



U.S. Department of Energy
Office of River Protection

P.O. Box 450, MSIN H6-60
Richland, Washington 99352

OCT 31 2012

12-WTP-0306

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DNFS SAFETY BOARD

The Honorable Peter S. Winokur
Chairman, Defense Nuclear Facilities Safety Board
625 Indiana Avenue, NW, Suite 700
Washington, D.C. 200004-2901

Dear Mr. Chairman:

TRANSMITTAL OF DEFENSE NUCLEAR FACILITIES SAFETY BOARD (DNFSB)
RECOMMENDATION 2010-2 IMPLEMENTATION PLAN (IP) DELIVERABLE 5.5.3.6
(THIRD DOCUMENT)

This letter provides the deliverable responsive to Commitment 5.5.3.6 of the U.S. Department of Energy, Waste Treatment and Immobilization Plan (DOE-WTP) plan to address WTP Vessels Mixing Issues, IP for DNFSB 2010-2.


An attachment provides the third of three test plans to establish Tank Farm performance capability. Testing will be conducted to determine the range of waste physical properties that can be retrieved and transferred to WTP and determine the capability of Tank Farm staging tank sampling systems to provide samples that will characterize waste and determine compliance with the waste acceptance criteria.

This test plan identifies and describes testing activities that will be performed to address the technical risks associated with solids accumulation in waste feed delivery tanks over the duration of the mission. The plan has been prepared separately from the first two plans so that initial test results can inform this testing.

Large-Scale Integrated Mixing System Expert Review Team review comments and resolution are also included with this transmittal.

If you have any questions, please contact me at (509) 376-8830, or your staff may contact Ben Harp, WTP Start-up and Commissioning Integration Manager at (509) 376-1462.

Sincerely,


Scott L. Samuelson, Manager
Office of River Protection

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Attachment

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Attachment
to
12-WTP-0306

TRANSMITTAL OF DEFENSE NUCLEAR FACILITIES
SAFETY BOARD (DNFSB) RECOMMENDATION
2010-2 IMPLEMENTATION PLAN (IP)
DELIVERABLE 5.5.3.6 (THIRD DOCUMENT)

Total No. of Pages, including coversheet: 134



September 27, 2012

WRPS-1204232-OS

Ms. S. E. Bechtol, Contracting Officer
U.S. Department of Energy
Office of River Protection
Post Office Box 450
Richland, Washington 99352-0450

Dear Ms. Bechtol:

CONTRACT NUMBER DE-AC27-08RV14800 – ONE SYSTEM - WASHINGTON RIVER PROTECTION SOLUTIONS LLC TRANSMITTAL OF DEFENSE NUCLEAR FACILITIES SAFETY BOARD RECOMMENDATION 2010-2 IMPLEMENTATION PLAN REQUIREMENTS FOR COMMITMENT 5.5.3.6 (THIRD DOCUMENT)

One System transmits the enclosed documents to support the U.S. Department of Energy, Office of River Protection (ORP) transmittal of the commitment requirements to the Defense Nuclear Facilities Safety Board (DNFSB). In accordance with the Washington River Protection Solutions LLC 2010-2 Commitment Document Review Plan, we have completed the third document associated with DNFSB Recommendation Commitment 5.5.3.6 and are providing the appropriate documents to ORP. Support documents include the following:

- RPP-PLAN-53193, Rev. 0, “One System Waste Feed Delivery Mixing and Sampling Program Solids Accumulation Test Plan” (Enclosure 1)
- WRPS-1203839-OS, WRPS Large-Scale Integrated Mixing System Expert Review Team (ERT) Comment Response Letter to L. M. Peurrung, ERT Chair. Letter also includes ERT comment dispositions and draft document with ERT review comment incorporations (Enclosure 2)
- ERT Comment Response Concurrence Letter (Enclosure 3)

As previously discussed with ORP and DNFSB staff, this test plan is the last of three test plans associated with DNFSB 2010-2 Commitment 5.5.3.6. This change to a sequential delivery of multiple test plans will be reflected in the proposed revision to the DNFSB 2010-2 Implementation Plan currently being developed.

Ms. S. E. Bechtol
Page 2
September 27, 2012

WRPS-1204232-OS

If you have any questions concerning this matter, please contact Mr. M. G. Thien at 372-3665 or Mr. S. A. Saunders at 372-9939.

