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Identification of Potential Biases in the Characterization Sampling and Analysis Process

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Abstract: This report provides a summary of the issues and concerns associated with biases of the characterization data in the Sampling and Analysis Process. It addresses the potential of biases to be introduced at each step of the process.

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Identification of Potential Biases in the Characterization Sampling and Analysis Process

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EXECUTIVE SUMMARY

The Tank Waste Remediation System (TWRS) Characterization Project is responsible for providing quality characterization data to TWRS. Documentation of sampling and analysis process errors and biases can be used to improve the process to provide that data. The sampling and analysis process consists of removing a sample from a specified waste tank, transporting it to the laboratory and analyzing it to provide the data identified in the Tank Characterization Plan (TCP) and Sampling and Analysis Plan (SAP). To understand the data fully, an understanding of errors or biases that can be generated during the process is necessary. Most measurement systems have the ability statistically to detect errors and biases by using standards and alternate measurement techniques. Only the laboratory analysis part of the tank sampling and analysis process at TWRS has this ability. Therefore, it is necessary to use other methods to identify and prioritize the biases involved in the process.

The sampling and analysis process is thus functionally described based on the existing systems engineering breakdown and enhanced with the use of existing operating procedures. This is used as a documentation foundation where the sampling and analysis process could be systematically looked at to evaluate the potential for, and issues associated with, biases at each functional step of the process. Documents listed in the reference section were used as a basis for existing knowledge of a known phenomenon that could cause errors or biases. These

phenomena were segregated into the functional area in which they occur. Putting this information together created a summary of each functional step along its potential error/bias sources.

The importance of each error/bias depends upon the data being measured and its relationship to the waste. Current Data Quality Objectives (DQO's) do not provide sufficient information to evaluate analysis measurements against DQO needs adequately. It was therefore necessary to use as a basis the probability of an "IDEAL" situation. The more likely an error/bias source will produce a result that significantly deviates from the "IDEAL," the higher it was placed in importance.

From the information received, a list of prioritized tasks is developed that focuses on the least understood or most important error/bias sources. From these tests, more information about the potential for error/bias can be determined.

Two additional preliminary summary tables were constructed. One table categorized errors/biases by physical waste type. The other table addressed the data needs of the safety screening DQO and the impact the errors/biases had on those parameters.

It is envisioned that this document will be revised periodically to include new findings and describe plans for addressing those issues.

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LIST OF ACRONYMS

AF	area factor
ARC	adiabatic reaction calorimetry
CN	Cyanide
DQO	data quality objective
DSC	differential scanning calorimetry
HASQAP	Hanford Analytical Services Quality Assurance Plan
HHF	hydrostatic head fluid
HTCE	Historical Tank Contents Estimate
ICP	Inductively coupled plasma atomic emission spectroscopy
LIMS	Laboratory Management Information System
OTC	onsite transport cask
PIC	person-in-charge
RSST	Reactive System Screening Tool
SAP	Sample and Analysis Plan
TCP	Tank Characterization Plan
TCR	Tank Characterization Report
TGA	thermogravimetric analysis
TWAP	Tank Waste Analysis Plan
TWRS	Tank Waste Remediation System

1.0 INTRODUCTION

The Tank Waste Remediation System (TWRS) Characterization process consists of multiple functions or process steps that include planning and coordinated field and laboratory activities. Each of these functions has the potential of introducing error, which can either be random or directional, into the final data output. Directional errors are commonly referred to as biases and are the primary focus of this document.

The process to characterize Hanford Site tank waste supports safety evaluations for storage, treatment, and/or remediation of the waste. The information needs of each program element are defined in the Data Quality Objective (DQO) process. The DQOs may apply to one or many tanks and multiple DQOs may apply to a single tank. The DQOs are used as the basis to define the data requirements for each of the 177 waste tanks at Hanford. These individual tank needs are documented in a Tank Characterization Plan (TCP). From the TCP a Sampling and Analysis Plan (SAP) is prepared to carry out the data acquisition and analysis process. Samples are taken through various sampling methods, transported to the laboratory, and analyzed to provide the data identified in the TCP and SAP. These data along with all other available information, are collected and maintained for each tank in tank characterization reports (TCR).

Each step of taking and analyzing these samples has the potential of introducing significant errors or biases to the resultant data. It is unclear whether all potential error/bias sources have been identified and evaluated. This document will summarize past work on this subject and evaluate new ideas on the existence of sample biases.

This document uses a systems analysis functional breakdown of the TWRS characterization function to find the potential of process equipment and procedural biases. This document also establishes a connection to the functions and requirements' document drafted by TWRS to store, treat, and immobilize highly radioactive Hanford waste in an environmentally sound, safe, and cost-effective manner (WHC-SD-WM-FRD-020).

The TWRS Characterization Project is funding this work. Its purpose is to document what is currently known of sampling and analysis process errors and biases, and then to use this information to improve the process.

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2.0 BACKGROUND

2.1 SCOPE

This document includes an investigation and documentation of sources of error or bias that impact TWRS characterization data. The analysis will be limited to the functions of acquisition and analysis of the waste tank samples that deal with physical manipulation of the sample material.

Biases addressed in this document are narrowly focused on how well the samples collected represent actual material distribution at the identified sample location. For instance, an item that is not addressed is the additional error or bias imposed by the restriction of sampling tanks only from available risers.

2.2 OBJECTIVE

The objective is to identify and evaluate the potential for errors and data biases in the sampling and analysis process used by TWRS characterization. The following three activities will be addressed.

- Evaluate the current sampling and analysis process and any bias relationships identified between a sample analysis and the tank waste region sampled.
- Describe probable mechanisms involved in any error/bias sources.
- Identify additional work required to adequately determine and to quantify the potential errors/biases for which information is lacking.

2.3 NEED

The characterization process is documented in *"Tank Characterization Process"* (Dove et al. 1995). To meet the needs of the program elements described in the DQOs, the characterization process strives toward the following objectives.

- A tank sample must represent the region sampled.
- Sample material to be analyzed must be representative of the waste material from which the sample was extracted.
- Analysis results must be directly applicable to end user needs.

This document describes under what conditions the preceding objectives cannot be met and how far from the objective the process is.

To achieve these objectives, the high-level functions of the current process to get chemical and physical information about tank waste involve:

- preparation of data needs and quality definition in DQO reports
- the preparation of tank characterization and sample analysis plans
- acquisition of tank wastes as core segments, grab samples, and/or auger samples
- analysis of tank waste samples at the analytical laboratories
- report preparation and validation activities.

Whenever a measurement is made, an error is also made (except for small discrete counts). Usually, taking a measurement implies a comparison with a standard. The laboratory analysis process does have standards to evaluate the laboratory process, however, a suitable standard to use for the complete TWRS characterization process which includes sample acquisition does not exist.

No measurement is free from error. The accuracy and precision of a measurement must be known to interpret the data properly. To preclude a false sense of accuracy, the nature of the errors and their relationship to the measurement, the source types, and the magnitude must be investigated for each of the various stages of the measurement operation. In addition, the interrelationship of the errors must also be known. Only after these parameters are established is it possible to predict the size of the errors in the result and the implications on the interpretation of the data.

To help narrow the scope this report will focus on the ability of the sampling process to meet the requirements of the Safety Screening DQO, "WHC-SD-WM-SP-004, Rev. 2," excluding vapor sampling. This DQO was established to classify the 177 waste tanks at Hanford as safe or unsafe. As such, particular importance is placed on evaluating how well the sampling and analysis process meets these needs.

The primary tank waste parameters required for the safety screening of a tank are energetics, total alpha activity, and flammable gas concentrations (not addressed in this report). If one of these parameters is outside the prescribed safety criteria, further analysis of a secondary set of analytes is done. If it can be established, with a 95 percent confidence level, that the tank meets the criteria, then the tank becomes a candidate for removal from the safety watch list.

The tank can become a candidate to be removed from the safety watch list if it meets the following criteria:

Energetics < 480 joule per gram (J/g) [115 calories per gram(cal/g)]
Total alpha concentration < 41 microcurie per gram (μ Ci/g)

In order to further narrow the scope of this analysis, the determination of the effects of the sampling and analysis process biases on these measurements will be its focus..

2.4 SYSTEM PROCESS AND EVALUATION STRATEGY

The TWRS personnel have developed a systems engineering functional breakdown of the process to clean up Hanford Site tank waste. This breakdown was used as a starting point to create a functional breakdown of the characterization process. From this systems engineering functional breakdown, the process procedures were then used to create the level seven breakdown. Only selected components of the TWRS systems engineering study were deemed to be appropriate to this document. For the complete functional analysis, refer to the Systems Engineering Document WHC-SD-WM-FRD-020.

Each function was used to postulate the potential for characterization bias during accomplishment of the function. This approach was used because of the inherent lack of information that could be used to decide the extent of any system biases that may be present. Other than formal tests designed to address sampling bias, no determination of a bias condition can be directly made. Past measurements will also have been subject to similar biases such that any comparisons between sampling events will be insensitive to the bias issue.

2.5 APPROACH

The TWRS system engineering approach is a sequence of activities that transform a mission or need into a description of the system performance parameters and a preferred system configuration. A first step is to prepare a function and requirements document.

Functions are statements of purpose which define specific actions or processes that the system must achieve or do to support the fulfillment of specific mission objectives and the total mission.

Requirements are criteria that define the acceptable performance limits of the functions and their products. Requirements can be general in nature and apply to the entire system. These requirements may also be decomposed and allocated to the appropriate subfunctions. Requirements are qualitative or quantitative measures specifying boundaries on how a function must be done. Requirements may be constraints or performance requirements. Constraints

are imposed on a function by the external environment. Performance requirements are imposed on a function to meet the total system's criteria.

In this document error/bias sensitivity has been added for each function. Each function is examined for its potential to bias the data needed in the sampling and analysis process.

For the purposes of this document, the following definitions and Figure 2-1 are provided to clarify the term bias.

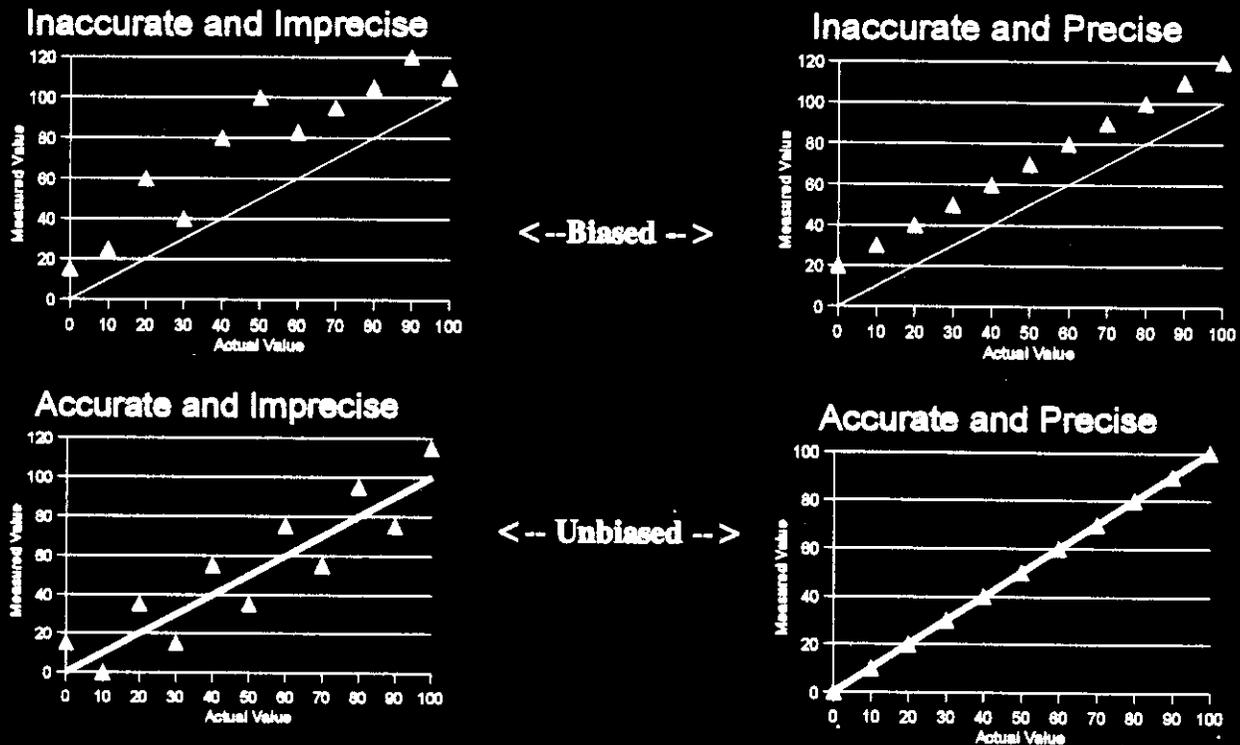
Accuracy - The extent to which a measurement approaches the true value of the measured parameter. Usually used as a measurement of the size of bias.

Precision - A measure of repeatability. Usually the relative standard deviation.

Bias - The difference between the average value of the measured parameter and the true value of the parameter.

The impact of these parameters can be seen in the graphs in Figure 2-1. The graphs plot actual values versus measured values and shows how the data would appear for different values of accuracy and precision.

Figure 2-1. How Accuracy and Precision Affect Actual Versus Measured Values.



In all cases, without the ability to know the real contents of the tank it is impossible to know

for sure if bias exists within the sampling and analysis process. However, a measure of precision can be obtained by using the standard deviation of the analysis of replicate samples taken in close proximity to one another. Tank heterogeneity, both vertical and horizontal, will affect precision measurement if data evaluation is not carefully controlled.

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3.0 BIAS NOT COVERED BY THIS DOCUMENT

Ideal statistical sampling schemes rely on obtaining random samples. The physical limitations of the waste storage tanks prevent this possibility. Access to tank material is limited and combined with the limitations of the sampling equipment, retrieved samples may not represent the entire contents of the tank.

Pictures of tank interiors show a difference in the waste at the edge of the tank and under the risers. Potential heterogeneity sources include:

- **Different fill mechanisms** - Filling the tank, from either the central fill port, or from a cascade overflow or a combination of these could potentially impact the heterogeneity/representativeness of particular samples under tank risers.
- **Local heterogeneity** - Disposal of selected wastes and debris (measurement tapes, gloves, unknown chemicals, etc.) into the tanks will have an impact on different samplers' ability to retrieve representative samples beneath risers.
- **Equipment insertion** - Past equipment that was removed from the tank left holes in the waste under risers which will affect sampling, provides a mechanism for vertical contamination, and affect the representativeness of samples to the bulk tank material.
- **Enhanced cooling around edges of the tank** - Cooling of the waste at the edges of the tank has produced a visible ring that is physically, and likely chemically, different from the rest of the waste matrix.
- **Condensation dripping off risers** - Tank pictures have shown where condensate that has dripped from the riser extensions has visibly changed the waste directly underneath them.
- **Past sluicing or water lancing activities** - These activities add water to the waste under the riser and also change the structure.
- **Repeated sampling in the same location** - Repeated sampling operations in the same riser location can have various effects on the sample material.

