-13	Cubical 4573, between barrels of slotted canister with damaged fuel.

Note: The KES samples are from 1995 sampling, see WHC-SP-1182.

Table 3. Summary of K Basins Floor and Pit Sludge Sampling Projections.

Location	Sample Location Identification	Similar Samples from 1995 Campaign	Isolation Tube Diameter (in.)	Assumed Sludge Depth (in.)	Calculated Volume in Sample (mL)	Projected Recovered Sludge Volume (mL)	Projected Recovered Sludge Weight (g)	Projected Recovered Sludge Weight (lb)	Number of Consolidated Sample Containers	Estimated Number of Sample Bottles	Identification for Final Sample Compositing at Laboratory
K Basin											
Main/West Bay	KW-11-C1		Consolidated	0.075	350	263	341	205	0.5	N/A	FE1
Main/Central Bay	KW-12-C1		Consolidated	0.125	350	263	341	205	0.5	N/A	FE1
Main/West Bay-North	KW-13-C1		Consolidated	0.271	350	263	341	205	0.5	N/A	FE1
Main/West Bay-Center	KW-4		4	0.077	141	80	104	63	2	2	FE2
Main/West Bay-South	KW-15-C2		Consolidated	0.400	350	263	341	205	0.5	N/A	FE1
North Loadout Pit-1	KW-6		2	17.717	912	519	675	405	4	4	FE5
North Loadout Pit-2	KW-7		2	15.746	811	461	600	360	4	4	FE5
North Loadout Pit-Tra-1	KW-8		2	8.800	453	258	335	201	2	2	FE5
North Loadout Pit-Tra-2	KW-9		2	8.800	453	258	335	201	2	2	FE5
Dumpy Elevator Pit	KW-10-C3		Consolidated	0.300	350	263	341	205	0.5	N/A	FE4
Tech View Pit South Channel	KW-11		4	0.600	125	94	122	75	2	2	FE5
Tech View Pit Main	KW-12-C3		Consolidated	0.105	350	263	341	205	0.5	N/A	FE4
Subtotal K West					1695	1245	1619	1031	3	16	
K Basin											
Main/West Bay	KE-1	KE5-K-12	4	5.600	1168	665	864	519		4	FE1
Main/West Bay-South	KE-2	KE5-K-05	4	2.500	521	297	386	231	2	2	FE1
Main/West Bay-13M	KE-3	KE5-K-08	4	3.700	772	429	571	343	4	4	FE2
Main/West Bay	KE-4	KE5-K-15	4	4.600	960	546	710	426	2	2	FE1
North Loadout Pit	KE-5		2	35.000	1699	967	1257	754	4	4	FE3
North Loadout Pit-Tra	KE-6		1	24.000	309	176	229	137	2	2	FE3
Dumpy Elevator Pit-1	KE-7		2	5.000	257	146	190	114	2	2	FE4
Dumpy Elevator Pit-2	KE-8		4	5.000	1643	594	772	463	4	4	FE4
Wessel Pit-East	KE-9	R-18/E-19	2	31.000	1594	908	1181	708	4	4	FE5
Wessel Pit-West	KE-10	P-16/B17	2	21.000	1081	615	800	489	4	4	FE5
Tech View Pit North Channel	KE-11		4	1.250	261	149	193	116	2	2	FE4
Main Basin East-13M-2	KE-12	KE5-K-08	4	3.700	772	429	571	343	2	2	FE2
Between Barrels	KE-13	KE5-K-09	1	3.000	39	22	29	17	1	1	FE7
Blank	TELC										TELC
Subtotal K East					10478	5933	7751	4651	9	39	
Grand Totals					15473	9637	11970	7182	3	55	11 Composites

Table 4. Overall Comparison of Proposed Targets from Data Quality Objectives to Present Sampling Projections for K East and K West Floor and Pit Sludge.

	DQO					SAP		
	Number of Sample Locations	Target Dry Weight per Location (g)	Target Total Dry Weight per Area (g)	Target Total As-Settled Weight (g)	Target Total Volume As-Settled (mℓ)	Calculated Total Recovered Volume As-Settled (mℓ)	Calculated Total Sludge Dry Weight (mℓ)	Number of Sample Locations
KE Floor	3	400	1200	2000	1538	1530	1193	4
KE Floor, IXM	1	400	400	667	513	878	685	2
KE Weasel Pit	2	500	1000	1667	1282	1523	1188	2
KE North Loadout Pit	2	350	700	1167	897	1143	891	2
KE Dummy Elevator Pit	2	300	600	1000	769	740	577	2
KE Tech View Pit	2	200	400	667	513	148	116	1
KW Floor	5	100	500	833	641	1130	882	5
KW North Loadout Pit	5	200	1000	1667	1282	1496	1167	4
KW Tech View Pit	2	200	400	667	513	356	278	2
KW Dummy Elevator Pit	1	200	200	333	256	263	205	1
Total	25		6400	10667	8205	9208	7182	25

Notes: Assumed conversion factors:

Density g/mℓ as-settled 1.3

Factor from wet to dry 0.6

Recovery factor cores 0.57 (based partially on 1995, total recovery to laboratory)

Recovery factor consolidated 0.75 Estimate

DQO for the K West Basin North Loadout Pit was deleted. This was done because sufficient volume of material could be obtained from four samples (conserving resources) and four samples was judged sufficient to characterize the limited area accessible.

To maximize the number of locations sampled within the resources available, sampling is planned at 25 locations but this material will be composited into 12 samples prior to starting detailed analyses in the laboratories. The DQO calls for not combining material from different pits. This was followed except for very shallow areas. Here it was judged much more advantageous to obtain a composite than have to ignore the area because of resources precluding so many analyses. Further, the only such composite (from K West Dummy Elevator Pit and Tech View Pit) is from pit areas that are in open communication (i.e., no doors or screens in between) with the main basin.

The conversion factors and constants used throughout the present SAP for sludge density; relating wet and dry sludge; and expected recovery factors for the samples is provided in the notes of Table 4. These are based on past experience from sampling floor sludge in K East Basin (Makenas 1996c).

Sludge depths in Table 3 are based on the historic data (K East Basin measurements made in 1994 and 1995, K West Basin measurements made in 1997). Operations at K Basin may have affected sludge depths noted here and current actual depths could vary. As sludge samples are taken during the campaign the sludge depths of the sampled area will be confirmed using the scales provided on the outside of the isolation tubes used in the core sampler or measured with an auxiliary measurement scale when the consolidated sampler is used. Generally, it is intended to take samples where local area depths of sludge are greatest (reflecting the greatest amounts of material in the basins). It will be an option for the SNFCP Test Engineer to move the location of a sample in a specific area if the sludge depth appears more appropriate at a nearby location. Final sampling locations will be documented in the logbook and video taping will be performed as samples are taken. As with prior campaigns (e.g., Makenas 1996c) the final evaluation report for these sampling activities will document the locations and condition of the sludge in the areas sampled.

K West Basin--Twelve sample locations, Figure 4, were selected from K West Basin, Tables 2 and 3. Six of these will be taken with the primary floor sludge sampling device using the isolation tubes to isolate cores of sludge (i.e., single pull core sampler). Six locations will be sampled with the consolidated sampling equipment (because of the comparatively shallow depth of sludge in these areas). With the consolidation sampler the sludge from two similar physical locations in the basin will be drawn into a single sampling container (i.e., they will be consolidated), see Section 2.5.1. For the K West Basin campaign this will result in three consolidated sample containers containing the material from the six K West Basin and open pit locations. See Tables 5 and 6 for summary of which sampler is being used to draw specific samples.

Main Basin Floor: Samples will be drawn from five locations, Figure 4, of the main basin floor at K West Basin. Figure B1 of Appendix B indicates the depths involved, based on measurements made in

Table 5. Summary of Sample Primary Uses, Sampler Used, and Compositing.

