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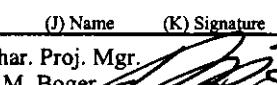
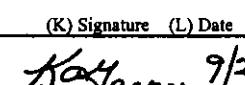
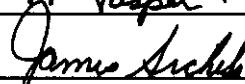
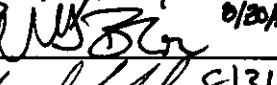
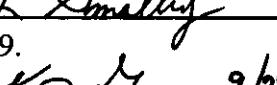
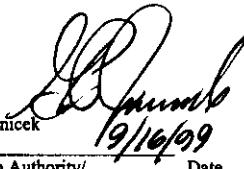
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## PHASE II TEST PLAN FOR THE EVALUATION OF THE PERFORMANCE OF CONTAINER FILLING SYSTEMS

R.M. Boger

Prepared for  
Lockheed Martin Hanford Corporation, Richland, WA 99352  
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### Abstract:

The PHMC will provide tank wastes for final treatment by BNFL from Hanford's waste tanks. Concerns about the ability for "grab" sampling to provide large volumes of representative waste samples has led to the development of a nested, fixed-depth sampling system. Preferred concepts for filling sample containers that meet RCRA organic sample criteria were identified by a PHMC Decision Board. These systems will replace the needle based sampling "T" that is currently on the sampling system. This test plan document identifies cold tests with simulants that will demonstrate the preferred bottle filling concepts abilities to provide representative waste samples and will meet RCRA criteria. Additional tests are identified that evaluate the potential for cross-contamination between samples and the ability for the system to decontaminate surfaces which have contacted tank wastes. These tests will be performed with kaolin/water and sand/water slurry simulants in the test rig that was used by AEAT to complete Phase I tests in FY 1999.

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**PHASE II TEST PLAN  
FOR THE EVALUATION OF THE  
PERFORMANCE OF CONTAINER FILLING SYSTEMS**

**Prepared for  
Lockheed Martin Hanford Corporation  
Characterization Engineering Group  
Richland, Washington**

**by**

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**August 1999**

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**PHASE II TEST PLAN  
FOR THE EVALUATION OF THE PERFORMANCE OF CONTAINER FILLING  
SYSTEMS**

### 1.0 Introduction

A fluidic-based sampling system is being developed for sampling radioactive waste that is stored in large underground storage tanks. The final treatment of Hanford's waste will be completed through a vitrification contract with BNFL, Inc. (BNFL). Hanford will provide the low level waste (LLW) and high activity waste (HAW) for BNFL per specifications that have been negotiated in the BNFL vitrification contract. Prior to transferring a waste batch to BNFL, waste samples will be taken to conform the specifications of the waste that includes physical and chemical properties (BNFL, 1999a, and 1999b).

In general, the tank waste sampling method (core sampling or grab sampling method) will be established for each tank waste based upon waste characteristics and capacity of the sampling system. The base line method for sampling liquid wastes is "grab" sampling, that utilizes the "bottle on a string" technique. Major concerns with the performance of this base-line sampling method and the sampling required to support the BNFL contract have resulted in an activity to develop a sampling system (LMHC 1998 and Ritter 1999).

The sampling system will be a nested, fixed-depth sampling system, based on fluidic pumping concepts developed by AEA Technology Engineering Services, Inc. (AEAT). This system will sample wastes from multiple depths in a waste tank. In FY-1998, initial tests were undertaken that showed the feasibility of this fluidic sampling system that used a sampling needle and sampling "T" container filling station. In FY-1999, Phase I tests were completed (Reich 1999) to evaluate the performance of the sampling system and to demonstrate that:

- the fluidic pumping system can extract and pump material that is representative (physically and chemically) of the material input to the pumping system over the lift distances required for underground tank deployment (24-ft to 57-ft distance range).
- the sampling needle and "T" sample container interface can extract large sample volumes (500-mL containers) of materials that are representative (chemically and physically) of the material being pumped to the sampling station within a time frame that supports the sample information and waste sample volume needs of the privatization contract.
- the sampling system can recover from a plugged condition and resume normal sampling operations without dismantling any hardware.

The Phase I testing has been completed, and the data is currently being analyzed.

The U.S. Department of Energy (DOE) has requested that this sampling provide samples that can meet Resource Conservation Recovery Act (RCRA) criteria for materials containing volatile and semi-volatile organic constituents (Kinzer 1999). The RCRA criteria, as identified in SW-846, that must be satisfied by a container filling system include the following:

- introduction of liquids/solids into the sample container gently to reduce agitation that might drive off volatile compounds and might introduce bubbles into the sample materials.
- 100% sample container filling, so that when the container is capped, sealed, and is inverted, no headspace (air space) is visible.
- the sampling process should avoid vacuum processes that apply a vacuum to the waste that may result in the out-gassing of dissolved gasses or volatile organics.
- hermetically seal the container immediately after filling.
- use container materials, such as glass, Teflon<sup>†</sup> and metals, that will not add trace constituents, especially organics, to the material in the container.

Other criteria, that are not associated with the sample container materials and filling process, include limited hold time (14 days) and preservation of the sample material (cool to 4°C to preserve the volatile organic constituents).

Although some of these criteria have been waived for other sampling tasks at Hanford, the current assumption is that this sampling system (sampling system and sample container filling system) will have to satisfy all of these RCRA criteria (Morant, 1994 and Bowman, 1997).

## 2.0 Scope

This test plan provides guidance for the proof-of-principle testing of container filling concepts that will:

- interface with the fluidic pumping system of the nested, fixed-depth sampling system.
- provide waste samples that can meet RCRA criteria for samples with volatile and semi-volatile organic constituents.

Potential container filling concepts were identified and compared in an Alternatives Generation and Analysis (AGA) study (Reich 1999b). A Decision Board made up of members of the PHMC Team (in FY 2000 to be known as the LMHC Team due to changing the Tank Waste Remediation Systems to River Protection Program) used the decision process outlined in the AGA to select two preferred container filling options. Input was solicited and received from AEAT. The selection was based on container filling criteria that were based on the BNFL privatization contract (BNF 1999a, and BNFL 1999b); the Level 2 Component Specification (Reich 1999c), and from the RCRA criteria for samples containing volatile and semi-volatile organic constituents (SW-846).

Teflon is a trademark of E.I. du Pont de Nemours & Co.

*KN*

The AGA and Decision Board process was initiated after it was determined that the current needle and "T" container filling system would not meet the RCRA criteria (Reich 1999b).

The performance of the sampling systems fluidic pumping capabilities and the sampling performance of a needle and "T" based container filling system were addressed in the Phase I testing that was completed by AEAT in FY-1999 (Reich 1999a). The tests outlined in this document for the container filling systems assume the following:

- the container filling system will interface with the fluidic pumping system in the nested, fixed-depth sampling system.
- the fluidic pumping system is capable of pumping materials to the container filling station that are physically and chemically representative of the materials at the inlets of the pumping system.
- the fluidic pumping system meets RCRA organic sample criteria so that the material input to the container filling station contains representative volatile and semi-volatile organic constituents.
- the fluidic delivery from the pumping system is in pulses.
- the volume of a single pump pulse is sufficient to fill a 500-mL container.

The container filling tests will use the optimized fluidic pumping system and operating parameters that were identified and verified in the Phase I testing, including the fluidic pumping system components and the supply tank with mixing pumps. It is anticipated that the container filling tests would use this same test rig except the full height (57) ft will not be required. The container filling system construction would also use representative materials, except where it is necessary to observe the fluidic action of the simulants in the container filling system (for example, metal valves and piping and a plexiglass container filling station).

### 3.0 Test Background and Guidance

The previous test plan (Reich 1999a) and the Level 2 Specification (Reich 1999c) included a description of the basic principles and components for the current sampling system concept being developed by AEAT.

#### 3.1 Conceptual Test System

The Phase I test rig was constructed with representative materials, including dimensions, piping runs, surface finishes, and bend radii, that are expected to be used in the prototype sampling system. Modular piping and a portable sampling "T" and jet-pump allowed testing at 24-ft and 57-ft heights. It is anticipated that this new test rig will use the test hardware from the Phase I testing, or similar hardware, and will be operated using the optimized parameters that were identified in the Phase I testing. Features of the Phase I test rig were described in the AEAT Phase I test implementation plan (*Implementation Plan for Proof of Principle Testing Supporting Design of a Prototype Nested Fixed Depth*

*Fluidic Sampler*, TFA/PF/28v1, April 1999, AEA Technology Engineering Services, Inc., Huntersville, NC) with other system data found in the Level 2 Specification (Reich 1999c) and in the previous test plan (Reich 1999a).