Altering these past disturbances or irregularities would be impossible but future disturbances can be minimized. Activities that have been initiated to attempt to understand the potential biases introduced by these effects include:

- Complete a review of the riser history before sampling to decide if off normal activities were done on that riser. An evaluation of available photos will also help determine if special circumstances are present under the riser.
- Complete a review of the data after sampling to evaluate differences in information from other risers or historical records exist.
- Evaluate the potential to install new risers in tanks that suggest problems of potentially unrepresentative material below the riser.

For the purposes of this document these potential bias sources are not addressed in detail beyond this section. The remainder of the report focuses narrowly on the ability of the sampling and analysis processes ability to collect properly and analyze a sample that represents the area sampled. The possibility that the sampled area may not represent the rest of the tank material is being dealt with elsewhere in the workscope of the TWRS Characterization Program.

4.0 SUMMARY

According to the *"DOE Methods for Evaluating Environmental and Waste Management Samples,"* (Goheen, S.C. et al. 1994) there are eight areas of concern involved in sampling tanks.

- **Sampling design adequacy** - This is related to the flexibility of access to tanks. Risers are not constructed in patterns that are conducive to systematic or random sampling of the contents.
- **Sampling access to tanks** - Current samplers are engineered to sample in areas proximate to entrance holes.
- **Sampler adequacy** - Past core-sampling equipment has not been effective in sampling hard salt cake or sludges, sampling the bottom three inches of a tank, or coping with the collection of multi-phase samples (e.g., samples that contain liquid and solid layers, or two immiscible liquid layers). Recent improvements in sampler design and the availability of the new rotary mode core trucks have helped mitigate this issue.
- **Multi-phase samples** - Multiple phase samples are particularly sensitive to sampler bias.
- **Sample representativeness** - Depending upon the heterogeneity of the tank, the collected sample may not represent the tank material as a whole.
- **Sample stability** - Sample manipulations normally considered routine in other environmental sampling activities in the field (e.g., compositing, in field analysis, preservation) cannot be done based on current technology. These limitations allow for samples to change between the tank and laboratory. Possible changes include loss of volatiles, sample drying, sample precipitation, etc.
- **Sample integrity** - Contaminating materials (e.g., water, silicone grease, normal paraffin hydrocarbons) are introduced when using some sampler designs. Sample manipulation in the laboratory may cause alterations (e.g., loss of volatiles and moisture) that affect data quality. Also, changes in the sample during collection and transportation may change the sample integrity.
- **Sample archiving** - Sufficient storage space is necessary.

4.1 HISTORICAL SAMPLING INDICATIONS

4.1.1 Auger Sampler

Based on hot cell observations, (summarized in Table C-1) the auger sampler experienced an expected recovery (recovery was assessed based on theoretical auger capacity) in cohesive material. Mixing occurred where there were several layers present. Auger performance was poor in hard salt cake material. Recovery was poor for phases exhibiting high moisture/low adhesion (high liquid content). Such phases separate from the auger during sampling, in the cask during transportation, or during the extrusion process in the hot cell. Subsequently, they are recombined with the remainder of the waste, or analyzed separately.

Differences in moisture contents within duplicates (such as reported for tank BY-103 and tank C-108) and within upper and lower portions of segments (such as for tanks TY-106, B-112, C-101, C-111, and C-108), could suggest sample inhomogeneity (sludge/salt cake mixtures, multi-layers) or procedural error(s). Inhomogeneity and under-saturation accentuate the driving capillary forces resulting in non-uniformity of moisture content within the waste matrix. Saturated, homogeneous matrices, such as that of tank TY-104, showed little variation in the moisture content results. In tanks with a salt cake crust material, such as C-101, drainable liquids were observed indicating weak capillary forces, and most likely, under-saturation of the upper portion of the waste matrix (crust material). Qualification and quantification of the species present in the waste matrix can provide clues about the phase separation and saturation moisture threshold. Unfortunately, such analytical data are only available for some tanks, none of which have been sampled by an auger.

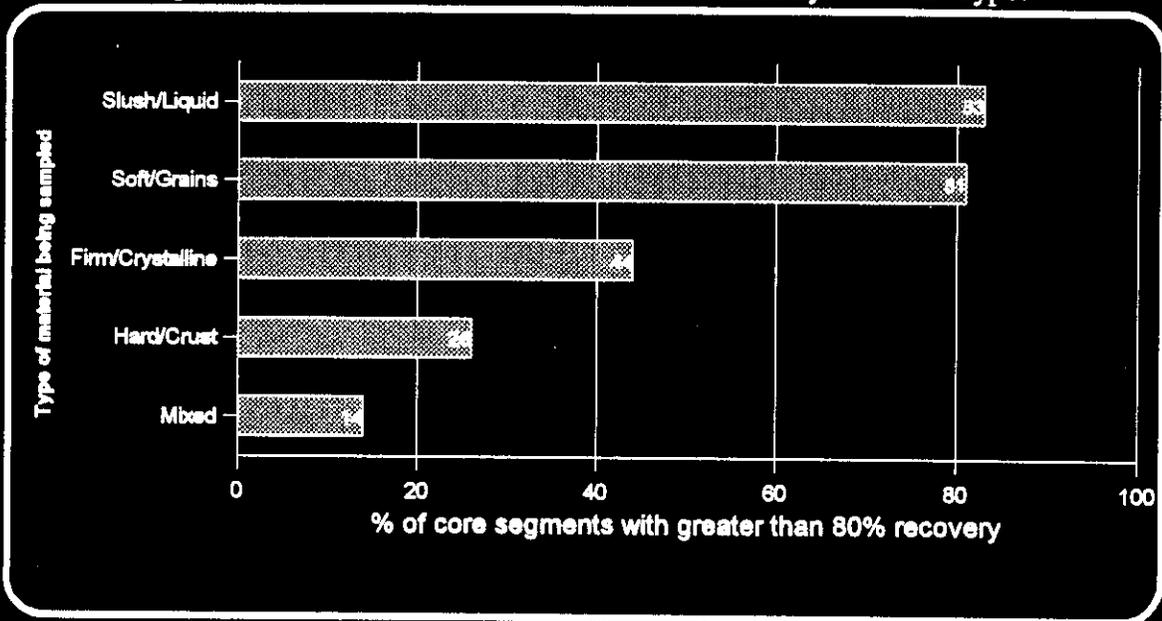
Possible modifications to the auger sampler have been evaluated and changes to the system configuration and operation are planned. Details of these design changes and findings from the tests can be found in *"Test Plan for Auger Sampler Improvements"* (Francis 1995) and *"Test Report for Auger Sampler Improvement Testing"* (Witwer 1995).

4.1.2 Core Sampling Recovery History

Based on the documents *"Further Investigation of Core Sampling Recovery History"* (Francis 1995) and *"Core Sampling Recovery History"* (Johns 1994), the primary reasons for poor recovery are equipment failure, waste location, and drill bit blockages.

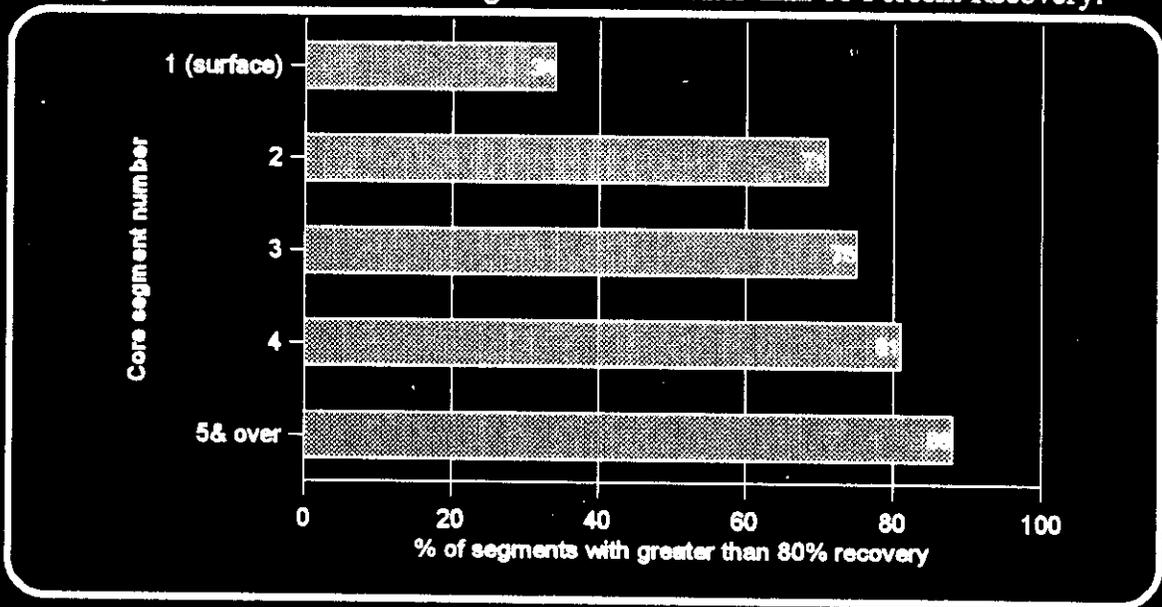
Figure 4-1 gives the percentage of core segments taken with greater than an 80 percent recovery for each of the different waste types.

Figure 4-1. Percent Recoveries Over 80 Percent by Material Type.



Another analysis was done by core segment and its relationship to the waste layer. Segment 1 is the first (surface layer) segment taken. Segment 2 is the next segment below that and so forth. A full tank can have as many as 19 core segments per core. The historical percentage of core segments with greater than 80 percent recovery for different segment numbers is shown in Figure 4-2.

Figure 4-2. Percent of Core Segments with Greater than 80 Percent Recovery.



As can be seen from the analysis data the first segment had a worse recovery history than subsequent samples. For reasons listed below, these low recoveries in the first segment may be because of procedural problems and therefore, they can be corrected. There is some question as to how the recovery was measured on past samples. Was the calculation was based on expected recovery, or a full 19-inch segment? Mixed phase samples caused the lowest recovery performance for recovery issues related to waste type.

In past operations, equipment failure accounted for approximately 8 percent of unacceptable segment recoveries. Most of the equipment that failed during these events has been redesigned and no longer counts as a significant factor in poor recoveries. The above recovery analysis did not include equipment failures in the analysis. The performance of the new rotary mode sample truck is not included in this analysis.

Sample recovery is historically worst for the first sample segment. Much of this poor recovery is attributed to failure to find the top of the waste. Surface level irregularities and errant level reading were attributed to zero recovery for many first segments. It also appears that recovery was worse in shallow tanks. The most likely cause of this is also the failure to find the top of the waste. The records varied dramatically even within the same tank.

New procedures established use zip cords, tank photos, and video cameras to help position the sampler should help to alleviate these types of problems.

Another possible reason for the low recovery of the first segment is past chain of custody records. Lab personnel were not aware of the expected recovery amounts and calculated the recovery based on a full segment. Most of the first segments are not expected to be full because of the practice of planning to get the last sample full. This error source has been corrected with the implementation of better record keeping.

The reasons just described may account for poor recovery of the first segment and this complicates the sample and analysis process but these issues have no relationship to future sample bias or quality.

Most other low recovery situations were attributed to a plugged bit. While this was probably the case, verification is difficult. Drill bit blockages came from many different sources. Most often, plugs occurred when the tank waste had a layered profile. Only 14 percent of the segments taken from mixed phase samples had a greater than 80 percent recovery.

Having a harder layer on top of a softer layer is the most difficult for the sampling system to capture. In these situations, no underlying resistance is available to force the top layer of the sample into the sampler. Hard, dry material is also more likely to break off chunks and block the drill bit opening.

For the push mode core sampling system a new bit was designed to achieve better sampling in crusted material. Results so far have been very promising. In addition, different sampling processes have been tried to improve recovery. These include variations in sampling speed

and slow rotation through sludges. Results from these efforts are inconclusive. Sometimes the changes in procedure seemed to help, in others it did not. Sometimes, no obvious explanation for plugging of the drill bit was discovered or it was simply not recorded.

4.1.3 Safety Screening Sample Analysis History

Based on Deichman (1995) 20 percent of the moisture analysis and 25 percent of the energetics analysis had data sets that exceeded the variance expected in the laboratory. There are several possible causes for these variances; however, the most likely is heterogeneity of the sample. Small subsamples [10-30 milligrams (mg)] are taken consistent with the maximum sample size of the instrument [thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC)]. Mixing is limited to 5 minutes to reduce moisture loss. These two processes contribute to the possibility of non-representative samples of the average waste composition being taken for analysis.

4.2 POTENTIAL SOURCES OF BIASES

Errors and biases exist as either systematic (cumulative) or random (compensatory). Random errors can be either plus or minus and usually attributed to errors of observation. They are truly random in size and occurrence and they have a tendency to compensate for one another. Systematic errors are usually cumulative in nature and are the type of error responsible for biases. A systematic error is one that will usually have the same size and the same sign (may also have a random component) under the same given conditions. There are three types of systematic errors.

- Common - These errors have to do with general factors related to site conditions (temperature, barometric pressure, humidity, etc.), waste layers, thickness of layers, waste depth, sampling riser location, and interactions between those and other factors.
- Direct - These errors are directly related to sampler specifications. For the auger sampler these would be flight type, shaft and pitch geometries, etc. For the core samplers, examples would be sampler geometry, opening diameter, etc.
- Indirect - These errors result from the sampling and analysis procedures that are executed. They include sampling speed, penetration velocity, vertical pressure force applied, retrieval speed, incremental and total sampling times, storage conditions (container and time), transportation time, preparation and analysis, and reporting procedures.

Qualitative variables include representativeness of waste layers and capture of a particular waste layer.

Quantitative variables include:

- Waste layer content (excess and equilibrium state, particularly regarding moisture)
- Overall average moisture concentration
- Waste physical properties such as compressive, tensile, and shear strength; yield stress; viscosity (cohesion/adhesion properties); conductivity; diffusion properties; hardness; particle size distribution; etc.
- Waste chemical properties such as pH, and composition (chemical and radiochemical, and inferences on reactivity, corrosivity, etc.).

Extrapolating this information to the sampling and analysis process, several possible sources for biases exist.

4.2.1 Whole Tank Estimate Bias Due to Incomplete Core Recovery

This issue deals with the effects of each sample segment analysis on whole tank estimates. In cases where core segments are not captured or minimally captured, estimates of whole tank concentrations may be biased. Based on reports by Jensen (1993) and Heasler and Jensen, (1993) it appears that the size of the effect is analyte and waste type dependent. While measuring the exact amount of the bias is impossible, the potential bias has been estimated to be significant. The primary variables involve vertical waste composition variability and sampler bias. If the vertical variability is great, missing one segment will have greater impact. If the sampler is biased (not all waste forms are collected equally) then whole tank estimates based on segment analysis are more dependant on complete recovery.