Sincerely,

(Signature Attached)

R. J. Skwarek, Project Manager
One System Integrated Project Team

(Signature Attached)

C. A. Simpson
Contracts Manager

MGT:MES

- Enclosures:
1. RPP-PLAN-53193, Rev. 0, "One System Waste Feed Delivery Mixing and Sampling Program Solids Accumulation Test Plan" (55 pages)
 2. Letter, R. J. Skwarek, WRPS, to L. M. Peurrung, PNL, "One System Technical Team Response to Review of Waste Feed Delivery Mixing and Sampling Program System Performance Test Plan (ERT-20)," WRPS-1203839-OS, dated September 24, 2012 (73 pages)
 3. ERT Comment Response Concurrence Letter, dated September 24, 2012 (1 page)

Ms. S. E. Bechtol
Page 3
September 27, 2012

WRPS-1204232-OS

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DOCUMENT RELEASE FORM

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Categories: Pending, Laura Solano, Gayla Bratton

From: Lee, Kearn P
Sent: Tuesday, September 25, 2012 11:04 AM
To: ^RIM DC; ^Information Clearance
Subject: RPP-PLAN-53193

The subject document RPP-PLAN-53193, One System Waste Feed Delivery Mixing and Sampling Program Solids Accumulation Test Plan, has been uploaded to the WRPS Records Holding Area for a clearance review and release. A clearance review is requested because information from an OUO document (RPP-RPT-50941) is cited. At the instruction of Information Clearance, I contacted the RPP-RPT-50941 clearance specialist for his guidance. A preliminary phone discussion with Gary Hulse, who made the determination on the cited document, said that the information I cited was okay to be released in a public document and I have treated this document as such. Although he is leaving his assigned duties at the end of this month, he did indicate that he was willing to review this document for the release process.

Kearn Patrick Lee (Pat)
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AREVA Federal Services
subcontractor to Washington River Protection Solutions,
contractor to the United States Department of Energy

RPP-PLAN-53193, Rev. 0

One System Waste Feed Delivery Mixing and Sampling Program Solids Accumulation Test Plan

KP Lee
Washington River Protections Solutions, LLC

Richland, WA 99352
U.S. Department of Energy Contract DE-AC27-08RV14800

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Cost Center: 2PD00 Charge Code: 201342
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Key Words: One System, Tank Farm Mixing and Sampling, Waste Feed Delivery, DNFSB Recommendation 2010-2, Solids Accumulation, Small Scale Mixing Demonstration

Abstract: This plan addresses the technical approach and test requirements for the Small-Scale Mixing Demonstration Solids Accumulation test activity being performed under the Mixing and Sampling Program to support waste feed delivery to the Hanford Waste Treatment and Immobilization Plant. Using a simulant that is typical of Hanford tank waste, testing will evaluate the propensity for fast settling solids to accumulate in the waste feed staging tanks as multiple fill and empty cycles deliver feed to the Hanford waste treatment plant.

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APPROVED
By Janis D. Aardal at 9:08 am, Sep 27, 2012

Release Approval

Date



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RPP-PLAN-53193, Rev. 0

EXECUTIVE SUMMARY

The primary purpose of the Tank Operations Contractor Mixing and Sampling Program is to mitigate the technical risks associated with the ability of the tank farms waste feed delivery systems to mix and sample High-Level Waste feed adequately to meet the Hanford Waste Treatment and Immobilization Plant Waste Acceptance Criteria. In a series of testing activities the Tank Operations Contractor will conduct tests to determine the range of waste physical properties that can be retrieved and transferred. Using two geometrically scaled tanks, testing and analysis will determine the propensity for the waste feed delivery mixing and transfer system to accumulate fast settling solids in the feed staging tanks. This test plan is the third of three test plan documents that are being prepared to address Defense Nuclear Facilities Safety Board DNFSB 2010-2, Sub-Recommendation 5, Commitment 5.5.3.6, "Test Plan to establish Tank Farm performance capability" and addresses the technical approach and test requirements for the Small-Scale Mixing Demonstration solids accumulation test activities being performed to support waste feed delivery. The solids accumulation tests are patterned after the duty cycle for double shell tank 241-AW-105, which is planned to have the greatest number of transfers to the Hanford waste treatment plant (ORP-11242 Rev. 6, *River Protection Project System Plan*).