Location	Sample Location Identification	Primary Sample Container Identification	Initial Characterization Sample?	Sludge Process Treatment Sample?	Type Sampling Equipment Used	Final Composite Sample Identification
KW Basin						
KW Basin Main/East Bay	KW-(1)-C1	WC1	Y	Y	Consolidated	FW1
Main/Central Bay	KW-(2)-C1	WC1	Y	Y	Consolidated	FW1
Main/West Bay-North	KW-(3)-C2	WC2	Y	Y	Consolidated	FW1
Main/West Bay-Center	KW-4	WB1	Y	Y	Core	FW2
Main/West Bay-South	KW-(5)-C2	WC2	Y	Y	Consolidated	FW1
North Loadout Pit-1	KW-6	WB2	Y	Y	Core	FW3
North Loadout Pit-2	KW-7	WB3	Y	Y	Core	FW3
North Loadout Pit Trs-1	KW-8	WB4	Y	Y	Core	FW3
North Loadout Pit Trs-2	KW-9	WB5	Y	Y	Core	FW3
Dummy Elevator Pit	KW-(10)-C3	WC3	Y	Y	Consolidated	FW4
Tech View Pit South Channel	KW-11	WB6	Y	Y	Core	FW5
Tech View Pit Main	KW-(12)-C3	WC1	Y	Y	Consolidated	FW4
KE Basin						
Main/East Bay	KE-1	EB1	N	Y	Core	FE1
Main/East Bay-South	KE-2	EB2	N	Y	Core	FE1
Main/West Bay-IXM	KE-3	EB3	N	Y	Core	FE2
Main/West Bay	KE-4	EB4	N	Y	Core	FE1
North Loadout Pit	KE-5	EB5	N	Y	Core	FE3
North Loadout Pit Trs	KE-6	EB6	N	Y	Core	FE3
Dummy Elevator Pit-1	KE-7	EB7	Y	Y	Core	FE4
Dummy Elevator Pit-2	KE-8	EB8	Y	Y	Core	FE4
Weasel Pit-East	KE-9	EB9	N	Y	Core	FE5
Weasel Pit-West	KE-10	EB10	N	Y	Core	FE5
Tech View Pit North Channel	KE-11	EB11	Y	Y	Core	FE6
Main Basin East-IXM-2	KE-12	EB12	N	Y	Core	FE2
Between Barrels	KE-13	EB13	N	Y	Core	FE7
Blank	KBLK	KBLK	N/A	N/A	Core	KBLK

Table 6. Summary of Composited Samples.

Sample Type	Sample Designation	Primary Samples Used to Make Composite	Area Represented
Core Sludge Sampler			
Normal	FW2	KW-4	KW deepest floor
Research	FW3	KW-6, KW-7, KW-8, KW-9	KW North loadout pit and channel
Normal	FW5	KW-11	KW tech view pit
Normal	FE1	KE-1, KE-2, KE-4	KE floor, general
Normal	FE2	KE-3, KE-12	KE floor, IXM beads
Research	FE3	KE-5, KE-6	KE North loadout pit and channel
Normal	FE4	KE-7, KE-8	KE dummy elevator pit
Normal	FE5	KE-9, KE-10	KE weasel pit
Normal	FE6	KE-11	KE Tech view pit
Normal	FE7	KE-13	KE floor between slotted barrels with damaged fuel
Normal	KBLK	Equipment blank water	KE equipment water sample
Consolidated Sludge Sampler			
Normal	FW1	KW-(1)-C1, KW-(2)-C1, KW-(3)-C2, KW-(5)-C2	KW floor, East bay and Center bay
Normal	FW5	KW-(10)-C3, KW-(12)-C3	KW dummy elevator pit and tech view pit

Figure 4. K West Basin Floor and Pit Sample Locations.

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1997 (Maassen 1997). The consolidated sampling equipment will be used to draw two approximately 350 ml (i.e., original wet sample volume) samples of sludge from one location in the East Bay and one in the Central Bay, into a single sample container. These locations were selected because they are the deepest observed (Maassen 1997) in these bays with very shallow sludge depths. The West Bay was observed to have deeper areas of sludge than the East and Central Bays. A second consolidated sample container will be used to draw similar quantities of sludge from two locations in the West Bay, one from the Northwest and one from the Southwest areas. These samples will be representative of two of the deeper (though still comparatively shallow) areas in the main basin. The consolidated sample target volumes were intentionally made larger than required by DQO to provide for contingency during sample recovery in the laboratory.

Finally, a fifth location will be sampled in the main basin using the primary (i.e., core) sampling device, drawing a sample using the largest diameter isolation tube (i.e., 4 in. diameter). This sample will be taken at the location in the West Bay where the sludge is indicated to be the deepest on the main floor (Maassen 1997) see Figure B1. It is intended that this sample provide any required data on slurry rheology properties for the main basin floor sludge, since it will have the material most representative for this type characterization (versus the consolidated sampler material that may not include a portion of the population of very fine, less than 5 microns, particle sizes). The combination of using the consolidated sampler to provide the two larger volume samples from four areas (primarily to support pretreatment process development) and one core sample (to provide a base check using single pull sample material) provides for the intended sampling objectives.

North Loadout (or Sandfilter Backwash) Pit: The North Loadout Pit will have a total of four samples taken from it, Figure B2. Sludge in this pit has not been previously characterized, see Section 2.1, and it represents the greatest volume of sludge in K West Basin, Appendix A. Sludge depth measurements, Figure B3, for this pit were reported by Dodd (1995). Routine operations have taken very small dilute samples of sludge to characterize potential fissile material inventory changes as backflushes have occurred. For the present characterization campaign two samples will be taken from the transfer channel; dividing the channel into a front (East) half and a back (West) half then taking samples near the middle of these areas. Two samples will be taken from the main pit area; spanning the available open area outside the floor drain valve restricted area, (a 6 ft radius circle encompassing the floor drain valves, see Figure B2). The primary intent is to collect sufficient sludge material for sludge treatment process development testing, since it is not expected that there will be significant variation in the sludge composition across the pit because of the way the material is backflushed into the pit with large volumes of water. However, it is expected that the sludge composition will vary by depth. To provide some spatial variation indicator, and since a limited number of sizes of isolation tubes are available, two samples are being taken in the relatively small area of the main pit.

South Loadout Pit: The sludge in the South Loadout Pit was pumped to the South Channel of the Tech View Pit in 1997, see Appendix A.

Dummy Elevator Pit and Tech View Pit: The Dummy Elevator Pit has an estimated sludge volume of only 1.4 ft³, with a maximum estimated depth of 0.3 in. (Maassen 1997). The Tech View Pit also has a relatively small quantity of sludge, total of 2.4 ft³. It was judged it would be adequate for these pits to use one consolidated sample including: approximately 350 ml of sludge from the deepest location in the Dummy Elevator Pit and a similar quantity from the deepest location in the main pit of the Tech View Pit. While the DQO recommended that sludge samples from different pits not be combined, in the case of these two "open" (to the main basin) pit areas with only shallow quantities of sludge, it was judged more important to obtain some information and significant material from both areas than potentially eliminating one due to resource constraints. Because the consolidated sampler equipment cannot be used in the South Channel of the Tech View Pit (area is screened off from main basin) a core sample is being taken in this shallow area. Figures B4 and B5 provide the sampling locations and Figure B1 provides selected sludge depths for reference.

Weasel Pit and Discharge Chute: It was judged with a maximum observed depth of 0.1 in., Figure B1, that neither of these relatively small areas warrant sampling (Makenas 1998a).

K East Basin--In K East Basin 13 samples will be taken, Figure 5. All the samples being taken in K East Basin will be drawn with the primary (i.e., core) floor sludge sampler.

Main Basin Floor: Cubicle locations in two of the three bays were selected for sampling based on sludge depth and past sampling locations, Figure 5. The sample locations were generally chosen from the deeper areas, Figure B6, so the sum of the samples represent the largest quantities of sludge on the K East Basin floor. Similarly, general locations to those used in prior (Figure B7) campaigns (i.e., opposite sides of the same empty cubicals sampled in 1995) were chosen in several cases; this was done to better assure accessibility and provide some linkage of the sludge from prior analysis results to the current material. Two samples, KE-3 and KE-12 were chosen specifically to provide samples with a high percentage (e.g., 75%) of IXM beads, similar to sample KES-H-08 from the sampling made in 1995. These samples with beads seek to provide the quantity identified in DQO as well as providing, on a best effort basis, some additional material for testing as identified in Delegard (1998) as needed.

Sample KE-13 was selected, as requested by the sludge treatment process development subproject staff, to be between the barrels of a canister with damaged fuel. This will provide a second data point under these conditions (one taken in 1995, "KES-0-09"). KE-13 is being taken from the floor in the Center Bay. The overall volume of sludge in this bay is less than the other two bays and a portion (i.e., the central area) of the sludge originally located in this bay was pumped to the Weasel Pit after 1995. Therefore, the two samples to come from the Weasel Pit in the present campaign should include some material from this bay.

Figure 5. K East Basin Floor and Pit Samples.

North Loadout Pit: A sample will be drawn from the center of the non-restricted area of the main pit of the North Loadout Pit. A second sample will be taken from the transfer channel near its center, Figures B8 and B9. These samples are primarily to provide material for the sludge treatment process development. Since the material in these areas should be reasonably mixed as backflushes are made of the sandfilter, only a single sample from each general location is required.