The bottle filling test rig shall be setup with a 24-ft high lift (24-ft between the reverse flow diverter (RFD) inlet and top of the sample container). The container filling stations, that are described in Appendix A, will be used in place of the sampling "T" that was evaluated in the Phase I testing (Reich 1999b).

The test setup shall be constructed and operated so that features impacting the ability to meet or perform the required sampling criteria, including the RCRA sampling criteria for volatile and semi-volatile organics, can be demonstrated and evaluated. The container filling stations will be complete to the extent that it can functionally demonstrate or perform the following functions:

- interface with the fluidic pumping system (a single sampling channel is adequate) and extract representative samples from it.
- fill 500-ml sample containers.
- fill a sample container without injecting air bubbles into the sample material.
- uncap, fill, and cap a filled sample container so that it is sealed at the end of the sampling operation. The method in which these operations are completed will depend on the type of filling system that is being tested; single-station or two-station filling systems.

The fluidic pump will be operated with the optimized operational parameters (drive and fill times and pressures, jet-pump pressure and flow, etc.) that were identified and validated in the Phase I testing. These include:

- a delivery time of approximately 8 to 10 seconds.
- a minimum delivery volume of 3 liters.

If other parameters are used in the sampling system (setup and operational), test data shall be obtained and analyzed to show that the performance of the sampling system is providing representative materials to the container filling station.

The container filling system shall be constructed of both metallic and semi-transparent components. These components shall have features (size, shape, and function) of a system that would be capable of being used in a full-scale, prototype sampling system. Metal valves and piping shall be used. The sampling system reservoir shall be constructed of clear materials (plexiglass\* etc.) in order to allow observation of the fluidic action during operation. Clear glass sample containers (dimensions the same as the amber 500-ml sample bottles) shall be used in some of the tests described below so that the status/condition of the simulant (air bubbles, froth, etc.) can be observed and photographed through the container wall.

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*KN*

Bottle handling operations, such as bottle insertion and removal, can be manual operations. However, all features of the filling concepts that are critical for meeting the above requirements shall be provided for in the test rig. The sample containers can be manually inserted into the filling station and all functions needed to position or hold the bottle in the station can be manual based operations. The uncapping/capping operations can be manually operations, provided the manipulators or end effectors that interface with the sample containers are the same that are envisioned to be used in the prototype filling station (Note: This will depend on the type of filling system being tested.).

For a two station filling system, where an open container is moved between stations, an option (if it can be economically completed) is to include a mockup of the mechanical hardware that will transfer a container between a filling station and a uncapping/capping station. This would provide data on the ability to move a "full" uncapped bottle and provide data on the level of contamination from spillage that might be expected with a two station container filling concept.

#### 4.0 Analysis and Measurement Methods/Techniques

##### 4.1 Measurement Methods

This testing will use the data acquisition, measurement, and analysis methods that were developed and used in the Phase I testing (*Implementation Plan for Proof of Principle Testing Supporting Design of a Prototype Nested Fixed Depth Fluidic Sampler*, TFA/PF/28v1, April 1999, AEA Technology Engineering Services, Inc., Huntersville, NC). Any deviations from these methods must be approved by the PHMC Team. The following definitions apply:

Particle size distribution -	This will be measured by the sieve based particle size analysis method used by AEAT in the Phase I test analysis and based on ASTM C92-95. (See also ASTM E276-93 and ASTM D4513-97 for additional information on sieve based particle sizing. ASTM E1638-94 also provides a summary of terminology for sieve based analysis.)
Solids wt% content -	This will be measured by the weighing and evaporation based method used by AEAT in the Phase I test analysis (at 105 °C per EPA 160.3) for total solids content measurements.
Viscosity -	Viscosity will be measured using rotating bobbin and cup apparatus. Contraves, Haake, or Brookfield apparatus may be used, depending upon shear range.

All measurements shall be made using calibrated sensors and instrumentation. The measurement of "blanks", or standards, shall be used to establish measurement

performance before and after completing the measurement campaign. The calibration data for the sensor and/or measurement systems shall be included with the measurement data. If calibration data or performance data is not available, then additional testing shall be completed that establishes the performance of the measurement/sensor system. The performance data established in the previous Phase I testing for the various measurement methods can be used. However, if procedures or equipment are changed, the calibration shall be redone and new performance data obtained. This additional testing shall be completed in a manner such that the resulting data satisfies accuracy rule-of-thumb criteria for measurement/sensor systems where the accuracy of the measurement/sensor system shall be at least an order of magnitude better than the accuracy that is required for the measurement data being produced by the system. In addition, the sensor/measurement systems shall be selected such that the expected or anticipated measurement value will fall within the central 75% of the sensor or measurement systems sensing/readout range.

#### 4.2 Validation of Test Simulant Properties

The properties of the simulants will be measured (characterized) and validated for each of the system tests using two independent methods. This reference data shall be obtained by:

- Accurate measurement (to 0.1 percent) of the simulant make-up constituents (volume or weight of each constituent, particulate distributions, etc.). This simulant makeup data will be used to assess the potential presence and impact of other, non-sampling system error sources such as incomplete mixing in the simulant vessels. The simulant makeup and the reference sample data sets will provide validation data that the test simulant properties are in conformance with the simulant specifications.
- “Grab” sampling at the inlet of the sampling channel during operation of the sampling system. These reference samples shall be taken very close to the inlet of the RFD and within the same time interval that a bottle sampling is being taken in the sampling station. The sampling shall be completed in a manner that minimizes the impacts of system induced errors, such as a lack of homogenous mixing in the vessels holding the simulant.

The performance of the container filling station will be assessed by comparing the bottle samples with the grab reference samples. The same analyses methods and techniques will be used for all samples and will be the methods developed and validated in the Phase I testing completed by AEAT. The required measurements in this test plan specifically identify particulate size distribution and wt% solids as the two main parameters for indicating the performance of the sampling system. Other measurements may be necessary to ensure that a simulant meets specifications.

Samples of the pumping system effluent shall also be taken at the same time sample containers are filled. The need to analyze these samples will depend on the results of the Phase I testing to characterize the RFD based pumping system. However, if the sampling

system or its operation is different from that of the Phase I testing, then these "grab" samples shall be analyzed to characterize the RFD based pumping system performance.

#### 4.3 Reference Material Sampling

Reference samples, will be obtained using a grab sampling process. The ASTM standard, *Standard Practice for Sampling Industrial Chemicals (ASTM E300-92)*, provides guidance for this grab sampling, that is the current baseline method for obtaining liquid and slurry tank waste samples. Care and diligence should be used in the acquisition of the grab samples. Section 10.0 of the ASTM standard covers the sampling of "Simple Liquids" while Section 35.0 covers "Slurry Sampling." The practices, procedures, limitations and cautions, etc., described in these sections should be used as guidance in the planning and execution of grab sampling. The grab sampling should use a 100-500-ml size sample bottle/flask with other recommendations and physical characteristics as described in ASTM E300-92 (mouth size, total sample volume, etc.). The grab sampling shall be carefully done and timed so that any simulant content biases from the operation of the sampling (such as the pulsed pumping action of the pumping system) does not affect sample content.

#### 4.4 Measurement Statistics and Accuracy/Precision

The performance of the sampling system will be assessed by comparing the sample materials with that from the grab samples. The reference materials for this performance assessment will be the material samples obtained directly adjacent to the sampling channel inlet. In assessing performance, following definitions, from the previous test plan, will apply:

- **Sensitivity:** This is the relationship between an instrument's output and the parameter it is responding to. It is used to specify the minimum detectable change in a parameter that the instrument will respond to (most often this is the signal-to-noise ratio of the instrument).
- **Accuracy:** This is an indication of how close a measured value (or a group of measured values) is to the "true" value or an accepted standard. How close together the group of measurements are with respect to each other is not an issue. With a group of repeated measurements, there is high accuracy if the mean value of the measurement group is very close to the "true" value. However, there can be a significant span (see definition below) between the individual measurement values.
- **Precision:** This is an indication of how repeatable a measurement is (the spread of the grouping of repeated measurements). Precision is different from accuracy in that it is defined without reference to what the "true" value or accepted standard value may be. For a group of repeated measurements, if the span of the measured values is relatively small (tightly grouped measurements), the measurements would have high precision. If there is a wide span between the values, the precision will be low. Precision can be expressed in a number of ways. One of the most common is the Standard Deviation from the mean value.