The tests being conducted to define the capabilities of the mixing, sampling, and transfer system are focused on three areas: limits of performance, solids accumulation, and scaled/system performance. Solids accumulation testing at two scales is described in this test plan. Solids accumulation testing is exploratory and is being conducted to understand the potential to concentrate dense fissile material in a waste feed staging tank that is subjected to repeated waste feed fill and empty cycles. Solids accumulation work will demonstrate mixing, sampling, and transfer performance using simulants representing a typical Hanford waste. Testing will be performed with base particulate solids in a Newtonian suspending fluid that are characteristic of Hanford waste in terms of bulk particle density, particle size, solids loading, supernatant density, supernatant viscosity, and slurry density. The slurry will contain dense particles (8 g/cm^3) having particle sizes exceeding 100-microns for assessing the propensity to accumulate fast settling solids in the waste feed staging tanks. A tungsten alloy powder with a particle density of approximately 9.6 g/cm^3 will be included in the simulant beginning with the third fill and empty cycle. The potential to concentrate fissile material in the tank will be evaluated with this spike particle. Core samples will be taken from the mounds to determine if the spike component migrates to the bottom of the mounds during subsequent fill and empty cycles. In addition, the spike particles will also be used to determine the capability of the system to transfer fast settling spike particles for comparisons to waste feed characterization requirements for uranium (U) and plutonium (Pu) and to requirements for waste treatment processability; (e.g., Pu and U unwashed solids concentration). These tests will use the Small-Scale Mixing Demonstration test platforms used in previous Waste Feed Delivery Mixing and Sampling Program test activities; however, the operating conditions and simulants tested will be expanded to collect additional performance data.

For the test activity covered in this test plan, the test objectives along with success criteria are identified. The necessary equipment to conduct the tests and collect the necessary data is identified and described. This work is follow-on work to the solids accumulation scouting studies performed at the Savannah River National Laboratory where measurement techniques

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and parameter sensitivity were first investigated (RPP-PLAN-52005, *One System Waste Feed Delivery Mixing and Sampling Program Limits of Performance and Solids Accumulation Scouting Studies Test Plan*). The simulants that are appropriate for testing are identified and qualified in accordance with the recommendations in RPP-PLAN-51625, *Waste Feed Delivery Mixing and Sampling Program Simulant Definition for Tank Farm Performance Testing*. The most important properties identified for solids accumulation work include variations to the mixer jet nozzle velocity and the sequential fill and empty cycles that simulate the multiple uses of the waste feed staging tanks during the waste feed delivery mission.

Small-Scale Mixing Demonstration solids accumulation testing will be conducted to:

- Use fast settling base particulate and spike solids in a Newtonian supernatant to determine at small scale how fast settling solids are spatially distributed in mounds left in a waste feed staging tank after the feed has been delivered.
- Evaluate how fast settling solids could be spatially distributed in a full-scale double-shell tank.
- Explore if fast settling spike particles can be concentrated at the bottom of full-scale double-shell tank.
- Evaluate the reliability of the collected data for predicting full-scale performance when the scaling relationship is uncertain.

Mixing, transfer, and heel accumulation data at two scales will be collected and analyzed to determine if the fast settling solids accumulate in the tank after ten fill and empty cycles are performed. The first fill cycle fills an empty tank with the waste simulant. The first transfer cycle uses the mixer jet pumps to mix the tank contents at one of two nozzle velocities and a pump to transfer material from the tank in 6.5 sequential transfer batches. Batch transfer samples are collected to quantify the amount of material transferred. After the final transfer of the first cycle, it is expected that there will be mounds (also called piles) of solids that accumulate along the perimeter and on the bottom of the tank in the area that is outside the area of influence of the two mixer jet pumps. Heel samples will be collected from these solids and the volume of solids in the mounds will be estimated. The tank is then filled to volume with additional, fresh simulant made to the same composition as the first cycle. Care will be taken when refilling the tank with fresh simulant so that the solid piles that accumulated in the tank are not disturbed. The process is repeated, estimating the volume of the solid mounds after each tank volume transfer (i.e., 6.5 transfer batches). Beginning with the third fill cycle a fraction of the fast settling solids will be replaced with a higher density spike solid that is chemically different from the other simulant components. The fill and empty cycles are repeated until ten cycles are completed. Heel samples are collected from the mounds after the first, fifth and tenth tank volume transfer. The spatial distribution of fast settling solids in the heel is determined by comparing component concentrations in the mound from the known sample locations. In the deepest parts of the mounds, the collected samples will be segmented to capture coarse vertical partitioning. The results will be mapped to show where the fast settling solids tend to accumulate in the tank. In addition, the potential to concentrate dense fissile material on the bottom of a mound will be evaluated by noting whether the spike particulate, which is added