South Loadout Pit and Discharge Chute: No significant sludge remains in the South Loadout Pit or the Discharge Chute. The South Loadout Pit sludge was pumped to the Weasel Pit after 1995. The Discharge Chute was pumped to the Weasel Pit prior to the 1995 sampling campaign (Welsh 1995).

Dummy Elevator Pit: The Dummy Elevator Pit was not sampled during the 1995 sampling campaign because there was too much debris in it to allow access to the floor by the sampling equipment. Some of the debris has now been removed and significant sludge is projected to reside in the pit (though this has not been directly measured, Appendix A). Two samples will be taken from the Dummy Elevator Pit on a "best effort" basis (there is still significant debris) during the present sampling campaign. One sample will be attempted near the center of the East half of the pit and one in the West half of the pit, Figure B10.

Weasel Pit: Since the 1995 characterization campaign the Weasel Pit has had sludge from the South Loadout Pit and the center area of the Center Bay added to it. Figure B11 indicates the sludge depths in 1995 and Figure B12 the sample locations used in 1995. For the current sampling campaign of the Weasel Pit two samples will be taken, Figure B13. One sample will be taken between the location of the samples designated KES-Q-17 and KES-P-18 in the 1995 campaign, and similarly the second sample will be taken between the locations of the KES-S-19 and KES-R-18 samples. Because harder layers were encountered at different depths during the 1995 sampling of this pit (Makenas 1996c), it is planned that the particular locations previously sampled will be avoided during the current sampling to better ensure representative core samples are obtained (i.e., from undisturbed areas).

Tech View Pit: The Tech View Pit sludge was not sampled in the 1995 campaign. One sample will be drawn during the current campaign. This will be located, Figure B13, where the deeper area of sludge was observed previously in the North Channel (Baker 1995a).

2.3.5 Sample Recovery, Final Compositing, and Preparation at Hot Laboratory

Procedure(s) or test instructions for the recovery of the sample material from the shipping containers (i.e., either 4 liter bottles or 10 liter sludge containers) and subsequent sample preparation and handling will be prepared by the designated Hanford Laboratory. These procedures will be similar to those used for the K East Basin floor sludge samples (Bechtold 1994). The sample preparation and handling procedure will contain the detailed instructions

necessary to safely and properly record observations, monitor, settle, decant and subsample (as needed), digest (if necessary), handle, package, and label each sample and its derivatives in the hot cell/hood. The SNFPC organization Project Coordinator will concur with the procedures or test instructions prior to their application.

For the present floor and pit sludge sampling campaign, it is planned to combine or composite sample material from similar basin locations (e.g., same floor area, same pit, etc.) prior to initiating the detailed analyses or use of the sample sludge materials for testing. In the case of the samples coming from the primary (i.e., core) sampler, this means combining the sludge sample materials from several locations together into a single initial container (e.g., combining sludge from sample bottles from several locations into a graduated cylinder) at the laboratory hot cell. Then proceeding with subsequent characterization analyses and pretreatment testing from this composite sample. In the case of the consolidated sampler the sludge sample material will be combined as it is collected from the two or three basin locations sampled. Therefore, the material need only be removed from the sample container and transferred to laboratory container to begin analyses (e.g., settling, etc.). Tables 3, 5, and 6 show the samples that are currently planned for compositing, this may be revised in the SOWs if the condition of the actual sample material recovered warrants a different approach.

Samples will be designated either "normal" or "research" after settling, the handling of 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 prior K East Basin 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. Typically two or three layers were found in the K East Basin sludge samples. Table 5 indicate those samples initially selected for research samples, however, this selection could be revised in subsequent SOWs after the material has gone through settling studies.

Analysis of all the sludge samples by layers was considered. It was, however, concluded the limited number of research samples would be sufficient. Factors involved in this decision (not analyzing by layers in all samples) included: the sludge feed streams will be mixed during recovery and transport to the sludge treatment equipment, the difficulty in objectively determining layers, settling differences between the evolving material in the basin versus using all the material at once 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. The research samples were chosen from the North Loadout Pit because these represent the largest volumes of sludge that have not been characterized related to fluid properties and information on layering (such as densities).

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 subsequent SOWs, the liquid portion and the wet sludge should be sampled for the various analyses. Table 7 indicates the condition of the sample material for the various analyses.

It is very important that the analytical laboratories measure and record the evolving densities (i.e., volumes and weights) of the sludge samples (i.e., as-settled, centrifuged, partially dried, dried, etc.). These densities will allow both comparison to past work and the state of sludge as collected.

Table 7. Proposed Responsible Laboratory and Sample Condition for the Floor and Pit Sludge Samples.

Analysis ¹	Proposed Laboratory ²	Acceptable to Acid Digest ?	Acceptable to Dry ?	Comments
<u>Rad-Chemistry</u> ³				
Pu Isotopes ⁴	222-S	Yes	Yes	²³⁸ Pu, ^{239/240} Pu
²⁴¹ Am, ^{243/244} Cm	222-S	Yes	Yes	
U Total	222-S	Yes	Yes	
U Isotopes	325	Yes	Yes	²³³ U, ²³⁴ U, ²³⁵ U, ²³⁶ U, ²³⁸ U
GEA	222-S	Yes	Yes	γ, Cs, and Eu Isotopes
Total Alpha	222-S	Yes	Yes	
Total Beta	222-S	Yes	Yes	
⁹⁹ Tc, ⁹⁰ Sr, ²³⁷ Np	222-S	Yes	Yes	
<u>General Chemistry</u>				
ICP Metals	222-S	Yes	Yes	
IC Ions	222-S	No	No	
TOC/TIC/TC	222-S	No	No	
SVOA/PCB	325	No	No	
pH	222-S	No	No	
Caustic Demand	222-S	No	No	
<u>Physical Properties</u>				
Settling Rate	325	No	No	Report Full Curves
Appearance	325	No	No	
Viscosity	325	No	No	
Density (ρ)	325	No	No	
Settled	325	No	No	
Dry Particle	325	No	No	
XRD				
Pyrophoric Material	325	No	Yes	
Residuals (digest)	325	No	Yes	
Particle Size	325	No	No	
Particle Shape	325	No	No	
Zeta Potential	325	No	No	
<u>Thermal Properties</u>				
TGA/DSC	325	No	No	Report Full Thermogram
<u>Process Testing Samples</u>	325, 222-S	No	No	

¹Some sludge materials have PCB levels that may require a TSCA permit for further process testing.

²222-S is the Hanford 222-S Laboratory facility, 325 is the Hanford/PNNL 325 Building Radiochemistry Process Development Laboratory.

³Shipping between laboratories may require 325 Building to run selected radiochemistry to support shipments.

⁴Report ²³⁸Pu, ²³⁹Pu, ²⁴⁰Pu, and ²⁴¹Pu if generated as part of other analyses.

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3.0 LABORATORY ANALYSIS INSTRUCTIONS

3.1 GENERAL

Specific instructions on processing of each sludge sample and composite sludge samples in the analytical laboratories will be provided by Statements of Work (SOW) from the SNFCP organization prior to shipment of samples or after the observation of the recovered samples. For shipping subsamples between laboratories sludge samples may be analyzed initially to ascertain properties such as fissile and fission product content for criticality, shielding and accountability.

Each sludge shipping container (either 4 liter bottle or 10 liter container) will be received by the analytical laboratories. The sludge samples will then be recovered from the shipping containers and prepared for settling studies, Section 2.3.5. Certain samples will be combined to provide composites, either as indicated in Tables 5 and 6, or as directed by subsequent SOWs. During the settling studies, any observations of gas generation from the sludge material should be immediately noted to the SNFCP organization. If significant, an evaluation will be conducted to determine if gas monitoring is required. High resolution video taping will be made of samples during settling studies and if gas generation is observed.

Selected sludge physical properties (e.g., settling rate, density, etc.) will be obtained prior to any subsampling efforts. Other properties (such as viscosity and particle size/shape) must be determined from aliquots taken prior to any other preparation steps. Prior to heating or acid digestion, aliquots of both the liquid and the solids will be obtained from the appropriate samples. These aliquots will be used by the laboratories 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 to minimize sample heterogeneity, will then be processed through an acid digest procedure. An aliquot of the acid digested sludge shall be prepared and sent to the laboratory. Any residual solids which remain after the acid digestion step will be analyzed by XRD. If there is a sufficient quantity the residuals will also be subjected to caustic fusion and further ICP, radiochemistry, and XRD analyses. The acid digest (i.e., hot plate and beaker) used will be the same as that previously used for the K East Basin canister sludge analyses (Makenas 1997) or as specified in subsequent SOWs. The general condition of the sample for the different analyses is provided in Table 7.