4.2.2 Sampler Selectivity to Physical Characteristics

Tank waste exists as soft and hard solids, liquid, and slurry forms. It has been found as liquid/slurry (with varying degrees of suspended solids) and solids (ranging from the consistency of gelatin, peanut butter, or honey, to hard concrete or fine grains). Building a single sampler that will handle every one of these potential sample forms and in any combination has never been accomplished. Currently, four possible sampling mechanisms exist for the condensed phase waste (non-vapor). Efforts are currently made to select the proper sampling tool for the expected waste form in the tank to be sampled. However, because of the heterogeneity of the tanks, the sampler that may be best suited for the waste at the top of the tank may not be the best selection for the waste at the bottom.

Selectivity of waste form will affect sample representativeness in that each form may have a different chemical makeup. For example selectively sampling sludges over liquids will bias the analysis toward the chemical makeup of the sludge.

Analytical results from each waste form, combined with knowledge of the amount of each waste form, could be used to predict waste tank contents accurately. However, if sampler performance is biased for or against a particular waste form, inventory estimates based on the quantity of that waste form would be suspect.

4.2.3 Contamination of Sample by Foreign Material

The contamination of the sample by other material is another source of bias. Possible sources of foreign material are leftover greases and solvents from the sampler's construction. Samples may leach elements from reactive components or atmospheres. In addition, samples may become cross-contaminated during handling. Moisture may be added during other events or tank conditions. These events include washing of equipment being removed, contamination from past sampling events, or dumping of waste material in the riser. Hydrostatic head fluid (HHF) used on core truck #1 is another example of possible contamination.

4.2.4 Sample Losses Due to Changes in Environment

The sampling systems are not designed to contain gaseous material. Therefore significant concentrations of low vapor pressure compounds (such as organics and moisture) could be enhanced by exposure to the atmosphere or conditions other than those in the tank. Losses of volatiles and trapped gases will also be enhanced by the changes in pressure between the tank contents and the atmosphere.

4.2.5 Sample Instabilities Due to External Forces

Mixing of the sample either during (by the sampling mechanism) or after (by the transportation mechanism) the sample collection could cause enhanced chemical reactions and physical changes within the sample matrix. Changes include modifications in the amount of drainable liquid in the sample and enhancement of off gassing. The use of nitrogen gas as a HHF and core bit cooling agent can also affect the waste matrix. The large flow of gas required to cool the rotary bit could have a drying or other chemical effect on the waste.

4.3 SAMPLING AND ANALYSIS PROCESS

Figure 4-3 shows the current Systems Engineering Breakdown for the Level 4 characterization function. This information was retrieved from the Systems Engineering Group Requirement Management System Browser. Levels 4, 5, and 6 exist in the database. Level 7 was created for the purposes of this document by using the field procedures associated with this work. Function definitions of each of level 4 through 6 functions are included in Appendix A. Some level 5 functions listed have a level 6 breakdown but were not included because they were not

pertinent to this document. The level 7 functions shown were primarily used as the basis for the analysis and were derived from the operating procedures used.

This breakdown is currently undergoing revision. When this revision is complete, the systems engineering basis of this document can be reviewed to decide if those changes influence the findings of this document.

Within the Characterize Waste function, two level 5 functions involve the physical handling of the waste samples. These functions, acquiring and analyzing waste tank samples, are considered the most likely to inject biases into the characterization data.

Figure 4-3. Characterization Function Hierarchy.

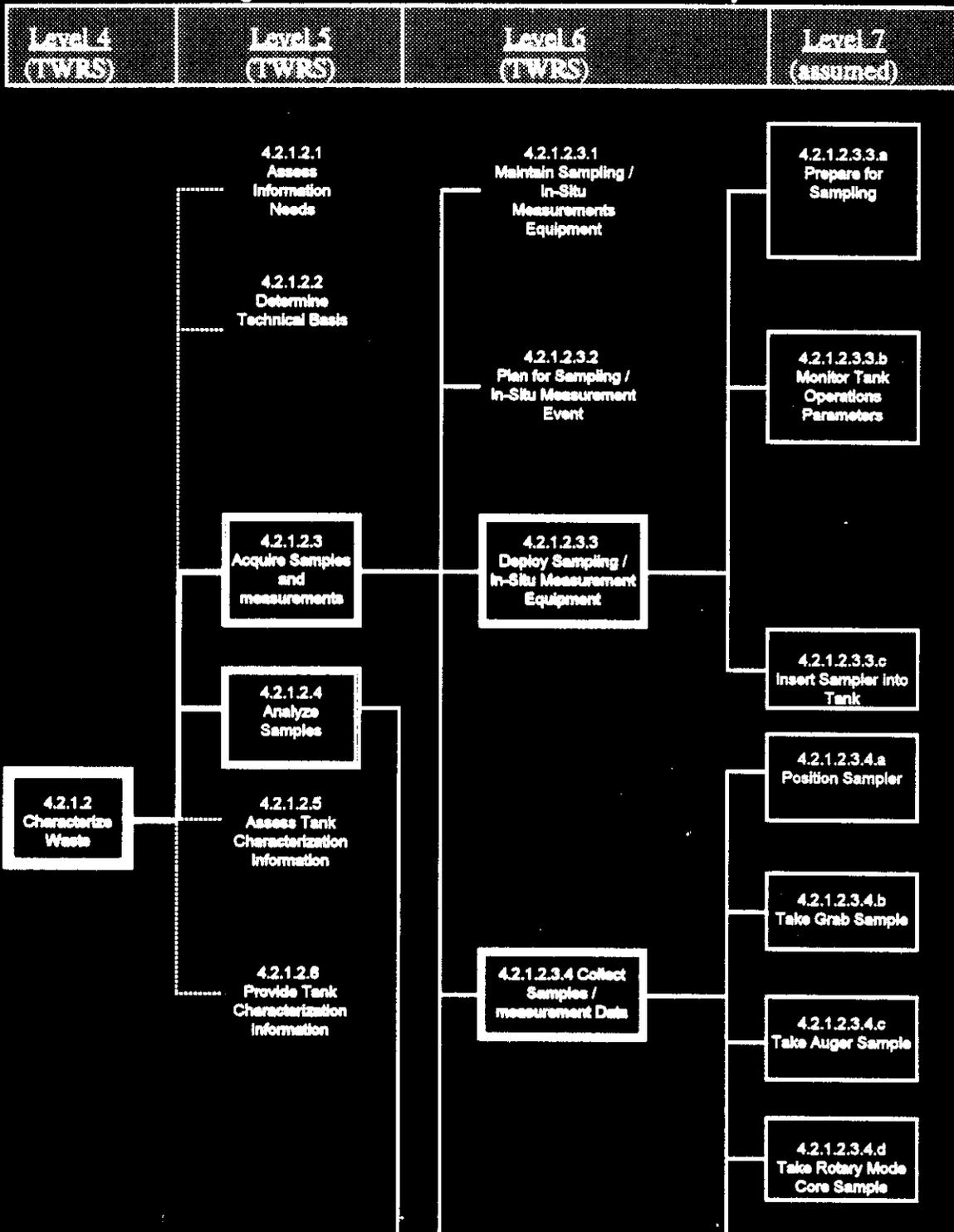


Figure 4-3. Characterization Function Hierarchy.

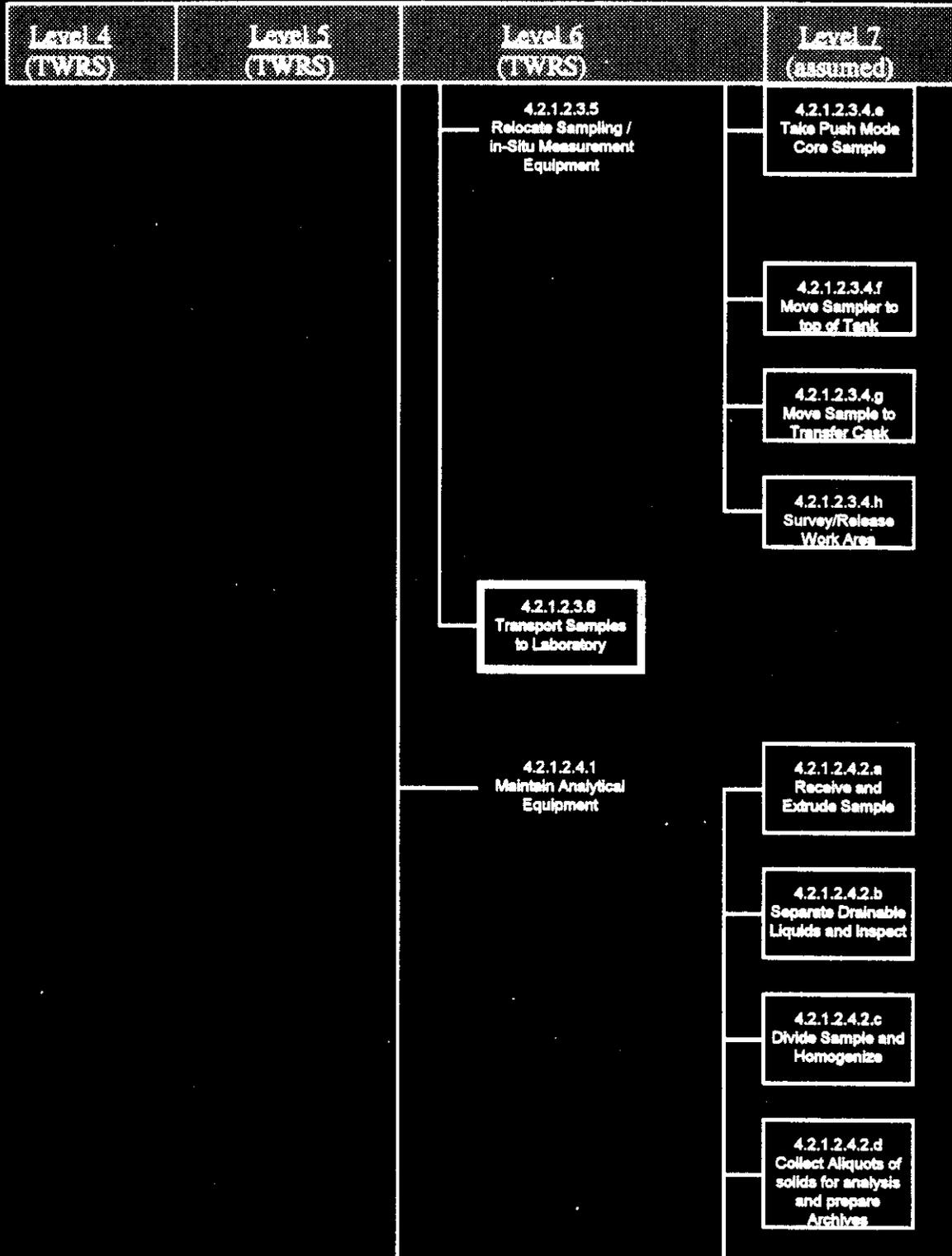
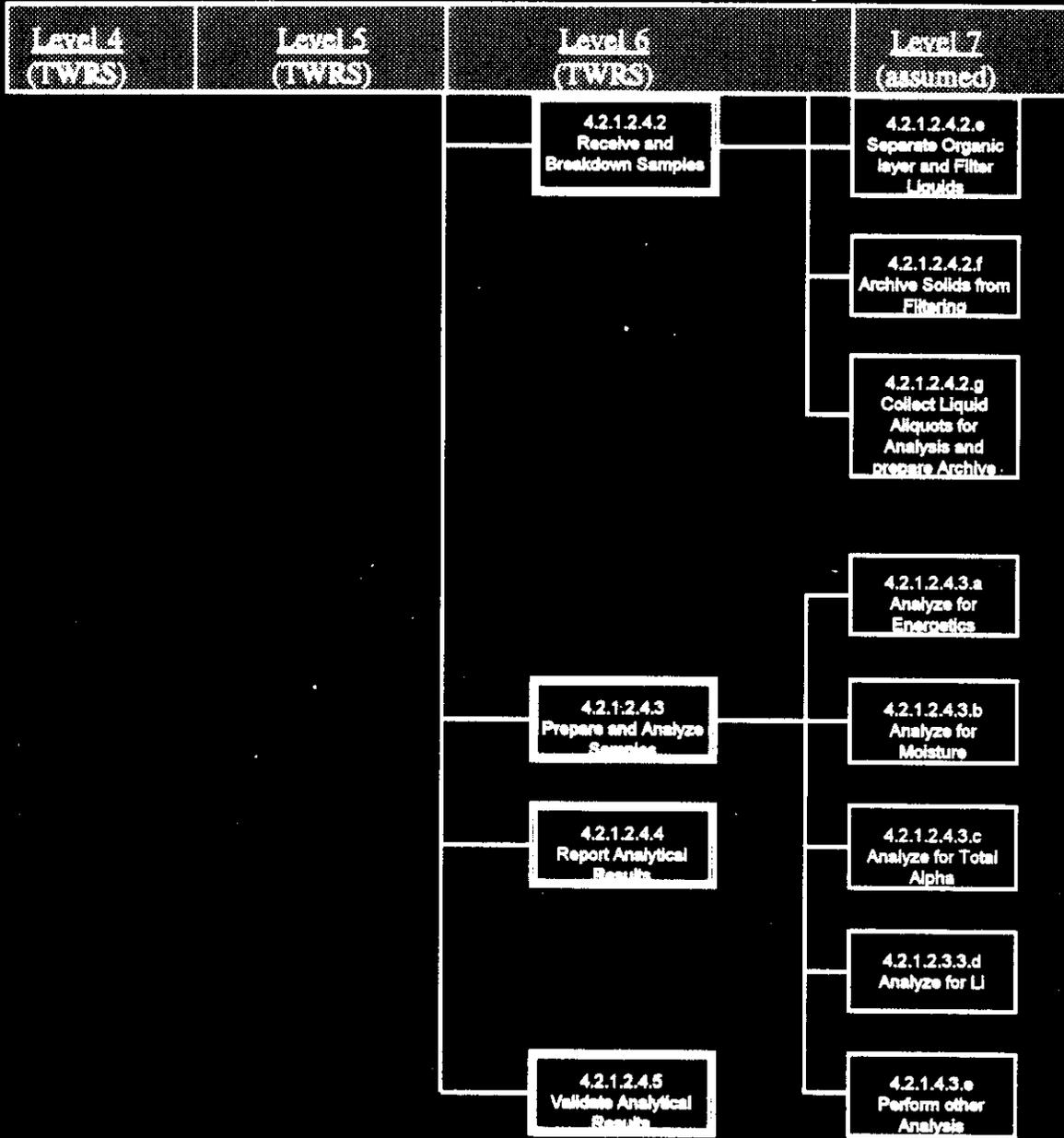


Figure 4-3. Characterization Function Hierarchy.



A tank sampling function is directed by the development of a sampling and analysis plan. The plan will specify the sampling and analysis needs for a particular tank. This plan includes specifications for which sampler to use, how many samples to take, and the analysis of the samples.

