0080241

Tri-Party Agreement

Change Notice for Modifying Approved Documents/ Workplans
In Accordance with the Tri-Party Agreement Action Plan,
Section 9.0, *Documentation and Records*

Change Number	Document Submitted Under Tri-Party Agreement Milestone	Date:
TPA-CN-258	M-34-30	**January 28, 2009**

Document Number and Title: **KBC-33786, Quality Assurance Project Plan/Sampling and Analysis Plan for Sludge in the KW Engineered Containers**

Date Document Last Issued: Revision 0, December 2008

Originator: J. L. Westcott **Phone:** 373-9800

Description of Change:

THOMAS K. TEYNOR (**DOE**) and Rod Lobos (**Lead Regulatory Agency**) agree that the proposed change modifies an approved workplan/document and will be processed in accordance with the Tri-Party Agreement Action Plan, Section 9.0, *Documentation and Records*, and not Chapter 12.0, *Changes to the Agreement.*

The document has been updated to incorporate the new path forward for K Basin sludge to be removal from the 100-K area, store as wet sludge at a 200 area facility, and then treat for eventual disposal at WIPP. The following changes have been made to the document:

- Revise text to incorporate the new path forward including deletion of the gas generation test on grouted sludge (Tables 2-1, 3-4a, and 3-8 and pages 2-4, 2-5, 2-10, 3-27)
- Update references and sludge volume and equipment blank information in document with recent references and information
- Correct editorial and other errors identified in revision 0 of the document.

The affected pages: iv, 1-1, 1-9, 2-4 to 2-7, 2-9 to 2-11, 2-13, 2-16, 3-1 to 3-3, 3-6, 3-7, 3-10, 3-14 to 3-18, 3-20 to 3-22, 3-24, 3-26 to 3-32, 3-34 to 3-38, 3-40, 3-43, 5-3, 5-5, 5-8, 6-2, 6-3, 6-6, and A-5

Justification and Impacts of Change:

The path to remove and treat sludge currently being stored in the K West Basin has recently been changed. The subject document is being changed to align with the new path. The document is also being corrected of errors that have been indentified in revision 0 of the document and updated with recent references (e.g., revised DQO) and updated information.

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Approvals:

DOE Project Manager	1-30-2009 Date	X Approved	__ Disapproved
Lead Regulatory Project Manager	1-30-09 Date	X Approved	__ Disapproved

Once all the above steps have been completed, the originator sends a copy of the signed change notice to the FH TPAI organization (H8-12), the Administrative Record (H6-08) (refer to TPA Action Plan, Section 9.3), lead regulatory agency, affected Hanford

Executive Summary

The purpose of this *Quality Assurance Project Plan / Sampling and Analysis Plan for Sludge in the KW Engineered Containers* is to define the strategy, processes, and quality control (QC) activities associated with the sampling and characterization of the sludge in the five K West (KW) Basin Large Engineered Containers. These five containers (SCS-CON-210, -220, -240, -250 and -260) are used to consolidate the Floor and Pit sludge from the KW and K East (KE) Basins. Sampling and analysis of the sludge in these containers will address two sets of K Basin Closure (KBC) and Sludge Treatment Project (STP) objectives: 1) to establish the nuclear material (NM) Accountability values for the KW Basin Floor and Pit sludge inventory and 2) to provide specific characterization data to support the final design of a process to retrieve, treat, store, and package this sludge prior to disposal at the Waste Isolation Pilot Plant (WIPP).

The Data Quality Objective (DQO) documents (Makenas and Baker 2007; ~~Westcott 2008~~Westcott 2009) for this initiative have determined that four representative vertical core samples of the sludge in each of two Large Engineered Container containing KW sludge are required to provide the basis for determining the required NM Accountability values and sludge characterization in support of process design. The Data Quality Objective (DQO) document (~~Westcott 2008~~Westcott 2009) has determined two representative vertical core samples of the sludge in each of three Large Engineered Container containing KE sludge are required to provide the basis for determining the sludge characteristics to support process design. Each sludge core will result in a set of 4-liter sample bottles containing portions of the sludge solids plus related basin water used to draw the samples from the basin pool to the grating level (up to 22 ft). The resulting sample bottles will be transferred from the KW basin to the analytical laboratory using a PAS-1 cask and a system of Shielded Sample Containers developed for these sample bottles. The number of sample bottles per core sample will vary depending on sludge depth at the container location being sampled.

At the laboratory, the cask will be unloaded and the bottles transferred to the processing hot cell. The sludge solids for each core will be recovered from the set of bottles and consolidated into a single container. The sludge is then mixed and settled, and as-settled density is measured. Finally, sub-samples are obtained and analyzed for the required NM Accountability analytes and characterization / processing parameters. The laboratory results will be reported to the STP Project and the Safeguards Accountability group. The laboratory will provide a final data package with required QA/QC information. The NM content of the KW Floor and Pit sludge in the related Large Engineered Containers will be determined from the analytical results along with the measured volume of sludge in the Large Engineered Containers. In addition, consistent with the data needs of the STP established in the DQO document (~~Westcott 2008~~Westcott 2009), the characterization parameters supporting sludge treatment, storage, and final disposition to WIPP will be determined. Subsequently, KBC/STP Project staff will evaluate, over-check, and document the overall sampling campaign results issuing a final topical report summarizing the sample collection activity and final laboratory results.

1.0 Introduction

The K Basin sludge will be removed, treated, stored, and disposed of in support of the execution of the *Interim Action Record of Decision for the 100-KR-2, Operable Unit, Hanford Site, Benton County, Washington* (K Basins ROD) (EPA 1999) as amended (EPA 2005). The selected action will remove sludge from containers located in the 105-K West (KW) Basin; treat the sludge onsite (as defined by CERCLA) to comply with transportation and disposal requirements; store the sludge at a Hanford 200 area facility; and send the treated sludge to the Waste Isolation Pilot Plant (WIPP) for disposal. The WIPP is the national transuranic (TRU) waste disposal facility located near Carlsbad, New Mexico.

This Quality Assurance Project Plan/Sampling and Analysis Plan (QAPjP/SAP) provides information necessary to implement sludge removal, treatment, storage, and disposal consistent with the following:

- DOE/RL-2006-06, *Remedial Design Report and Remedial Action Work Plan for the K Basins Interim Remedial Action: Sludge Treatment and Interim Storage* (work plan)
- Correspondence No. 0800842, 2008, *Contract NO. DE-AC06-96RL13200-K Basins Sludge Disposition Direction*
- Correspondence No.0702389A, 2007; *Contract NO. DE-AC06-96RL13200-K Basins Sludge Disposition Direction*

Sample collection and analysis of container sludge is the subject of this QAPjP/SAP. Container sludge is the sludge that was collected from the floors and pits of the KE Basin, which currently resides in three of the engineered containers in the KW Basin, and the sludge collected from the floors and pits of the KW Basin, most of which currently resides in two other engineered containers in KW. This QAPjP/SAP implements the data collection specified in two separate Data Quality Objectives (DQO) reports, *Data Quality Objectives for Sampling of K West Basin Floor and Pit Sludge in Large Engineered Containers* (Makenas and Baker 2007) and *Data Quality Objectives Report for Sampling and Analysis of K Basin Sludge* (~~Westcott 2008~~Westcott 2009).

Sludge sampling and collection will be conducted in accordance with *Sludge Treatment Project Quality Assurance Program Plan* (Mata 2008) and the *Environmental Quality Assurance Program Plan* (McCallum 2008). This QAPjP/SAP report has been written in accordance with the requirements of *EPA Requirements for Quality Assurance Project Plan,* EPA QA/R-5 (EPA 2001). These requirements are further clarified in *Guidance for Quality Assurance Project Plans,* EPA QA/G-5 (EPA 2002). This QAPjP/SAP includes the applicable requirements of *Test Methods for Evaluating Solid Waste, Physical and Chemical Methods,* SW-846 (EPA 1997), and *Hanford Analytical Services Quality Assurance Requirements Document* (HASQARD 2007).

Table 1-1. Estimates of Sludge in KW Large Engineered Containers (February 2008)

Container Number and Sludge Source		Average Sludge Depth Reading in Container[a], ft	Average Depth of sludge in Container[b], ft	Estimated Sludge Volume, m^3	Date of Observation
SCS-CON-210	KW Floor and Pit Sludge	3.4	2.9	2.8	1/29/08
SCS-CON-220	KW Floor and Pit Sludge	2.3	1.8	1.0	1/30/08
SCS-CON-230	Empty	---	---	---	---
SCS-CON-240	KE Floor and Pit Sludge	3.2	2.7	2.6	~~7/15/07,~~ 10/~~29~~7/08
SCS-CON-250	KE Floor and Pit Sludge	6.2	5.7	7.7~~6~~	~~7~~10/~~15~~7/0~~7~~8
SCS-CON-260	KE Floor and Pit Sludge	6.5	6.0	8.1	~~7~~10/~~15~~7/0~~7~~8

[a]Values shown are taken from level markings in each Large Engineered Container and are referenced to the basin floor and the average height of sludge observed.
[b]The depth of sludge in container is 0.5 ft (6") off the basin floor which is the bottom of the container hopper, hence the actual maximum physical sludge depths are scale depths minus 0.5 ft (6").

1.3.4 Composition and Parameters of KW and KE Floor and Pit Sludge

The KW Basin Floor and Pit sludge in containers SCS-CON-210 and SCS-CON-220 includes material from the KW Basin West Bay where fuel cleaning and packaging occurred. This sludge may be higher in uranium residues. The STP sludge databook (Schmidt 2006a) incorporates this possibility when estimating the KW Basin West Bay sludge composition as described in Table 5-1 of the databook. Analyses for the handling and transport of this sludge shall use the STP sludge databook composition parameters.

Sub-streams and design parameters for the KE Basin Floor and Pit sludge in containers SCS-CON-240, -250 and -260 are directly available in the Sludge Databook (Schmidt 2006a).

Waste and Fuel Management Project (WFMP)/Transportation Logistics and Shipping
WFMP Transportation Logistics and Shipping management has the following responsibilities:

- Prepare shipping paperwork and mark and label the transport to ship samples to the 325 laboratory and empty casks from the 325 laboratory back to KW Basin
- Schedule shipments and shipment resources

WFMP/Project Engineering
WFMP Engineer management is responsible to qualify a cask system as a transportation package to ship samples (One Time Shipping Request).

Project Hanford Management Contract (PHMC)/Safeguards
Safeguards management has the following responsibilities:

- Prepare a 741 form to accompany each shipment of sludge sample
- Review the quality of the KW sludge sample analysis data
- Reconcile analysis data results with user requirements in concert with STP Characterization personnel

PHMC/Closure Services and Infrastructure (CS&I)
PHMC/CS&I management has the responsibility to provide personnel and equipment necessary to transport samples and empty casks between the KW Basin the 325 laboratory.

Each organization named above will maintain qualifications of personnel performing the listed activities and manage corrective actions associated with work performed by each organization.

2.2 Problem Definition and Background

This section identifies the problem to be solved, decisions to be made, and outcome to be achieved. Background information that provides a historical, scientific, and regulatory perspective is provided is Section 1.0.

2.2.1 Statement of the Problems

This document addresses two separate problems that are identified in their respective data quality objectives reports (~~Westcott 2008~~Westcott 2009; Makenas and Baker 2007). The sludge treatment DQOs (~~Westcott 2008~~Westcott 2009) are an estimate type of problem; that is estimation of sludge characteristics is necessary to address the problem statement. The sludge nuclear accountability data objectives established in Makenas and Baker 2007 only apply to KW Basin sludge and are a decision type of problem; that is, a decision is made depending on the results of the sludge characteristics that are measured.

Sludge Treatment Design
A process is being designed to remove K Basin container sludge from its current locations in the KW Basin~~for storage~~, store the sludge at a Hanford facility, ~~and treatment on the Hanford site pending its~~for eventual shipment to and disposal at WIPP. The design requires information

about the characteristics of the sludge. More specifically, sludge stored in the KW Basin engineered containers shall be characterized sufficiently to support the following activities:

- design a system to retrieve the sludge from containers located in the KW Basin,
- transport the untreated sludge to a Hanford storage facility,
- store untreated sludge onsite pending treatment and packaging for shipment to the national repository,
- design a system to treat and package the sludge in compliance with applicable requirements,
- operate the sludge treatment and packaging system safely and compliantly,
- ~~transport the sludge to an onsite storage facility,~~ and
- store treated sludge onsite prior to shipment to the national repository.

The characterization information may also be used to certify the waste for disposal at WIPP and other purposes such as KW Basin facility deactivation.