- **Range:** This is defined as the interval over which a parameter is measured--the lowest and highest values of the parameter.
- **95% Confidence Interval:** This is the measurement interval (or delta on either side of the sample mean) within which we will find 95% of the members of a group of samples. In other terms, this is the interval around the sample mean in which there is a 95% probability that a member of a sample population will be found.

When comparing statistically the mean values of two sets of measurements, such as the grab samples with the bottle samples, there are three factors that impact the comparison:

- the standard deviation of the two populations,
- the number of samples in each population, and
- the confidence that we wish to make our comparison with.

The Student's t-distribution shall be used to identify the number of samples that will be acquired for each measurement. For example, if we have two sample populations that each have a standard deviation of  $\pm 5\%$  of their means, then statistically these mean values can be considered to be indistinguishable, within a 95% confidence level, if they are within  $\pm 5\%$  of each other. The Student's t-distribution requires that each population include at least 15 independent samples/measurements to support this statistical comparison. The Student's t-distribution indicates that fewer samples are required for smaller standard deviations. However, in each test outlined below, an adequate number of samples should be taken based on an anticipated maximum estimate of what the sample population's standard deviation. It may not be necessary to measure all samples, based on the outcome of the sample population's standard deviation, when the samples are analyzed.

## 5.0 Container Filling System Tests

This section describes tests, test setups, simulants, measurements, and performance criteria that will be used to verify the performance of the container filling system.

### 5.1 RCRA Compliant Sampling

This test will demonstrate and verify that the container filling system can fill sample containers that are capable of meeting the volatile and semi-volatile RCRA criteria that are defined in SW-846.

#### 5.1.1 Test Objective

The objective is to complete tests that demonstrates or verifies that the samples filled by the container filling system can meet RCRA criteria, as defined in SW-846, for sample materials containing volatile or semi-volatile organic constituents. The RCRA criteria include:

- No visible head-space (100% fill) in a filled, sealed container.

- No air bubbles in the sampled material.
- A fully sealed sample container after the filling operation is completed.

### 5.1.2 Test Setup

The fluidic pumping system for these tests shall be the Phase I test rig, setup as a 24 ft fluidic pumping system as described in Section 3.1. The fluidic pump system will be operated with the optimized operating parameters (drive and fill times and pressures, jet-pump pressure and flow, etc.) that were identified and validated in the Phase I testing.

The valve and lead-in piping to the sample filling chamber shall be constructed of similar components and materials anticipated in a prototype container filling system. The reservoir of the container filling system can be constructed of transparent components, as indicated in Section 3.1, to allow visual observation and photographic recording of the fluidic action.

The test rig will be used with both the single-station and two-station filling systems described in Appendix A. The bottle handling operations, such as bottle insertion and removal, can be manual operations for a single station filling system as per Section 3.1. The uncapping/capping operations can be manually completed provided the manipulators or end effector design that are envisioned for the prototype sampling system are used. The single-station option will use a single station for uncapping, filling, and capping. The two-station filling system shall be setup with two separate stations; filling station and uncapping/capping station. The two-station system may also include a mockup of the container transfer manipulator that will move the container between the two stations. This can be manually operated provided the end-effectors, anticipated for a prototype system, are used. The transfer mockup will allow the simulation of the expected motion and hand-off between stations, generating data on the expected level of spillage and subsequent contamination that may be expected.

To baseline the performance of filled sample container closures, reference samples shall be obtained by manually filling and capping sample containers with simulant. These filled containers shall also comply with the RCRA criteria. To provide a baseline for bubbling or frothing and head space evaluations, some of the reference samples shall contain a small void space.

### 5.1.3 Test Simulants

The performance of the container filling system will be tested and evaluated with two simulants:

- Kaolin/water slurry with:
  - 25 to 35 wt% solids content
  - no dispersant to lower the specific gravity, as this has shown it makes the simulant overly sticky
- Sand/water slurry with:

- 10 to 15 wt% sand content
- modified particulate distribution as identified in HNF-3402, Rev. 1

These two simulants cover the range of waste viscosity expected with the tank waste. The high viscosity of the kaolin/clay will provide a better indication of bubbling/frothing in the container and issues related to material flow. The low viscosity of the water/sand slurry will provide a better means to evaluate the potential for container leakage after being capped/sealed and the potential to assess impacts of particle settling. In order to enhance the ability to assess the sealed status of a closed/capped container, a dye (fluorescent or plain coloring agent) may be added to the simulants. The dye concentration should be low enough that there is no impact on the physical properties of the simulants.

In order to visually observe the materials in the samples through the container walls, clear glass sample containers should be used that have the same physical dimensions as that of the amber containers that are anticipated to be used in a hot tank deployment with the sampling system. For each of the container filling systems and simulants being evaluated, waste samples and reference samples will be obtained. The total samples that shall be taken during this test is:

- kaolin/water simulant:
  - 8 single-station samples
  - 18 two-station samples
  - 18 reference samples
- sand/water simulant
  - 18 single-station samples
  - 18 two-station samples
  - 18 reference samples

#### 5.1.4 Test Measurements

The sample containers will be filled, capped, and sealed in the filling stations. For the kaolin/water simulant samples:

- Immediately after filling and capping/sealing kaolin/water samples, wipe the excess simulant from the container surfaces, immediately visually examine the container for the presence of bubbles and record with photographs. Invert the container and visually examine for the presence of a head space (void) in the bottle. Estimate and record the approximate bubble size range and the void space. Photograph through the container wall for a permanent record.
- Let the sample bottles set in an inverted position for 24 to 48 hours (hold the simulant/container temperature at approximately the same temperature as when sampled) and again visually examine for the presence of a head space (void) in the bottle. Record the approximate size of the void space and photograph it through the container wall for a permanent record.

For the sand/water slurry samples:

- Immediately after filling and capping/sealing the sand/water samples, wipe dry the outer surfaces of the filled containers and visually examine for the presence of bubbles in the simulant and the presence of a head space (void) in the bottle. Estimate and record the approximate void space and bubble size range and photograph for a permanent record.
- Shake the containers vigorously for 5 to 10 minutes and immediately visually inspect the container seal areas for the presence of liquid. If leakage results, locate the leak position, estimate the leaked quantity and record. Record the leak area with a photograph. If a fluorescent dye is used, examine the exterior of the sealed container with fluorescent readout methods. Wiping and shaking should be repeated to verify the leakage. Wiping the outer surface of the sealed container and examining the wipe may also be used as a method of sensing leakage.

To baseline the performance of the filling system and sample containers, the above operations will be completed with the manually filled sample containers and record the results.

The above analysis shall be completed with all of the samples identified in Section 5.1.3.

#### 5.1.5 Performance Criteria

The sampling station and its filled sample containers shall meet the RCRA sample criteria for materials that contain volatile organic constituents as per SW-846. The samples must have the following:

- No visible head space, with 100 % of the container filled with simulant, when inverted
- No visible air bubbles or waste froth in simulant material
- No leakage of simulant from the closure seals on a filled container

The manually filled/sealed reference samples, undergoing similar manipulations, shall be used as the reference to assess the performance of the sample containers reference samples and to baseline the simulants in the sample containers. The container filling system will be considered RCRA compliant if it passes visual examination/tests for these criteria.

#### 5.2 Representative Waste Sampling

The sample material shall be physically and chemically representative of the tank waste material that is being sampled. The container filling system shall extract and fill containers with sample material that is representative (particle distribution and total solids content) of the sample materials that the fluidic sampling system is delivering to the container filling system.

### 5.2.1 Test Objective

The objective is to complete testing that verifies the ability of the container filling system to fill containers with representative sample materials that the RFD fluidic pumping system is providing to the container filling station. A comparison of the sample particle size distribution and total solids content with that of the material being pumped to the filling station shall be used to verify that the samples are representative.

### 5.2.2 Test Setup

The test setup will be as per the description in Sections 3.1 and 5.1.2, including described component/system materials, dimensions, and shapes. The test setup will include all hardware components that are part of the sample extraction, container filling, and uncapping/capping processes for each container filling system. The test rig will be used with both the single-station and two-station filling systems that are described in Appendix A. It is critical that all components have the dimensions, shapes, and surface features that will be used in the prototype sampling system. Remote manipulation of the container and the container filling hardware is not required for this test. The inclusion of the capping/sealing operation should not impact the representative properties of an extracted sample.