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after the initial mounds are formed, is found at the bottom of a mound. Two campaigns of ten fill and empty cycles will be performed at each scale. The composition of the simulant used will be the same in all tests but different nozzle velocities will be set for each campaign. For the first campaign in the 1:21-scale tank the nozzle velocity will be set at the equal power per volume scaling condition. For the first campaign in the 1:8-scale tank the nozzle velocity will be set so that similarly proportioned mounds are attained. The nozzle velocity for the second campaign in the 1:21-scale system will be determined based on the performance in the first campaign. Similar to the first campaign, the nozzle velocity for the second campaign in the 1:8-scale tank will be set to match the mound proportions from the second campaign in the 1:21-scale tank. Therefore, twenty tests will be conducted in the 1:21 and 1:8 scale mixing tanks in the Small-Scale Mixing Demonstration test platform.

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TERMS

Abbreviations and Acronyms

ASME	American Society of Mechanical Engineers
BNI	Bechtel National, Inc.
DOE	U.S. Department of Energy
DNFSB	Defense Nuclear Facilities Safety Board
DST	double-shell tank
DQO	data quality objective
HLW	high-level waste
ICD	Interface Control Document
MDT	SRNL mixing demonstration tank
ORP	Office of River Protection
Pu	plutonium
PNNL	Pacific Northwest National Laboratory
RPP	River Protection Project
RSD	Remote sampler Demonstration
SRNL	Savannah River National Laboratory
SSMD	Small-Scale Mixing Demonstration
TOC	Tank Operations Contractor
U	uranium
WAC	waste acceptance criteria
WFD	Waste Feed Delivery
WRPS	Washington River Protection Solutions, LLC
WTP	Hanford Waste Treatment and Immobilization Plant

Units

°C	degrees Celsius
cm	centimeter
cP	centipoise
ft	feet
in	inch
g	gram
gpm	gallons per minute
l	liter
ml	milliliter
rpm	revolutions per minute
s	second

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1.0 INTRODUCTION

The Office of River Protection (ORP) has defined the interface between the two prime River Protection Project (RPP) contractors, Bechtel National, Inc. (BNI) and Washington River Protection Solutions (WRPS), in a series of interface control documents (ICDs). The primary waste interface document is 24590-WTP-ICD-MG-01-019, *ICD-19-Interface Control Document for Waste Feed* (also known as ICD-19). Section 2.3 of ICD-19 states, that the Tank Operations Contractor (TOC) baseline sampling plans and capabilities are not currently compatible with WTP sample and analysis requirements.

The primary purpose of the TOC Waste Feed Delivery (WFD) Mixing and Sampling Program is to mitigate the technical risks associated with the ability of the tank farms feed delivery systems to adequately mix and sample High Level Waste (HLW) feed to meet the Hanford Waste Treatment and Immobilization Plant (WTP) Waste Acceptance Criteria (WAC). Initial work for the WFD Mixing and Sampling Program demonstrated that the concept functionality for the first feed tank to deliver consistent feed delivery batches was viable. However, uncertainties related to scale-up, simulant representativeness, data uncertainty, optimizing system performance, applicability to all feed tanks, feed conditioning, and understanding emerging WTP solids handling risks still need to be addressed. The TOC has identified two critical risks TOC-12-64 and TOC-12-65 per the TFC-PLAN-39, Rev. G, *Risk Management Plan*, which address sampling methods and emerging changes to WAC requirements. The root of the mixing and sampling risk is the ability to collect samples that are characteristic of the tank waste, including the rapidly settling solids in the HLW for the purpose of demonstrating compliance with the WTP waste acceptance requirements. In addition, in November 2011, the U.S. Department of Energy (DOE) issued the implementation plan for the Defense Nuclear Facility Safety Board (DNFSB) Recommendation 2010-2 (DOE Rec. 2010-2, Rev. 0, *Implementation Plan for Defense Nuclear Safety Board Recommendation 2010-2*), which addresses safety concerns associated with the ability of the WTP to mix, sample, and transfer fast settling particles. DOE developed the implementation plan to resolve these issues and a related TOC issue concerning the capability of the mixing and transfer system to adequately mix the tanks to minimize the buildup of waste solids in the waste feed staging tanks that are re-used during the feed delivery mission.