Nuclear Material Accountability
The container sludge contains nuclear material that is controlled and protected. The sludge is to be treated and disposed as waste. The nuclear material control of sludge will be terminated because the sludge will be disposed as waste. The termination of nuclear material control requires that the concentration of nuclear materials in sludge be measured. The concentration of nuclear materials in KE sludge has been measured, but has not been measured in KW sludge. This campaign shall sample the two engineered containers that contain KW sludge and analyze the samples to establish the concentrations of nuclear material.

2.2.2 Sludge Treatment Design Estimation Statements

The applicable six principal study questions (PSQs) and estimate statements (ESs) associated with sludge are reproduced from the DQO report (~~Westcott 2008~~Westcott 2009) in Table 2-1. The analysis data needed to address each of these PSQs and associated ESs is also listed in Table 2-1. The analysis results produced will result in establishing sludge characteristics to be used to design the sludge treatment process.

Table 2-1. DQO Principal Study Questions and Data Needs to Support STP Process Design

#	Principal Study Question	Estimation Statement and Data Collected
1	Does sufficient characterization information exist about the sludge to support design and safe and compliant operation (e.g., nuclear safety, criticality, and environmental impacts) of sludge retrieval, ~~treatment, packaging~~ and onsite transport processes, storage at Hanford with subsequent retrieval from storage, treatment, and packaging?	Parameters that affect the design of the processes ~~a~~ sludge will be subjected to ~~treatment process~~ will be measured. For the identified design parameters, both an average and the variability associated with the average will be established. The data to be collected are: • Settled sludge density • Weight percent solids in settled sludge • Physical characteristics important to sludge retrieval, pumping, and mixing: mass, particle density, x-ray diffraction spectra, uranium metal, and uranium speciation • Rheology testing • Uranium metal content in settled sludge and size fractionated sludge ~~•Hydrogen generation rate from grouted sludge~~ • Total Uranium • U-233, U-234, U-235, and U-238 • Pu-238, Pu-239, Pu-240, Pu-241, and Pu-242 • Cs-137 (report others found e.g., Eu-154, Co-60) • Am-241 • Sr-90 • Np-237, Am-243, and Am-242m • Chemical composition of sludge
2	Does sufficient characterization information exist about the sludge to support demonstration of compliance of the sludge packaged for storage ~~final waste form~~ with the on-site storage facility requirements in the *Hanford Site Solid Waste Acceptance Criteria* (Ramirez 2008)?	Parameters that will be used to characterize and package ~~treated~~ sludge for storage at a Hanford 200 area facility will be measured in a manner compliant with the waste acceptance requirements. For the identified parameters, both an average and the variability associated with the average will be established. The data to be collected are: • Settled sludge density • Uranium metal content in settled sludge and size fractionated sludge ~~•Hydrogen generation rate from grouted sludge~~ • Total Uranium • U-233, U-234, U-235, and U-238 • Pu-238, Pu-239, Pu-240, Pu-241, and Pu-242 • Cs-137 (report others found such as Eu-154, Co-60) • Am-241 • Sr-90 • Np-237, Am-243, and Am-242m • Chemical composition of sludge

Table 2-1. DQO Principal Study Questions and Data Needs to Support STP Process Design

#	Principal Study Question	Estimation Statement and Data Collected
3	Does sufficient characterization information exist about the sludge to support demonstration of compliance with the RH TRAMPAC requirements applied to the treated waste form?	Parameters that will be used to characterize and package treated sludge for transportation to WIPP in accordance with the RH TRAMPAC requirements will be measured in a manner compliant with the requirements. For the identified parameters, both an average and the variability associated with the average will be established. The data to be collected are: • Settled sludge density • Uranium metal content in settled sludge and size fractionated sludge • ~~Hydrogen generation rate from grouted sludge~~ • Total Uranium • U-233, U-234, U-235, and U-238 • Pu-238, Pu-239, Pu-240, Pu-241, and Pu-242 • Cs-137 (report others found such as Eu-154, Co-60, that contribute to 95% or more of the radioactive hazard) • Am-241 • Sr-90 • Np-237, Am-243, and Am-242m
4	Does sufficient characterization information exist about the sludge to support demonstration of compliance with the RH TRU Waste Characterization Implementation Plan requirements?	Parameters that will be used to characterize and package treated sludge for disposal at WIPP in accordance with the RH TRU Waste Characterization Program Implementation Plan (WCPIP) requirements will be measured. For the identified parameters, both an average and the variability associated with the average will be established. The data to be collected are: • Uranium metal content in settled sludge and size fractionated sludge • Total Uranium • U-233, U-234, U-235, and U-238 • Pu-238, Pu-239, Pu-240, Pu-241, and Pu-242 • Cs-137 (report others found such as Eu-154, Co-60 that contribute to the curie loading of the waste) • Am-241 • Sr-90 • Np-237, Am-243, and Am-242m
5	Does sufficient characterization information exist about the sludge to support demonstration of compliance with the New Mexico WIPP Hazardous Waste Facility Permit	Parameters that will be used to characterize treated sludge for disposal at WIPP in accordance with the New Mexico WIPP Hazardous Waste Facility permit requirements must be measured prior to certification of the waste for shipment to WIPP[a]. For the identified parameters, both an average and the variability

2.2.3 Sludge Nuclear Accountability Decision Statements

The PSQ and decision rule (DR) associated with KW sludge developed from the DQO report Makenas and Baker 2007 are presented in Table 2-2. The analysis data needed to address the PSQ and DR is also listed in Table 2-2. The analysis results produced will result in establishing sludge nuclear material concentrations to be used to determine the attractiveness level necessary to terminate Safeguards for the KW sludge.

Table 2-2. Nuclear Accountability Principal Study Question and Data Needs

#	Principal Study Question	Decision Rule and Data Collected
1	Does sufficient characterization information exist about the KW sludge to establish the concentration of nuclear material in sludge and thereby the attractiveness level for the sludge?	The decision rule is: If the total plutonium concentration in KW sludge is < 0.20 weight percent then the sludge has an attractiveness level of "E." If the total plutonium concentration in KW sludge is ≥0.20 weight percent, then the sludge has an attractiveness level of "D." The data to be collected are: • Pu-238, Pu-239/240, Am-241 • U total • Pu total • U and Pu Isotopes including U-235, U-238, Pu-239, Pu-240, Pu-241, Pu-242 • Np-237

2.3 Project/Task Description

Samples of sludge will be collected from each of five engineered containers of sludge located under water at the KW Basin after completion of sludge transfers into each container. Two core samples will be collected from each of three KE sludge containers, SCS-CON-240, SCS-CON-250, and SCS-CON-260. Four core samples will be collected from each of two KW sludge containers, SCS-CON-210 and SCS-CON-220. These samples will be submitted to the 325 laboratory for analysis. The activities to be performed to execute this sampling and analysis campaign ~~is~~ are described as follows:

- KW Basin operating personnel collect representative samples (two or more bottles) of a complete core at locations within each container as identified herein.
- KW Basin operating personnel stage the bottles of sample at the KW Basin until transport.
- KW Basin operating personnel package bottles of sample in a cask for transport to the 325 laboratory.

- 325 laboratory personnel receive the bottles of sample at the laboratory then reconstitute each core.
- 325 laboratory personnel remove sub-samples from the reconstituted cores for analysis and preparing composite samples representing each of the five tanks.
- 325 laboratory personnel sieve the composite samples and analyze the sieved fractions.
- ~~325 laboratory personnel prepare grouted coupons from core samples then monitor the grouted coupons for hydrogen generation. Baseline grout formulation will be provided by STP.~~
- 325 laboratory personnel return the transportation cask to the KW Basin.

The 325 laboratory will produce and deliver to the STP data packages that document the laboratory analysis performed. These data packages will be reviewed, verified, and validated as described in Section 5.0. The validated data will be compiled, calculations performed as necessary, and a report written documenting the results of the analysis. The report will evaluate data to determine that the DQOs and supporting assumptions identified in ~~Westcott 2008~~Westcott 2009 and Makenas and Baker 2007 have been achieved or confirmed.

The high-level working schedule for the sampling and analysis of the Container sludge is shown in Table 2-3. The schedule outlines the sample collection and analysis process and is shown for planning purposes. The actual detailed schedule governing the work may deviate from what is shown in Table 2-3 and will be maintained and updated by the STP.

Table 2-3. Container Sludge Activity Schedule

Activity	**Start**	**Finish**
Collect samples from 3 KE sludge containers (CON-240, CON-250, and CON-260) and deliver to laboratory	4/9/09	7/30/09
Receive sample and perform analysis of samples at laboratory	4/14/09	8/5/09
Collect samples from 2 KW sludge containers (CON-210 and CON-220) and deliver to laboratory	8/3/09	9/17/09
Deliver expedited KE sludge analytical data package to project	---	9/23/09
Evaluate KE expedited result to determine if additional samples from the KE sludge containers is necessary	9/24/09	10/08/09

Activity	Start	Finish
Deliver the balance of KE sludge analytical data package(s) to project	---	11/23/09
Deliver KW sludge analytical data package(s) to STP and PHMC/Safeguards	---	7/16/10
Prepare Data Report	7/19/10	9/20/10

2.4 Quality Objectives and Criteria

The objective of this QAPjP/SAP is to obtain data of known and appropriate quality to support,

- the termination of safeguards for the KW sludge in the Large Engineered Containers and
- the design of a safe and effective process to retrieve, treat, and package sludge so that it will meet the WIPP transportation and disposal requirements.

The specific data objectives reproduced from the two DQO reports (Makenas and Baker 2007; Westcott 2008Westcott 2009) are listed in Tables 2-1 and 2-2. The data criteria discussed below are established to ensure that these objectives are achieved.

The goal of sample collection and analysis supporting nuclear material accountability requirements is the determination of the concentration of nuclear material in KW sludge with variability minimized to the extent practical. Four samples for each of the KW sludge containers was determined to provide acceptable data as discussed in Section 2.6 of Makenas and Baker 2007.

The goal of sample collection and analysis supporting sludge retrieval and treatment design requirements are the establishment of sludge characteristics identified in Table 2-1. An acceptable variability in key radionuclide ratios to Cs-137 analysis results is established in Section 7.0 of Westcott 2008Westcott 2009 as ≤72 percent of the Relative Standard Deviation associated with the mean (% RSD[mean]). Variability of the key radionuclide ratios to Cs-137 will be calculated and compared to the 72% RSD(mean) performance criteria. If the variability exceeds the acceptable performance criteria then corrective action will be evaluated and action taken as necessary. For KE sludge containers, the sample design assumes that all three containers are one population; this assumption will be confirmed as described in Section 5.3.3.

Measurement performance criteria for sample analysis data are stated in terms of the desired level of uncertainty in the data that will be used to address the data quality objectives. The measurement performance criteria are stated as data quality indicators (DQIs) and, where possible, in quantitative terms. The data quality indicators (DQIs) applied to this sample and analysis campaign are defined in Table 2-4. Table 2-4 also describes methods that may be used

Table 2-4. Data Quality Indicators

Data Quality Indicator	Definition	Determination Methodologies
	If the magnitude and direction of the systematic error are known, the error is a known bias, and the data are typically corrected for the known bias.	equipment blank(s). The equipment blank will consist of KW basin water being pulled through the clean sample system.
Representativeness	Representativeness is a qualitative term that expresses the degree to which data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, a process condition or an environmental condition.	Representativeness is achieved through the collection of unbiased samples as described in the DQO report (~~Westcott 2008~~Westcott 2009). The ability of the sample collection apparatus and process to collect representative samples is demonstrated using surrogate material. Work documents implement the demonstrated sample collection equipment and process.
Comparability	A qualitative term that expresses the measure of confidence that one data set can be compared to another for the decision(s) to be made.	The comparability of the samples and analysis data will be maintained by using standard work documents/methods/procedures and trained/qualified personnel to collect, handle, transport, and analyze samples.
Completeness	A measure of the amount of valid data needed to be obtained from a measurement system.	The completeness required for all analyses performed on sample cores is specified in Table 3-8 or is 100% for those analyses listed in Table 3-7. The completeness required for all analyses performed on sample composites shall be ≥ 50%. All analyses that require 100% laboratory duplicates have a completeness

Results of any field calibrations and surveys shall be recorded as prescribed in work documents. References to any forms that were used, other data records, and the work documents followed in conducting the calibrations and surveys shall be recorded.

Chain-of-custody (COC) forms shall be prepared and maintained to identify and control samples in accordance with the requirements of Section 4.4 of HASQARD Volume 2. The COC will accompany the sample throughout its life-time.

2.6.2 Field Sampling Deviations

Changes from the sample collection, staging, and transportation described herein may be made in the field by the Lead Sampling Engineer with the concurrence of the STP Characterization manager or designated alternate. All deviations shall be recorded in the field log.