### 5.2.3 Test Simulants

The representative sampling testing will be completed with two simulants:

- Kaolin/water slurry with:
  - 25 to 35 wt% solids content
  - no dispersant to lower the specific gravity, as this has been shown to make the simulant overly sticky
- Sand/water slurry with:
  - 10-15 wt% sand content
  - modified particulate distribution as identified in HNF-3402, Rev. 1

The low viscosity sand/water slurry will test the ability for the sampling station to handle materials that are very prone to settling. The high viscosity kaolin/water will verify representative sample acquisition with materials that are not prone to settling but other physical properties that make sample extraction and container filling difficult.

### 5.2.4 Test Measurements

Waste samples will be obtained at three points in the test setup:

- sample containers will be filled, capped, and sealed with the sample container filling system
- grab samples will be obtained adjacent to the RFD inlet of the pumping channel.

- in addition, grab samples will be obtained from the waste pumped past the sampling station and being return to the waste supply vessel.

Although the analysis of the performance of the RFD fluidic pumping system was part of the previous Phase I testing completed in FY-1999, the results were not currently available. Therefore, it is necessary to provide samples of the material that the RFD fluidic pumping system is providing to the container filling station. If the Phase I results become available, it may not be necessary to analyze these samples. However, a conservative path will be taken and these samples shall be taken at the same time as sample containers are filled.

Standard grab sampling methods will be used to obtain reference samples adjacent to the RFD inlet and from the simulant material being pumped to the sample filling station (sample the simulant effluent being drained back into the supply tank).

Adequate sample numbers shall be obtained during testing to satisfy the Students-t criteria for a 95% confidence level as indicated in Section 4.4. The number of sample below is a recommended minimum of the number of samples to be taken:

- kaolin/water simulant:
  - single-station system samples
    - 18 filled container samples
    - 18 RFD inlet samples
    - 18 RFD fluidic pump effluent samples
  - two-station system samples
    - 18 filled container samples
    - 18 RFD inlet samples
    - 18 RFD fluidic pump effluent sample
- sand/water simulant:
  - single-station system samples
    - 18 filled container samples
    - 18 RFD inlet samples
    - 18 RFD fluidic pump effluent samples
  - two-station system samples
    - 18 filled container samples
    - 18 RFD inlet samples
    - 18 RFD fluidic pump effluent sample

As indicated in Section 4.1, the methods used in the acquisition and analysis of samples for the Phase I testing shall be used. All samples will be analyzed for:

- total solids content
- particle distribution

The number of samples analyzed will depend on the standard deviation of each sample population as per the Student's-t distribution. The performance objective of the sampling system is to provide samples that have physical properties that are within  $\pm 5.0\%$  of the tank waste, with a 95 percent confidence level. The Student's t-distribution will be used to identify the number samples that are needed to meet this precision and confidence level criteria. Section 4.4 indicates that if the sample populations have standard deviations that are  $\pm 5\%$  of their mean values, then at least 15 samples are required to satisfy the 95% confidence level in determining if the mean values are within  $\pm 5\%$  of each other. However, all do not need to be analyzed if the standard deviation of a population is smaller than  $\pm 5\%$ . However, adequate sample numbers must be taken during the testing to cover uncertainties about the standard deviations of the sample populations and to assure that the Student's-t distribution criteria are met for the worst case expected standard deviations within each sample group.

Sample analysis will be completed by comparing the contents of the bottle samples with the grab samples (RFD inlet and fluidic pump effluent). For the sand/water simulant samples:

- Compare particle distributions using the graded sieve technique
- Dry the samples and compare the weight of the total solids content

For the kaolin/water simulant samples:

- Dry the samples and compare the weight of the total solids content with the reference samples.

#### 5.2.5 Performance Criteria

The container filling station will be considered able to provide representative waste samples if:

- The mean values of the total solids content of the samples are within  $\pm 5\%$  of the grab sample mean values, within a 95% confidence level, and the standard deviation of the samples is approximately equal to that of the grab samples standard deviation (within  $\pm 25\%$ ).
- The particle distributions of the samples are within  $\pm 5\%$  of the particle distributions of the grab samples with a 95% confidence level and the standard deviations in each particle grouping are approximately equal to that of the grab samples standard deviation (within  $\pm 25\%$ ).

### 5.3 Container Filling System Cross-Contamination

A single sampling container filling system will be used to obtain waste samples from a number of depths in the waste tank. It is important that the cross-contamination between samples be minimized in order to have samples that are representative of the waste in the tank. The sampling must be completed with a minimum of cross-contamination between samples where the sources of cross-contamination include waste materials from previous samples and flush water from flushing and decontamination operations.

#### 5.3.1 Test Objective

This test will analyze the flushing (water and waste) that is required for producing filled sample containers with acceptable cross-contamination levels. The testing will quantify the potential level of material cross-contamination in filled sample containers and will validate/verify the waste and water flushing required to maintain this level of cross-contamination.

#### 5.3.2 Test Setup

The test setup will be as per the descriptions in Section 5.1 and 5.2 where the simulants are kaolin/water and sand/water materials. The test setup will include all hardware components that are part of the sample extraction and container filling process for a filling system. It is critical that these components have the dimensions, shapes, and surface features that will be used in the prototype sampling system. The inclusion of the capping/sealing operation should not impact the representative properties of an extracted sample and should be considered an optional test system feature.

For this test two different setups can be used:

- a two channel sampling system with each channel having its own supply vessel and simulant. This will provide cross-contamination data for only the container filling station.
- A single pump channel setup with a single supply vessel where the simulants can be quickly changed. This test setup will require purging of the pumping system to remove its potential impact on sample cross-contamination. This setup will also allow the potential for cross-contamination from the sampling system to be evaluated.

The supply vessel volume must be large enough such that the residue coming from the simulant being pumped past the sampling station is not a factor in the cross-contamination measurements and the removal of supply vessel material via sampling has a negligible impact on the simulant. The fluidic pump system will be operated using the optimized operating identified and verified in the Phase I testing completed by AEAT in FY-1999. These operating parameters will provide the fluidic conditions in the test rig that are expected to be experienced in the prototype, field deployed sampling system.

The test setup shall be modified with a sampling port (valve) between the sampling station and the container filling station. This port will be used to obtain reference samples of the materials being pumped to the sampling station. The materials in these samples will be used to assess cross-contamination from the container filling station to be assessed independently from the cross-contamination caused by the RFD pumping system.

### 5.3.3 Test Simulants

The cross-contamination tests will be completed using two simulants:

- Kaolin/water slurry with:
  - 25 to 35 wt% solids content
  - no dispersant to lower the specific gravity, as this has shown makes the simulant overly sticky
- Sand/water slurry with:
  - 10 to 15 wt% sand content
  - modified particulate distribution as identified in HNF-3402, Rev. 1

These simulants cover the range of waste viscosity and potential for coating and settling with the sampling system hardware. The kaolin/clay will provide a better indication of cross-contamination from adherence to surfaces while the sand/water simulant will provide a better indication of cross-contamination from settling of solids within the container filling and sampling systems.

To help detect cross-contamination, a dye may be added to the simulants. The dye concentration should be low enough that there is no impact on the physical properties of the simulants.

### 5.3.4 Test Measurements

Cross-contamination levels will be determined by measuring the amount of kaolin or sand residue (the dye is expected to be a qualitative indicator) that is in the system after flushing with water. The kaolin/water simulant and the sand/water simulant will be used to setup (fully coat all surfaces) the sampling system. A typical test procedure will be:

- Pump the simulant (kaolin/water or sand/water) through the sampling station until a steady-state condition is reached.
- Fill a sample container to assure that the system is at steady-state and all surfaces are coated with simulant.
- Start the system flush with water. Pre-measure the flow rate from the flushing system.
- Periodically “grab” sample the flush water passing through the system. The residue within the grab sample is an indication of the cross-contamination.
- Take samples at the onset of flushing and then at pre-determined time intervals to provide a time dependent cross-contamination measurement.

- Continue flushing and sampling until the grab sample is clear of simulant (kaolin, sand, or dye).

Visually examine the filling system hardware for the presence of kaolin and sand residue and record the presence or lack of these materials with photography.

This cross-contamination test may be combined with either the representative sampling test or the RCRA tests.