The laboratory will analyze each aliquot in duplicate or as directed in Section 8.0 of the present SAP or subsequent SOWs. 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 SOW 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 SNFCP organization Project Coordinator.

A flowchart showing the general analysis scheme for the sludge samples in the laboratory is presented in Figure 6. The analytical tests for the analytical laboratory work are shown in Table 8. The reason or need for each of these analyses is defined in the DQO (Makenas 1998a). In general the samples designated as "normal" will receive all the analyses in Table 9 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 each in general receive all the analyses in Tables 8 and 9 including rheology measurements (also see Sections 2.3.5 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 knowledge." As discussed in the DQO document, pyrophoricity of the sludge is being assessed from the XRD measurements for metallic zirconium, metallic uranium, and uranium hydrides. Exothermic reactions may be evaluated by differential scanning calorimetry (DSC).

An additional objective of this sampling campaign is to provide representative sludge material to the K Basin sludge treatment process development subproject testing activities. Any material not directly used for characterization analyses will be kept as much as possible in an "as-collected" state during any storage in the hot cells and shipping activities. The material should not be allowed to dry out (oxidizing some materials and causing physical changes), be mixed with non-prototypic materials (e.g., de-ionized water, etc.) or subjected to extremes in environment (i.e., higher than room temperatures, high pH, contact with potentially contaminating surfaces, etc.). The disposition of the sample material will be the subject of a SOW after the samples are obtained and initially observed. This SOW will provide more direction on storage environment requirements. The actual testing and analyses related to sludge process development testing itself will be covered under separate SOWs from the sludge treatment process subproject. The exception to this is for scoping process testing done with the initial samples in direct cooperation with the sludge treatment process subproject, in order to improve the economics of the overall project.

3.2 ANALYSES

Table 9 shows the analytes, methods, and the laboratory procedures required for the analysis of the samples. The table also lists 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 the 1995 K East Basin floor sludge sampling (Miller 1995), the detection levels, precision, and accuracy values shown in Table 9 will have to be re-evaluated. The SNFCP Project Coordinator will be notified of the condition and a decision will be made and documented on how to proceed. One reagent blank will be run if required by the laboratory procedure for each analytical

Table 8. 105-K West and K East Basins Floor and Pit Sludge Analyses.

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, ²²⁶ Ra, ⁹⁵ Nb, 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 suite of analyses)
IC Analyses	IC	PO ₄ ³⁻ , SO ₄ ²⁻ , F ⁻ , Cl ⁻ , Br ⁻
Tc-99	Solvent extraction with liquid scintillation counter	⁹⁹ Tc
Organics	SVOA	Any TICs found in the analysis,
Endothermic/Exothermic Reactions (Optional)	DSC/TGA (use TWRS method with nitrogen and air cover gas)	DSC/TGA include annotated thermogram
Settling Rate	Settling columns and video camera	Volume settled versus time, video tape

Table 8. 105-K West and K East Basins Floor and Pit Sludge Analyses. (Continued)

Requested Analysis	Analysis Technique	Constituents to be Reported
Polychlorinated Biphenyls (PCB)	PCB as near EPA methods as reasonable	PCB concentration in indicated solids (1 ppm) and water (0.2 ppb)
Settled Densities	Gravimetric/sample preparation procedure	Settled density, volume, and weight
Residual from Digest	Gravimetric/sample preparation procedure	Weight percent of original material left after acid digest (wet and dry)
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_2 , and FeO_x
Particle Size Distribution	Particle size analyzer and sieves	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 (Optional)	Potentiometric titration	Buffering capacity
Gas Generation from Settlers (Optional)	Capture measured volume of gas over specific time and analyze for composition	Rate of gas volume generated (as a function of hours), composition of gas released, and temperature of sludge sample
pH	pH probe	pH units
Cyanide	Standard methods	Cyanide concentration

Table 9. Analytical Requirements for the 105-K West and East Basins Floor and Pit Sludge Samples (222-S and 325 Laboratories). (Continued)

Process	Constituents	Procedure	Required MDL ¹	Precision ²	Accuracy ³
Separation, AEA					
	²³⁸ Pu	LA-953-104 PNL-ALO- 417/496/422	8.0 μ Ci/g	$\pm 20\%$	--
	^{239/240} Pu	LA-953-104 PNL-ALO- 417/496/422	3.0 μ Ci/g	$\pm 20\%$	$\pm 25\%$ Sp
	²⁴¹ Am ^{243/244} Cm	LA-953-104 PNL-ALO- 417/496/422	15.0 μ Ci/g 15.0 μ Ci/g	$\pm 20\%$	$\pm 25\%$ Sp Am only
Phosphorescence or Fluorimetric	U total	LA-925-009 PNL-ALO-445	10 μ g/g	$\pm 20\%$	SPC
Thermal Ionization Mass Spectroscopy (TIMS) Plasma Mass Spectroscopy (ICPMS)	U Isotopes, ²³³ U, ²³⁴ U, ²³⁵ U, ²³⁶ U, ²³⁸ U, ²³⁶ Pu, ²³⁹ Pu, ²⁴⁰ Pu, and ²⁴¹ Pu ⁷	PNL-ALO-455 (TIMS) LA-506-101 (ICPMS)	25 μ g/g	$\pm 20\%$	SPC
GEA		LA-548-121 PNL-ALO-450			
	¹³⁴ Cs		0.20 μ Ci/g	$\pm 20\%$	--
	¹³⁷ Cs		0.015 μ Ci/g	$\pm 20\%$	SPC
	⁶⁰ Co		0.010 μ Ci/g	$\pm 20\%$	SPC
	²⁴¹ Am		15.0 μ Ci/g	$\pm 20\%$	--
	¹⁵² Eu		1.5 μ Ci/g	$\pm 20\%$	--
	¹⁵⁴ Eu		1.5 μ Ci/g	$\pm 20\%$	--
	¹⁵⁵ Eu		2.0 μ Ci/g	$\pm 20\%$	--
	²¹² Bi		10.0 μ Ci/g	$\pm 20\%$	--
	²⁰⁸ Tl		30.0 μ Ci/g	$\pm 20\%$	--

¹ Required Method Detection Limit (MDL) 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., $-20\% < \text{RPD} < 20\%$. $\text{RPD} = [(\text{result1} - \text{result2})/\text{mean}] * 100$.

³ All spike recoveries should be within $\pm 25\%$. 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 using same methods as used for K East canister sludge samples or as directed in LOI/SOW. All Aroclor compounds should be identified and quantified.

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 9. Analytical Requirements for the 105-K West and East Basins Floor and Pit Sludge Samples (222-S and 325 Laboratories). (Continued)

Process	Constituents	Procedure	Required MDL ¹	Precision ²	Accuracy ³
	¹²⁵ Sb		10.0 $\mu\text{Ci/g}$	$\pm 20\%$	--
	¹⁰⁶ Ru/Rh		35.0 $\mu\text{Ci/g}$	$\pm 20\%$	--
	¹⁴⁴ Ce/Pr		25.0 $\mu\text{Ci/g}$	$\pm 20\%$	--
	⁹⁵ Nb		1.0 $\mu\text{Ci/g}$	$\pm 20\%$	--
	²²⁶ Ra		45.0 $\mu\text{Ci/g}$	$\pm 20\%$	--
Gross Alpha	Total α	LA-508-101 PNL-ALO-420/421	3.0 $\mu\text{Ci/g}$	$\pm 20\%$	$\pm 25\%$ Sp
Gross Beta	Total β	LA-508-101 PNL-ALO-430/431	0.50 $\mu\text{Ci/g}$	$\pm 20\%$	$\pm 25\%$ Sp
ICP	Metals	LA-505-161 PNL-ALO-211			
	Al		100 $\mu\text{g/g}$	$\pm 20\%$	$\pm 10\%$ SD
	Cd		15 $\mu\text{g/g}$	$\pm 20\%$	$\pm 25\%$ Sp
	Sm		200 $\mu\text{g/g}$	$\pm 20\%$	$\pm 25\%$ Sp
	B		100 $\mu\text{g/g}$	$\pm 20\%$	$\pm 25\%$ Sp
	Fe		100 $\mu\text{g/g}$	$\pm 20\%$	$\pm 10\%$ SD
	Ba		100 $\mu\text{g/g}$	$\pm 20\%$	$\pm 25\%$ Sp
	Cr		20 $\mu\text{g/g}$	$\pm 20\%$	$\pm 25\%$ Sp
	Pb		200 $\mu\text{g/g}$	$\pm 20\%$	$\pm 25\%$ Sp
	Ag		20 $\mu\text{g/g}$	$\pm 20\%$	$\pm 25\%$ Sp
	Be		10 $\mu\text{g/g}$	$\pm 20\%$	$\pm 25\%$ Sp
	Tl		400 $\mu\text{g/g}$	$\pm 20\%$	$\pm 25\%$ Sp
	Zn		20 $\mu\text{g/g}$	$\pm 20\%$	$\pm 25\%$ Sp
	Cu		20 $\mu\text{g/g}$	$\pm 20\%$	$\pm 25\%$ Sp
	K		1000 $\mu\text{g/g}$	$\pm 20\%$	$\pm 25\%$ Sp
	Se		200 $\mu\text{g/g}$	$\pm 20\%$	$\pm 25\%$ Sp

¹ Required Method Detection Limit (MDL) 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., $-20\% < \text{RPD} < 20\%$. $\text{RPD} = [(\text{result1} - \text{result2})/\text{mean}] * 100$.