2.6.3 Laboratory Data Packages

The laboratory shall provide STP Characterization group with the following data packages:

- An expedited data package of the KE sludge core sample analyses for settled sludge density, total uranium, and the following radionuclides listed in Table 7-1 of the DQO (~~Westcott 2008~~Westcott 2009): Cs-13~~1~~7, U-235, Pu-238, Pu-234, Pu-240, Pu-~~240~~241, and Am-241.
- A data package containing the results of all analyses performed on core and composite samples from a given sludge container.
- ~~A data package containing the results of hydrogen generation tests.~~

Each data package shall include the information described in Sections 3.10.2 and 3.11.2. Documentation of the analysis of samples performed by the laboratory will be maintained in accordance with the laboratory QA program.

3.0 Data Generation and Acquisition

This section addresses data generation, acquisition and management activities, including the following:

- Sampling Process Design (Section 3.1)
- Sampling Methods (Section 3.2)
- Sampling Handling and Custody (Section 3.3)
- Analytical Methods (Section 3.4)
- Quality Control (Section 3.5)
- Instrument/Equipment Testing, Inspection, and Maintenance (Section 3.6)
- Instrument/Equipment Calibration and Frequency (Section 3.7)
- Inspection/Acceptance of Supplies and Consumables (Section 3.8)
- Non-Direct Measurements (Section 3.9)
- Data Management (Section 3.10)
- Final Reports and Communications (Section 3.11)

3.1 Sampling Process Design

The sampling of the sludge in the KW Sludge Containers will satisfy the requirements of the corresponding DQO documents (Makenas and Baker 2007 and ~~Westcott 2008~~Westcott 2009) by providing the required Safeguards/Accountability data plus characterization data supporting STP engineering/process and equipment development for disposition of the sludge to WIPP.

Consistent with SW-846 and WIPP requirements (WIPP 2003; WIPP 2008; NMED 2007) for characterization., a probabilistic sample design was used to select the locations of samples to be collected. As described in Section 3.1.2., sample locations available to collect core samples from each engineered container are constrained to the eight locations shown in Figure 3-1. These same eight locations are available for each of the engineered containers (SCS-CON-210, -220, -240, -250, and -260), because all of the engineered containers are of the same design.

Full vertical cores of sludge will be collected from each randomly selected sample location identified in each of the engineered containers using the apparatus discussed in Section 3.2. The apparatus does not collect an intact core (the sludge varying from compact to slurry like), but collects a mixture of sludge into a series of sample bottles that composes the entire core. Each core sample will produce between two and six sample bottles, each containing portions of the core, depending on the depth of the sludge in the container. Because the sampling apparatus does not provide an intact core, the expected original vertical variability of the sludge cannot be determined by analysis. However, the possible bounding vertical variability has been evaluated and estimated based on existing information about sludge and the process used to transfer the sludge from basin floors and pits as described in ~~Westcott 2008~~Westcott 2009, Appendix C. The bounding vertical variability was estimated to be approximately 25% and will be accounted for in the application of the final data to design analyses (e.g., variability used in WIPP drum estimates ~~Westcott 2008~~Westcott 2009, Section 7).

All of the sample bottles containing core material will be delivered to a laboratory for analysis. After receipt at the laboratory, the laboratory will reconstitute each core by mixing all the sample bottles that constitute one core into one bottle or equivalent. This is referred to as the core sample. Excess water will then be removed from each reconstituted core. Sub-samples from this reconstituted sludge core sample will then be subjected to analyses using the methods identified in Section 3.4.1, Table 3-4. The analyses of core samples will be used to establish the required sludge characteristics, including the key parameters required for the DQOs.

For each engineered container a composite sample will also be prepared by combining equal amounts of all core samples taken from each container into a single composite sample for that container. Sub-samples from each of these container composite samples will be subjected to analyses using the methods identified in Section 3.4.1, Table 3-5. Each container composite sample will be analyzed to measure corresponding parameters required by the DQOs (~~Westcott 2008~~Westcott 2009).

3.1.1 Basis for Selection of the Sampling Equipment and Shipping Cask

As part of the initial preparation for the current sampling campaign, Energy*Solutions* personnel experienced in both K Basin sludge characterization sampling and transportation methods for radioactive samples were contracted by the STP to review the initial proposed sampling campaign requirements of the Safeguards DQO document (Makenas and Baker 2007) and provide recommendations of the best sampling methods and sample transportation option. The conclusions of the Energy*Solutions* options study (Johnson 2007) were:

1. Sampling should be performed using the KBC Floor Sludge Sampler equipment used successfully in three previous sludge sampling campaigns at KE Basin (Makenas 1996, Pitner 1999, and Mellinger 2004).

2. Transport of the sludge samples to the laboratory should be made with a PAS-1 Cask system and supporting sample shipment system that was used in the three prior KE Basin Floor and Pit sludge sampling campaigns.

Both recommendations were made based on the knowledge that some minor modifications to prior methods and equipment would be needed. The STP management accepted these recommendations and applied them to subsequent considerations including sampling the containers with KE Basin sludge.

3.1.2 Sampling Locations in Each Container

As noted in Section 5.3 of the DQO document (~~Westcott 2008~~Westcott 2009), the construction of the engineered container and the need to design openings in the work grating mounted above the basin pool constrain the locations where sludge core samples can be collected. Each engineered container has a clear polycarbonate cover mounted on its top and is fabricated with internal piping, discharge heads, cover seams and lifting pins, flush water injectors, and supports mounted inside the container. The isolation tubes for this sampling campaign will be placed to avoid these external and internal interferences.

Holes must be drilled in the polycarbonate covers of each container to gain access to insert the sample isolation tubes at identified sampling locations. Further, special supporting guide frames must be mounted on the top of the containers and access holes must be designed into the grating above the basin pool (where the operators and shielded sample cart weighing ~1000 pounds are located) for the sampling campaign (see Appendix A, Figure A-3). Because of the engineering and construction effort needed to place and relocate the sample apparatus to new sample locations, the number of sample locations is limited to the eight identified in Figure 3-1.

The DQO documents (~~Westcott 2008~~Westcott 2009; Makenas and Baker 2007) discuss the selection of sampling locations in context with the basic objectives, including representativeness. In selecting these locations, careful consideration was made of the way sludge was loaded in the containers (refer to the Wescott 2008 DQO for a more detailed discussion). The sludge was pumped into each container through two distribution heads mounted symmetrically inside each container at a level of about 8 feet from the basin floor (see Section 1.3.2 and Appendix A). The eight sample locations selected were sited to vary the distance from the two distribution heads, thereby ensuring a representative horizontal cross-section of the sludge in each container was considered. Four of the sample locations were identified where sludge is somewhat more likely to contain larger and denser sludge particles (i.e., near distribution heads); these locations are identified with an "A" on Figure 3-1. Four of the sample locations were identified where sludge is somewhat less likely to contain larger and denser sludge particles (i.e., away from distribution heads); these locations are identified with a "B" on Figure 3-1. The locations from which samples will be collected were then randomly selected from among these eight locations.

The number of core samples required from each container to meet the data objectives is:

- Four core samples from each of the two engineered containers with KW sludge (SCS-CON-210 and SCS-CON-220) are needed to satisfy the DQO document for Safeguards information for sludge (Makenas and Baker 2007). This set of core samples will also satisfy the requirements of the DQO document for STP engineering/process -support (~~Westcott 2008~~Westcott 2009).

- A minimum of two core samples from each of the three engineered containers containing KE sludge (SCS-CON-240, SCS-CON-250 and SCS-CON-260) need to be collected and analyzed to satisfy the DQO document for STP engineering/process support (~~Westcott 2008~~Westcott 2009). This DQO document states that more than two core samples from each of these containers may be collected if needed. This decision will be based on evaluation of the data from the initial two core samples per container.

The following section addresses the random selection of the specific core sample locations from the eight potential locations for each container.

Table 3-2. Selection of Isolation Tube Diameter Based on Sludge Depth

Container Location Identification (see Figure 3-1)	Estimated Isolation Tube Diameters Container, SCS-CON-				
	210	220	240	250	260
A1	2"	2"	--	--	1"
A2	--	--	2"	--	--
A3	--	2"	--	--	--
A4	1"	--	--	1"	--
B1	1"	2"	1"	--	--
B2	--	--	--	1"	--
B3	1"	--	--	--	1"
B4	--	2"	--	--	--

Table 3-3a. Core Sample Estimates for Container with K West Floor and Pit Sludge (Based on Sludge Depths Measured in Containers as per Table 1-1)

Core Sample Identification (Based on Section 3.2.1)	General Location in Container (Fig. 3-1)	Total Sludge Depth to Local Bottom Plate (in)	Assumed Isolation Tube Diameter (in)	Theoretical Calculated Volume of Sludge Isolated in Core(ml)	Conservative Estimate of Sludge Volume Recovered* at Lab (ml)	Estimated Numbers	
						Sample Bottles	PAS-1 Shipment
SCS-CON-210							
KW210A1	A1	~~15.6~~17.4	2	~~859~~958	~~515~~574	6	3
KW210A4	A4	~~19.4~~21.1	1	~~276~~301	~~165~~180	2	1
KW 210B1	B1	~~22.6~~24.4	1	~~321~~346	~~192~~208	2	1
KW210B3	B3	~~24.6~~26.4	1	~~349~~374	~~209~~224	2	1
Total				~~1805~~1979	~~1083~~1186	12	6
SC-CON-220							
KW220A1	A1	4.2	2	232	140	2	1
KW220A3	A3	8.0	2	442	265	4	2
KW 220B2	B2	11.2	2	618	371	4	2
KW220B4	B4	13.2	2	726	436	6	3
Total				2018	1211	16	8

* Estimate is conservatively based on overall sample solids capture and laboratory recovery factors derived from prior core samples taken at KE, where the sludge was commingled with debris (> ¼ in material) as it lay on the basin floor. For the current campaign, the overall recovery is expected to be higher because materials have been handled multiple times and passed through ¼" screens prior to containerization.

Table 3-3b. Core Sample Estimates for Engineered Container with K East Sludge (Based on Sludge Depths Measured in Containers as per Table 1-1)

Core Sample Identification (Based on Section 3.2.2)	General Location in Container (Fig. 3-1)	Total Sludge Depth to Local Bottom plate (in)	Assumed Isolation Tube Diameter (in)	Calculated Volume of Sludge Isolated in Core (ml)	Conservative Estimate of Sludge Volume Recovered* at Lab (ml)	Estimated Numbers	
						Sample Bottles	PAS-1 Shipments
SCS-CON-240							
KW240A2	A2	14.515.0	2	800862	480469	6	3
KW240B1	B1	21.522.0	1	31205	1837	2	1
Total				11051138	6683	8	4
SC-CON-250							
KW250A4	A4	54.58	1	7727	463466	6	3
KW250B2	B2	65.03	1	9216	5526	6	3
Total				16921703	10151022	12	6
SC-CON-260							
KW260A1	A1	55.154.6	1	781774	468	6	3
KW260B3	B3	64.163.6	1	908901	5405	6	3
Total				16885	10131004	12	6

* Estimate is conservatively based on overall sample solids capture and laboratory recovery factors derived from prior core samples taken at KE, where isolation tubes may have included materials greater than ¼" and materials were originally settled (not previously handled/pumped). For the current campaign, the overall recovery may be higher because materials have been handled multiple times and passed through ¼" screens prior to containerization..

3.1.5 Basin Facility Preparations for Sampling

The KW Large Engineered Containers to be sampled and the area around the containers shall be prepared prior to the sludge sampling consistent with the final approved KW Basin work documents. The Lead Sampling Engineer shall establish the specific preparations as part of the overall sampling evolution and consistent with KW Basin Operations requirements. Example preparations include:

- Special grating sections over the containers will be prepared consistent with the sampling equipment requirements including holes for isolation tubes and the use of the shielded sampling cart (consideration for the cart's weight, approximately 1000 pounds, will be factored into all preparations).

- Building utilities and related support equipment, including: electrical power, service water and a viable underwater video camera system with local monitor and video/DVD/disk recorder, will be prepared.

- Sample Bottle Volume Measurement Method: A method to measure the approximate sludge solids volume in the 4-liter sample bottles after a sampling event shall be used. The range of volume increments shall be from 100 ml to 1200 ml in increments of not more than 200 ml. The method shall be reference volume marks placed on the sides of the bottles (such as with permanent ink) or use of a provided calibrated fixture that the bottles can be placed in for evaluation or a functional equivalent of one these two methods. The STP Sampling and Testing group shall provide a documented basis for the method used as part of the final sample data package (Section 3.11.1). Except for the 600 ml volume level, which will be used to confirm a shipping criterion is met, the volume measurements are for general reference for the sample team and laboratory. The sludge solids will be checked to be below the 600 ml indication prior to a bottle being shipping, per the PAS-1 OTRS (CHPRC, 2008~~FH 2008~~) shipping analysis.