Complete this testing with the single-station and two-station container filling systems. Although the systems are fundamentally identical in design, the repeat testing will be used to confirm the results of the cross-contamination levels.

### 5.3.5 Performance Criteria

Performance shall be assessed by measuring the solids content of the grab samples (drying and weighing). Plot the solids content (wt%) as a function of flush volume (time multiplied by flow rate). The system shall have an acceptable level of cross-contamination when:

- the mean values of the total solids content of the bottle and grab samples are less than 1% of the initial solids content of the simulant that was previously pumped through the sampling system, within a 95% confidence level.

## 5.4 Container and Filling Station Hardware Decontamination

The hardware in the container filling station and some areas of the outer surfaces of the sample container will contact tank waste during the filling and capping operations. To support ALARA (as low as reasonably achievable) and to limit the spread of contamination, these areas will be flushed with water (Reich 1999c). The container must be decontaminated in the filling chamber before it is moved into the buffer chamber in order to control the spread of contamination

### 5.4.1 Test Objective

This test evaluates the effectiveness of the water sprays, that will be incorporated into the container filling (and potentially capping) station, in cleaning waste from the filling system and sample container surfaces. The testing will also identify additional hardware, processes, and cleaning needs that may be required to limit the spread of contamination in the sampling system. The objective is to complete tests with water spray systems that clean these waste contaminated surfaces and to assess the effectiveness of cleaning.

#### 5.4.2 Test Setup

The test setup will be as per the description in Section 3.1 and for the tests in Sections 5.1, 5.2, and 5.3. The test setup shall include all of the container filling station hardware components that are anticipated to make contact with the tank waste during a sampling operation. It is critical that the sample bottle and the sample bottle filling station's components have dimensions, shapes, and surface features as close as possible to the prototype sampling system in order for this testing to be valid. Spray nozzles will be setup to clean residual waste from these areas. The testing will not address the RFD pump piping or the container filling chamber areas. These are addressed in the above test (Section 5.3).

The spray nozzles used in this testing shall be designed also for use in the prototype sampling system. The test setup will include pressure and flow meters to record flushing water flow and pressures used in the testing. In addition, the dimensions of the spray patterns will also be recorded (photography is recommended). All hardware that makes contact with the tank waste will be included in this test, including the moveable drain that set under the container filling system reservoir.

#### 5.4.3 Test Simulant

The surface decontamination features of the sample container filling station will be tested using:

- Contaminating materials:
  - Kaolin/water slurry with:
    - 25 to 35 wt% solids content
    - no dispersant to lower the specific gravity, as this has shown makes the simulant overly sticky
  - Sand/water slurry with:
    - 10-15 wt% sand content
    - modified particulate distribution as identified in HNF-3402, Rev. 1
  - Tap water flushing to flush contaminated surfaces

A dye can also be added to the "contaminating" simulants to help identify residual materials on the container and on the container filling hardware.

#### 5.4.4 Test Measurements

The testing will use the simulants to "contaminate" the sampling system components and then use water flushing nozzles to flush contamination from these surfaces. Visual analysis with photography will be used to assess the performance of the flushing. A typical test procedure will be:

- after filling the sampling system with a simulant, circulate/pump until a steady state condition is reached.

- complete all filling and capping/sealing operations for two sample containers. The first container will be used as the reference to assess effectiveness of flushing.
- visually inspect the surfaces of the sample container and filling system for simulant materials and record with photography.
- for the second sample, activate the water spray systems to flush residual material from the container.
- activate water spray nozzles and flush the contaminated surfaces of the container filling hardware.
- visually inspect the sample surfaces and record/photograph the flushed areas
- repeat the filling/flushing cycle.

The use of swabs to collect and assess residual contamination should be considered. If a dye is used, the color of the dye and swab should be chosen to maximize contrast and enhance visual detection.

Test data will be recorded and test setups documented to provide input data for the prototype container filling system. The reference samples will be used to identify the container areas where contamination is expected to accumulate. The effectiveness of the decontamination will be assessed by comparing the water flushed containers with the reference containers. Other information to be recorded includes:

- configuration of spray nozzles
- nozzle spray patterns
- areas where contamination will collect/reside
- results of water flushing

#### 5.4.5 Performance Criteria

None.

#### 6.0 Reporting

The results of this testing will be documented in a written report. This report will include test objectives, a description of the test setups, test measurement and calibration data, laboratory measurement and calibration data, and a brief analysis of the data from each test that includes an assessment of how well its performance criteria were met. The report that AEAT sends to the PHMC Team will be a formal document that can be submitted for public release by the PHMC.

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*Fluidic Sampling and At-Tank Analysis Systems*, Lockheed Martin Hanford Corporation, Richland, Washington.

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## **APPENDIX A**

### **PREFERRED CONTAINER FILLING OPTIONS**

## APPENDIX A

### PREFERRED CONTAINER FILLING OPTIONS

#### A.1.0 Preferred Option

The single-station and two-station concepts that are to be tested are shown in Figures 1 to 6 below. These concepts were selected as the preferred container filling concepts by the PHMC Team Decision Board (Reich 1999a). These container filling stations will be used in place of the sampling "T" that was evaluated in the Phase I testing that was completed by AEAT in FY 1999.

In the testing, the hardware for the container filling stations will be complete to the extent that it can functionally demonstrate or perform the following functions:

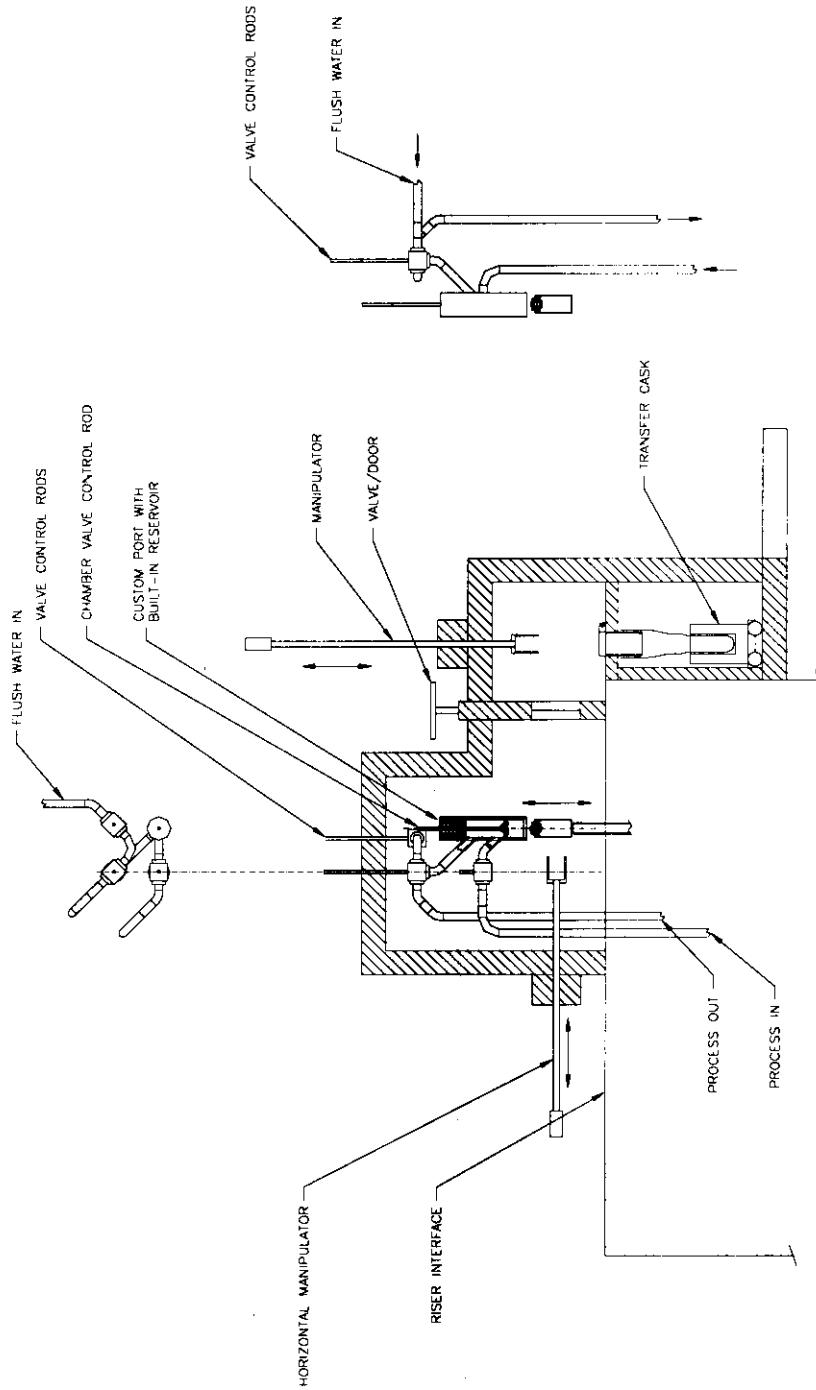
- interface with the fluidic pumping system (a single sampling channel is adequate) and extract representative samples from it.
- fill 500-ml sample containers.
- fill a sample container without injecting air bubbles into the sample material.
- uncap, fill, and cap a filled sample container so that it is sealed at the end of the sampling operation. The method in which these operations are completed will depend on the type of filling system that is being tested; single-station or two-station filling system