³ All spike recoveries should be within $\pm 25\%$. 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 using same methods as used for K East canister sludge samples or as directed in LOI/SOW. All Aroclor compounds should be identified and quantified.

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 9. Analytical Requirements for the 105-K West and East Basins Floor and Pit Sludge Samples (222-S and 325 Laboratories). (Continued)

Process	Constituents	Procedure	Required MDL ¹	Precision ²	Accuracy ³
	Mn		20 µg/g	±20%	±25% Sp
	Mg		200 µg/g	±20%	±25% Sp
	Ca		200 µg/g	±20%	±25% Sp
	Na		200 µg/g	±20%	±25% Sp
	Zr		20 µg/g	±20%	±25% Sp
	Ni		40 µg/g	±20%	±25% Sp
	P		400 µg/g	±20%	±25% Sp
	Bi		200 µg/g	±20%	±25% Sp
6	Hf		TBD	±20%	±25% Sp
6	Sn		TBD	±20%	±25% Sp
	U		1000 µg/g	±20%	±25% Sp
IC	Anions--F ⁻ , Cl ⁻ , NO ₂ ²⁻ , NO ₃ ⁻ , PO ₄ ³⁻ , SO ₄ ²⁻ , Br ⁻	LA-533-105 PNL-AL0-212	20 µg/ml	±20%	±25% Sp
Solvent Extraction with Liquid Scintillation Counter	⁹⁹ Tc	LA-438-101	0.05 µg/g	±20%	SPC
Exothermic/Endothermic	DSC (with air and nitrogen cover gas)	LA-514-114 PNL-AL0-508	--	±20%	SPC
Drained Solids (TWRS)	Percent water (TGA)	LA-560-112 PNL-AL0-508	--	±20%	SPC
Hot Persulfate/Coulometry	TIC (CO ₂ ²⁻) and TOC/TIC/TC	LA-342-100 PNL-AL0-381	40 µg/g	±20%	±25% Sp
Separation and Beta Counting	⁹⁰ Sr	LA-220-101 PNL-AL0-430/431	1.0 µCi/g	±20%	±25% Sp

¹ Required Method Detection Limit (MDL) 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., -20% < RPD < 20%. 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 using same methods as used for K East canister sludge samples or as directed in LOI/SOW. All Aroclor compounds should be identified and quantified.

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 9. Analytical Requirements for the 105-K West and East Basins Floor and Pit Sludge Samples (222-S and 325 Laboratories). (Continued)

Process	Constituents	Procedure	Required MDL ¹	Precision ²	Accuracy ³
Extraction and Alpha Counting	²³⁷ Np	LA-933-141 PNL-ALO-415/422	5 µCi/g	±20%	±25% Sp
Gravimetric (before and after acid digest, etc.)	Settled and dry density/volume	Sample preparation procedure ⁴ LA-519-132	N/A	N/A	N/A
Caustic Demand (optional)	Buffering capacity	LA-211-104	0.04 mole/kg	±20%	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	N/A	N/A	N/A
XRD--Digestate Residue	Any identifiable compounds (analytes) after acid digest and caustic fusion	PNL-ALO-268	N/A	N/A	N/A
Settling Rate	Volume settled versus time	Sample preparation procedure	N/A	N/A	N/A
Particle Size 1. Analyzer 2. Sieving	Particle size (submicron to 0.25 in.) and distribution	1. LT-519-101 PNL-ALO-530 2. Test instruction approved by SNFCP	N/A	N/A	N/A
Particle Shape	Shape	PNL-ALO-530	N/A	N/A	N/A
Viscometer	Viscosity	LT-519-115 PNL-ALO-502	N/A	N/A	N/A

¹ Required Method Detection Limit (MDL) 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., -20% < RPD < 20%. $RPD = [(result1 - result2)/mean] * 100$.

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 using same methods as used for K East canister sludge samples or as directed in LOI/SOW. All Aroclor compounds should be identified and quantified.

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 9. Analytical Requirements for the 105-K West and East Basins Floor and Pit Sludge Samples (222-S and 325 Laboratories). (Continued)

Process	Constituents	Procedure	Required MDL ¹	Precision ²	Accuracy ³
Organics	SVOA--all TICs	LA-523-131/406 PNL-AL0-120/ 344/345	Report TICs >10 ppm	N/A	N/A
	Polychlorinated biphenyls (PCB) ⁵	PNL-AL0-346 ⁵ LA-523-136	1 ppm (solids) 0.2 ppb (water)	-- ⁵ -- ⁵	-- ⁵ -- ⁵
Zeta Potential	Zeta potential	TWRS-95-5.6a-2, Rev. 0	TBD	TBD	TBD
pH (electrode)	pH	LA-212-106 PNL-AL0-225	0.1 pH units	±15%	SPC
Gas Generation Rate (optional)	Gas volume as rate of data time (e.g., hours), volume, and temperature of sludge	Test plan approved by SNFCP	TBD	TBD	TBD
Gas Composition (optional)	H, He, Ar, N ₂ , Xe, Kr, tritium, and any other significant gases	PNL-AL0-284	TBD	TBD	TBD
Cyanide	LA-695-102 PNL-AL0-287/289	LA-695-103 PNL-AL0-287/289	5 µg/g	±20%	±25% Sp

¹ Required Method Detection Limit (MDL) 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., -20% < RPD < 20%. $RPD = [(result1 - result2)/mean] * 100$.

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 using same methods as used for K East canister sludge samples or as directed in LOI/SOW. All Aroclor compounds should be identified and quantified.

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.

Figure 6. Flow Chart of Sludge Sample Processing and Analyses.

batch. An equipment blank from the K Basin sample collection mockup may be analyzed for each set of new sampling equipment. At least one hot cell blank (if the hot cell is used) will also be analyzed. One method control standard will be run with each batch. A matrix spike will be run per matrix, where applicable, as indicated in Table 9. Also see Section 8.0 on QA requirements.

Generally analyses noted in Table 9 may be performed at either the 325 Building laboratory or the 222-S laboratory depending on sludge treatment process subproject logistics and past support in the K Basins sludge characterization efforts (for consistency of data), follow-on test needs, specific laboratory capabilities, sample quantities, schedule, and contractual and HAMTC restrictions. The SOW including the detailed analyses requirements will define which laboratory will perform which analyses. All analyses will be performed consistent with doing work at the Hanford Site and as part of the Project Hanford Management Contract (PHMC).

Additional analyses of the liquid fraction of the sludge samples, beyond those noted in Figure 6, are potential options to support (1) calculation of maximum theoretical leachate concentration for RCRA heavy metals, and/or (2) bench scale water/sludge processing tests. These will be addressed in the SOW 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.

As noted earlier, additional scoping characterization analyses or combined characterization and process development tests, supporting the development and validation of treatment process may also be performed if warranted. If performed, these will be consistent with the DQO and the sludge processing subproject objectives in order to utilize limited sample and maximize the use of resources (i.e., funding, laboratory waste disposal, ALAR, etc.). Such analyses will be documented in SOWs to 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 section). The QA/QC standards must conform to Table 9 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 9. 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 acceptable results are not obtained, report the QC failure to SNFCP organization) or (2) report QC failure and suggested actions immediately to SNFCP Program Coordinator for evaluation.

The method detection level (MDL) 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 (Makenas 1998a).

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 1995 K East Basin sludge samples were also considered in determining the requirements presented in Table 9. The DQOs for the present sampling effort (Makenas 1998a) 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.