Deploying the sampling equipment consists of collecting all necessary equipment, procedures, and information required to accomplish the sampling event. Functional and safety documentation is reviewed to ensure proper sample operating conditions. Equipment is inspected to ensure it is clean and in good operating condition. Tank monitoring equipment is used to detect tank conditions. This equipment usually includes the use of in-tank video equipment to help in the determination of the level of the waste surface and the position of the sampler.

The tank is then accessed through an existing riser and the appropriate equipment installed to interface the sampler with the tank. The details of equipment installation and the collection of the sample depend on the type of sample being taken.

Removing samples from the tank is currently performed with one of four samplers: the grab sampler, the auger sampler, the push mode core sampler, and the rotary mode core sampler.

4.3.1 Grab Sampler (Bottle on a String)

This sampler comprises a 100-milliliter (mL) bottle (wide and narrow mouths) fitted with a rubber stopper in a weighted holder. The sampler is lowered into the tank by a nylon-coated wire rope. When the bottle reaches the desired depth, the stopper is removed from the opening remotely using the same rope, allowing waste material to flow into the bottle. The sampler is raised to the top of the tank in the next functional operation.

The grab sampler is limited to a single-elevation sample. It can obtain samples within five inches of the bottom of the tank. The sampler is limited to low viscosity sludges and liquid samples. Access is limited to directly under the tank opening used to deploy the system. Tank waste must be sufficiently liquid throughout to allow the sampler to be lowered to the desired depth.

4.3.2 Auger Sampler

Auger sampling equipment consists of an outer guide tube assembly and an inner auger sampler assembly. The auger guide tube assembly consists of a steel tube, 2-inch pipe, and a threaded flange and kamlock connector on the top. The guide tube is assembled such that when it is lowered into the tank and bolted to the riser it is about 13 mm (0.5 in.) above the waste. The threaded flange on the top is then adjusted to hold the sleeve just above the waste. The auger is contained within a metal sheath inside the guide tube, except during the sampling

operation. The waste material is penetrated by turning the auger and is contained on the auger flights. The auger can then be removed to the top of the tank with the waste material contained on the auger within the sheath.

The auger sampler is a manually operated system. Auger samplers come in two different sizes (10 and 20 inches long). The maximum capacity for either auger is approximately 11.8 cc/cm (30 cc/in.) of depth with an expected recovery of 7.8 cc/cm (20 cc/in.). The auger sample is open to the atmosphere within the shipping container.

4.3.3 Core Samplers

Four sampling trucks can be used to obtain core samples. The trucks are equipped with LongYear Model 34 drilling platforms. The drill assembly hydraulically lowers the drill rod assembly into the waste with a core sampler latched inside the bottom of the drill rod assembly. A downward 48 cm (19 in.) stroke loads the sampler with material. Then the drill assembly and the shielded receiver, which are mounted on a rotating platform, are rotated 180° to position the shielded receiver above the drill rods for sampler retrieval. The sampler is pulled into the receiver and the platform is then rotated with shielded receiver and positioned above the on-site transport cask (OTC). The sampler is then lowered into the OTC for shipment to the laboratory. A clean sampler is retrieved from another OTC, the truck platform is rotated to lower the next sampler into the drill rod assembly and the process is repeated.

Samples can be taken in either rotary mode or push mode. In rotary mode, a cutting bit is attached to the bottom of the drill rod assembly and the drill rod assembly is rotated as it is pushed into the waste. This process cuts a core sample out of the waste and draws it into the sampler. Push mode simply pushes the drill string into the waste using a tapered push bit. The push bit is constructed of stainless steel, with a one-inch sample opening and a 60° outside taper to ease the sampler's penetration into the softer waste material.

Core Sample Truck #1 is not able to use nitrogen purge gas to cool the bit and clean the hole during rotary sampling. It is therefore used exclusively for push-mode sampling. In addition, water laced with 0.3 Molar (*M*) lithium bromide (LiBr) solution is used as a HHF instead of nitrogen gas. Trucks 2, 3 and 4 use nitrogen as the HHF but LiBr may still be introduced to the waste when necessary to unplug or unstick a drill bit.

The rotary mode sampler was designed to obtain salt cake, sludge, and liquid samples. It is used in both push and rotary mode core sampling. The sampler is loaded into the drill string and either pushed or rotated through the waste using the LongYear drill rig. This sampler can obtain a complete profile of tank waste in 48.3 cm (19 in.) segments. The maximum volume is 300 mL for liquids and sludges and 245 mL for salt cake samples. This sampler can obtain samples within 7.6 cm (3 in.) of the bottom of the tank.

The rotary mode sampler contains a bearing assembly in the bottom of the sampler that supports the sampler and allows the outer barrel to rotate while the sampler remains stationary relative to the waste. The seal between the sampler and the inside of the bit is a chevron type seal.

The push mode sampler is a modified version of the rotary mode sampler used in push mode core sampling. The main differences between the two samplers are that there is no bearing assembly at the bottom and the seal is a standard O-ring rather than a chevron seal.

4.3.4 Sample Removal and Transportation

Once samples are captured in the sampler, they are raised to the top of the tank. For the grab sampler, the bottle is then sealed with a screw lid and washed off. The core samplers are retrieved into a receiving cask and then X-rayed to detect recovery.

Samples are then moved to an OTC, loaded on a truck and transported to the laboratory. Currently no provisions are made, other than freeze protection, to control the temperature of the samplers once they leave the tank.

4.3.5 Analysis of Samples

The samples are received at the laboratory and prepared for analysis. This involves processing the samples as defined and in the sampling and analysis plan and archiving samples as applicable.

Once at the laboratory, samples are removed from the shipping cask and inserted into the hot cell. Core samplers are loaded into the extruder where the sample is removed by using a piston to push it out of the sampler and onto a sample tray. With auger samplers, the waste material contained on the flutes is scraped off and placed in a suitable sample container.

The sample is visually inspected and observations are recorded. These include color, texture, homogeneity, and consistency. Color photographs and videotapes are also used to document the core segment.

Any drainable liquid is separated and the volume measured. The liquids are retained for further processing. The mass of the segment and the approximate length or mass is recorded.

Each sample is divided and homogenized according to the sampling and analysis plan. Homogenization is performed so that aliquots removed for analysis will represent the sub-segment composite. If a sufficient sample exists, a homogenization test can be done. This test compares duplicate assays of homogenized material. If the analytes from the aliquots are within a relative percent difference (RPD) of 10 percent, the samples are considered homogenized. The cognizant person decides if a homogenization test is required.

Aliquots of the solid portion of the sample material are taken in duplicate to do the required analysis and at least 20 mL and up to 40 mL of each homogenized sub-sample is archived (Bratzel 1994).

Any liquid portion of the sample is handled separately. If an apparent organic layer is present, it is separated from the sample and analyzed separately. The remaining liquid is filtered to remove suspended solids. If the filtered solids exceed one gram then the filtered solids are archived for possible future analysis.

Enough aliquots of liquid sample material are taken to do the analysis specified in the sampling and analysis plan. Remaining sample material is placed in archive containers and stored for possible future analysis or use.

Once the sample aliquots are taken, some analysis can be done directly on the sample material. Other analysis required a sample dissolution before the analysis can be done. Analyses are reviewed for consistency according to the laboratory quality assurance plan and the data are entered in the Laboratory Information Management System (LIMS).

.3.6 Energetics Analysis

To analyze for energetics, differential scanning calorimetry (DSC) is used. The sample pan and the reference pan sit on a ceramic sensor in the sample chamber of a DSC furnace. The differential heat flow between the sample and reference pan is monitored by the thermocouples placed beneath the pans. The system provides qualitative and quantitative data of endothermic and exothermic reactions occurring within the sample. Most sampling and analysis plans call for a required analytical uncertainty of the measurement is ± 10 percent of the decision threshold of 480 J/g (115 cal/g) dry weight percent (wt%) basis. The sample sizes are limited to 35 milligrams (mg) or a 10-microliter (μL) volume to ensure temperature uniformity throughout the sample. An exothermic reaction on a sample of a particular volume can only represent a reaction for that size of a sample. Samples containing a large percentage of volatile organics may not be analyzed by this procedure. Samples with a high explosion potential below 500°C also may not be analyzed by this procedure.

4.3.7 Moisture Analysis

Moisture (water wt%) is used to make a correction to the total fuel content dry basis criteria. The TGA is the method used to make this measurement. The TGA measures the changes in sample weight as the sample is heated from 35°C (95°F) to 500°C (932°F) at a constant rate. The percentage weight loss evaporated off the sample is calculated from the thermogram, and assumed to be primarily water. Samples used are as small as possible. As the amount of sample increases, the distribution of heat in the sample becomes uneven, and the exchange of gas with the surroundings is impeded. Low boiling point organic compounds will evaporate off the sample with the water. This will cause an artificially high water content to be calculated. In addition, a minimal sample size is recommended because of a slight possibility

of an explosion and as low as reasonably achievable (ALARA) considerations.

The TGA and DSC measurements are used collectively to determine the thermal stability of the sample material. These analysis are semi-quantitative and require considerable experience and judgement on the part of the analyst.

4.3.8 Total alpha Analysis

A total alpha measurement is done to detect the fissile material's content. Sample aliquots are dissolved by a fusion method. A measured volume of the liquid sample is placed into a stainless steel dish and evaporated to dryness. The determination is made by measuring the count rate on a proportional counter. Total alpha activity is considered directly related to the disintegration rate by an efficiency factor. There is no apparent difference in efficiencies because of differences in alpha energy.

Using best laboratory practices a precision of \pm percent of the decision threshold of 1 gram per liter (g/L) of fissile materials is required. This translates into the 41 $\mu\text{Ci/g}$ decision limit usually specified in the sampling and analysis plan.

4.3.9 Li Analysis

For cases where contamination of the sample was possible, either by a HHF or the need to unblock/unstick a drill bit, a lithium analysis is done. Lithium is detected through analysis by ion chromatography (IC). If greater than 100 $\mu\text{g/mL}$ is detected then a secondary analysis for bromium by ion chromatography is done. These measurements can then be used to provide compensation parameters to calculate the amount of intrusion..

4.3.10 Other Analysis

Other analyses are done as needed. Secondary analyses are required when the primary safety screening measurements have exceeded the preset limits. Also secondary analyses are done when the relative standard deviations of the duplicate measurements exceed preset limits. These analyses include distillation for cyanide (CN), counting for plutonium^{239/240}, energy analysis by reactive system screening tool (RSST), total organic carbon, and IC analysis for bromine..

4.3.11 Reporting Results

Analytical reports are then written to integrate, evaluate and distribute the results of the sample analysis as specified by the tank waste analysis plan and/or the specific tank characterization plan or other supporting documents, if applicable. Reporting requirements vary depending upon user requirements and intended data use. Reporting formats include, but are not limited to, letter reports, LIMS printouts, data packages with raw data, and reports/results in electronic format for input to the tank characterization database or other

electronic characterization databases. Copies of those data results are relinquished to the Characterization Data Management function, and are also used as an input to the Tank Characterization Report Development function.

Verification and validation are done on the analytical results in the data packages prepared by the laboratory. This activity includes:

- reviewing the data package to ensure the laboratory has performed and properly documented the requested analyses by the requirements set forth in the Tank Waste Analysis Plan and Tank Characterization Plan (or other appropriate working documents), and the appropriate quality assurance documentation such as the Hanford Analytical Services Quality Assurance Plan (HASQAP).
- preparing a validation report that documents the results of the validation. Validation is only performed when the Tank Waste Analysis Plan and Tank Characterization Plan (or other documentation) requires that the data be validated.

4.4 EVALUATION OF SAMPLING AND ANALYSIS PROCESS BIAS POTENTIAL

The potential of each step in the sample and analysis process to contribute to these potential bias sources was evaluated. The key sampling and analysis biases and errors identified in this initial review are presented in Table 4-1.

For functions where the bias is dependant upon the waste phase the Table 4-2 summarizes the issues associated with each material type.

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Table 4-1. Sampling and Analysis Functions with Potential Bias Sources.

Function (function numbers are preceded with 4.2.1.2)	Potential Bias Phenomena		
	a. Grab Sampler b. Auger Sampler	c. Push Mode Core Sampler d. Rotary Mode Core Sampler	
	Common	Direct	Indirect
.3.3.a Prepare for Sampling			
.3.3.b Monitor Tank Operations Parameters	*Real time video monitoring of sampling operations should significantly enhance the understanding of waste physical properties and sampling constraints.		
.3.3.c Insert Sampler into Tank	✓Fixed location of sampling risers		
.3.4.a Position Sampler			
.3.4.b Take Grab Sample	✓Sample exposure to air in bottle	✓Selective bias toward lower viscosity liquid	
.3.4c Take Auger Sample		✓Modification of physical properties ✓Mixing of phases	✓Sampling speed affects mixing
3.4d Take Rotary Mode Core Sample		<ul style="list-style-type: none"> ✓Selective bias toward lower viscosity liquids ✓Lack of sufficient HH pressure ✓N₂ Flow prevents sample capture ✓uncertainty of interval selected ✓Potential compaction of partial segments ✓Lack of integrity of obstructive causes low confidence in layering information ✓Bit plugs with waste layering 	<ul style="list-style-type: none"> ✓Addition of H₂O for bit unplugging/ unsticking ✓Possible capture of purge gas ✓Loss of fines from cuttings and N₂ flow ✓Drying of sample from N₂ Flow
.3.4e Take Push Mode Core Sample	✓Volatility of sample with ppressurechanges	✓Selectively sample different viscosity materials	<ul style="list-style-type: none"> ✓sampling speed ✓HHF contamination ✓Smearing/compressing of sample material

Table 4-1. Sampling and Analysis Functions with Potential Bias Sources.

Function (function numbers are preceded with 4.2.1.2)	Potential Bias Phenomena		
	a. Grab Sampler b. Auger Sampler	c. Push Mode Core Sampler d. Rotary Mode Core Sampler	
	Common	Direct	Indirect
.3.4f Move Sampler to Top of Tank	<ul style="list-style-type: none"> ✓a. b. Sample exposure to atmospheric air 	<ul style="list-style-type: none"> ✓a. b. Loss of interstitial gases. ✓b. Loss of liquid phase ✓b. Bias to insolubilities on multiple sample ✓c, d. Partial failure of ball valve cause loss of liquid ✓c. Pressure changes 	<ul style="list-style-type: none"> ✓c. Water added as HHF ✓a. Mbdng with other waste material. ✓a. Dilution from bottle washing
.3.4g Move Sample to Transfer Cask	<ul style="list-style-type: none"> ✓Temperature changes 		<ul style="list-style-type: none"> ✓Disturbance of sample from movement
.3.4.h Survey Release Work Area	<ul style="list-style-type: none"> ✓Temperature changes 		<ul style="list-style-type: none"> ✓Disturbance of sample from movement
.3.6 Transport samples to laboratory	<ul style="list-style-type: none"> ✓Temperature changes 	<ul style="list-style-type: none"> ✓Loss of volatiles 	<ul style="list-style-type: none"> ✓Disturbance of sample from movement
.4.2.a Receive and Extrude Sample	<ul style="list-style-type: none"> ✓Exposure to atmosphere 	<ul style="list-style-type: none"> ✓c. d. Physical effects of extruder on sample 	<ul style="list-style-type: none"> ✓Pressure changes
.4.2.b Separate Drainable Liquids and Inspect	<ul style="list-style-type: none"> ✓Exposure to atmosphere 	<ul style="list-style-type: none"> ✓Nonquantitative transfers 	<ul style="list-style-type: none"> ✓Modification of physical properties
.4.2.c Divide Sample and Homogenize	<ul style="list-style-type: none"> ✓Exposure to atmosphere 	<ul style="list-style-type: none"> ✓Sample changes from homogenization process 	<ul style="list-style-type: none"> ✓Alter physical properties ✓Subjective determination of segment facies
.4.2.d Collect Aliquot of solids for analysis and prepare Archives	<ul style="list-style-type: none"> ✓Exposure to atmosphere 	<ul style="list-style-type: none"> ✓Selection of sub samples 	
.4.2.e Separate Organic layer and Filter Liquids	<ul style="list-style-type: none"> ✓Exposure to atmosphere 		

Table 4-1. Sampling and Analysis Functions with Potential Bias Sources.