- Sample Bottle Labels: The Lead Sampling Engineer shall assure each 4-liter sample bottle is labeled or marked in a durable, waterproof manner with an easily read unique sample number. The nomenclature for the sample number shall be "KW ZZZ-XX-N" [where "ZZZ" = the KW Large Engineered Container number (e.g., "210"), "XX" = the particular core sample number (e.g., "A1"; see Figure 3-1) and "N" = the particular sample bottle (A, B, C, D, E, etc.)]. The sample numbers shall be included on the Chain of Custody (COC) along with other information. The labels shall not interfere with basic ALARA and decontamination considerations (e.g., covered with smooth tape, etc.).

Sampling Team Preparations

The Lead Sampling Engineer and the STP Sampling and Test group will collaborate with the KW Basin Operations staff to prepare a trained sampling team. This team shall include subject matter experts and skilled crafts persons, who shall follow the final approved work documents when collecting the sludge core samples, packaging the resulting sample bottles, and shipping the bottles to the Laboratory using a PAS-1 cask.

Readiness

Prior to taking the first samples, a readiness review (Management Self-Assessment), including both KW Basin Operations staff and STP Sampling and Testing staff, shall be performed. Any identified issues resulting from this review shall be resolved prior to starting actual sampling. A readiness review plan (Dobson 2008b) for the assessment (e.g., including general basin preparations, sample transport, and the readiness to receive at the laboratory) has been developed.

3.2.3 Sampling Equipment Needed

The sampling system (generally illustrated in Figure 3-2) and its design requirements are described in the *System Design Description for the K West Basin Container Sludge System* (Snow 2008; see also Baker 1995b). The sampling system utilizes an "isolation tube," which is a special metal tube (or pipe) that is inserted into the sludge isolating a representative vertical core of the sludge from the sludge bed. The isolation tube has water inlet ports high above the bulk sludge bed to allow ingress of basin water during the sampling process. The isolated core of sludge is then vacuum transferred with a special extraction tube/wand into a set of 4-liter sample

Additional background drawings of the KW containers and sampling equipment are provided in Appendix A, including overviews of the KW Large Engineered Containers, the interface of sampling isolation tubes with the containers and basin operating grating, and a general schematic of the PAS-1 Cask used for transporting the sample bottles.

3.2.4 Planning for Off-Normal Operations and Sampling Problems

The System Design Description (Snow 2008) for the sampling campaign includes a review of off-normal operational events and the corresponding responses that should be followed during sampling evolutions. These off-normal conditions consider such things as plugging of the system and receiving higher dose material than considered in the ALARA report (Greenborg 2008).

- A primary contingency built into the sampling campaign is that, if the Lead Sampling Engineer finds a particular core sampling location has become compromised or not possible to obtain for some reason (e.g., through plugging, etc.), there are in-place hardware provisions for taking contingent core samples at other positions in each container (i.e., 4 to 6 contingent locations are available; Table 3-1 includes several randomly selected contingent locations for each container). The specific situation will be assessed by the Lead Sampling Engineer and, if necessary, a contingent sampling location substituted (with the concurrence of the STP Characterization manager)[2].

- The sampling system cart has several built-in safety features including the use of a third shielded "reserve" sample bottle that acts as an in-place backup for the functioning of a check valve system (Section 3.2.4). Should this bottle, due to a problem with the valve (which is very unlikely), end up with water and sludge sample in it, the Lead Sampling Engineer will assure it is shipped to the laboratory as part of subject core sample.

3.2.5 Performance Requirements

Section 3.2.3 includes summary performance core recovery requirements for the sampling equipment to be used in the campaign. The System Design Description (Snow 2008) provides additional background on performance requirements. Prior "cold" testing of the equipment (Bridges 1998) and recent supplemental testing (Lysher 2008) have confirmed the primary basic core recovery requirements have been met.

During actual sampling events, the Lead ~~Test~~Sampling Engineer shall monitor the performance of the equipment and will confirm performance indicators that are part of the K Basin sampling

[2] Similarly, if operational problems result in sample bottles containing sludge samples where the balance of the core material cannot or will not be drawn, the resulting bottles do not represent a valid core sample but still need to be dispositioned. The operational disposition of such bottles shall be decided through evaluation and a plan agreed to by the manager of STP Characterization, the manager of STP Sampling and Testing, and K Basin Operations.

procedure (e.g., pump vacuum gauge indicates at least 20 in Hg vacuum on the bottle system prior to the initial opening the sampling valve, etc.).

At the completion of sampling for each core, the Lead Sampling Engineer shall confirm that the total sludge core has been acquired by confirming the extraction wand has reached the bottom of each isolation tube (i.e., the bottom of the engineered container). A permanent mark on the extraction wand that can be compared to the top of the isolation tube will be used to confirm the extraction wand is at the fully inserted level. All final material drawn shall be deposited into the sample bottles, as evidenced by all significant sludge clearing the sampling tubing. These two indicators having been reached shall be noted in the sampling log by the Lead Sampling Engineer.

3.2.6 Information to be Collected

The Lead Sampling Engineer shall confirm the following information is recorded in the sampling log or supporting work documents for each sampling event.

The following critical information results from the field sampling activities:

- Container core sample identification and the corresponding identification numbers of the resulting sample bottles,
- Date and time each sample bottle was drawn,
- Initiated Chain of Custody tracking forms, and
- Completion and acceptability of the visual inspection and cleanliness of sampling equipment and sample bottles.

In addition, dose rate measurements on the sample bottles and shielded sample containers should be performed and reported to the 325 Building 24 hours prior to shipping the PAS-1 cask.

Related supporting general information includes the following:

- Corresponding confirmation for each core sample that extraction tube reached the bottom of the isolation tube at completion of sampling,
- Observed sludge depths from the two sets of markings in container,
- Confirmation of acceptable sampling system vacuum gauge pressure prior start of sampling,
- The room temperature when sample bottles are collected and stored at the basin, and
- If obtained, local depth of sludge noted on the isolation tube.

3.2.7 In Situ Monitoring Instruments

No in situ monitoring instruments will be used to generate data in this sampling campaign. Therefore, there are no requirements for instrument placement, operation, maintenance or corresponding data handling.

3.3 Sampling Handling and Custody

3.3.1 Sample Transport in the PAS-1 Cask

Once filled with water and sludge, each pair of 4-liter sample bottles will be moved from the shielded sampling cart and staged for shipment in a PAS-1 cask (Section 3.1.2 and Appendix A, Figure A-4). K Basin work documents will detail the preparations and shipment of the cask. Prior to shipment in the cask, the 4-liter sample bottles will be placed into Shielded Sample Containers, SSCs (Figure A-4), that in turn will be placed into the PAS-1 cask. The cask will be assembled and transported from the KW Basin to the Laboratory.

The shipping of the sample bottles in the cask shall be made in accordance with the shipping safety document (i.e., the One Time Request for Shipment or OTRS, CHPRC, 2008FH 2008). A criterion of the OTRS is a limit on the volume of settled sludge solids in each sample bottle. Based on previous campaigns, a maximum volume was conservatively set at 600 ml. The basis for this limit is provided in the System Design Description for the sampling equipment (Snow 2008). No bottles approaching this level of solids are expected from this sampling campaign. A method shall be provided for measuring the 600 ml volume level in each of the 4-liter sample bottles (Section 3.2.2). This volume shall be checked against observed significant solids layers in each bottle prior to shipment consistent with K Basin work documents.

Due to washing and packaging operations performed on degraded spent fuel elements in the KW Basin, there is the potential for higher levels of metallic uranium particles to be present in the containerized KW floor sludge than is found in KE floor sludge. As a precaution to avoid any flammability concerns related to the potential build-up of hydrogen (through generation from uranium metal oxidation) during sample transport, the OTRS for the PAS-1 cask requires the cask to be backfilled with inert gas (e.g., helium) prior to being sealed for shipment. This criterion will be incorporated into the K Basin work documents and checklists used for shipping.

3.3.2 Recording of Information in Field Notes

The sampling team will follow KW Basin work documents to collect the sludge core sample bottles. The Lead Sampling Engineer, under the cognizance of the STP Sampling and Testing group manager, shall direct the sampling team with respect to the sampling objectives and process. The Lead Sampling Engineer shall ensure the sample bottles are properly identified with unique sample numbers recorded on permanent labels (Section 3.2.2) and the sampling locations are verified/recorded in accordance with HASQARD Volume 2, Section 4.2, prior to the sample bottles being moved from the sampling cart. The Lead Sampling Engineer will assure the sampling log entries and procedure entries are maintained as work progresses. Since the sampling events will occur in a radiation zone, accommodations to avoid contamination of the sample log will be made consistent with ALARA considerations.

As each set of bottles is taken the container, sample location, bottle numbers, date, time, temperature, depth of sludge and any observations shall be recorded for each core sample. (See Section 3.2.6.)

3.3.3 Sample Bottles

The sample bottles used for this campaign to contain and transport the sludge samples are heavy walled polypropylene 4-liter bottles (i.e., Nalgene 002126 or equivalent). Closure lids used for shipping are vented. Handling bails are attached to outside of the bottles to provide for ease of handling consistent with ALARA methods. Sample bottles ~~and lids~~ shall be cleaned and ~~sealed~~ protected in accordance with the requirements of Section 3.2.2~~3~~.

3.3.4 Sample Tracking

The Lead Sampling Engineer shall ensure documented custody traceability for each sample bottle is started, and plans are in place for tracking during handling and transportation using COC protocol. Chain-of-Custody shall start once a filled bottle is removed from the cart. The COC form used shall be CHPRC/FH form A-6003-432 or an equivalent approved by the manager of the STP Characterization group. After the samples are accepted by the Laboratory, custody is maintained by assuring the samples are in the possession of an authorized individual, in that individual's view, in a sealed or locked container controlled by that individual or in a secure controlled-access location. Sample custody shall be maintained until the sample is released by the STP Characterization manager or until the sample is expended.

Unique sample identification numbers (see Section 3.2.2) shall to be used to track the sample bottles.

Custody seals shall be used on the sample bottle lids, if feasible. The potential highly radioactive nature of the samples and potential contamination spread may preclude the use of any custody type seals (i.e., ALARA concerns). The Lead Sampling Engineer with K Basin Operations staff shall assess the field situation and, if custody seals are inadvisable, confer with the manager of STP Characterization group to define administrative controls to use to ensure samples are not tampered with.

During normal operations, the third bottle in the sampling cart is a reserve (Section 3.2.4) and is not filled nor shipped to the Laboratory. In the unlikely event this bottle receives a portion of the core sample, the bottle is to be handled in the same manner as the other sample bottles. The Lead Sampling Engineer shall maintain the status of this bottle in the log.

3.3.5 Holding Times and Preservation

The filled sample bottles shall be shipped to the laboratory and processed expeditiously. The analyses for metals (i.e., ICP analyses) identified in Section 3.4 shall be completed within six (6) months from when the sample bottle of a core sample is taken from the K Basin container.

These sludge samples will also be analyzed for uranium metal content. To minimize calculated adjustments to the resulting measured data due to uranium reactions during storage and handling, samples shall be shipped to the laboratory as soon as feasible. As a target, sample bottles should be shipped from KW Basin to the laboratory within 12 days of being collected. The Lead Sampling Engineer shall monitor and inform the manager of STP Sampling and Testing and the manager of STP Characterization of storage periods extending beyond this for their evaluation.

Once the samples are at the laboratory, they shall be stored and handled to minimize water evaporation. The samples shall be monitored to assure sludge samples remain covered with water during storage.

Chemical preservation of sample sludge will not be performed because it does not make allowance for slurries and might adversely affect the sample (e.g., pH change, etc.). No temperature preservation steps are specified for these radioactive samples, given the physical constraints to transport and work with the samples in the hot cell.

Handling Caution
While significant hydrogen gas generation is not expected with these samples at room temperature, safety precautions should be reviewed at the laboratory when sealing small containers with this sludge. In past campaigns, no visual evidence of gas generation was noted in the KE Basin Floor and Pit sludge samples; however, KW floor and pit sludge may include more canister and related sludges, which have been observed to generate hydrogen at times. Loading and shipment of the PAS-1 cask, which is used for sample transport, will follow the requirements of the OTRS (CHPRC, 2008FH 2008) and use a helium backfill.

3.3.6 Handling After Initial Analyses Completed

Once all laboratory analyses noted in this plan are complete, documented, validated and accepted by the STP, a decision will be made regarding the disposition of the sludge samples. This decision will be made by the appropriate CHPRC STP contract Buyer's Technical Representative (BTR) and communicated to the Laboratory. The laboratory may be asked to 1) dispose of any remaining excess sludge samples and water samples from this campaign or 2) transfer the excess sludge and water samples to be archived under the CHPRC sample archive contract. Archived samples are held in case there is a need for additional future analyses supporting STP. Because of the investment in resources to obtain this sludge sample material, the use of the excess sludge sample material for other STP objectives (e.g., process validation, disposal option verification, etc.) will be evaluated by the BTR prior to disposal.