4.0 LABORATORY REPORTING REQUIREMENTS

The laboratories will be required to submit a final report within a period defined in the SOW from SNFCP 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 SOW. The laboratory will be required to submit interim preliminary data reports to the SNFCP Project Coordinator as requested.

The final reports submitted to the SNFCP Project Coordinator for technical review will conform to the requirements for a "full" data package (e.g., similar to Silvers 1997; Miller 1997). If a laboratory uses its own set of internal sample identification numbers in the data reports, a clear cross-reference of these numbers to the original sample identifications will be provided. A narrative that summarizes the results and their quality will also be included, as well as the signature of the individual(s) responsible for the report. 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, chain of custody forms, and 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.

The laboratory performing the analyses of the PCB concentrations in the sludge samples will inform the SNFCP Project Coordinator as soon as definitive data are available. This information will also be transmitted to the laboratories, in case there are any impacts on managing waste. The information will also be provided to K. L. Pearce, NHC, for assessment of impact on sludge process treatment feed stream assumptions.

4.1 DATA PACKAGING

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

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5.0 FINAL SUMMARY REPORT REQUIREMENTS

The completed data packages from the analytical laboratories will be sent to the SNFCP Project Coordinator who will coordinate data review and final summary data report preparation. The data will be reviewed specifically against the criteria set in the DQO document (Makenas 1998a). The Project Coordinator will ensure the data and reports become a part of the permanent Project Records.

5.1 STATISTICAL ANALYSIS REQUIREMENTS

A statistical analysis of the data will be performed as a separate activity from the overall laboratory final data package. This analysis will be reported as part of the final summary evaluation report to be completed by the SNFCP. 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 samples)
- Estimate the analytical variability (precision)
- Compute confidence intervals (e.g., 95% 90%, etc.) on the mean concentration for each analyte consistent with the requirements of the DQO and sludge process treatment subproject at the time of the report
- Compare present sludge data to past campaigns.

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6.0 SAFETY PLANS

6.1 RADIATION/INDUSTRIAL SAFETY

An industrial safety assessment will be performed prior to the start of these activities consistent with the requirements of each performing facility or laboratory. The requirements of HSRCM-1, Hanford Site Radiological Control 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 requirements of each facility or laboratory involved [e.g., K Basin Master Work Plan (MWP-95-TBD)], prior to the start of each activity. The requirements determined by these assessments shall be adhered to during the performance of the sampling activities.

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7.0 GENERAL ALARA PLAN

The sampling procedure for the activities discussed in Section 2.3.1 will take into consideration exposure reduction techniques which will minimize the radiation exposure to the sampling team.

ALARA considerations will be an integrated part of the design effort for the sampling and sampling equipment to minimize personnel exposure. The application of the sampling equipment will be made under specific ALARA plans developed and applied at the K Basins during the campaign. Each laboratory will apply its own review and ALARA plan consistent with operation on the Hanford Site (e.g., the Hanford Site Radiological Control Manual, HSRCM-1).

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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 10CFR830.120 and FDH QAPD (HNF-MP-599, Rev. 2), 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 Building 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 9. Sample quality control (duplicates, spikes, and standards) are identified in Section 3.2 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 the SOW that reflect the number of duplicates and spike analyses that should be run. The categories are:

Category

- 1 Duplicate analyses will be performed on all "first-time" characterization samples (i.e., all samples from K West Basin, plus those from K East Basin containing locations KE-7, KE-8, and KE-11). There should be at least four samples receiving duplicate analyses if there is an insufficient number of samples from those indicated, the laboratory will select enough additional samples to provide a total of four. One of the samples used for duplicate analyses will be chosen by the laboratory for spike analyses. The balance of the samples will be handled as normal laboratory batches with QA as per each laboratories internal QA plan.
- 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 9, the applicable laboratory procedures, or as called for in the final SOW.

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A P P E N D I X A

SUMMARY LETTER (DESH-9857199) DOCUMENTING
ESTIMATED VOLUMES OF SLUDGE IN K BASINS

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Subject: REVISED ESTIMATES OF SLUDGE VOLUMES IN K EAST AND K WEST BASINS

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August 28, 1998

DESH-9857199

Ms. K. L. Pearce
Spent Nuclear Fuel Project, Process Design
Numatec Hanford Corporation H7-20
Post Office Box 1300
Richland, Washington 99352-1300

Dear Ms. Pearce:

REVISED ESTIMATES OF SLUDGE VOLUMES IN K EAST AND K WEST BASINS

Reference: Internal Memo, R. B. Baker to K. L. Pearce, "Current Estimates of Sludge Volumes in K East and K West Basins," dated March 19, 1997.

In March 1997, a review was made of the volume estimates of sludge from the K East and K West Basins (Reference). Considered were the volumes of sludge (1) on the main basin floors, (2) in remote pits, (3) in fuel storage canisters, and (4) that expected to be generated from washing the storage canisters and fuel elements prior to packaging the fuel elements in the Spent Nuclear Fuel (SNF) multi-canister overpacks (MCOs).

Per your request, and with your support, we have reviewed these original estimates and their sources. Many of them still remain the best values available. There have been, however, some changes (such as additional measurements from K West Basin and improved estimates based on additional characterization data) which have resulted in revision of some of the previous sludge volume estimates. The revised sludge volume estimates and those still applicable are summarized herein (Attachments 1 and 2).

Please contact me if you have questions or you need additional information.

Very truly yours,

R. B. Baker, Senior Engineer
SNF Characterization Project

jms

Attachments 2

K EAST AND K WEST BASINS REVISED SLUDGE VOLUME ESTIMATES

R. B. Baker
August 28, 1998

1.0 SUMMARY

The revised estimates of sludge volumes in the K Basins based on this review are provided in Tables 1 and 2. Figures 1 through 3 provide overviews of the volume fractions of sludge for both basins together and separately. As used throughout the Spent Nuclear Fuel (SNF) Project, the physical definition of sludge used herein was particulate materials in K Basins (i.e., sand, fuel, corrosion products, cladding, IXM beads, insects, etc.) with no pieces greater than those expected to pass through a screen with 0.25 inch (6350 microns) openings.

2.0 SUMMARIES OF SLUDGE VOLUMES MADE PREVIOUSLY THAT ARE STILL VALID

A general review was made of the previously provided K Basins sludge volume estimates (Baker 1997). It was determined that the volume estimates for sludge reported in Baker (1997) remain unchanged for the following areas (the primary references these data come from is also indicated):

- K East (KE) Basin main floor (Baker 1995)
- KE Basin remote pits (Baker 1995; Makenas et al. 1996)
- KE Basin canisters (Pitner 1996)
- K West (KW) Basin North Loadout Pit (Baker 1995; Dodd 1995)

Attachment 2 of the current letter provides the text from Baker (1997) for reference with respect to the assumptions made for these areas. The KW Basin North Loadout Pit (or Sandfilter Backwash Pit) values had a minor error in Baker (1997), the sludge depths were incorrect, the sludge volumes quoted were correct. In addition, for consistency it is suggested you refer to the more detailed Dodd (1995) for the sludge measurement data for this pit, as well as Baker (1995).

3.0 REVISED ESTIMATES OF SLUDGE VOLUMES AT K BASINS

Fuel Wash Sludge

Recent estimates of volumes of sludge have just been completed for the material generated during washing of canisters and fuel elements at both KE and KW Basins (Pearce and Pitner, 1998a and 1998b, respectively). These estimates replace prior fuel wash sludge estimates and are being revised based on the availability of additional data from laboratory evaluations of material from the K Basins fuel element surface and subsurface examinations (Pitner 1997; Silvers 1998). Additionally, it was previously assumed that

fuel wash sludge was generated by metal fuel pieces being granulated (crushed) in the fuel washing machine and thus, turned into additional sludge. This assumption has been revised as it is now judged that the washing machine does not have the capability to significantly "granulate" the fuel pieces. However, it is expected that the washing process will cause further breakage of the elements and dislodge corroded uranium pieces, zirconium cladding, and loosely adhered material (such as coatings and internal sludge). This dislodged material is referred to as the "fuel wash sludge" and is assumed comprised of three components; pieces of coatings, fuel pieces and internal sludge (i.e., sludge originating directly from within the fuel elements). The fuel wash sludge component volume calculations are documented in Pearce and Pitner (1998a and 1998b). The calculated volumes for the KE Basin and KW Basin fuel wash sludge components are provided in Tables 1 and 2, respectively.