Function (function numbers are preceded with 4.2.1.2)	Potential Bias Phenomena		
	a. Grab Sampler b. Auger Sampler	c. Push Mode Core Sampler d. Rotary Mode Core Sampler	
	Common	Direct	Indirect
.4.2.f Archive Solids from Filtering			
.4.2.g Collect Liquid Aliquots for Analysis and Prepare Archive	✓Exposure to atmosphere		
.4.3.a Analyze for Energetics		✓Small Sample size (DSC) Secondary analysis (RSST) uses a larger sample	✓Moisture measurement to interpret results ✓baseline determination effects results
.4.3.b Analyze for Moisture		✓Small Sample Size ✓Presence of Volatile Organics	✓Definition of relevant moisture type
.4.3.c Analyze for Total Alpha		✓Buildup of solids	✓Fusion process yield ✓Fusion process bias
.4.3.d Analyze for Li		✓Li may not remain in solution ✓Diffusion into solids	✓Unquantifiable secondary effects of HHF ✓Large amounts of HHF intrusion
.4.3.e Perform Other Analysis			

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Table 4-2. Bias Potential by Sample Material Type.

Functions (function numbers are preceded with 4.2.1.2)	Material Type / Bias Potential				Mixed
	Slush/Liquid	Soft/Grains	Firm/Crystalline	Hard/Crust	
.3.3.a Prepare for Sampling					
.3.3.b Monitor Tank Operation Parameters					
.3.3.c Insert Sampler into Tank					
.3.4.a Position Sampler		a.NA	a. NA	a. NA	
.3.4.b Take Grab Sample		NA	NA	NA	Possible bias if material is chunky
.3.4.c Take Auger Sample	NA	Bias towards material with sufficient cohesion to remain on the bit	Bias towards firm material with cohesion sufficient to remain on bit	Grinding of material into fines with minimal cohesion	Drainage of liquids from sample
.3.4.d Take Rotary Mode Core Sample	N ₂ flow will selectively effect recovery	Cohesion of samples on sides of sampler may smear sample.	Low angle drill bit has difficulty cutting material Crystalline material may be redistributed in sampler.	N ₂ flow will tend to blow material away from the sampler that has been ground up by the drilling	Sampler may selectively sample material with lower viscosity Hard layers over soft layers may cause bit plugging
.3.4.e Take Push Mode Core Sample	HHF may displace sample material	HHF may dissolve sample material and selectively collect dissolution product	HHF may dissolve sample material and selectively collect dissolution product	HHF may dissolve sample material and selectively collect dissolution product	Sampler may selectively sample material with lower viscosity Hard layers over soft layers may cause bit plugging

Table 4-2. Bias Potential by Sample Material Type.

Functions (function numbers are preceded with 4.2.1.2)	Material Type / Bias Potential			
	Slush/Liquid	Soft/Grains	Firm/Crystalline	Hard/Crust
3.4.f Take Push Mode Core Sample				Mixed
3.4.g Move Sample to Transfer Cask				
3.4.h Survey/Release Work Area				
3.6 Transport Samples to Laboratory				Distribution of sample is suspect after agitation from transport
4.2.a Receive and Extrude Sample				
4.2.b Separate Drainable Liquids and Inspect				
4.2.c Divide Sample and Homogenize				
4.2.d Collect Aliquot of solids for analysis and prepare Archives			Crystal sizes on the order of subsample volume can make subsampling representativeness questionable	Crystal sizes on the order of subsample volume can make subsampling representativeness questionable
4.2.e Separate Organic layer and Filter Liquids				

Table 4-2. Bias Potential by Sample Material Type.

Functions (function numbers) preceded with are (4.2.1,2)	Material Type / Bias Potential				Mixed
	Slush/Liquid	Soft/Grains	Firm/Crystalline	Hard/Crust	
4.2.f Archive Solids from Filtering					
4.2.g Collect Liquid Aliquots for Analysis and prepare Archive					
4.3.a Analyze for Energetics					
4.3.b Analyze for Moisture					
4.3.c Analyze for Total Alpha					
4.3.d Analyze for LJ					
4.3.e Perform other Analysis					

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Table 4-3. Sampling and Analysis Functions with Bias Magnitudes for Specific Analysis.

Function (Function numbers are preceded with 4.1.2)	Analysis Method/Sample type											
	H2O Analysis			Energetic Analysis			Alpha Analysis			Analysis Method/Sample type		
	Direction	Magnitude	Cause	Direction	Magnitude	Cause	Direction	Magnitude	Cause	Direction	Magnitude	Cause
3.3.a Prepare for Sampling				+	Small	Foreign Solvents and Greases						
3.3.b Monitor Tank Operations Parameters												
3.3.c Insert Sampler into Tank												
3.4.a Position Sampler												
3.4.b Take Grab Sample	- +	small ?	exposure Preferential sampling of liquids	- ±	? ?	Preferential sampling of liquids	±	??	±	??	Sampling Selectivity	
3.4.c Take Auger Sample				±	?	Sample Mixing	±	?	±	?	Mixing of waste	
3.4.d Take Rotary Mode Core Sample	+ ± -	? ? ?	Bit plug removal Lack of sample capture Drying from N ₂ flow	±	?	Lack of sample capture Selective Capture	±	?	±	?	Sampling selectivity	
3.4.e Take Push Mode Core Sample	+	??	HHF (except first segment)	-	??	HHF (except 1st segment)	-	??	-	??	HHF	

Table 4-3. Sampling and Analysis Functions with Bias Magnitudes for Specific Analysis.

Function (number) numbers are presented with (3.1.2)	Analysis Method/ Sample type											
	H ₂ O Analysis				Energetics Analysis				Alpha Analysis			
	Direction	Magnitude	Cause	Direction	Magnitude	Cause	Direction	Magnitude	Cause	Direction	Magnitude	Cause
.3.4.f Move Sampler to Top of Tank	-	<6%/hour	a. Exposure to air	+	?	a. Loss of moisture	±	?	a. Loss of moisture	±	?	b. Sampling selectivity
	-	<6%/hour	b. Loss of liquid /exposure to air	+	?	b. Loss of moisture	+	?	b. Loss of moisture	+	?	
.3.4.g Move Sample to Transfer Cask	±	?	Agitation to redistribute H ₂ O	-	?	Agitation increases chemical breakdown						
.3.4.h Survey Release Work Area												
.3.6. Transport Sample to Laboratory	±	?	Agitation to redistribute H ₂ O	-	?	Agitation increases chemical breakdown						
.4.2.a Receive and Extrude Sample	-	<6%/hour	Exposure caused drying	+	?	Compensation effect from loss of moisture						
.4.2.b Separate Drainable Liquids and Inspect												
.4.2.c Divide Sample and Homogenize	-	<6%/hour	Exposure caused drying	+	?	Compensation effect from loss of moisture						

Table 4-3. Sampling and Analysis Functions with Bias Magnitudes for Specific Analysis.

Function Function numbers are preceded with 4.1,2	Analysis Method/Sample type											
	H ₂ O Analysis			Energetics Analysis			Alpha Analysis			Other		
	Direction	Magnitude	Cause	Direction	Magnitude	Cause	Direction	Magnitude	Cause	Direction	Magnitude	Cause
4.2.d Collect Aliquot of solids for analysis and prepare Archives		<6%/hour	Exposure caused drying	+	?	Compensation effect from loss of moisture						
4.2.e Separate Organic layer and Filter Liquids												
4.2.f Archive Solids from Filtering												
4.2.g Collect Liquid Aliquots for Analysis and Prepare Archive												
4.3.a Analyze for Energetics												
4.3.b Analyze for Moisture												
4.3.c Analyze for Total Alpha												

Table 4-3. Sampling and Analysis Functions with Bias Magnitudes for Specific Analysis.

Function (source numbers are provided with 4.2.1.2)	Analysis Method/Sample type											
	H2O Analysis			Energetics Analysis			Alpha Analysis			Other Analysis		
	Direction	Magnitude	Cause	Direction	Magnitude	Cause	Direction	Magnitude	Cause	Direction	Magnitude	Cause
.4.3.d Analyze for Li												
.4.3.e Perform other Analysis												

5.0 TESTING REQUIRED TO QUANTIFY POTENTIAL SIGNIFICANT BIASES

Testing needed to evaluate the effects of potential sampling and analysis biases are listed below in order of priority. This prioritization is subjective in that a complete analysis would be based on which analysis was the most important. Primarily the tasks were prioritized to gain knowledge in specific areas and identify conditions under which bias occurs.

1. Waste tanks samplers appear to sample different types of waste forms with different levels of success. Determining the sample bias for or against various types along with waste type interfaces could lead to the quantification of typical losses of materials under different circumstances. It should be possible to use simulated waste that was made to model the different waste physical properties. Examples include, layers of slush/liquid, soft/grains, firm/crystalline, hard crust, and mixed in various orders as expected in the tanks.
2. An evaluation of the amount of mixing and contamination by the HHF for different types of material is needed. Infusion of HHF into the sample material will vary with sample material, depth in tank and specific gravity of the waste. Current knowledge of HHF infusion, and secondary effects on the waste is limited. Specific tests need to be constructed to determine the limits of HHF intrusion, ways of monitoring intrusion, evaluation of secondary effects
3. Evaluate alternative methods for energetics analysis. Evaluation will need to consider moisture loss during sample preparations and ease of analysis. Possible alternatives include the analysis of additional sub-samples to enhance statistical treatment of the data. Another alternative is to use DSC/TGA system, which is capable of handling larger samples.
4. Evaluate alternative methods for moisture analysis. Evaluation will need to consider moisture loss during preparation and ease of analysis. Possible alternatives include using larger samples and performing the analysis in the hot cell. Another potential alternative is performing more measurements to enhance the statistical treatment of the data. This will not affect any bias that is present but it will help determine if one exists.
5. The amount of moisture loss associated with the auger sampler has not been completely studied. Testing to determine the moisture retention loss curve associated with waste forms collected with the auger sampler is needed. Moisture retention is associated with waste form and molecular species of the waste. The outcome of this testing would be the possible development of a model to predict a saturation moisture range or threshold for a particular tank waste.

6. Water is added during rotary mode core drilling to unplug drill strings. The effects of this process on the sample being taken are uncharacterized. Testing to determine the direction of flow and potential for the water to enter the sample area needs to be considered.
7. As the sample enters the core sampler and then again as the sample is extruded from the sampler friction of the sample with the sampler walls will distort the sample. The extent of this distortion is unknown. Testing to determine the extent of distortion experienced by the sample is needed. First of all however, the degree of smearing that would be allowable and still obtain the data needed needs to be determined.
8. The effects of temperature variation and agitation during sample transportation are unknown. Testing to determine the possible sample changes that potentially exist and the magnitude of those changes are needed.
9. Further evaluate the correlation between DSC and ARC energetics measurements. A method for calibration and verification of results should be developed that is specific to the Hanford waste matrix variations. System accuracy and precision need to be determined and evaluated to the needs of the safety program.

6.0 DETAILS OF FUNCTION BIAS SENSITIVITY

The following is a summary of the specific potential biases associated with each process function. Appendix A contains a description of each function listed. More detailed System Engineering breakdown of the functions can be found in Systems Engineering database and referenced. All of the System Engineering breakdown numbers begin with 4.2.1.2.

.3.3.a Prepare for Sampling: The improper selection of sampling method could directly affect the quality of the resultant data. The introduction of foreign material used in the maintenance or cleaning operations that would contaminate sample material. Equipment fabrication greases and solvents may affect sample analysis. Procedures are in place to clean and verify samplers to minimize the possibility of this event.

.3.3.b Monitor Tank Operations Parameters: The use of video monitoring of tank sampling events has greatly improved the ability of the operators to determine sample quality. Issues such as sampler interface with the waste surface, and debris from previous related activities that may cause interference are just examples of information now available.

.3.3.c Insert Sampler into Tank: When more than one sample is taken from the same riser the possibility exists for cross contamination. Water used to wash the drill string and other activities performed during sampling could affect the next sample to be taken. Limited riser locations, which limit sample, selection can create a whole tank estimate bias.

.3.4.a Position Sampler: Waste stratification and physical properties inhomogeneity can cause biases. In some cases, these can be detected and mitigated.

.3.4.b Take Grab Sample: The use of a sample bottle containing air allows the mixing of sample bottle air with the liquid sample as it enters the bottle. Solids/liquids mixtures may not be accurately represented as material with less viscosity will more easily enter and fill the bottle.

.3.4.c Take Auger Sample: The auger sampler is designed to penetrate hard materials by cutting and grinding as it is lowered and rotated into the waste material. Thus, physical makeup of the retrieved sample is considerably disturbed. Significant mixing of waste layers can occur because of the motion and operation of the sampler.

The auger sampler will not capture free liquid.

.3.4.d Take Rotary Mode Core Sample: For liquids and viscous samples the drill string is usually operated in a push mode and as such should have minimal selectivity to waste heterogeneities. It is possible that a lack of capture could result from inadequate hydrostatic pressure to overcome the exiting nitrogen gas stream. This event may also result in restricted collection of less-fluid components of the waste.

For multiphase wastes, the interaction of the drill string rotation, nitrogen gas flow, and sample collection is complex. It is very possible that a sample collection bias toward the more liquid components of a multiphase sample would exist. This is particularly true during the setting of the pintle rod for sampler removal when a slight vacuum is formed and additional sample may be drawn into the sampler. When a liquid sample is being taken in rotary mode (as would be the case during a hard to soft transition), the sample can be contaminated by the capture of purge gas.

Solid salt cake wastes could exhibit a lack of capture because of loss as cuttings. The action of drilling into the salt cake produces fine particles that are carried away by the nitrogen gas flow. This could also result in a substitution of sample by material that does not degrade into fine particles.