3.3.7 Regulatory Considerations During Handling of Sludge Samples

The following background information related to the handling the K Basin sludge samples during transport and at the laboratory is provided for reference:

Sludge is a CERCLA waste. As such the following applies: *CERCLA wastes may not be transferred back to the CERCLA site (i.e., 100 K Area) unless the Remedial Project Manager or OSC (i.e., on-scene coordinator) assures the proper management of the CERCLA waste samples*

Section 3.4.6 provides the specific analyses and tests the laboratory needs to perform on each core sample. Section 3.4.7 provides the specific analyses and tests the laboratory needs to perform on the each of the five container composite samples.

3.4.1 Overview of Requirements for Sample Compositing

This section summarizes and reviews the general objectives and laboratory analyses/tests required by the controlling DQO documents for this sampling campaign. It also provides the background logic for additional sample compositing, fractionating, and sub-sampling. The subsequent sections in Section 3.4 then provide the specific requirements to the laboratory needed to accomplish these general objectives.

The DQO document supporting STP engineering/process needs (Westcott 2008Westcott 2009) concluded in the analytical approach development step (Section 6.0) the parameters that will be calculated from the analysis of sludge samples are to come from two sources:

1. Analyses made on sub-samples taken directly from the individual core samples taken from each container. These analyses are summarized here in Table 3-4a and 3-4b and illustrated in Figure 3-4 (a generalized flow chart, not intended to show all operational handling steps).

2. Analyses made on sub-samples taken from composite samples made for each of the five containers. Equal quantities of all cores taken from a container are combined to make the composite sample representing that container. These analyses are summarized in Table 3-5 and illustrated in Figure 3-5 (a generalized flow chart, not intended to show all operational handling steps).

The parameters required for the Accountability DQO (Makenas and Baker 2007) are a sub-set of the parameters noted in Item 1 above. The Accountability DQO document focuses on the characterization of specific nuclear material concentrations in the sludge of core samples from SCS-CON-210 and -220. While these nuclear material parameters are already required to be analyzed as part of Item 1, there is an additional set of requirements stemming from the Accountability considerations that need to be considered as part of these Laboratory analyses to satisfy the subject Data Quality Objectives. A FH Safeguards group representative must review and concur with the subject laboratory procedures prior to use, and the balances used for weighing must be checked for linearity and accuracy on the day the balance is used.

Table 3-4a. Analyses Performed on Each Core Sample

Sample Preparation Step	Measured Parameter	Analysis Method
Settle sample and remove excess water	Settled sludge density[1]	Measure mass and volume of settled sludge
Collect settled sludge sub-samples for testing	Weight percent solids in settled sludge	Sub-sample of settled sludge weighed wet then dried and weighed again
	Uranium metal concentration	Selective dissolution for uranium metal, with U by Kinetic Phosphorescence Analysis or Inductively Coupled Plasma Analysis
	Total Organic Carbon	Hot persulfate oxidation, combustion oxidation or both
Collect settled sludge sub-samples, dry and perform acid dissolution	Cs-137 (and other measured gamma emitters) concentrations[1]	Gamma energy analysis
	Pu-238, Pu-239/Pu-240, Am-241, Np-237 concentrations	Separations and Alpha energy analysis
	Pu-239, Pu-240, Pu-241, Pu-242, U-233, U-234, U-235, U-238 isotopic abundance[1]	Thermal ionization mass spectroscopy
	Total uranium concentration[1]	Kinetic Phosphorescence Analysis or Inductively Coupled Plasma Analysis
	Metals (including Table 3-4b except mercury) concentration	Inductively coupled plasma
	Sr-90 concentration	Separation and beta counting
Rinse, dry, and weigh, then subsample solids remaining from acid dissolution	Mass fraction not digested	Dry and weigh un-dissolved sample and use sub-sample volume or mass that was digested to calculate the fraction
	Gamma count	Dry and subject to gamma energy analysis
	Identify crystalline structures	Dry and subject to x-ray diffraction
~~Collect settled sludge sub-sample then grout. The grouted waste form is sealed and gas samples are drawn off and submitted for analysis at specified times.~~	~~Hydrogen gas concentration in head space over time~~	~~Mass spectroscopy~~

[1] These measured parameters comprise the data required in the KE sludge expedited data packages. (See Section 2.6.3.)

Table 3-4b. WIPP Hazardous Waste Facility Permit Metals

Antimony (Sb)	Cadmium (Cd)	Nickel (Ni)	Vanadium (V)
Arsenic (Ar)	Chromium (Cr)	Selenium (Se)	Zinc (Zn)
Barium (Ba)	Lead (Pb)	Silver (Ag)	--
Beryllium (Be)	Mercury[a] (Hg)	Thallium (Tl)	--

[a]As described in the DQO Section 3.8 ~~Westcott 2008~~Westcott 2009, mercury will not be analyzed for in this sample and analysis campaign.

Figure 3-4. Generalized Core Sample Analysis Flowchart.

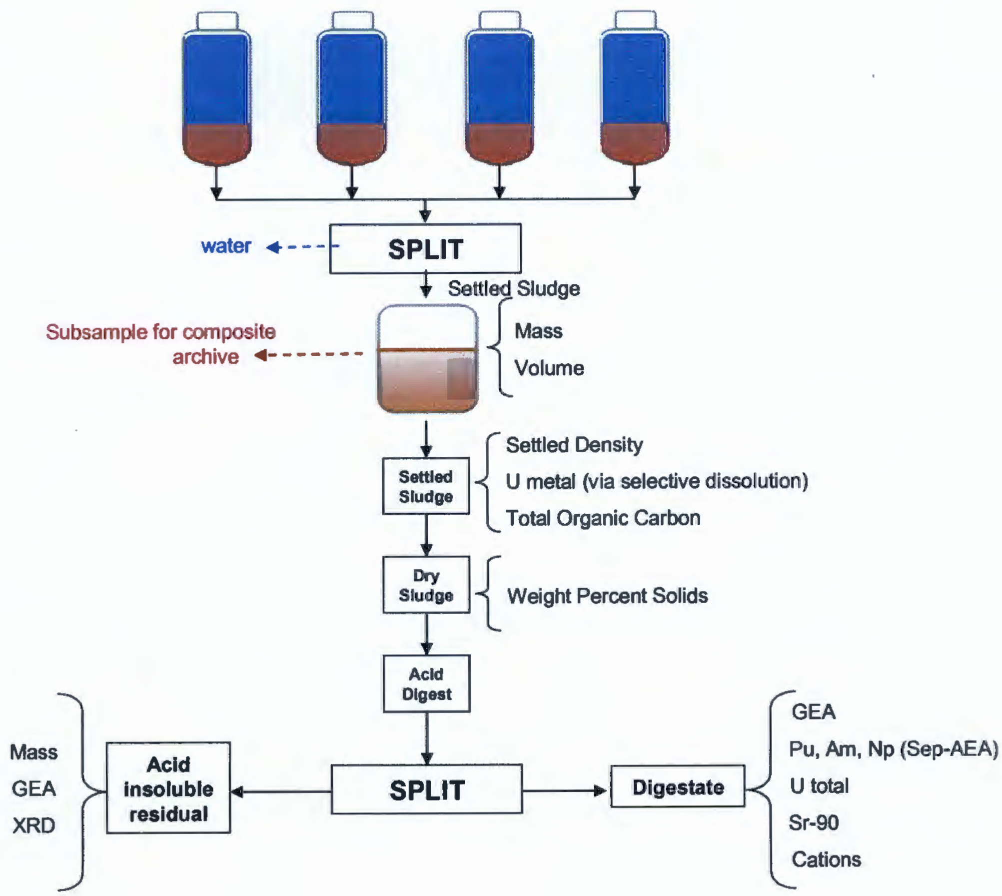
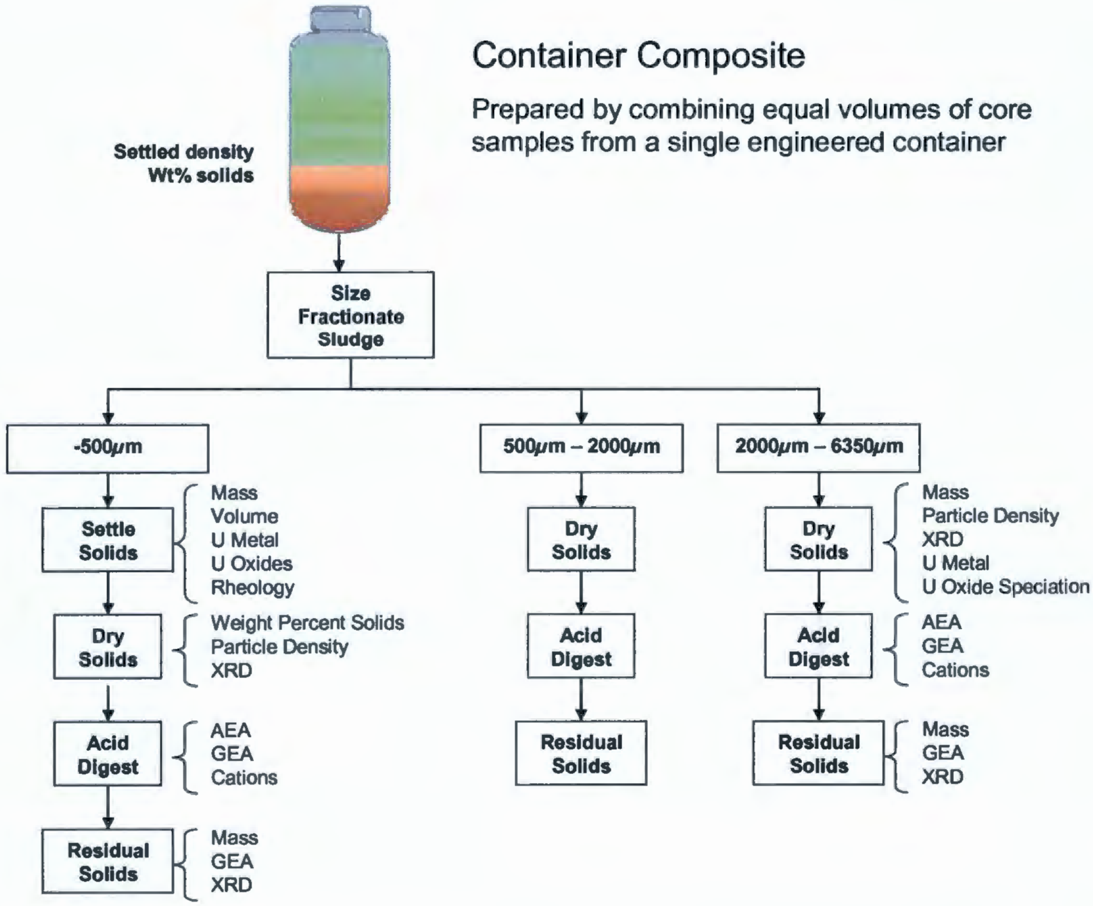


Figure 3-5. Generalized Container Sludge Composite Sample Analysis Flowchart.



3.4.2 Laboratory Requirements for Sample Handling

During each step of handling and storage of the sludge samples at the laboratory, the sludge samples shall be maintained in a saturated state and not be allowed to dry. Samples shall be maintained wet using the basin water provided with the sludge samples, if possible; otherwise clean laboratory de-ionized water may be used. Past laboratory samples of K Basin sludge have demonstrated rapid evaporation of cover water, even when sample bottles are apparently tightly capped. Laboratory Test Instructions (approved by the STP BTR) for handling the samples will include specific criteria on initial periods for monitoring samples for water loss. Samples can be stored at room temperature, as noted in Section 3.3.5.

The sample bottle shipped from KW Basin containing in-basin equipment blank samples (i.e., basin water) shall be analyzed ~~similar to~~as specified in Section 3.5.2 ~~core sludge samples~~ with 500 ml of the excess stored as a contingency for future analyses. These samples shall not be disposed of except with the concurrence of the STP contract BTR.

- Archived for use in maintaining the sludge in a wet condition through its analysis and storage.
- Archived for potential analyses until all planned analyses of the solids are completed.
- Disposed of through appropriate methods (given the sludge is a PCB remediation waste).

Because unforeseen situations may develop during sampling, it may be necessary for the STP to provide the Laboratory additional instructions and/or information with respect to the condition of the samples, basic handling and/or analysis noted herein. If this becomes necessary, the Manager of STP Characterization and the STP contract BTR will provide additional information by written instructions to the Laboratory.