K West Basin Canister Sludge

Volume estimates for KW Basin canister sludge made previously were based on laboratory measurements of KE canister sludge density and measured ¹³⁷Cs content found in the water of the closed canister barrels in KW Basin (Trimble 1997). These prior estimates were revised (Pearce and Pitner 1998b) based on additional characterization data from KW Basin fuel storage canisters (Makenas et al. 1998). The revised volumes are provided in Table 2.

K West Basin Remote Pits and Main Basin Floor

The volume of sludge in the KW Basin remote pits and on the main basin floor provided in Baker (1997) were based on a quick general survey of these areas with sludge depths approximated through the full depth of water in the pool. A much better estimate is now available based on direct underwater measurements of sludge depths in the remote pits (except for the North Loadout Pit) and on the main floor of KW Basin (Maassen and Roe 1997). After these measurements were made the sludge in the South Loadout Pit was pumped to the Tech View Pit South Channel. The quantity of sludge originally reported located in the South Loadout Pit was therefore added to the volume of sludge reported for the Tech View Pit. The final estimates of sludge in the KW Basin remote pits and on the floor of KW Basin are provided in Table 2.

K East Basin and K West Basin North Loadout Pits

The depths and volumes of sludge in the North Loadout Pits (also referred to as, Sandfilter Backwash Pits) in both KE and KW Basins are based on direct measurements made at the end of 1995 (Baker 1995; Dodd 1995). Some minor increase in sludge depths has likely occurred because of additional backwashing of the sandfilters. It is judged (based on input from K Basin staff), however, that this has had little influence on the overall volumes (i.e., within the uncertainties noted). Therefore, the volumes of sludge in both pits are still reported the same as in Baker (1997) with exception of the comments in Section 2.0. Additional depth measurements are planned in the future for these pits during acquisition of sludge characterization samples.

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Table 1. Estimates of Sludge Volumes for K East Basin Floor, Pits, and Fuel Wash Components.

Location or Source	Sludge Average Depth (in.)	Area (ft ²)	Sludge Volume		Positive Percent Uncertainty	Upper Bound Sludge Volume		Reference
			ft ³	m ³		ft ³	m ³	
Tech View-North Channel	1.0	49.4	4.0	0.1	50	6.0	0.2	Baker (1995)
Tech View-South Channel	0.2	49.4	0.9	0.0	50	1.3	0.0	Baker (1995)
Tech View Pit	0.9	141.5	10.4	0.3	50	15.6	0.4	Baker (1995)
Weasel Pit Channel	12.0	53.8	53.8	1.5	20	64.5	1.8	Baker (1995) Makenas (1996)
Weasel Pit	32.3	112.3	302.5	8.6	20	362.9	10.3	Baker (1995) Makenas (1996)
North Loadout Pit	33.0	66.5	183.0	5.2	20	219.6	6.2	Baker (1995)
North Loadout Channel	20.0	23.2	38.6	1.1	20	46.4	1.3	Baker (1995)
South Loadout Pit	0.0	64.6	0.0	0.0	0	0.0	0.0	Baker (1997)
South Loadout Channel	0.0	23.0	0.0	0.0	0	0.0	0.0	Baker (1997)
Dummy Elevator Pit	6.0	97.0	48.5	1.4	50	72.8	2.1	Baker (1995)
Discharge Chute	0.0	316.5	0.0	0.0	0	0.0	0.0	Baker (1995)
Main Basin Floor (Open)	1.9	4848.4*	760.4	21.5	20	912.5	25.8	Baker (1995)
Canisters--Empty			13.8	0.4	20	16.6	0.5	Baker (1995)
Canisters--Fueled			106.1	3.0	147	261.4	7.4	Pitner (1996)
Subtotals			1522.0	43.1		1979.6	56.0	
Fuel Wash Components:								
Fuel Pieces	NA	NA	5.3	0.149	NA	13.2	0.373	Pearce (1998a)
Coating	NA	NA	2.2	0.061	NA	3.1	0.092	
Internal Sludge	NA	NA	18.3	0.518	NA	45.7	1.294	
Grand Total			1547.8	43.8		2041.6	57.8	

*Open area around canisters set on floor.

Table 2. Estimates of Sludge Volumes for K West Basin Floor, Pits, and Fuel Wash Components.

Location or Source	Sludge Average Depth (in.)	Area (ft ²)	Sludge Volume		Positive Percent Uncertainty	Upper Bound Sludge Volume		Reference
			ft ³	m ³		ft ³	m ³	
Tech View-North Channel	0.066	49.35	0.27	0.008	417***	1.13	0.032	Maassen (1997)
Tech View-South Channel**	0.319	49.35	1.27	0.035	Combined**	3.76	0.107	Maassen (1997)
Tech View Pit	0.066	141.50	0.78	0.022	417	3.24	0.092	Maassen (1997)
Weasel Pit Channel	0.066	50.67	0.282	0.008	379	1.07	0.0302	Maassen (1997)
Weasel Pit	0.066	116.46	0.647	0.018	379	2.45	0.0694	Maassen (1997)
North Loadout Pit	20.7	65.2	112.3	3.18	20	134.8	3.82	Dodd (1995)
North Loadout Channel	8.8	22.7	16.2	0.46	20	19.4	0.550	Dodd (1995)
South Loadout Pit**	0.0	65.00	0.0**	0.0	NA	0.0	0.0	Maassen (1997)
South Loadout Channel**	0.0	22.30	0.0**	0.0	NA	0.0	0.0	Maassen (1997)
Dummy Elevator Pit	0.16	97.10	1.31	0.0371	385	5.05	0.143	Maassen (1997)
Discharge Chute	0.052	526.30	2.28	0.0647	432	9.88	0.280	Maassen (1997)
Main Basin Floor (Open)	0.064	5479.2*	29.0	0.822	229	66.3	1.88	Maassen (1997)
Canisters--Fueled	NA	NA	35.7	1.01	77	55.8	1.58	Pearce (1998a)
Subtotals			200	5.67		303	8.58	
Fuel Wash Components:								
Fuel Pieces	NA	NA	5.26	0.149	150	13.2	0.373	Pearce (1998a)
Coating	NA	NA	14.3	0.406	150	21.6	0.613	
Internal Sludge	NA	NA	18.3	0.518	150	45.7	1.294	
Grand Total			238	6.74		383	10.9	

*Open area around canisters set on floor.

**The sludge measured in Maassen (1997) in the South Loadout Pit was transferred to the Tech View Pit South Channel in September 1997 (Maassen 1997; Lodwick 1997). The volume of sludge measured in Maassen (1997) in South Loadout Pit (1.003 ft³, 0.0263 m³) has been added to the Tech View Pit South Channel (0.271 ft³, 0.00766 m³) in this table. The uncertainties used were from Maassen (1997) for Tech View-South Channel 417%, and for the South Loadout Pit sludge volume 263%.

***Uncertainties taken from Maassen (1997) were based on upper limits of the 95% confidence interval including random variabilities and systematic measurement variability.

Figure 1. Comparison of Sludge Volume Fractions Versus Locations or Sources at K Basins.

Figure 2. Comparison of Sludge Volume Fractions Versus Locations or Sources at K East Basin.

Figure 3. Comparison of Sludge Volume Fractions Versus Locations or Sources at K West Basin

DESH-9857199

ATTACHMENT 2

Background Information Text from Reference Baker 1997

Consisting of 5 pages,
including cover page

From: Spent Nuclear Fuel Evaluations
Phone: 376-5109 HO-40
Date: March 19, 1997
Subject: CURRENT ESTIMATES OF SLUDGE VOLUMES IN K EAST AND K WEST BASINS

To: K. L. Pearce R3-11

cc: C. J. Alderman	X3-85	M. J. Packer	X3-85
D. W. Bergmann	R3-85	A. L. Pitner	HO-40
K. H. Bergsman	X3-85	A. N. Praga	R3-85
S. L. Hecht	HO-40	D. R. Precechtel	X3-85
P. G. LeRoy	R3-15	D. S. Takasumi	X3-85
B. J. Makenas	HO-40	D. J. Trimble	HO-40
R. P. Omberg	HO-40	RBB File/LB	HO-40

- References: (1) Internal Memo, A. L. Pitner to D. S. Takasumi, "K West Basin Sludge Volume Estimates for Integrated Water Treatment System," dated January 29, 1997. [Also provided in DESH EDT 620777 dated February 1997.]
- (2) Internal Memo, D. J. Trimble to D. S. Takasumi, "K West Basin Sludge Volume Inventory," dated January 26, 1997.
- (3) D. J. Trimble, *Cesium-137 in K West Basin Canister Water*, HNF-SD-SNF-ANAL-014, Rev. 0, dated January 24, 1997.
- (4) Internal Memo, A. L. Pitner to D. S. Takasumi, "K East Basin Sludge Volume Estimates for Integrated Water Treatment System," dated January 23, 1997. [Also provided in DESH EDT 620776 dated February 1997.]
- (5) A. L. Pitner, *K East Canister Sludge Survey*, WHC-SD-SNF-TI-034, Rev. 0, dated August 28, 1996.
- (6) B. J. Makenas, et al., *Analysis of Sludge from Hanford K East Basin Floor and Keasel Pit*, WHC-SP-1182, dated April 1996.
- (7) S. M. Short and M. M. Beary, *Spent Nuclear Fuel Project Technical Databook*, WHC-SD-SNF-TI-015, Rev. 0, dated August 11, 1995 (ECN 191395 February 1996).
- (8) R. B. Baker, *Summary Status of K Basin Sludge Characterization*, WHC-SD-SNF-TI-005, Rev. 0, dated January 1995.