Inaccuracies in the moisture measurements from core samples are incurred because of the use of a nitrogen gas flow in the rotary-mode core sampling system, which cause the drying of sample material (as it is contained in the sample) as well as potential drying of samples in hot cells. The present method provides conservative values (e.g., potentially lower moisture than in tank), and may result in unnecessary safety restrictions.

In all cases, the waste sample can potentially smear the components because of adhesive forces between the waste and sampler walls. Due to the nature of the sampler designs, the material at the top of the sample travels up through the bottom of the sampler and can possibly smear on the bottom of the sample tube and then attach itself to the lower portion of the sample. The difference in diameter of the sampler mouth and internal cavity could cause the loss in preserving the individual waste layers and phases in the sample. It could also cause a preference for collection of more fluid material over more solid material.

After a sample has been taken, and the valve closed on the sampler, the bit is raised about an inch and a half. The material below the valve is supposed to stay in place (relative to waste) so it will not be sticking up when the next sampler is inserted. If it stays with the sampler, the next sample could come up short and if it falls out or remains in place relative to the bit, it will be squeezed flat when the next sampler is inserted or may prevent the sampler from being fully lowered into the bit.

The core sample bit transfers mechanical energy to the waste. This energy transforms the waste by changing its shear strength, viscosity and other physical parameters.

.3.4.e Take Push Mode Core Sampler: For homogenous samples of liquid or mostly liquid waste, the push mode sampler should not exhibit significant bias. For multi-phase samples, the push mode sampling can bias the retrieved sample towards material that can more easily enter the sampler. Solid samples may result in incomplete capture due to cohesive forces in the waste that are beyond the drill bits ability to cut through them. This could also result in solid wastes being pushed down into softer material below rather than being sampled. This situation has lead to the development of a 60 degree push mode bit that more effectively pushes most material in a lateral direction rather than downward.

Failure to capture the top 14 cm of waste will directly affect the ability to determine the safe condition of the tank.

Smearing of the sample may occur under certain conditions. Because of the nature of the sampler designs, the material at the top of the sample travels up through the bottom of the sampler and can possibly smear on the bottom of the sample tube and then attach itself to the lower portion of the sample.

The addition of hydrostatic head fluid during sampler change out could bias all of the core segments below the first segment. LiBr tracer is added to the HHF to allow for determination of the amount that was added. However, the model used to perform this calculation is a simple one and it has many secondary effects that are not quantified. The calculation assumes that the fluid will be well-mixed. It assumes that the waste matrix remains constant with only an addition of moisture (differences in the solubility of different compounds in the waste matrix would make this assumption untrue). It assumes that the LiBr remains in solution and does not precipitate. It also assumes that additional sample bias is not included due to additional liquidity of certain forms of the waste.

Bias because of evaporative effects should be minimal because of the use of a closed sampler. The sampler is closed and sealed before it is removed from the tank.

The core sample bit transfers mechanical energy to the waste. This energy transforms the waste by changing its shear strength, viscosity and other physical parameters.

.3.4.f Move Sampler to Top of Tank:

Core Samplers

Push mode and rotary mode sampler systems are sealed before they are raised to the top of the tank and should not exhibit any significant form of bias. If the valve does not fully close, drainage of any liquid in the sample could occur biasing the sample towards the captured solids. The sampler is not air tight and therefore volatile compounds and trapped gases could escape due to pressure changes.

In push mode core sampling, where LiBr laced water is used as a HHF, removing the sampler too fast could potentially cause a vacuum to be formed in the core barrel. This would tend to draw waste material into the core barrel. When the sampler was raised sufficiently, the HHF

would then pass by the sampler and into the bottom of the drill string with sufficient force to wash out the end into the waste material causing contamination.

An accurate measurement of the specific gravity of the waste is usually not available. This leads to the practice of using more HHF than calculations indicate is needed to assure that no waste material enters the core barrel. This means that the chances of HHF leaving the core barrel and entering the waste matrix are almost assured.

Auger Sampler

For the auger sampler, this is the primary function in the process of taking an auger sample where sample loss occurs. Samples that are relatively dry and crumbly have low cohesive forces and will fall out of the bit. Mixed phase samples will tend to let the liquid drain out while retaining solid material with sufficient cohesive force to remain on the auger fins. Both cases indicate a bias of the sampler towards samples that exhibit higher cohesive forces.

Determination of the amount of bias would require a significant amount of data. To predict a saturation moisture range or threshold for a particular tank waste the following information would be needed.

Molecular species

concentration data for ions

metallic elements

other relevant chemical properties

physical properties data such as particle size, pH and density

If prior moisture data are available on that waste, such knowledge information could be used to estimate the bias associated with the data. If no prior moisture data is available while the tank is about to be sampled, such information can project the magnitude of the moisture contents bias, and thus determine if the sampling system being utilized, in this case the auger, is appropriate.

The sampler is not confined until it is removed from the tank and secured in a shipping cask. This process can take up to an hour and the sample to void volume ratio in the cask may be greater than ten. Both of these factors may allow for moisture to evaporate from the sample.

Grab Sampler

The grab sampler is not sealed until it reaches the top of the tank. Movement of the sample bottle through the waste could cause mixing with waste above the sampled location. Gases generated by or associated with the waste are free to escape during the sample transit through the tank head space. The sample will be contaminated by air from the bottle and the vapor in the tank. In some cases the sample has been diluted by water used to clean the wire rope, bottle and sampler exterior.

.3.4.g Move Sample to Transfer Cask: At this point all samples become exposed to environmental conditions different than interior to the tank. No temperature or environmental controls are present to control waste chemical reactions.

Grab Sampler

The stopper is placed back in the mouth of the sampler bottle effectively sealing it before transfer to the OTC.

Core Samplers

The core samplers remain closed (not gas tight). The X-ray imagery is an external energy source that may be a catalyst for additional chemical reactions. Mixing from movements could also change the characteristics of the sample.

.3.4.h Survey Release Work Area: No potential biases identified.

.3.6 Transport Samples to Laboratory: As samples are loaded, transported and unloaded they experience agitation and vibration from the movement. This effect could cause mixing and settling of the sample that would change its appearance and contents from how it existed in the tank.

From the time samples leave the tank to when they get archived they are not temperature controlled. The effects of heating, cooling or cycling of temperatures is unknown. This may be a very important parameter for precipitating reactions.

Samples that experience low recovery, void spaces, or solid/liquid separation may experience rearrangement of the sample sections. To combat this, in some cases the sampling operations have taken only partial segments. This assures that when they are extruded in the hot cell that the location of the waste is known to within the length of core taken.

.4.2.a Receive and Extrude Sample: The extrusion process can potentially have many effects on the sample.

For Salt Cake material, the effect of fines generated during sampling and redistributed during transportation is unknown.

For multi phase samples, it is very possible for the extrusion process to push the liquids out in ahead of the solids. In addition, the normal draining when the sampler valve is opened may cause a redistribution of the solid material within the sampler. Exact indication of sample layering information may be lost.

Moisture loss studies indicate that up to 6 % relative per hour of moisture can be lost while the sample is exposed during hot cell operations. The recording of the time that the sample is open could be used to correct for moisture loss during this time (Winters 1994).

.4.2.b Separate Drainable Liquids and Inspect: Moisture loss studies indicate that up to 6 wt% per hour of moisture can be lost while the sample is exposed during hot cell operations.

.4.2.c Divide Sample and Homogenize: The homogenization process changes the characteristics of the sample. Past experience has shown that moisture that was bound becomes free during the process. The process enhances the exposure to atmospheric air increasing the potential for drying and oxidation to occur as well as enhanced loss of volatiles.

Homogenization will also change sample cohesion and particle size distribution.

Moisture loss studies indicate that up to 6 wt% per hour of moisture can be lost while the sample is exposed during hot cell operations.

.4.2.d Collect Aliquot of solids for analysis and prepare Archives: The scale of homogeneity is unknown. Therefore the size of the aliquot removed may not be sufficient to represent the average value of that analyte in the waste tank material.

Depending upon how much liquid has remained in the solids, whether wash water was used, etc. At times, centrifugal separation will give two solids layers, one less dense than liquid.

.4.2.e Separate Organic layer and Filter Liquids: No potential biases identified.

.4.2.f Archive Solids from Filtering: No potential biases identified.

.4.2.g Collect Liquid Aliquots for Analysis and Prepare Archive: The scale of homogeneity is unknown. Therefore the size of the aliquot removed may not be sufficient to represent the average value of the waste tank material.

Over time, some of the highly caustic waste has been known to dissolve the glass jars. This indicates that chemical processes are taking place in the archive environment.

.4.3.a Analyze for Energetics: A moisture determination by TGA is required to interpret the energetics measurement. If total endothermic/exothermic calculations are made any errors from the TGA measurement will propagate to errors in determining energetics.

Because of the small sub-sample size used with the DSC, sample representativeness is in question. For a secondary analysis a ARC(RSST) instrument is used. This instrument can use

a much larger sample size. However, RSST measurements are made on a dry weight basis.

Interpretation of the baseline determination can effect interpretation of results.

Comparison of results from samples analyzed with both DSC and ARC have shown poor correlation. In general, the ARC procedure is felt to have better accuracy but this has not been substantiated for this waste matrix.

.4.3.b Analyze for Moisture: Due to the small sample size (10-30 mg) being analyzed, representativeness of the complete sample is questionable.

The presence of volatile organics in the sample will give positive bias towards moisture measurement.

The definition of what type of moisture is important to the safety issues addressed in the safety screening DQO is unclear. No distinction is made between free, drainable, bound, or waters of hydration is made. In order to determine bias, one must first know what information is desired. From this the instrument can be analyzed as to what it measures and any biases can be determined.

.4.3.c Analyze for Total Alpha: A build up of solids on the counting dish can create a problem. These solids can result from dissolved material in the sample, such as the potassium salts from a fusion preparation or corrosion of the counting dish by hydrochloric acid from the fusion dissolution. The amount of solids in the dish should be kept to a minimum or self-absorption of the activity, especially alpha, can result and lead to low results.

Relative activity levels such as high beta or gamma to low alpha may cause detector crosstalk depending on the ratios, this bias can be positive or negative.

Possible biases exist in the fusion process for sample preparation. The yield for the process is unknown.

In most cases the measurement is biased low and the calculation is biased high attempting to give a conservative answer.

.4.3.d Analyze for Li: LiBr was used as a tracer in HHF and other water used to unplug or unstick the drill string bit because it was not expected to be in tank waste.

Using the concentration of the tracer element is a correction for the amount of added water in the sample could be performed.

Past experience has shown that Li cannot be expected to remain in solution when contacted with tank wastes. Bromide is however expected to be chemically inert under alkaline tank conditions and will not encounter significant amounts of precipitating cations in tank waste.

In addition, there are some unquantifiable secondary effects of HHF added to the tank waste.

The HHF is not prevented from convectively leaching soluble analytes from the waste medium in places other than the sampled segment portion. This would act to enrich the as-found associated liquid and subsequently shift the corrected values of liquid analytes to higher values. The portion that is leached from the sampled segment should not affect calculated total segment content, but any portions leached from material outside the waste core will. The amount of HHF introduced into the sample, the solubility of the waste type and the amount of convection will determine how much bias is produced. LiBr is also known to be corrosive to steel.

Large amounts of HHF can be expected to produce unquantifiable enriched soluble analytes in the corrected drainable liquid values.

In addition, the HHF tracer found in the homogenized solids could be infused there in ways concurrent to simple wetting. If the waste is sufficiently rigid, the HHF could conceivably displace interstitial liquid from the pores of the solid: not merely adding to the water content, but rather displacing it. As a result the final corrected water content will be excessively low. This type of error would add conservatism from a safety assessment point of view.

Diffusion of tracer ions from HHF to water-saturated solids could also occur and once again give a low corrected water content.

"Analytical results having manageable uncertainties may be recovered from HHF-contaminated segments if the contamination is not too severe. This means that to do this depends on a simple model whose secondary effects can be ignored, and on laboratory efforts to secure analysis on homogenized or multiply-sampled segment sections".¹

.4.3.e Perform other Analysis: No biases identified.

¹. Bechtold, D. B. 1995, *Use of LiBr as Tracer for HHF and Correction of Results*.

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APPENDIX A

TANK WASTE REMEDIATION SYSTEM

FUNCTION AND REQUIREMENTS

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TWRS FUNCTIONS AND REQUIREMENTS

The process of developing functions and requirements for tank waste characterization was initiated by TWRS and published in Tank Waste Remediation System Functions and Requirements. Table A-1 provides the breakdown structure and function descriptions for the TWRS level four function to, characterize waste. The TWRS breakdown consisted of level four to level six functions. Level 7 was added to provide more detail in the analysis of process steps and their impact on the data. The level 7 breakdown was based on the operating procedures used to perform those functions.

Table A-1 Breakdown Summary of TWRS Characterization Function.

TWRS Level 4 Functions	TWRS Level 5 Functions	TWRS Level 6 Functions	Proposed Level 7 Functions	Function Definitions
4.2.1.2 Characterize Waste				Characterize waste by provide characterization information to satisfy TWRS Manage Tank Waste and Process Tank Waste functions. Tank Waste consists of all Double-Shell, Single-Shell, and Miscellaneous Underground Storage Tanks. Determine information needs through the Data Quality Objectives Process and assessment of information needs. Determine technical basis through the development and optimization of the Tank Waste Analysis Plan and Tank Characterization Plans and preparation of an integrated schedule. Acquire samples and measurements including samples transportation to the laboratory. Analyze samples including data validation and reporting.; Summarize and interpret integrated data by developing predictive layering and inventory models, preparing Historical Tank Contents Estimates, and Tank Characterization Reports. Provide Tank Characterization Information by compiling, maintaining, and providing access to characterization information databases. Treat and prepare r of liquid, gaseous, and solid wastes generated during the characterization of tank wastes.

Table A-1 Breakdown Summary of TWRS Characterization Function.

TWRS Level 4 Functions	TWRS Level 5 Functions	TWRS Level 6 Functions	Proposed Level 7 Functions	Function Definitions
	4.2.1.2.3 Acquire Samples / Measurements			Sample tank waste, collect waste characteristics measurements, and deliver the samples to the laboratory. Maintain the sampling / in-situ measurements equipment, and plan for the sampling / in-situ measurement event. Select riser, prepare work plans, deployment and relocate sampling / in-situ measurements equipment, collect the waste samples or measurements, transport the waste samples to the laboratory's hot cell(s), and providing in-situ measurement reports. The physical samples consist of solids, liquids, or gases, or possibly a combination of all three. Grab samples refer to samples of the supernatant liquid phase tank layer. Vapor samples refer to tank dome gas phase samples. Push or rotary mode samples are mostly a combination of liquid (supernatant or waste interstitial) and solids (sludges and salt cake). This function also includes the treatment of gaseous effluents and the packaging of solid wastes generated during the sampling and measurements acquisition event.
		4.2.1.2.3.1 Maintain Sampling / In-Situ Measurements Equipment		Maintain sampling / in-situ measurement equipment to maintain optimum working conditions. This task also includes equipment development and improvement.
		4.2.1.2.3.2 Plan for Sampling / In-Situ Measurements Event		Plan for sampling / in-situ measurements Event by developing the necessary work plans and safety documentation such as Environmental Impact Statements, Operational Readiness Reviews, Plant Engineering Procedures, and Safety Analysis Reports. This function also includes riser selection and other related tasks.