The uncapping/capping operations can be manually completed provided the manipulators or end effector designs that are envisioned for the prototype sampling system are used. The single-station option will use a single station for uncapping, filling, and capping. The two-station filling system shall be setup with two separate stations; a filling station and an uncapping/capping station. The two-station system may also include a mockup of the container transfer manipulator that will move the container between the two stations. This can be manually operated provided the end-effectors, anticipated for a prototype system, are used. The transfer mockup will allow the simulation of the expected motion and hand-off between stations, generating data on the expected level of spillage and subsequent contamination that may be expected.

#### A.2.0 References

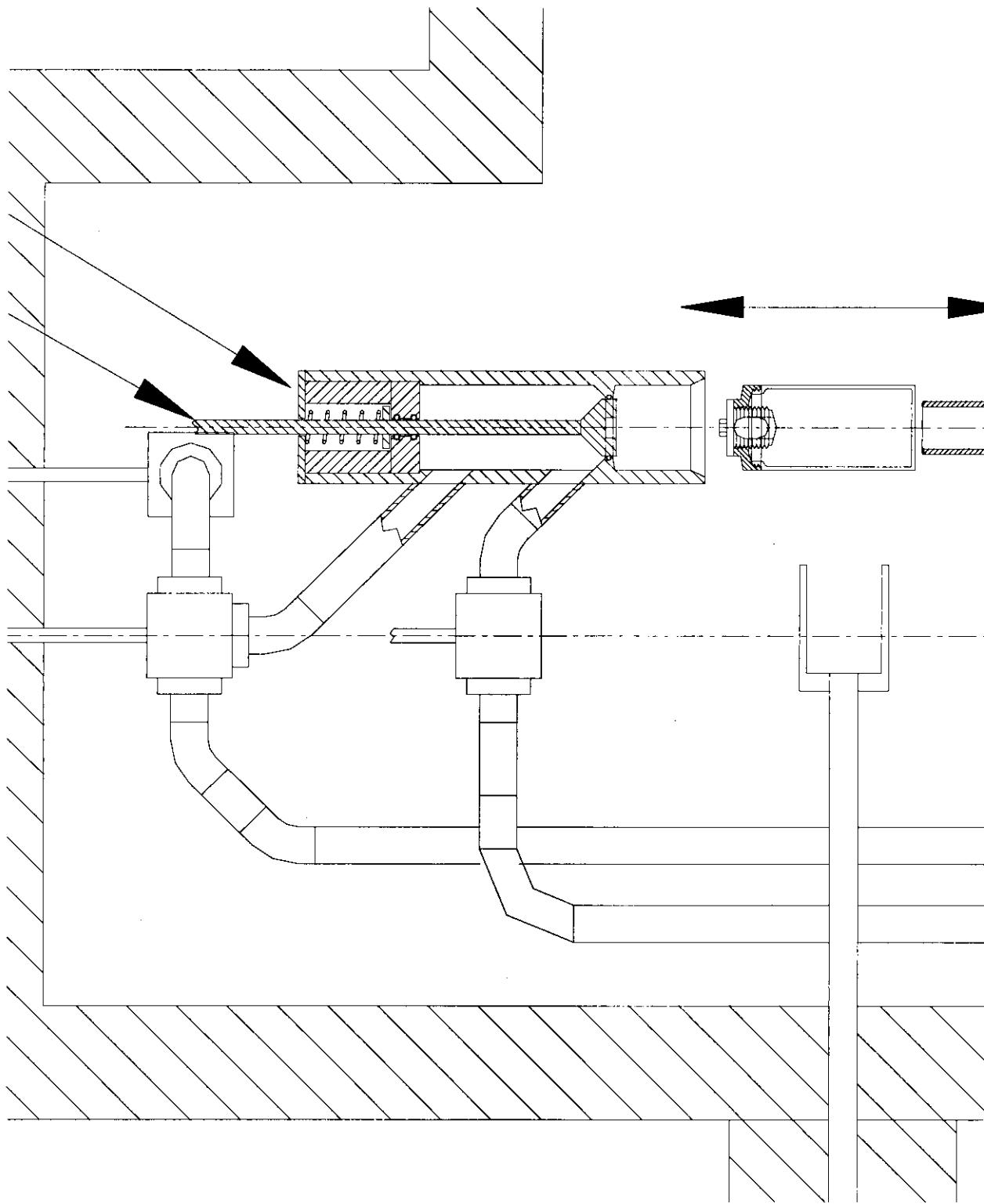
Reich, F. R., 1999, HNF-4545. Rev. 0, *Alternative Generation and Analysis Study for a Waste Sample Container Filling System for the Nested, Fixed-Depth Sampling System*, 1999, Lockheed Martin Corporation, Richland, Washington.

**Figure A- 1 General arrangement of bottle filling system Option 4, showing interfaces to the sampling system waste stream.**



OPTION 4: SINGLE-STATION W/CUSTOM PORT & BUILT-IN RESERVOIR (2.2.2-B)

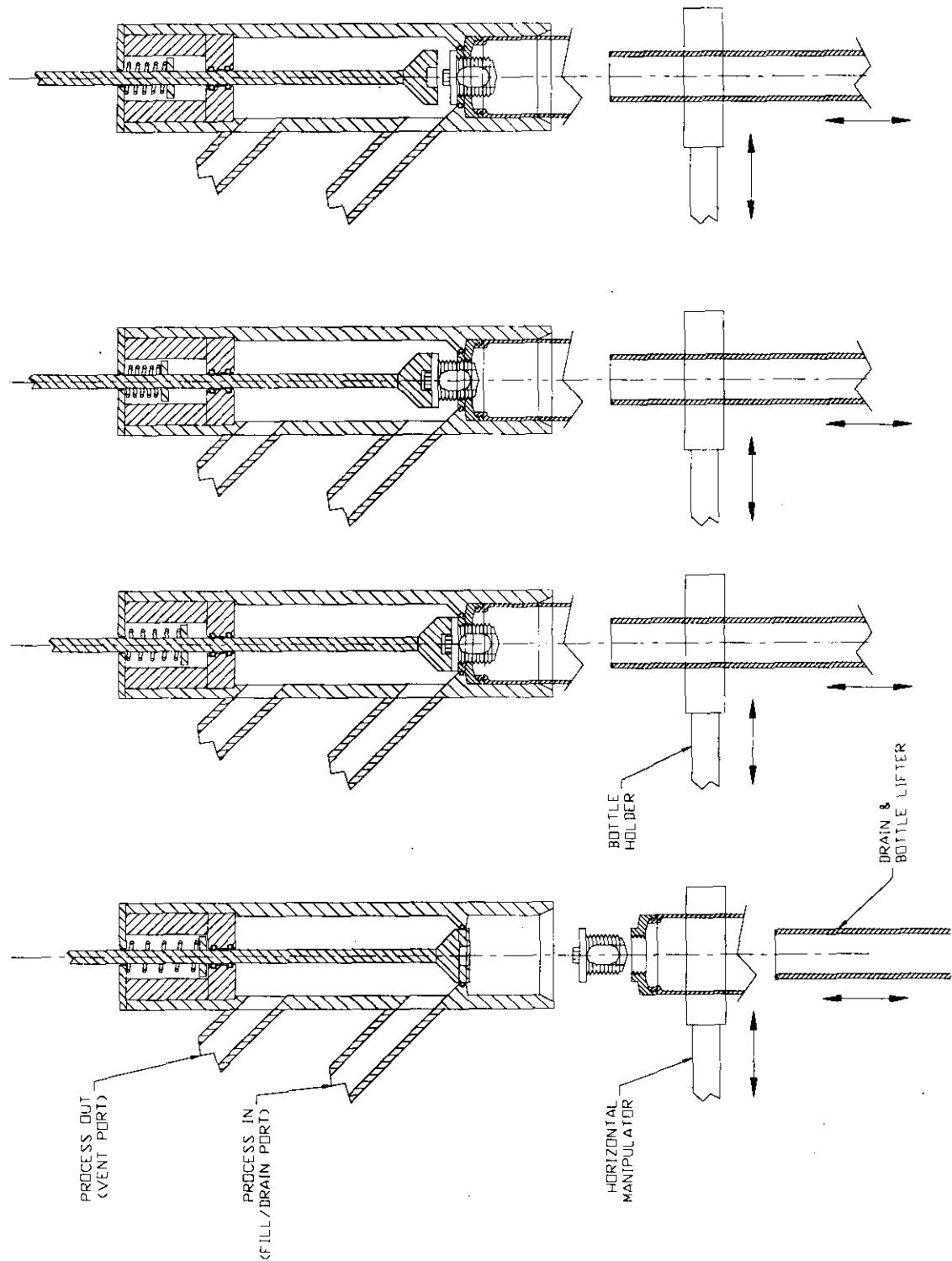
**Figure A- 2 Zoomed view of container filing chamber for Option 4.**