K. L. Park
Page 2
March 19, 1997

Per your request, a review was made of the current estimates for volumes of sludge in the K East Basin and K West Basin. Considered was the sludge on the main basin floor, in remote pits, in fuel canisters, and the potential for generation of sludge from washing storage canisters/fuel prior to the fuel being packaged in multi-canister overpacks (MCOs). Attachments 1 and 2 provide a summary of these results.

Past Summaries of Sludge Volumes

An original summary of sludge volumes in the Basins was provided in Reference 8. These were based primarily on measurements of sludge depths made at the K East and K West Basins just prior to the report being issued (i.e., 1994). These sludge volumes, with uncertainty estimates, were later used both in the Spent Nuclear Fuel (SNF) project feed document and the SNF Project Databook, Reference 7. In many cases these estimates remain the best values available. However, there have been additional measurements, improved estimates, and changes affecting some of these sludge estimates. For these instances the improved values should be used and these are described in the following sections and attachments.

Because of recent questions, it is re-emphasized here that the estimates of sludge volumes made in Reference 8 for the floor of the K East main basin account for the fact that the fuel storage canisters sit directly on the basin floor, with the "floor sludge" built-up around them. Any sludge under the canisters is assumed insignificant or accounted for in the uncertainty of the sludge volumes projected as residing in the canisters themselves.

Revised Estimates of Sludge Volumes at K Basins

Revised estimates of volumes of sludge in the fueled canisters in K East Basin were made based on ultrasound measurements of sludge depths made in a set of selected fuel canisters in 1996, Reference 5. These represent a much better estimate of this sludge volume than that provided in Reference 8, because there is accurate direct data as a basis rather than the secondary observations used in Reference 8. Attachment 1 provides these revised values. Note the magnitude of the volumes provided in these revised estimates are similar to those provided in the original estimates of Reference 8 within the bounds identified (e.g., Reference 8: total volume in fueled canisters was about 7.5 m³ with a bounding estimate of 12.7 m³, and in Reference 5: total volume is 3.0 m³ bounding estimate of 7.4 m³).

Measurements of sludge volume in the fueled (sealed) canisters in K West Basin were also attempted using ultrasound equipment to make depth measurements of the sludge. However, these measurements were found to be inconsistent, as opposed to the valid measurements resulting from the measurements in K East Basin storage canisters. The problem with the application in K West Basin canisters was probably caused by (1) less sludge being present and (2) complications arising from pieces of the material used

K. L. Park
Page 3
March 19, 1997

to seal the canister lids falling between the fuel elements during the process of removing the canister lids, see Reference 2. Given this, it was judged a better estimate of sludge volume was possible, from measured cesium concentrations in the water samples analyzed from a selected set of sealed canister barrels in K West Basin. This approach is fully described in Reference 2, and the resulting volumes given in Attachment 2 of the current memo.

The volume of sludge indicated in Reference 8 as contained in the Weasel Pit at K East Basin, was based on an inference of sludge depth measured from the shadow of sludge observed on the screen separating the Weasel Pit from the main basin. A much better estimate was later made available based on measurements made in August and September 1995. These are discussed in Reference 6, which concluded an integrated sludge volume for the pit was 7.4 m³ (264 ft³). Since these measurements were made in 1995 at least two activities have added sludge to this pit. First, the South Loadout Pit was cleaned and this sludge pumped to the Weasel Pit. Second, an area of the Center Bay of the K East Basin was cleaned of sludge, to support processing and cleaning empty canisters, again the sludge was pumped to the Weasel Pit. Estimates of the sludge moved to the Weasel Pit were made based on the sludge estimated in these areas from Reference 8 data. These quantities were then added to the volume of sludge estimated from measurements made in September 1995 (Reference 6) for the Weasel Pit. For the South Loadout Pit a volume of 3.6 m³ was assumed to have been moved, and for the cleaned portion of the Center Bay the estimate of the associated volume of sludge was 0.1 m³. The later volume was subtracted from the main basin floor value. The final estimates of sludge in the K East Basin Weasel Pit and on the main basin floor are shown in Attachment 1.

A quick survey was made of the K West Basin remote pits and main basin floor as a result of your request. From this the estimates of sludge depths were made that are shown in Attachment 2. Direct physical measurement of sludge depths on the K West main basin floor and in the pits (except the North Loadout Pit) is planned to be completed by the Spent Nuclear Fuel Evaluations group within the next 2 months.

The depths and volumes of sludge in the North Loadout Pits (i.e., Sandfilter Backwash Pits) in both K East and K West Basins are based on direct measurements made at the end of 1995, Reference 8. Some minor increase in sludge depths has no doubt occurred due to further backwashing of the sandfilters. It is assumed here this has had little influence on the volumes (i.e., within the uncertainties noted). This will be verified in the next year when samples are taken from these pits for characterization.

Similar to the North Loadout Pits, there has likely been some increase in sludge volume on the K East main basin floor since 1995 when depth measurements were measured. Here again this is assumed to be within the uncertainties indicated.

K. L. Park
Page 4
March 19, 1997

To provide a comprehensive set of sludge volumes, estimates are also included in Attachment 1 for the future washing of fuel and canisters at the K East Basin; these estimates were taken from Reference 4. K West Basin was also reviewed to make a similar estimate for fuel washing, Reference 1. Here it was estimated that at this time a value similar to the K East Basin value should be used. This is reflected in Attachment 2.

Please contact me if you have questions.

RBB Baker

R. B. Baker, Fellow Engineer
Spent Nuclear Fuel Evaluations

jmn

Attachments 2

PEER REVIEW:

A. L. Pitner

A. L. Pitner
Spent Nuclear Fuel Evaluations

Date: 3/19/97

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A P P E N D I X B

**SLUDGE DEPTH ESTIMATES AND SAMPLE LOCATIONS DETAIL
FOR K WEST AND K EAST BASINS**

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Figure B1. Floor Sludge Depth (in inches) at Locations Measured in 1997 at K West Basin (Maassen 1997). (Prior to Sludge Being Pumped from South Loadout Pit to Tech View South Transfer Channel.)

Figure B2. K West Basin North Loadout Pit Sample Locations.

Figure B3. Surface Fit of Sludge Depth Data in K West Basin
North Loadout (Sandfilter Backwash) Pit in 1995.

Figure B4. K West Basin Weasel Pit and Tech View Pit Sample Locations.

KW Basin's Tech View and Weasel Pits' overview of the Exclusion Zone, Plywood Covering, and Sample Locations (all units are in inches)

Figure B5. K West Basin Dummy Elevator Pit Sample Locations.

Figure B6. Fit of Sludge Depth Data in the Three Bays of K East Main Basin in 1995. (Locations relate to Figure 4)

Figure B7. Sample Locations Used in 1995 Campaign in K East Basin.

R027040018.1

105-KE Basin North Loadout Pit's Exclusion Zone,
Plywood Covering, and Sample Locations (all units are
in inches)

Figure B8. K East Basin North Loadout
(Sandfilter Backwash) Pit Sample Locations.

Figure B9. Surface Fit of Sludge Depth Data in K East Basin North Loadout (Sandfilter Backwash) Pit in 1995.

Figure B10. K East Basin Dummy Elevator Pit Sample Locations.

Figure B11. Sludge Depth Data for K East Basin Weasel Pit in 1995.

Figure B12. Sample Locations Used in the K East Basin Weasel Pit for the 1995 Campaign.

Figure B13. K East Basin Weasel Pit and Tech View Pit Sample Locations.

KE Basin's Tech View and Weasel Pits' overview of the Exclusion Zone, Plywood Covering, and Sample Locations (all units are in inches)

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