Table A-1 Breakdown Summary of TWRS Characterization Function.

TWRS Level 4 Functions	TWRS Level 5 Functions	TWRS Level 6 Functions	Proposed Level 7 Functions	Function Definitions
		4.2.1.2.3.3 Deploy Sampling / In-Situ Measurements Equipment		Deploy sampling / in-situ measurements equipment including riser preparation, positioning of crew(s), sampling vehicles, tank access platform / in-situ measurements delivery platform, crane(s), casks and cask stands, support generators, and all other ancillary equipment.
			4.2.1.2.3.3.a Prepare for Sampling	Prepare all paperwork and assure all required materials are available for use during sampling event. Evaluate tank environment for personnel safety issues and establish appropriate procedures. Set up sampling equipment to prepare for sampling event. Open tank and install tank opening interface equipment.
			4.2.1.2.3.3.b Monitor Tank Operations Parameters	Monitor appropriate tank operating parameters for changes during the sampling process. Install and operate video equipment to monitor and record sampling event.
			4.2.1.2.3.3.c Insert Sampler into Tank	Position sampling device above the waste and ready to move to the position in the tank where the sample will be taken.
		4.2.1.2.3.4 Collect Samples / Measurements Data		<p>Collect samples and measurement data by collecting waste samples / in-situ measurements from single shell tanks, double shell tanks, and other miscellaneous Underground storage tanks. This function also includes reporting in-situ measurement data.</p> <p>Collect a waste sample, insert the sampler into the tank waste strata (or dome in case of a vapor sample), retrieve and withdraw the sample, store into a HLRW cask, and seal the cask.</p> <p>In-situ measurement are made by inserting the cone Penetrometer into the waste data relayed by the probes or sensors installed on that cone.</p>

Table A-1 Breakdown Summary of TWRS Characterization Function.

TWRS Level 4 Functions	TWRS Level 5 Functions	TWRS Level 6 Functions	Proposed Level 7 Functions	Function Definitions
			4.2.1.2.3.4.a Position Sampler	Use the deployment mechanism to place sampler at desired location in the tank. For Grab samples the sample bottle is lowered to desired height. The Auger sampler is positioned above the waste surface. For core sampling, the equipment is positioned ready to take the next segment.
			4.2.1.2.3.4.b Take Grab Sample	Get sample in sampler by jerking on the bottle tether and dislodging the stopper. Allow sample material to drain into the bottle and displace the air.
			4.2.1.2.3.4.c Take Auger Sample	Rotate auger into waste material until pre-defined stop is reached.
			4.2.1.2.3.4.d Take Rotary Mode Core Sample	Rotate Coring bit while pushing sampler into waste and holding pintle rod stationary to collect a core length of 19 inches maximum per operation.
			4.2.1.2.3.4.e Take Push Mode Core Sample	Push sampler without rotating sampler into waste to collect a core length of 19 inches maximum per operation.
			4.2.1.2.3.4.f Move Sampler to Top of Tank	Retrieve sampler to top of tank receiver cask.
			4.2.1.2.3.3.4 Move Sample to Transfer Cask	Move top of tank receiver cask to the On-Site Transfer Cask (OTC) and move sampler to OTC.
			4.2.1.2.3.4.h Survey Release Work Area	Close tank opening at end of sampling event. Survey area for contamination. Package reusable sampling equipment. Perform post ALARA review.

Table A-1 Breakdown Summary of TWRS Characterization Function.

TWRS Level 4 Functions	TWRS Level 5 Functions	TWRS Level 6 Functions	Proposed Level 7 Functions	Function Definitions
		4.2.1.2.3.5 Relocate Sampling / In-Situ Measurements Equipment		Remove sampling / in-situ measurement equipment from the site for relocation. Restore the sampling site and the tank riser to a pre-sample condition. Package the solid waste and treat the gaseous effluents generated during the sampling / in-situ measurement event.
		4.2.1.2.3.6 Transport Samples to Laboratory		Transport samples to the laboratory by performing radiation surveillance of the casks, loading the sample casks (including cask stands) onto the transportation truck, delivering the casks to the laboratory's hot cell, and relinquishing custody of the samples and casks.
	4.2.1.2.4 Analyze Samples			Analyze the sample to provide physical, chemical, and radiochemical information on waste samples (liquids, solids and gases). Maintain laboratory capabilities for sample analysis and processing of in-situ measurement data. (This includes the necessary equipment and instrumentation maintenance and upgrades. Receive samples / in-situ measurement data, and break those samples into the appropriate aliquot as specified in the Tank Characterization Plan. Prepare those samples for analysis, and perform the analysis as specified. Transfer samples between laboratories if necessary. Report, validate and distribute the analysis / in-situ measurement results. Treat, prepare and package liquid, gaseous, and solid wastes generated during the analysis of waste samples.
		4.2.1.2.4.1 Maintain Analytical Equipment		Maintain analytical equipment by completing the necessary equipment and instrumentation maintenance tasks including upgrades, repairs, and modifications to meet the required capabilities.

Table A-1 Breakdown Summary of TWRS Characterization Function.

TWRS Level 4 Functions	TWRS Level 5 Functions	TWRS Level 6 Functions	Proposed Level 7 Functions	Function Definitions
		4.2.1.2.4.2 Receive and Breakdown Samples		Receive and breakdown samples to prepare for analysis. Receive samples from the field or from other laboratories. Log-in samples and chain of custody transfer. Perform extrusion, sub-sampling, homogenization of samples to meet the analysis needs outlined in the Tank Characterization Plan or other appropriate work document. Store and archive samples, package and ship samples to other laboratories for analysis. Treat gaseous effluents, and prepare solid wastes generated during the receipt and breakdown of waste samples.
			4.2.1.2.4.2.a Receive and Extrude Sample	Prepare sample for transfer to extruder in Hot Cell. Load sampler into Hot Cell and extrude sample from core sampler. Scrape Auger samples off the auger.
			4.2.1.2.4.2.b Separate Drainable Liquids and Inspect	Inspect extruded sample and record information as specified by the applicable Sample and Analysis Plan. Recordable observations may include a Sketch, description of color, texture, homogeneity, and consistency. Take a color photograph and or videotape. If extruded core sample contains drainable liquids, drain them into a separate sample container.
			4.2.1.2.4.2.c Divide Sample and Homogenize	Core sample solid materials are subdivided into half or quarter segments. These subdivided sections are then homogenized according to the current appropriate procedure. Auger samples are not subdivided.
			4.2.1.2.4.2.d Collect Aliquot of Solids for Analysis and Prepare Archives	Remove enough aliquot from solid sample material to perform the required preparations and analysis called for in the SAP in duplicate. Remove at least 20 mL and up to 40 mL of each homogenized sub-sample and archive according to Bratzel, 1994, Letter, "Archiving Requirements"
			4.2.1.2.4.2.e Separate Organic layer and Filter Liquids	Inspect liquid samples for the presence and approximate volume of any potential organic layers. If present, separate and retain the possible organic layer for future analysis. Filter the remaining liquid through .45 micron filter.

Table A-1 Breakdown Summary of TWRS Characterization Function.

TWRS Level 4 Functions	TWRS Level 5 Functions	TWRS Level 6 Functions	Proposed Level 7 Functions	Function Definitions
			4.2.1.2.4.2.f Archive Solids from Filtering	If there is greater than 1 gram of solids present from filtering the liquid, and archive them for possible future analysis.
			4.2.1.2.4.2.g Collect Liquid Aliquots for Analysis and Prepare Archive	Remove sufficient aliquot from the segment-level liquid sample to perform the appropriate analysis listed in the SAP in duplicate. Archive at least 20mL of the liquid and up to 40mL of the liquid per Bratzel, 1994, Letter, "Archiving Requirements".
		4.2.1.2.4.3 Prepare and Analyze Samples		Prepare and analyze samples by performing sample dissolutions and extractions, sample analysis or re-analysis, if necessary. Perform the necessary quality control activities and computations, review the analytical / in-situ measurement results, and store those results in a Laboratory Information Management System. Treat liquid and gaseous effluents, prepares and packages solid wastes generated during the analysis of waste samples.
			4.2.1.2.4.3.a Analyze for Energetics	Perform a Differential Scanning Calorimeter measurement directly on sample aliquots removed from the main sample.
			4.2.1.2.4.3.b Analyze for Moisture	Perform Thermal Gravimetric Analysis measurement directly on sample aliquots removed from the main sample.
			4.2.1.2.4.3.c Analyze for Total Alpha	Perform Total Alpha particle measurements on a fusion or acid dissolved aliquot.
			4.2.1.2.4.3.d Perform other Analysis	Analysis other than for safety screening may be called for in the SAP. If any of the Safety Screening Analysis exceeds set limits, then a secondary set of analysis is performed. These analysis may include RSST for energetics, ICP analysis for Fe, Mn, U and Br. Hot per sulfate for TOC and distillation for CN analysis.

Table A-1 Breakdown Summary of TWRS Characterization Function.

TWRS Level 4 Functions	TWRS Level 5 Functions	TWRS Level 6 Functions	Proposed Level 7 Functions	Function Definitions
		4.2.1.2.4.4 Report Analytical Results		Report the analytical results by integrating, evaluating and distributing the results of the sample analysis as specified by the Tank Waste Analysis Plan, the specific Tank Characterization Plan or other supporting documents if applicable. Reporting requirements varies depending upon user requirements and intended data use. Reporting formats include but are not limited to letter reports, Laboratory Information Management System printouts, data packages with raw data, reports and results in electronic format for input to the Tank Characterization Database or other electronic characterization databases. Copies of those data results are relinquished to the Characterization Data Management function, and are also used as an input to the Tank Characterization Report development function.
		4.2.1.2.4.5 Validate Analytical Results		Validate analytical results by verifying and validating data packages prepared by the laboratory. Review the data package to ensure the laboratory has performed and properly documented the requested analyses in accordance with the requirements set forth in the Tank Waste Analysis Plan and Tank Characterization Plan or other appropriate working documents, and the appropriate quality assurance documentation such as HASQAP. Prepare a validation report that documents the results of the validation. Validation is only performed when the Tank Waste Analysis Plan and Tank Characterization Plan or other appropriate documentation requires that the data be validated.

APPENDIX B

RMCS TRUCK 2 ATP SAMPLE RECOVERY RESULTS

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APPENDIX B: RMCS TRUCK 2 ATP SAMPLE RECOVERY RESULTS.

This is a summary of the information obtained from sample recoveries obtained during acceptance testing for core samples. The information was taken from section A2 and A3 of WHC-SD-WM-ATP-048, "Acceptance Test Report for Core Sample Truck (Rotary Mode, H0-68K4345".

The following tables summarize the test sample data sheets and provide percent recovery for saltcake (Sulfur K-Mag), sludge (Bentonite clay), and liquid (water) mediums. The table of recoveries was used to determine whether the sampling system met the criteria of 90% recovery 90% of the time which is contained in "Development Criteria for the Rotary Mode, Universal Sample & Bit, and NPH Elimination System" WHC-SD-WM-CR-044 Rev. 1. The following excerpt from the criteria document establishes the method used in obtaining the percent recovery:

The sampler shall obtain and retain for:

- **Liquids and Sludges:** Ninety percent by volume of the designed capacity of the sampler 90% of the time. The designed capacity for liquid and sludge samples is 310cc. For acceptance and operational testing purposes, the sludge sample recovery may be calculated by the percent length of recovery. The sludge sample must meet the 90% recovery 90% of the time standard.
- **Hard Salt Cake:** Ninety percent of the expected length of the sample 90% of the time for hard salt cake samples. The designed length is 19 inches. The expected length may vary depending on drilling procedures.

Core and Segment Numbers

The segment numbering in the table and on the test sample data sheets provides an alphabetic letter referring to the core sample followed by a numeral referring to the segment. For example, C6 would refer to the sixth segment in the "C" core sample. Core samples D, F, H, and J are included in the table for evaluation of the sample recovery. Core samples A, B, C, E, G, and I were used as part of the functional testing and are not included in the acceptance testing. The ATP core samples D, F, H, and J were comprised of the following materials in a 20 inch diameter column:

- | | |
|----------------------|--|
| Core Sample D and F: | 7 ½ ft of water on top
7 ½ ft of Bentonite Clay on the bottom |
| Core Sample H: | 5 ft of Sulfur K-Mag on top
7 ½ ft of water in the middle
7 ½ ft of Bentonite clay on the bottom |

Core Sample J:

20 ft of Sulfur K-Mag

In core samples D, F, H, and J the following segments are not included in the table as part of the recovery calculation because of the reasons listed.

Segment F1: This segment was not included because considerable liquid was leaking from the sampler when retrieved. The amount of liquid lost was not known. This segment was excluded from the recovery calculation. Sampler leakage only occurred after their repeated use in testing and is not expected to occur in actual operation where samplers are only used once.

Segment H8: This segment was not included due to failure of the sampler valve which blocked off the sampler inlet. This failure occurred once after repeated uses of that specific sampler and is therefore not expected to occur in actual operation where samplers are only used once.

Segments D5, F5, and H4: These segments were not included in the recovery calculation due to transitioning between different sampling mediums. Section 8.0.7 of the ATP excludes transition zones from the sample recovery requirement.

- **Sampling Length:** The distance the drill string was penetrated into the simulated waste during each segment of sampling operations. This length is used as the basis for "expected sample length" or "expected sample volume".
- **Measured Sample:** The raw measurement of the sample length or volume obtained from the segment. For liquids, this measurement was taken by pouring the sample from the sampler into a milliliter graduated cylinder. Sludge measurements were taken by extruding the sample onto a tray and measuring the length of the extruded slug, end to end. Salt cake simulant measurements were taken by measuring the length of the sample while still in the sampler tube. This was accomplished by measuring the linear change in sampler piston position during extrusion from the point from the sample starts to exit the sampler to the point where the bottom of the piston is positioned at the sampler exit.
- **Adjusted Recovery:** The measured length of salt cake simulant samples adjusted by an area factor (AF). AF is defined in revision one of the criteria document as an adjustment factor required to account for the difference in cross sectional areas of the bit and sampler. For the ATP samples the AF varies from 1.04 (unit of measurement?) to 1.266 due to the proportion of fines to chunky material. Chunks were defined as any cylindrical slug, one half inch or greater in length, and were noted as a data sheet comment. This adjustment reflects the decrease in sample length (inside the sampler) when fines expand to fill the 1 1/8 inch diameter sampler. Chunky material will remain at the one inch diameter size of the drill bit inlet hole. Adjusted recovery is used in Tables A3.4 and A3.5.
- **Percent Recovery:** For liquids, percent recovery is the measured sample divided by

the expected sample. Where the expected sample is 310 mL (sampler design volume), multiplied by the ratio of sampling length to 19 (length of complete segment).