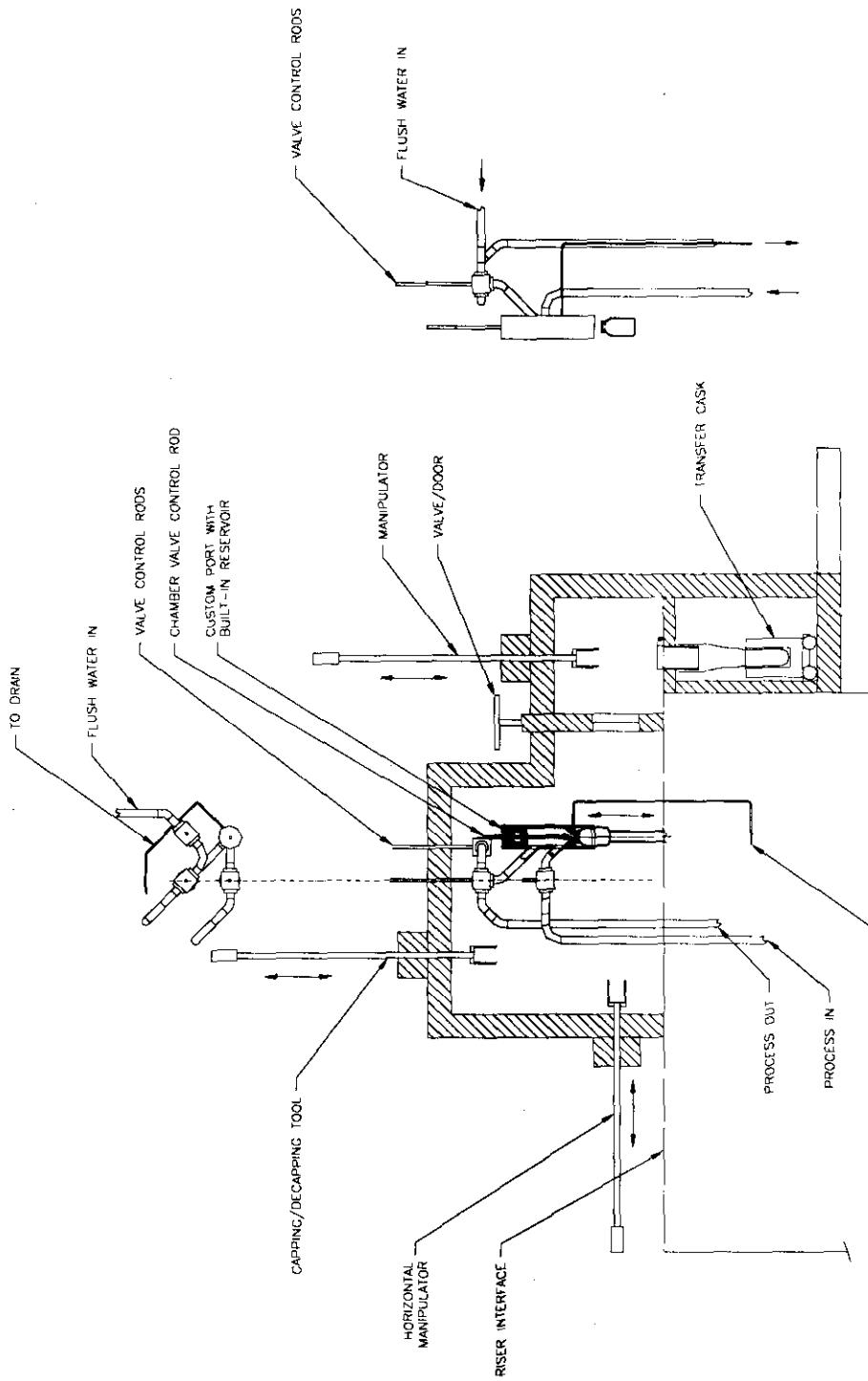
**Figure A-3 Sampling sequence and details for container filling Option 4.**

OPTION 4: THREADED PLUG WITH FLOW CHANNELS (2.2.2-B) MATED TO CUSTOM PORT WITH RESERVOIR (SINGLE STATION)

EXPLODED VIEW (1) INSERT BOTTLE (2) OPEN CAP AND FILL (3) CLOSE CAP AND FLUSH



**Figure A- 4 General arrangement of bottle filling system Option 2, showing interfaces to the sampling system waste stream.**



OPTION 2: TWO-STATION W/CUSTOM PORT & BUILT-IN RESERVOIR (2.2.2-B)