3.4.4 Sub-sampling of Individual Core Samples from Each Container

Once each sludge core sample has been consolidated from its source 4-liter sample bottles and the requirements of Section 3.4.3 are complete, the Laboratory will proceed to mix the sample thoroughly to homogenize the material of each core sample and obtain the representative sub-samples of the sludge for the required analyses described in Section 3.4.6. The analyses address the parameters and tests previously summarized in Tables 3-4a and 3-4b and Figure 3-4. The steps for this sub-sampling shall be defined in a Laboratory Test Instruction approved prior to use by the Manager of STP Characterization or the STP contract BTR.

Two independent sub-samples (a main sample and Client Duplicate sample) will be collected from each of the core samples as identified in Table 3-6. For characterization analyses in which sample preparation is required, each sub-sample (main and duplicate) shall go through separate sample preparation.

After the sub-sampling per this section is complete, the additional sub-sampling required in Section 3.4.5 can be taken or cover water should be added back to the core sludge samples to prevent samples from drying out.

3.4.5 Compositing Samples for Each Container

As noted in Section 3.4.1, a requirement of the process DQO document (~~Westcott 2008~~Westcott 2009) is the creation of a single composite sample representing each of the five containers, in addition to the analyses of the individual core samples. Once each individual sludge core sample has been consolidated from its source 4-liter bottles and the requirements of Section 3.4.4 are completed, the Laboratory can proceed to assemble the single composite sample for each container using equal volumes of each core sample taken from that container. If equal volumes of each core sample do not provide ample material for analysis, the manager of STP characterization shall be notified and another methodology may be proposed to develop composite samples. The steps for this composite development shall be defined in a Laboratory Test Instruction approved prior to use by the Manager of STP Characterization or the STP contract BTR. The Test Instruction will address the volumes of sub-sample required for subsequent analyses as per Section 3.4.7.

The sub-samples used to make this composite can be taken from the core samples at the same time each is handled as part of the tasks in Section 3.4.4 or at a separate time. In either case, all sludge samples shall be kept in a wet condition during handling. Client duplicates of composite samples are not required.

Using the approved Test Instruction, the Laboratory shall then fractionate (sieve) the resulting composite samples into three particle size population range sub-samples, as follows:

1. 6350 to 2000 microns,
2. 2000 to 500 microns, and
3. less than 500 microns.

Sub-samples of these will then be taken for the analyses and tests prescribed in Section 3.4.7 as per the requirements noted in Section 3.4.1. With approval of the STP Characterization manager, minor adjustment of the sieve sizes may be made in order to respond to the needs of STP Engineering. Any such changes must be part of the approved Test Instruction.

3.4.6 Laboratory Compositional Analyses of Individual Core Samples

The required laboratory analyses for the individual core samples are summarized in Section 3.4.1 of the current document Tables 3-4a and 3-4b and Figure 3-4. The following section provides specific laboratory requirements for the compositional analyses of the core samples as well as supporting information. The specifications of analytical evaluations are divided into two parts to assist in responding to the specific objectives of the two subject DQO documents:

- Part 1 Analyses: Initial handling/recovery/consolidation of the multiple 4-liter sample bottles containing each sludge core sample and the laboratory analyses required for measuring radioactive concentrations are described in Tables 3-6 and 3-7. These analyses address the basic physical parameters required for the sludge samples, the Accountability objectives (Makenas and Baker 2007) and radioactive analyses required for STP sludge engineering/processing objectives (Westcott ~~et al. 2008~~2009)

- Part 2 Analyses: Laboratory analyses and tests required for the balance of specific STP sludge engineering/processing objectives (~~Westcott 2008~~Westcott 2009) are provided in Table 3-8. These analyses address measurements of uranium metal fuel concentrations, radioisotopes from fission and activation products, overall specific dose rates, and characterization of metals present~~, and hydrogen gas generation of the sludge in a defined STP prototypic grout matrix~~.

Table 3-9 further defines terms in the tables provided in this section on analytical requirements.

The laboratory analyses are to be performed to the requirements noted in Tables 3-6 through 3-8, and in accordance with the HASQARD, Volumes 1 and 4 (2007).

Table 3-6. Sample Laboratory Analyses and Testing
(Addressing Accountability and Engineering/Process Development Parameters)

Requested Evaluation or Analysis		Analysis Technique	Constituent to be Reported	Reporting Units[(1)]	Duplicate Analysis[2]	
					Yes	No
1	Density of settled sludge	Gravimetric	Density[3]	g/ml	X	
2	Weight fraction solids (in wet sludge)	Drying at 105°C and gravimetric	Weight fraction solids[(3)]	$g_{dry}/g_{as\text{-}settled}$	X	---
3	During acid preparation digest and/or fusion, confirm full dissolution of sludge material for each sub-sample	Visual observation, gravimetric, and gamma count	Confirm by inspection; report if significant material remains after dissolution and/or fusion. Confirm no material remains. [(3, 4)]	$g_{dry}/g_{as\text{-}settled}$	---	X
4	U Total	KPA	Total Uranium	µg/g	X	---
5	U Isotopics and Pu isotopics	TIMS	U and Pu Isotopes including: ^{233}U, ^{234}U, ^{235}U, ^{236}U and ^{238}U, ^{238}Pu, ^{239}Pu, ^{240}Pu, ^{241}Pu, and ^{242}Pu	Isotope atom% of U & Pu, respectively	X	---
6	^{238}Pu, $^{239/240}$Pu, and ^{241}Am	Separation and Alpha Energy Analysis (AEA)	^{238}Pu, $^{239/240}$Pu, and ^{241}Am	µg/g (as applicable), µCi/g	X	---
7	Neptunium	Extraction and AEA	^{237}Np	µCi/g	X	---
8	Conversion of U and Pu atom% isotopics to sludge matrix units[(5)]	From Analysis #4, #5, and #6	Isotopic concentrations per gram of original as-settled sludge matrix	µg/g, µCi/g	X	---

General Note: Acronyms used in this table are defined in Table 3-9.

1 Units of weight fractions are to be based on original, as-settled sludge matrix.

2 Each core sample will have two independent samples taken (one main sample and a client duplicate) and analyzed

3 Weights will be determined using a calibrated balance. Balance checks (linearity and accuracy) are to be performed each day the balance is used. (Note: narrative provided in documentation on sample preparation will provide the calculation used to determine the sludge settled density.)

4 If any significant (>5 wt% of original material (as-settled) being analyzed or any high gamma material observed) material remains, confer with PNNL Project Manager and the STP Characterization manager-.

5 For the calculation, first convert atom% abundance to mass abundance. Using results from U total, ^{235}U, ^{238}U, ^{238}Pu, $^{239/240}$Pu, and mass abundance, determine mass and Curies of each isotope. The isotopic abundance values for ^{238}Pu (via TIMS) may be unreliable due to likely contamination of the much more abundant isobar ^{238}U. Therefore, ^{238}Pu via separation AEA will likely provide more reliable data.

Table 3-7. Quality Control for Sample Analyses from Table 3-6

Analysis[1]	Required Detection Limit	Preparative Duplicate[2] (Precision)	Post Preparative Matrix Spike[3]		Laboratory Control Standard ~~Recovery~~[4]	
			~~Accuracy~~Recovery	Frequency / Preparative Batch	~~Accuracy~~Recovery	Frequency / Preparative Batch
U by KPA	10 µg/g	± 20%	± 25%	1	± 20% ± 7.5%	1 – blank spike 2 – calib. check
U-TIMS	NA	± 20%	NA	NA	± 2%	1
Pu-TIMS	NA	± 20%	NA	NA	± 6%	1
^{238}Pu $^{239/240}$Pu ^{241}Am Separation, AEA	1×10^{-3} µCi/g 1×10^{-3} µCi/g 1×10^{-3} µCi/g	± 20% ± 20% ± 20%	NA ± 25% ± 25%	NA 1 1	NA ± 20% ± 20%	NA 1 1
^{237}Np-AEA	1×10^{-3} µCi/g	± 20%	± 25%	1	± 20%	1

General Note: Acronyms used in this Table are defined Table 3-9.

(1) All analyses will be performed to standard laboratory procedures.

(2) Note: For each core sample, two independent samples shall be prepared and analyzed; a Main sample and a Client Duplicate. For purposes of precision QC, the Main Sample and Client Duplicate from each core are to be evaluated for conformance with the "preparative duplicate" requirement. For TIMS analyses, the ± 20% precision applies to the isotopic abundance of major isotopes in the Main and Client Duplicate samples for each core (major isotope = isotope abundance greater than 1% of the total sample).

(3) For Pu analyses, ^{239}Pu will be used for post preparation matrix spike. Tracers (internal standards, ^{242}Pu and ^{243}Am) will be added to each sample analyzed for Pu and Am, respectively, and internal standard recovery results will be used to adjust reported data. Matrix spikes for Pu, Am, and Np analyses will be added after sample preparation, but before the Pu, Am, and Np chemical separations (i.e., post-preparation).

(4) The Laboratory Control Standard (LCS) for Pu, Am, and Np analyses will be processed through the Pu, Am, Np chemical separations (i.e., serves as a Method LCS).

Action if Criteria are Exceeded: If the precision between duplicates or the spike or LCS recoveries exceed the appropriate criteria, the following action shall be taken: Consult the PNNL Project Manager, who will communicate with the ~~KBC/~~STP Project Coordinator and Safeguards representative to determine if a rerun is needed. If needed, make one rerun to see if acceptable results are obtained (if acceptable results are not obtained, report the QC failure to PNNL Project Manager, who will contact the ~~KBC/~~STP Project Coordinator).

Action "Less Than" Result is Obtained: If a "less than" value for a key analyte is obtained, consult the PNNL Project manager, who will communicate with the ~~KBC/~~STP Project Coordinator and Safeguards representative. An evaluation of circumstances surrounding the result (sample size, dilutions, actual uncertainty of results etc.) will be performed to determine if the result is due to sample processing or an actual low value that is not measurable. If the result is due to processing, a rerun of the sample will be performed to provide the requested results. It is anticipated that all samples will be processed so as to provide usable data for the project.

Table 3-8. Additional Laboratory Analytical and Testing Requirements for Core Samples Addressing Specific STP Engineering/Process Development Parameters

Process	Parameter	Lab Procedure or Test Instruction?	Completeness Required	Required MDL(1)	Precision(2)	Accuracy(3)
Gamma Energy Analysis (GEA) (5)	^{134}Cs	Procedure	100%	0.20 µCi/g	±20%	---
	^{137}Cs			0.015 µCi/g	±20%	SPC
	^{60}Co			0.010 µCi/g	±20%	---
	^{241}Am			15.0 µCi/g	±20%	---
	^{152}Eu			1.5 µCi/g	±20%	---
	^{154}Eu			1.5 µCi/g	±20%	---
	^{155}Eu			2.0 µCi/g	±20%	---
Separation and Beta Counting	^{90}Sr	Procedure	50%	1.0 µCi/g	±20%	±25% Spike
Inductively Coupled Argon Plasma (ICP) ~~(8)~~(7)	Al	Procedure	50% or 100%~~(9)~~(8)	100 µg/g	±20%	±20% Spike
	As			20 µg/g	±20%	±20% Spike
	Cd			4 µg/g	±20%	±20% Spike
	Fe			100 µg/g	±20%	±20% Spike
	Ba			400 µg/g	±20%	±20% Spike
	Cr			20 µg/g	±20%	±20% Spike
	Pb			20 µg/g	±20%	±20% Spike
	Ag			20 µg/g	±20%	±20% Spike
	Be			20 µg/g	±20%	±20% Spike
	Tl			20 µg/g	±20%	±20% Spike
	Zn			20 µg/g	±20%	±20% Spike
	Se			20 µg/g	±20%	±20% Spike
	Mg			200 µg/g	±20%	±20% Spike
	Ca			200 µg/g	±20%	±20% Spike
	Na			200 µg/g	±20%	±20% Spike
	Zr			20 µg/g	±20%	±20% Spike
	Ni			20 µg/g	±20%	±20% Spike
	Sb			20 µg/g	±20%	±20% Spike
	U			1000 µg/g	±20%	±20% Spike
	V			20 µg/g	±20%	±20% Spike
	~~Mass, gamma emitters, and identify materials with crystalline structures of digestate residue~~			~~(6)~~	~~(6)~~	~~(6)~~

Table 3-8. Additional Laboratory Analytical and Testing Requirements for Core Samples Addressing Specific STP Engineering/Process Development Parameters

Process	Parameter	Lab Procedure or Test Instruction?	Completeness Required	Required MDL[1]	Precision[2]	Accuracy[3]
Recover and characterize any solids remaining after sample preparation by acid dissolution	Recover measure mass, gamma emitters, and identify materials with crystalline structures in digestate residue by XRD ~~TOC/TC~~	Procedure~~Test instruction and XRD procedure~~	50% or 100%~~(10)~~(9)	(6)~~200 µg/g~~	(6)~~±20%~~	(6) ~~±25% Spike~~
Hot Persulfate Oxidation or Combustion Oxidation	TOC/TC~~Density/weight % solids~~	procedure ~~Procedure~~	50%	200 µg/g~~N/A~~	±20%~~N/A~~	±25% Spike~~N/A~~
Gravimetric Analysis	Density / weight % solids~~Selective dissolution with uranium analysis –or– measurement through gas generation testing~~	Test Instruction [4]	100%	N/A~~0.01 wt% (settled sludge basis)~~	N/A~~±20% (when [U_{metal}] > 0.1 wt%)~~	N/A~~25% for LCS~~
Uranium Metal Content	Selective dissolution with uranium analysis –or– measurement through gas generation testing~~Gas volume unit of time (e.g., hours), volume, and temperature of sludge~~	~~Test Instruction~~Procedure	75%	0.01 wt% (settled sludge basis)~~(7)~~	±20% (when [U_{metal}] > 0.1 wt%)~~(7)~~	25% for LCS~~(7)~~
~~Gas Generation Rate in Grout Matrix~~	~~H, He, Ar, N_2, Xe, Kr, and any other significant gases~~	~~Test Instruction~~	~~75%~~	~~0.001 mol%~~	~~10%LCS~~	~~10% for LCS~~

Table 3-8. Additional Laboratory Analytical and Testing Requirements for Core Samples Addressing Specific STP Engineering/Process Development Parameters

Process	Parameter	Lab Procedure or Test Instruction?	Completeness Required	Required MDL(1)	Precision(2)	Accuracy(3)
~~Gas Composition of gas from grout matrix test~~		~~Procedure~~	~~75%~~			

(1) Required MDL has been established based on expected concentrations and use of the data, taking into account different capabilities of the laboratory's equipment and analysis methods.