For sludges, percent recovery is the measured sample divided by the expected sample, where the expected sample is the sampling length.

For salt cake simulant, percent recovery is the adjusted recovery divided by the expected sample, where the expected sample is the sampling length.

Recovery Rates Exceeding 100%

Recovery rates can exceed 100% as demonstrated in the following tables. This is possible when additional liquid or sludge material enter the sampler by vacuum pressure created by the piston during pintle rod removal, or when the breakup and settling of salt cake material fines does not fill the sampler void areas and offset the AF adjustment.

$$\text{Percent Recovery} = \frac{\text{Liquid Volume} + \text{Wet Solids Volume}}{\text{Expected Sample Volume}} \times 100\%$$

$$\% \text{ Length Recovery} = *AF \times \frac{\text{Salt Cake Sample Length}}{\text{Expected Sample Length}} \times 100 \%$$

$$\% \text{ Length Recovery} = \frac{\text{Sludge Sample Length}}{\text{Expected Sample Length}} \times 100 \%$$

* Note: AF = Area Factor. This is an adjustment factor to account for the different cross sectional areas of the bit and sampler. The AF will vary from 1 to 1.266 depending on the proportion of fines to solids material (buttons and cylinders). This is due to a decrease in length when fines expand to fill the 1 1/8 inch diameter sampler. Solid material will remain at the 1 inch diameter of the bit cutting head.

TABLE B-1. ATP Sample Information by Core.

ATP Sample Recovery - Core D				
Segment Number Data Sheet Item 1	Sampling Length Data Sheet Comment	Measured Sample Data Sheet Item 16	Percent Recovery Criteria Document	Comments
D1	17	295	106.4	Water
D2	19	326	105.2	Water
D3	19	324	104.5	Water
D4	19	314	101.3	Water
D5	19	176 6.75	92.3	Transition: Water - Sludge
D6	19	19	100	Sludge
D7	19	7.8	41.1	Sludge
D8	19	19	100	Sludge
D9	19	19	100	Sludge
D10	9	17	188.9	Sludge

ATP Sample Recovery - Core F				
Segment Number Data Sheet Item 1	Sampling Length Data Sheet Comment	Measured Sample Data Sheet Item16	Percent Recovery Criteria Document	Comments
F1	15.75	Not used	See Note 1.	Water
F2	19	312	101	Water
F3	19	327	105.4	Water
F4	19	350	113	Water
F5	19	176 10	109.4	Transition: Water--Sludge
F6	19	19.5	102.6	Sludge
F7	19	14	73.7	Sludge
F8	19	18	94.8	Sludge
F9	19	18.25	96.1	Sludge
F10	12	12.5	104.1	Sludge

ATP Sample Recovery - Core H					
Segment Number Data Sheet Item 1	Sampling Length Data Sheet Comment	Measured Sample Data Sheet Item 16	Adjusted Recovery Criteria Document	Percent Recovery Criteria Document	Comments
H1	17	15.5 in	18.8 in	110.8	Kmag
H2	19	17.25 in	18 in	95	Kmag
H3	19	17.5 in	22.2 in	117	Kmag
H4	19	Not Used -- See Note 2.			Transition Kmag-- water
H5	19	331 in	N/A	105.8	Water
H6	19	328 in	N/A	105.8	Water
H7	19	310 in	N/A	100	Water
H8	19	Not Used -- See Note 1.			Transition Water-- Sludge
H9	19	4.5 in	N/A	23.7	Sludge
H10	19	11.5 in	N/A	60.5	Sludge
H11	19	20 in	N/A	105	Sludge
H12	19	19 in	N/A	100	Sludge

Note 1. Segments F1 and H8 were not included in sample recovery calculations because test-unique conditions resulted in partial or total sample loss, rendering the sample measurement meaningless. Specifically, fluid was observed leaking past the cable seal o-rings as the sampler was recovered for segment F1. On segment H8, the sampler ball valve failed prior to sampler removal. These failures were due to repeated use of these samplers during ATP testing (new samplers were unavailable). The samplers are designed for one-time use in the field. Therefore, recovery measurements for segments F1 and H8 are not considered valid data for the ATP.

Note 2. Segment H4 was not included in sample recovery calculations due to the sampler transition from K-mag to water during this segment. The recovery requirement does not apply to transition zones according to step 8.0.7 of the ATP.

ATP Sample Recovery - Core J					
Segment Number Data Sheet Item 1	Sampling Length Data Sheet Comment	Measured Sample Data Sheet Item 16	Adjusted Recovery Criteria Document	Percent Recovery Criteria Document	Comments
J1	6	6.5	8.2	102.9	K-mag
J2	19	17.5	22.2	116.6	K-mag
J3	19	16.5	20.9	109.9	K-mag
J4	19	17.25	21.8	114.9	K-mag
J5	19	14.5	18.4	96.6	K-mag
J6	17.5	13	16.5	94.0	K-mag
J7	19	17	21.5	113.3	K-mag
J8	19	17.25	21.8	114.9	K-mag
J9	19	15.25	19.3	101.6	K-mag
J10	19	16.25	19.3	101.6	K-mag
J11	19	15.5	19.6	103.3	K-mag
J12	19	16.75	21.2	111.6	K-mag
J13	19	17.25	21.8	114.9	K-mag
J14	8	6.75	8.5	106.8	K-mag

Summary Of Core Sample Recoveries In Salt Cake, Sludge, and Liquid					
Test Medium	Segment Number	Segment Length	Sample Quantity	Adjusted Sample Quantity	Percent Recovery
Sulfur K-Mag	H1	17	15.5 in	18.8 in	111
Sulfur K-Mag	H2	19	17.25 in	18 in	95
Sulfur K-Mag	H3	19	17.5 in	22.2 in	117
Sulfur K-Mag	J1	6	6.5 in	8.2 in	103
Sulfur K-Mag	J2	19	17.5 in	22.2 in	117
Sulfur K-Mag	J3	19	16.5 in	20.9 in	110
Sulfur K-Mag	J4	19	17.25 in	21.8 in	115
Sulfur K-Mag	J5	19	14.5 in	18.4 in	97
Sulfur K-Mag	J6	17.5	13 in	16.5 in	94
Sulfur K-Mag	J7	19	17 in	21.5 in	113
Sulfur K-Mag	J8	19	17.25 in	21.8 in	115
Sulfur K-Mag	J9	19	15.25 in	19.3 in	102
Sulfur K-Mag	J10	19	16.25 in	19.3 in	102
Sulfur K-Mag	J11	19	15.5 in	19.6 in	103
Sulfur K-Mag	J12	19	16.75 in	21.2 in	112
Sulfur K-Mag	J13	19	17.25 in	21.8 in	115
Sulfur K-Mag	J14	8	6.75 in	8.5 in	107
Sulfur K-Mag Average Recovery =108%					
Bentonite Clay	D6	19	19	N/A	100
Bentonite Clay	D7	19	7.8	N/A	41
Bentonite Clay	D8	19	19	N/A	100
Bentonite Clay	D9	19	19	N/A	100
Bentonite Clay	D10	9	17	N/A	189
Bentonite Clay	H9	19	4.5	N/A	24

Summary Of Core Sample Recoveries In Salt Cake, Sludge, and Liquid					
Test Medium	Segment Number	Segment Length	Sample Quantity	Adjusted Sample Quantity	Percent Recovery
Bentonite Clay	H10	19	11.5	N/A	61
Bentonite Clay	H11	19	20	N/A	105
Bentonite Clay	H12	19	19	N/A	100
Bentonite Clay	F6	19	19.5	N/A	103
Bentonite Clay	F7	19	14	N/A	74
Bentonite Clay	F8	19	18	N/A	95
Bentonite Clay	F9	19	18.25	N/A	96
Bentonite Clay	F10	12	12.5	N/A	104
Bentonite Clay Average Recovery = 92%					
Water	H5	19	331	N/A	107
Water	H6	19	328	N/A	106
Water	H7	19	310	N/A	100
Water	F2	19	312	N/A	101
Water	F3	19	327	N/A	105
Water	F4	19	350	N/A	113
Water	D1	17	295	N/A	106
Water	D2	19	326	N/A	105
Water	D3	19	324	N/A	105
Water	D4	19	314	N/A	101
Water Average Recovery =105%					

Thirty-seven out of 41 segments (90%) had greater than 90% recovery. This meets the recovery criteria in WHC-SD-WM-CR-044 Rev 1. Lower than 90% recovery in certain sludge (Bentonite clay) segments is suspected to be caused by the repeated use of those samplers. Repeated use caused leakage around the sampler valve cables which allowed air to enter the sample chamber. The air in leakage is suspected to cause the less than optimum recovery in those specific sludge samples. If new samplers had been available for testing it is anticipated that all of the sludge samples would have had greater than 90% recovery.

APPENDIX C

AUGER SAMPLED WASTE INFORMATION

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Table C-1 Auger Sample Information.

Tank	Waste Depth	Waste Layers / Type	Current Temp.	pH	Bulk Density/void Fraction	Moisture Content/ Method/Date/Location	Material Consistency Color/Date
AX-102	9.5"	28Kgal saltcake (14 Kgal Drainable Interstitial Liquid) 7Kgal sludge 3Kgal supernatant (Purest/Blank/Evaporat or feed)	90 lb 114 °F 250 °F (1970)	11.3 (eq.) 1968	1.57 g/mL 0.99 g/mL (dry)	42.5% 1974 (Int.stab. 1968) 28.79% TGA 08/95 Riser 3A 32.21% TGA 08/95 Riser 0E	Riser 3A: No drainable liquid Riser 0E: Wetter material with little drainable liquid Both material had dark brown color 08/95
TY-104	1'	43Kgal sludge (12 Kgal DL) 3Kgal supernatant (Reduced plant (TBP))	85 °F	11.95 (1967)	1.41 g/mL (TLM) 1.625 g/mL (1967)	51.50% TGA 08/95 Riser 18 51.55% TGA 08/95 Riser 15 (1/4 segments)	No drainable liquid or crust. Material from both risers were thick and moist and had a dark brown color (Visc.=10,000cps, APS=6.1 µm) 08/95
TY-106	13"	17 Kgal sludge (diatomaceous earth added)	85 °F	NR	1.43 g/mL (1985)	30.54% TGA 08/95 Riser 7 34.69% TGA 08/95 Riser 6 (Upper half of segment) 39.17% TGA 08/95 Riser 6 (Lower half of segment)	Riser 7: sticky, paste-like brown colored material. Some of the sample was drier and not as sticky, but no drainable liquids were recovered. Riser 6: Moist and runny with a soft paste-like consistency. Color was a mixture of olive-green and light brown. No drainable liquid. 08/95
BY-103	151"	5 Kgal sludge 305 Kgal saltcake (180 Kgal DL) (Metal wash/Pure/Organic wash/57 tons of FS added in 1990)	75 °F	13.5 (1963)	NR	23.83% TGA 08/95 Riser 10B (range: 14.06 to 33.59%) 15.58% TGA 08/95 Riser 12A	Riser 10B: Brown-gray, dry and crumbly. Riser 12A: Yellow-brown, dry and crumbly. 08/95
B-112	≤ 1'	30 Kgal sludge (14/10-slugge/saltcake) 3 Kgal supernatant	63 °F	NR	1.41 g/mL (TLM)	48.9% (TLM) (S 1965) 30.63% TGA 08/95 Riser 7 (Upper half of segment) 41.65% TGA 08/95 Riser 7 (Lower half of segment) 46.36% TGA 08/95 Riser 3	Small amount of drainable liquid. Gray-brown solids distributed in layers on auger flights. 08/95
C-101	2'	88 Kgal sludge (3 Kgal DL) (MM/TBP/CW)	82 °F	NR	1.73 to 1.84 g/mL =0.38	15.43% TGA 05/95 Riser 8 (Crust-like material) 22.58% TGA 05/95 Riser 8 (Upper half of segment) 33.92% TGA 05/95 Riser 8 (Lower half of segment) 73.8% TGA 05/95 Riser 8 (Drainable liquid)	Thick and mud-like appearance. Lower half of segment was damper with tightly packed solids. Upper half contained off-white, crust-like material. 05/95 (phase separation?)

Table C-1 Auger Sample Information.

Tank	Waste Depth	Waste Layers / Type	Current Temp.	pH	Bulk Density/void Fraction	Moisture Content/ Method/Date/Location	Material Consistency Color/Date
C-111	16"	57 Kgal sludge (Purest/Ferr/Cy)	75 °F	NR	1.37 g/mL =0.65 (TLM)	68.49% (TLM) 31.02% TGA 08/05 Riser X (un-homogenized) 28.12% TGA 08/05 Riser XX (second quarter of segment) 41.66% TGA 08/05 Riser XX (third quarter of segment) 44.24% TGA 08/05 Riser XX (bottom quarter of segment)	Riser X: Brown, dry and crumbly Riser XX: Dark brown with tan streaks. 08/05
C-203	26"	5000 gals non-complexed MM	75 °F	NR	1.7 g/mL = 0.2 (TLM)	47% (TLM) 31.39% TGA 08/20/05 Riser 7 (upper half of segment) 37.15% TGA 08/20/05 Riser 7 (Lower half of segment) 35.16% TGA 08/21/05 Riser 7 (upper half of segment) 50.59% TGA 08/21/05 Riser 7 (lower half of segment)	Yellow cream colored overlaid with brown to dark brown sludge. 08/05
AW-101	18"	84 Kgal sludge (2 Kgal DIL) 1041 Kgal supernatant	80 °F	11.8 (kg) (1975)	1.134 g/mL (kg)	 41.17% TGA 08/05 Riser 13A (face/crust) 38.03% TGA 08/05 Riser 13A (upper half of segment) 40.26% TGA 08/05 Riser 13A (Lower half of segment) 42.02% TGA 08/05 Riser 12A (upper half of segment) 38.96% TGA 08/05 Riser 12A (lower half of segment) 33.6% TGA 08/05 Riser 24B (upper half of segment) 40.3% TGA 08/05 Riser 24B (lower half of segment)	Damp, dark brown sludge. Riser 13A had dispersed crust material (sampled separately) 08/05
C-108	18"	66 Kgal sludge	80 °F	NR	1.57 g/mL (TLM) =0.35 (TLM)	29.68% 9.17% TGA 08/12/05 Riser 7, (2.99 and 15.35%) 18.8% TGA 08/12/05 Riser 7 (9.94 and 27.66%) (top quarter of segment) 36.06% TGA 08/14/05 Riser 7 (second quarter of segment) 46.02% TGA 08/14/05 Riser 7 (third quarter of segment) ~50% TGA 08/15/05 Riser 4	Riser 7 Aug.12: Yellow-brown color, crumbly in ladure. Riser 7 Aug.14: Tan color, paste-like Riser 4: Tan, moist, paste-like. Off-white, hard material embedded throughout. 08/05

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