**Figure A- 5 Zoomed view of container filling chamber for Option 2.**

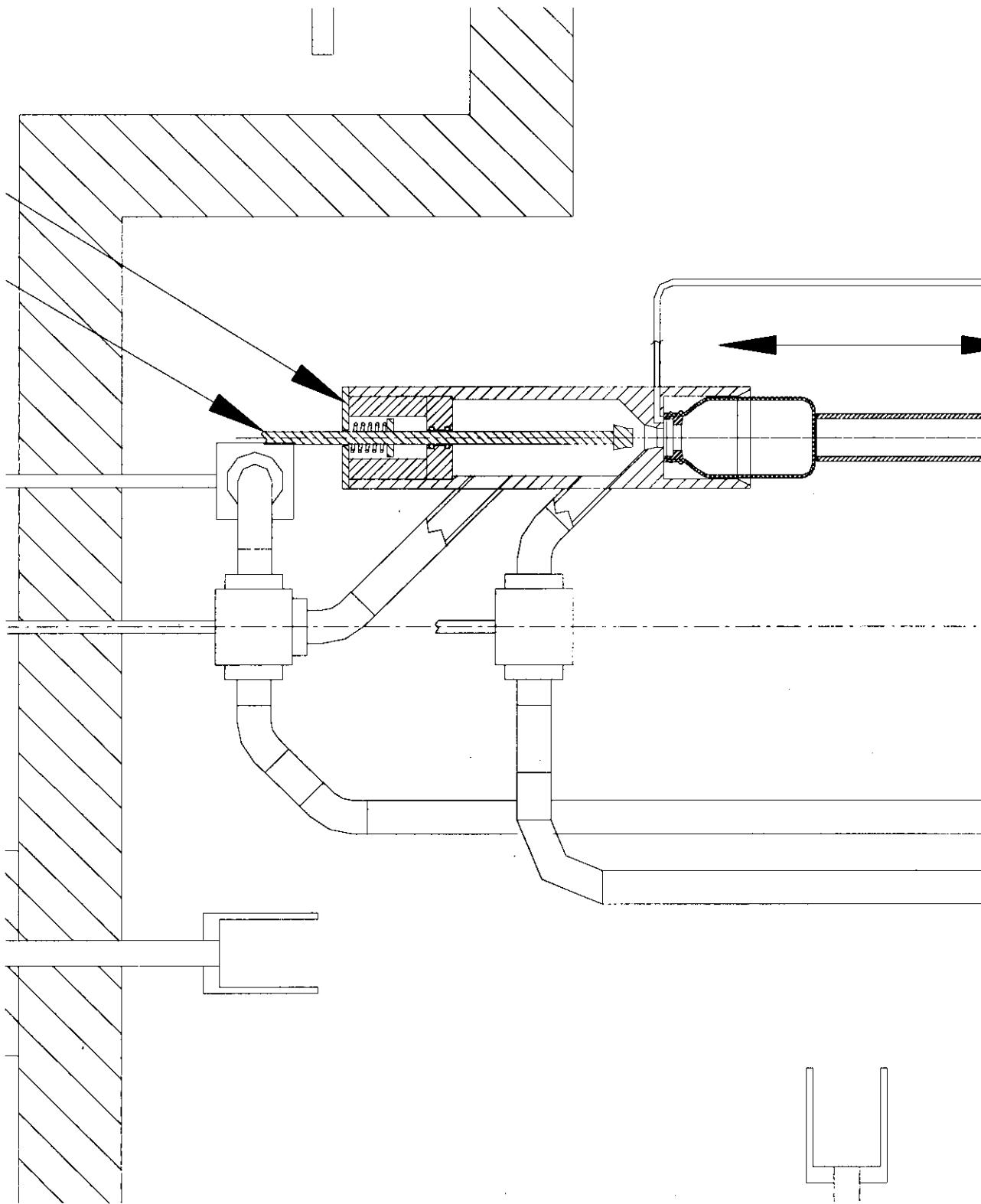
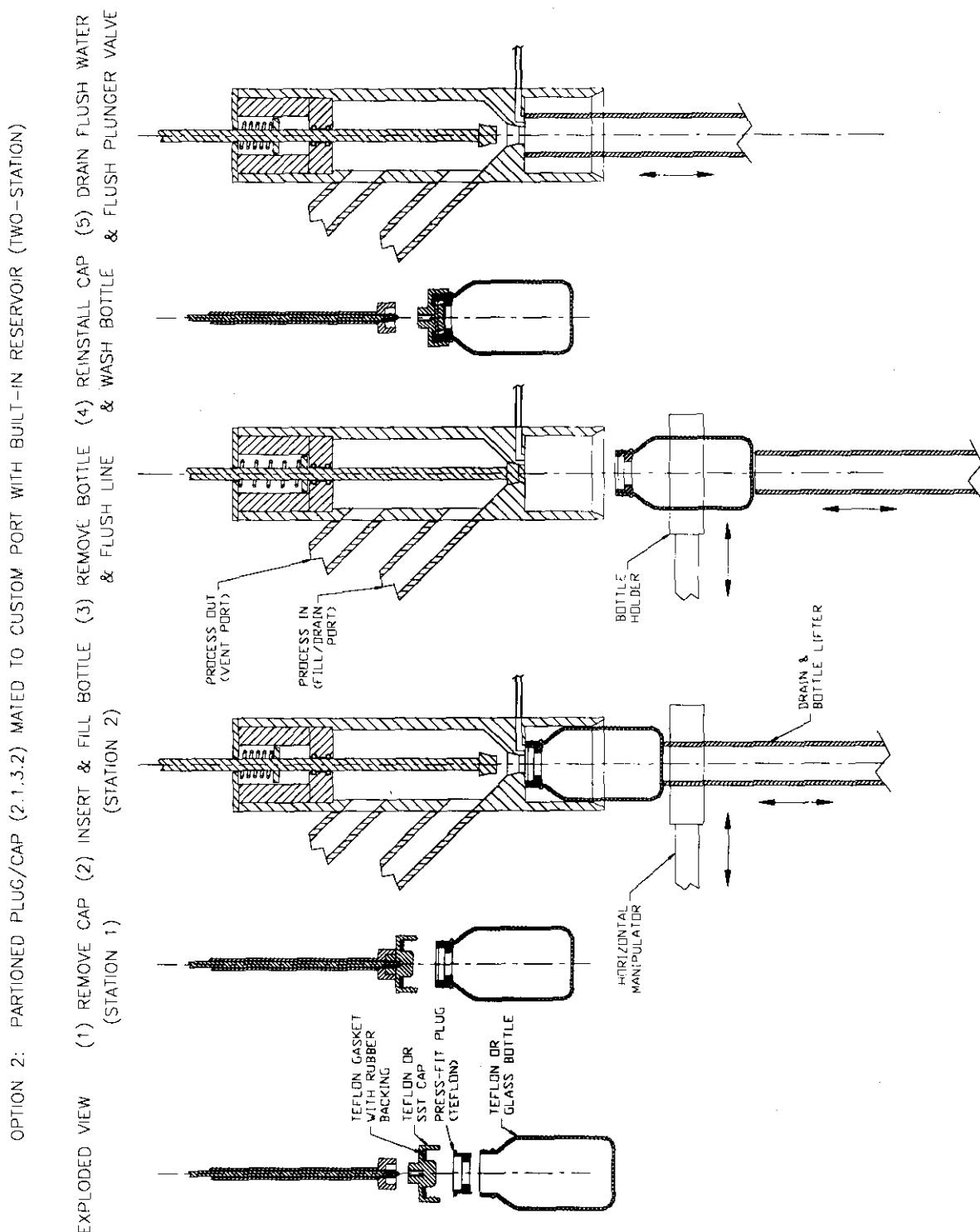


Figure A- 6 Sampling sequence and details for container filling Option 2.



**APPENDIX B**

**PARTICLE SIZE DISTRIBUTION  
FOR  
SAND/WATER SLURRY SIMULANTS**

## APPENDIX B

## PARTICLE SIZE DISTRIBUTION FOR SAND/WATER SLURRY SIMULANTS

**B.1** Introduction

The Level 2 Specification (Reich, 1999a) and the test plan (Reich, 1999b) for the sampling system contained particle size ranges that were expected to be in actual tank waste that would be addressed in the Phase 1 of the BNFL privatization contract. Although this simulates the tank waste properties, it does not provide good statistics in the tests with this sample materials. There is a significant lack of particle content for the larger particle sized to support good statistical assessments of sampling performance, as indicated in Table B-1 below.

**Table B-1** Tank waste particle size distributions (from Powell 1998a and 1998b).

Particle Size Range (microns)	Allowable Weight Percent Solids in Size Range
> 4000 $\mu\text{m}$	0 wt.%
500 to 4000 $\mu\text{m}$	< 1 wt.%
50 to 500 $\mu\text{m}$	< 5 wt.%
< 50	> $\approx$ 94 wt.%

AEAT identified three types of sand which were potential candidates for the sand/water simulants; "Silco-Sil 52" and two types (Type 1 and Type 2) of locally obtained sand materials (basically bags of children's play sand). The "Silco-Sil 52" was ground silica with a particle size less than 44 micron. The Type 1 sand has a significant number of large (greater than 425 micron) particles while the Type 2 sand is largely made up of particles that are in the 45 to 250 micron range. By blending combinations of these sands, a sand mixture can be obtained that has more desirable particle size ranges to better support representative sample testing.

Table B-2 shows the results of blending these three sand materials. The wt% ratios of the three sand types were established through an iterative process. The goal was a double digit particle count for a cubic centimeter of simulant. Table B-2 shows that if a mixture of 65 wt% Silica-Sel 52, 20 wt% Type 1 and 15 wt% Type 2 are blended, the resulting sand mixture will have the particle distributions as shown in column 5. For comparison purposes, the particle distribution expected in the tank waste, as estimated by Powell (Powell, 1998a and 1998b) is shown in column 6. The presence of a much higher number of larger particles will improve statistics during particle size distribution measurements.

**Table B- 2 Basic constituent sand particle size ranges.**

Size range	Weight percent (wt%)			Estimated wt%	Estimated Particles/cm <sup>3</sup>	Waste Estimated Particles/cm <sup>3</sup>
	Silica-Sel 53	Type 1	Type 2			
> 425 micron		42.4	1.8	8.75	37	1
250 to 425 micron		36.8	25.4	11.17	156	
150 to 250 micron		1.8	47.1	10.43	698	
45 to 150 micron		3.2	25.0	4.39	25793	5
< 45 micron	100	<1	<1	65.35	383955	94

Notes: Particle density calculations are based on an assumed spherical particle shape and silica sand density of 2.4 gm/cm<sup>3</sup>.

## B.2 References

Powell, M. R. and M. W. Rinker, 1998a, *Simulant Specification for Nested Fixed-Depth Sampler*, Correspondence No. 9854073, Pacific Northwest National Laboratory, Richland, Washington.

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