(2) Measured sample precision is to be 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% except for ICP which should be ±20%. All method standard recoveries should be within statistical process control (SPC).

(4) The sample preparation Test Instruction will provide the calculation used to determine settled density.

(5) Other opportunistic gamma emitters in library should be reported if available (e.g., ^{60}Co, ^{106}Ru/Rh, ^{241}Am, ^{244}Cm, ^{95}Nb, etc.).

(6) ~~Equivalent~~ The MDL, precision and accuracy values for ~~the parameters being determined by multiple types of sequential analyses based on Test Instructions (TI) shall be addressed in the final TI that will be approved by the manager of STP Characterization or the STP BTR~~the various analyses cannot be specified due the high variability in quantity and constituents that may exist in the residue. If >5.0% of residue is a crystalline phase then it is detectible by XRD.

~~(7) Required MDL depends on test vessel, sample size, test interval (time) and other related factors. Calculations have demonstrated results from planned tests can be scaled to 55 gallon drums. Precision and accuracy targets, of 20% and 25% respectively, shall be addressed in the approved Test Instruction.~~

~~(8)~~(7) While conducting Inductively Coupled Argon Plasma-Optical Emission Spectroscopy (ICP) analyses, results from the following analytes are to be reported on an opportunistic/best efforts basis: boron (B), bismuth (Bi), copper (Cu), potassium (K), manganese (Mn), and phosphorous (P). While not specifically identified as analytes of interest for the current characterization effort, these analytes have been requested in previous characterization campaigns and the results are of interest for consistency between data sets. Any other analyte present at a concentration greater than the instrument detection limit for that analyte is to be reported; however, specific QC criteria may not be applicable for such analytes

~~(9)~~(8)The metals Antimony, Cadmium, Nickel, Vanadium, Arsenic, Chromium, Selenium, Zinc, Barium, Lead, Silver, Beryllium, and Thallium have a 100% completeness requirement, all other metals have a 50% completeness requirement.

~~(10)~~(9)If significant gamma radioactivity is measured in the residue then 100% completeness is required, otherwise 50% completeness is applicable.

- In addition, for the fraction with particle sizes less than 500 microns, the samples will be characterized using the two rheology parameters of viscosity and shear strength per Table 3-10.

The laboratory analyses are to be performed to the requirements noted in Tables 3-10 and in accordance with the HASQARD, Volumes 1 and 4 (2007).

Table 3-10. Analytical and Testing Requirements for Composite Samples and Sub-Samples

Process	Parameter	Standard Procedure or Test Instruction?	Required MDL[(1)]	Precision[(2)]	Accuracy[(3)]
Gamma Energy Analysis (GEA)[(5)]	^{134}Cs	Procedure	0.20 μCi/g	±20%	---
	^{137}Cs		0.015 μCi/g	±20%	SPC
	^{60}Co		0.010 μCi/g	±20%	---
	^{241}Am		15.0 μCi/g	±20%	---
	^{152}Eu		1.5 μCi/g	±20%	---
	^{154}Eu		1.5 μCi/g	±20%	---
	^{155}Eu		2.0 μCi/g	±20%	---
Inductively Coupled Argon Plasma (ICP) for Metals[(7)]	Al	Procedure	100 μg/g	±20%	±20% Spike
	As		20 μg/g	±20%	±20% Spike
	Cd		~~1~~4~~5~~ μg/g	±20%	±20% Spike
	Fe		100 μg/g	±20%	±20% Spike
	Ba		~~1~~400 μg/g	±20%	±20% Spike
	Cr		20 μg/g	±20%	±20% Spike
	Pb		200 μg/g	±20%	±20% Spike
	Ag		20 μg/g	±20%	±20% Spike
	Be		~~1~~20 μg/g	±20%	±20% Spike
	Tl		~~40~~20 μg/g	±20%	±20% Spike
	Zn		20 μg/g	±20%	±20% Spike
	Se		200 μg/g	±20%	±20% Spike
	Mg		200 μg/g	±20%	±20% Spike
	Ca		200 μg/g	±20%	±20% Spike
	Na		200 μg/g	±20%	±20% Spike
	Zr		20 μg/g	±20%	±20% Spike
	Ni		420 μg/g	±20%	±20% Spike
	Sb		~~10~~20 μg/g	±20%	±20% Spike
	U		1000 μg/g	±20%	±20% Spike
	V		~~10~~20 μg/g	±20%	±20% Spike
Gravimetric Analysis	Density/weight % solids	Test Instruction approved by STP[(4)]	N/A	N/A	N/A
Dry Particle Density	Volume and mass of particles	~~Test instruction approved by STP~~Procedure	N/A	±5%	±1%

Table 3-10. Analytical and Testing Requirements for Composite Samples and Sub-Samples

Process	Parameter	Standard Procedure or Test Instruction?	Required MDL(1)	Precision(2)	Accuracy(3)
Particle Size Range Sieving with General characterization of resulting mass and volume fractions	Three Particle size ranges 6350 to 2000 microns, 2000 to 500 microns and less than 500 microns	Test instruction approved by STP	N/A	N/A	N/A
XRD—identify crystalline structures in each as-settled sieve fraction	Hydrate compounds Al hydrates, U hydrides, U oxides, and Zr metal, plus any other identifiable compounds	Procedure	N/A	N/A	N/A
Recover and characterize any solids remaining after sample preparation by acid dissolution	Recover measure mass, gamma emitters, and identify materials with crystalline structures in digestate residue by XRD	Procedure	(6)	(6)	(6)
Uranium Metal content	Selective dissolution with uranium analysis – or- measurement through gas generation testing	~~Test instruction approved by STP or approved laboratory test p~~Procedure	0.01 wt% (settled sludge basis)	±20% (when $[U_{metal}] > 0.1$ wt%)	25% LCS
Uranium speciation by Spectrophotometry	Relative abundance of uranium IV versus VI	~~Test Instruction~~Procedure	~~(6)~~N/A(8)	~~(6)~~N/A(8)	~~(6)~~ ±25% for LCS
The following two tests will be used only for the sub-samples for the sieve fractions less 500 micron					
Rheology: Shear strength	Shear strength	Procedure	N/A	N/A	N/A
Rheology: Viscometer	Viscosity	Procedure	N/A	N/A	N/A

Notes:

General: Acronyms used in this table are defined in Table 3-9.

(1) Required 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 is to be 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% except ICP which should be ±20%. All method standard recoveries should be within statistical process control (SPC).
(4) The sample preparation procedure will provide the calculation used to determine the sludge settled density.

(5) Other opportunistic gamma emitters in library should be reported if available (e.g., ^{60}Co, $^{106}Ru/Rh$, ^{241}Am, ^{244}Cm, ^{95}Nb, etc.).
(6) The MDL, precision and accuracy values for the various analyses cannot be specified due the high variability in quantity and constituents that may exist in the residue. If >5.0% of residue is a crystalline phase then it is detectible by XRD.Equivalent MDL, precision, and accuracy values for the parameters being determined by multiple types of sequential analyses based on STP-approved Test Instructions (TI) shall be determined on a best effort basis.
(7) While conducting Inductively Coupled Argon Plasma-Optical Emission Spectroscopy (ICP) analyses, results from the following analytes are to be reported on an opportunistic/best efforts basis: boron (B), bismuth (Bi), copper (Cu), potassium (K), manganese (Mn), and phosphorous (P). While not specifically identified as analytes of interest for the current characterization effort, these analytes have been requested in previous characterization campaigns and the results are of interest for consistency between data sets. Any other analyte present at a concentration greater then the instrument detection limit for that analyte is to be reported; however, specific QC criteria may not be applicable for such analytes.
(8)The objective of the uranium oxidation state measurement is to determine the ratios, or relative concentrations of U(IV) and U(VI). At sufficient uranium concentrations (i.e., concentrations greater than 0.001 M uranium in each oxidation state), the uranium oxidation state distributions should be measurable with 5%.

3.5 Quality Control

3.5.1 General

All sample handling, sample packaging/shipping, and analytical process activities will be performed in accordance with the requirements of this plan. For all analyses and tests, the analytical laboratory shall perform to its internal QA program plans and procedures. The 325 Building Laboratory shall follow the requirements delineated in the PNNL Standards Based Management System and the Radiochemical Processing Laboratory (RPL) Operations Manual for sample receipt, consolidation and sub-sampling. The PNNL Analytical Support Operations shall comply with the QA/QC requirements in their QA Plan.

All analyses and testing will be performed consistent with the base contract between PNNL and CHPRC and in accordance with HASQARD Revision 3 as applied at the laboratory. Additional QA requirements may also be imposed by the specific contract statement of work (SOW) or Letters of Instruction (LOI) provided by the STP contract BTR for these activities.

The procedures and methods for calculating QC statistics shall be performed consistent with HASQARD Volume 4, Section 7.0 (2007).

3.5.2 Field Quality Control

As in prior K Basin floor sludge sampling campaigns, an equipment blank will be taken by the sampling team at KW Basin (i.e., drawing a sample of basin water using the sludge sampling equipment in a manner prototypic of a sludge sample). At least one sample bottle containing an equipment blank shall be shipped to the laboratory in the PAS-1 cask like a standard sludge sample. All protocols, including chain of custody procedures, will be used (see Section 3.3.4 requirements). At the Laboratory, this equipment blank will be handled, processed, ~~analyzed~~subjected to the analyses listed in Table 3-7 and 3-8 except gravimetric analysis and analyses of residues, and reported as much as feasible as a typical sludge core sample (Section 3.4) as appropriate. No general blanks, splits, duplicates or spikes are to be prepared in the field.

At the time that an equipment blank is prepared, a sample of KW Basin water will be collected and shipped to the laboratory consistent with *Sampling and Analysis Plan for 105-K East and West Basins Wastewater* (Bolles 2006). The water sample shall be subjected to the same analysis as specified for the equipment blank.

3.5.3 Laboratory Quality Control Measures

Tables 3-7, 3-8, and 3-10 provide quality control requirements specific to the analytes, methods, and the laboratory procedures to be used for the sample analyses and tests. These control measures include detection levels, precision, and accuracy of resulting data as appropriate. The quality control criteria provided in these tables have been established as planning values. Based on actual conditions and compositions of the samples delivered to the laboratory and preparatory and analytical method constraints, some of the quality control criteria may not be achievable for all samples. Therefore, the quality control criteria in these tables are to serve as triggers for further actions. If exceeded, consultations between the Laboratory and BTR for the STP will be held to discuss the appropriate further actions (e.g. no action, rerun analysis, obtain and prep new sample aliquot, etc.). The resolution of quality control issues will be documented in the verification and validation of laboratory data process described in Section 5.2.

Analyses of client duplicates for precision shall be performed on each of the individual core samples received from each of the five subject Large Engineered Containers, as identified in Table 3-6, plus GEA and uranium metal content analyses listed in Table 3-10. As noted previously, these client duplicates are repeats of the identified analyses. If client duplicates are not specified, then quality control duplicates for each preparative batch will be run by the Laboratory as identified in Tables 3-7, 3-8, and 3-10.

Quality control parameters for tests and analyses designated to be performed under laboratory Test Instructions shall be addressed directly in the Test Instructions which shall be approved prior to use by both the STP Characterization Manager and STP Contract BTR.

One reagent blank and reagent blank spike will be analyzed if required by the laboratory procedure for each preparative batch. When appropriate, one method control standard (e.g., calibration control standard) will be run with each batch. One matrix spike will be analyzed per matrix, as indicated in Tables 3-7, 3-8, and 3-10 or by the laboratory procedure.

The method detection level (MDL) or minimum detectable activity (MDA) as used in this document (e.g., Tables 3-8 and 3-10) is the detection level that 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 for resolving data quality objectives of the Safeguards and

Accountability DOQ document (Makenas 2007) and the STP engineering/process DQO (Westcott 2008Westcott 2009). 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 subsequent handling, dilution, and analysis methods prescribed.

If the QC parameters are not within specifications, the following action will be taken: Consult with the PNNL Project Manager, who will communicate with the STP Characterization Manager or the STP contract BTR (who will confer with the Safeguards group representative as appropriate) to determine if a rerun is needed (e.g., major vs. minor constituent). If it is agreed a rerun is to be made, the Laboratory shall make one rerun to see if acceptable results are obtained. If acceptable results are not obtained, the Laboratory shall report the QC failure to the PNNL Project Manager, who will contact the STP Characterization Manager. Any QC failure shall be clearly noted and flagged in the data package. The resolution of quality control issues will be documented in the verification and validation of laboratory data process described in Section 5.2.

For analyses and tests where laboratory Test Instructions are used (and standard laboratory procedures are not used), quality control parameters shall be designated in the Test Instruction. Test Instructions shall be used only after they approved by PNNL, the STP Characterization Manager, and the CHPRC/STP contract BTR.

If no criteria are provided in the current plan or Test Instruction, the performing laboratory shall perform to its own quality assurance plan(s) or analysis procedures, and shall report the results in the final data package.

All measurement methods and procedures used to determine nuclear materials and related physical parameters (e.g., weights, density, etc.) for Safeguards and Accountability values shall be reviewed and accepted by the Safeguards group prior to the measurements being performed.

3.6 Instrument/Equipment Testing, Inspection, and Maintenance

3.6.1 Sampling Equipment

The System Design Description (Snow 2008) for the K West Container sludge sampling system provides the testing, inspection and maintenance requirements for the equipment. Prior to pulling each core sample the peristaltic pump drive tubing is to be replaced. In addition, prior to each use the Lead Sampling Engineer is responsible to perform a general inspection of the sampling equipment. This inspection shall include the setup of sampling equipment, and using a prepared checklist, confirming basic system readiness for application. Sampling will not proceed until any deficiencies found at that time are resolved to the Lead Sampling Engineer's and the K Basin sampling procedure requirements. The setup checklist shall become part of the sampling log book.

The critical supplies and consumables for the sampling system are

- 4-liter sample bottles (Section 3.3.3)
- Bottle transfer lids and corresponding hoses
- Transport lids for the bottles
- Sets of isolation tubes and extraction tubes and corresponding connecting flexible hoses
- Peristaltic pump drive tubes
- Miscellaneous hose clamps and valve fittings.

The majority of these items are required to be replaced as each new sampling event occurs. These supplies will be procured by the STP Sampling and Testing group prior to the campaign and stored at K Basins under controlled conditions as needed to maintain cleanliness (Section 3.2.2). K Basins Operations. The sampling team will retrieve the required materials as the sampling campaign progresses per the controlling work documents or as directed by the Lead Sampling Engineer.

Supplies obtained to support laboratory instruments used to take measurements shall be inspected and accepted in accordance with quality processes and work documents that satisfy the requirements of the laboratory QA program. The processes and work documents shall identify the supply inspection/acceptance requirements and the methods used to inspect/accept supplies. Correction of nonconformances shall be in accordance with quality processes and work documents that satisfy requirements of the laboratory QA program.

3.9 Non-Direct Measurements

3.9.1 Existing Data on K Basin Sludge

The data bases forming the basis of the current Sludge Databook (Schmidt 2006a), its basis document (Schmidt 2006b) and supporting characterization campaign documents (e.g., Makenas 1996) will be used in assessing the current sampling data and assist in data validation. The current sampling campaign will establish the applicability of the past characterization data to the current state of the sludge as it now resides in the KW containers, since it has not been characterized since being transferred in to these containers.

3.9.2 Values from Other Methods

Per the engineering/process DQO (Westcott 2008Westcott 2009) Am-243 and Am-242m may be calculated using ratios to other isotopes that are measured; these ratios will be derived from nuclear material calculation codes (e.g., ORIGIN) made based on the source nuclear fuel (N-Reactor).

3.10 Data Management

Data will be collected and reported in accordance with work documents governing the activity. The work documents shall describe data reduction, data reporting, and mechanisms to detect and

- Sampling data sheets, as appropriate
- Non-conformance reports, as appropriate
- Verification package as described in Section 5.1
- Any other associated documentation pertinent to the sampling tasks performed.

Any related quality assurance documentation or reference to where such data is stored should also be provided. A general draft package or its planned content shall be provided to the STP Characterization Manager for review prior to the package being issued.

3.11.2 Analytical Laboratory

Each Laboratory data package shall conform~~s~~ to the scope of a "comprehensive or full" data package with all supporting QA and QC information consistent with the information required by this QAPjP/SAP, the Laboratory's own Quality Assurance Plan for this campaign, and information required by HASQARD (2007). Review drafts of the final data packages from the analytical laboratory shall be provided to the STP Characterization Manager or CHPRC Contract BTR (who will also obtain the Safeguards group representative for review) prior to the reports being issued. These initial reviews of the data will confirm compliance of the data package with the information requirements set in the DQO documents (Makenas and Baker 2007, Westcott et al ~~2008~~2009) and this QAPjP/SAP. The data packages shall be provided to STP in both hard copy and electronic format.

A narrative that summarizes the analytical results and their quality shall also be included in the data package, as well as the signature of the individual(s) responsible. In addition to those elements identified in Section 3.10.2, each data package shall provide the duplicate sample results, deviations from any of the established requirements (i.e., procedures, QAPjP/SAP, standard Laboratory procedures, Test Instructions, etc.), and completed copies of any Test Instructions. Related copies of any video tapes, digital images, photographs and reference color cards shall also be provided. All related settled sludge density data shall be clearly defined to establish the state of sludge material that was analyzed at each analysis step reported. Each page of the data package shall include the report number and consecutive page numbers. The data package shall address the results of each sludge core sample provided for analysis, but it is not required to provide the average or statistical analysis of the group of sludge core samples provided unless requested to do so in the future by the Buyer's Technical Representative (BTR). The laboratory data package shall include full copies of all Test Instructions and Standard Laboratory Procedures used in the analyses and testing reported.

Distribution of the laboratory data package shall include the STP Characterization Manager, CHPRC Contract BTR, the Safeguards group, and other designated STP staff. Copies of any data packages shall also be provided to STP Project permanent Project Records (the CHPRC Contract BTR will provide the address to the Laboratory at the time of document generation).

Table 5-2. Records Used as Inputs to Data Validation

Operation	Records to be Reviewed	Source for Record Specifications
Sample Collection	Verified data and data verification records; field records generated from sample collection activities as specified in Section 3.11.1	• The requirements of this QAPjP/SAP, especially applicable DQI's in Table 2-4 and the requirements in Section 3, *Data Generation and Acquisition* • DOE/RL-96-98, HASQARD, Revision 3, Volume 2 *Sampling Technical Requirements* • Specific Work documents for sampling and sample transport
Analytical Laboratory Operations (Sample Receipt, Preparation and Analysis)	Verified data and data verification records, the Laboratory's Data Package(s) as specified in Section 3.11.2	• All requirements in the base contract with the laboratory and Statement of Work • This QAPjP/SAP, especially applicable DQI's in Table 2-4 and the requirements in Section 3, *Data Generation and Acquisition* • DOE/RL-96-98, HASQARD, Revision 3, Volume 4 *Laboratory Technical Requirements*, and the Laboratory's QA Plan. • Approved laboratory protocols and procedures; test instructions

5.2 Verification and Validation Methods

5.2.1 Verification Methods

Data verification is basically a two step process:

- Identifying the project needs for records, documentation, and technical specifications and assembling these records, and
- Verifying records that are produced against the requirements of the method, procedure, and/or contract, as applicable.

For this project, the project needs for records, documentation, and technical specifications are specified in the DQO documents Makenas and Baker 2007 and ~~Westcott 2008~~Westcott 2009,

Sample Preparation
Sample preparation records are produced as defined in laboratory QA and work documents. Once these records are identified, they are verified in much the same way as the records for sample receipt. Sample preparation records shall be checked for completeness, correctness, and technical compliance against project needs.

Sample Analysis
The laboratory organization shall verify its respective batch test reports documenting their test results and the assignment of any applicable laboratory data qualifiers before issuing them to the Project. Data verification performed on fixed laboratory results is primarily to confirm that sampling and chain-of-custody documentation are complete, sample numbers can be associated with client supplied samples, samples were analyzed within the applicable holding time limits, and analyses met the data QC requirements of Section 3.5 of this QAPjP/SAP. The results of the verification shall be incorporated in the data package provided by the laboratory technician to the Project in accordance with Section 3.11.2 of this QAPjP/SAP.

Data Verification Records Review
The first output of the data verification process is verified data that have been checked for a variety of factors including transcription errors, correct application of conversion factors, and the assignment of applicable laboratory data qualifiers. Any changes to the results as originally recorded by the laboratory shall be accompanied by a note of explanation from the data verifier or the laboratory.

The second output from data verification is the data verification record. The record should include a signed certification statement that the data have been reviewed and verified. The record may also include a narrative that identifies non-compliance issues, identifies the records involved, and indicates any corrective actions taken in response.

Records of the data review and verification shall be included in the Laboratory Data Package as specified in Section 3.11.2. The data package shall include documentation from sample receipt through sample analysis.

5.2.2 Validation Methods

Validation will be performed consistent with HNF-20433, *Data Validation Procedure for Chemical Analyses,* and HNF-20434, *Data Validation Procedure for Radiochemical Analyses,* as modified by project specific work instructions. These procedures include sections on records management, data package completeness, personnel requirements, technical validation requirements, reporting requirements, validation checklists and qualifiers.

The first step of data validation begins with review of the planning documents for the project including the two DQO documents (Makenas and Baker 2007, and ~~Westcott 2008~~Westcott 2009) and this QAPjP/SAP. The data validator should outline all of the planning document requirements in order to understand what documents and records should be reviewed during data validation.

5.3.2 Qualification Testing of Sampling Equipment

The ability of the sample collection apparatus to collect representative samples will be demonstrated before it will be used to collect samples (STP 2008, A21C-STP-TPR-0002, *Test Procedure for Qualification of K West Sludge Sampling System*). If one or more of the acceptance criteria specified in this test procedure is not met, then the consequences of this result will be evaluated and corrective action taken as appropriate.

Even if the acceptance criteria are met, the results of the sampler testing will be incorporated into the calculation of the total measurement uncertainty during the assessment of the data for use in STP process design.

5.3.3 Data Assessment for Use in STP Process Design

Table 2-1 summarizes the PSQs developed during the DQO process to support the design of the STP process. For each PSQ, this table further lists the data needed to satisfy that PSQ. The mean of each parameter's results will be calculated as an arithmetic mean. The parameter variability will be calculated as percent RSD(mean).

As described in the DQO (~~Westcott 2008~~Westcott 2009), the project has adopted a ≤72% RSD(mean) as the acceptance criterion for the variability of the following radionuclide ratios to Cs-137: U-235, Pu-238, Pu-239, Pu-240, Pu-241, and Am-241. If one of these key ratios is shown to exceed the 72% RSD(mean) variability acceptance criterion, then the consequences of this result would be evaluated and corrective action taken as appropriate. Corrective action may require additional sample collection and/or analysis.

Sampling equipment shall not be removed from the KW Basin until this assessment has been completed, and corrective actions, if any, have been determined.

5.3.4 Data Assessment for Use in Safeguards Accountability Decisions

Table 2-2 summarizes the PSQ developed during the DQO process to terminate safeguards controls (Makenas and Baker, 2007). This table further lists the data needed to satisfy that PSQ and the population parameters of interest.

For accountability purposes, a measured value of the nuclear material in each container of KW must be obtained; therefore, each container of KW sludge is considered as a single population.

If the total plutonium concentration in the KW sludge is <0.20 weight percent, then the KW sludge has an attractiveness level of "E". If the total plutonium concentration in the KW sludge is ≥0.20 weight percent, then the KW sludge has an attractiveness level of "D".

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Figure A-4. Exploded View of the Loaded PAS-1 Cask

(Assembled steel and lead cask weighs ~13,000 pounds. Primary containment is backfilled with helium and is shipped on a flat bed trailer, CHPRC, 2008FH 2008)