Through multiple test activities (see Figure 1-1), the TOC will determine the range of waste physical properties that can be retrieved and transferred to WTP, and determine the capability of tank farm staging tank sampling systems to provide samples that will characterize the tank waste to determine compliance with the WAC. These tests will reduce the technical risk associated with the overall mixing, sampling, and transferring of HLW feed to WTP so that all WAC requirements are met. Report RPP-PLAN-41807, *Waste Feed Delivery Mixing and Sampling Program Plan and Test Requirements* defined the three test requirements for continued WFD Mixing and Sampling Program testing to address DNFSB concerns. In accordance with DNFSB 2010-2 Sub-Recommendation Commitment 5.5.3.6, "Test Plan to establish Tank Farm performance capability", test plans are prepared to further refine testing requirements as follows:

- Limits of performance - determine the range of waste physical properties that can be mixed, sampled, and transported under varying modes of operation. These tests will use both the Remote Sampler Demonstration (RSD) platform and the Small-Scale Mixing

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Demonstration (SSMD) platform. In addition, a test using a full-scale slurry transfer pump will be performed. Specific test requirements and additional details for the limits of performance testing activities are documented in RPP-PLAN-52005, *One System Waste Feed Delivery Mixing and Sampling Program Limits of Performance and Solids Accumulation Scouting Studies Test Plan*.

- Solids accumulation - perform scaled testing to understand the accumulation and spatial distribution of the remaining solids in a double-shell tank (DST) during multiple fill, mix, and transfer operations that are typical of the HLW feed delivery mission. These tests include scouting activities at the Savannah River National Laboratory (SRNL) Mixing Demonstration Tank (MDT) and the SSMD platform. Specific test requirements and additional details for the SRNL solids accumulation testing activities are documented in RPP-PLAN-52005. Draft SRNL test results and recommendations used to develop this test plan are documented in SRNL-STI-2012-00508, *Solids Accumulation Scouting Studies* (in process). Specific test requirements and additional details for the SSMD solids accumulation testing activities are documented in this test plan.
- Scaled/system performance - demonstrate mixing, sampling, and transfer performance using a realistic simulant representing a broad spectrum of Hanford waste to meet WTP WAC Data Quality Objectives (DQO) sampling confidence requirements. These tests will use both the SSMD and the RSD platforms. The RSD platform is full scale; therefore, RSD system performance testing activities will collect additional system performance data at full scale. Specific test requirements and additional details for the SSMD scaled performance and RSD system performance testing activities are documented in RPP-PLAN-52623, *One System Waste Feed Delivery Mixing and Sampling Program System Performance Test Plan*.

A TOC simulant plan, RPP-PLAN-51625, *Waste Feed Delivery Mixing and Sampling Program Simulant Definition for Tank Farm Performance Testing*, was developed to define the simulant objectives for this testing. Simulants were developed to represent the complete range of physical properties for the broader spectrum of Hanford waste tanks, and to address specific testing requirements summarized above. This represents a broadening of objectives from earlier SSMD and RSD testing. The simulants and operating conditions in this earlier testing were intended to simulate the particle size, density distribution, and operating configuration of Hanford DST 241-AY-102, the first tank waste to be delivered to WTP. The particle size distribution for the SSMD simulant for DST 241-AY-102 (1% is 0.39 microns, 50% is 13.2 microns, 95% is 200 microns, and 99% is 394 microns) is documented in PNNL-20637, *Comparison of Waste Feed Delivery Small-Scale Mixing Demonstration Simulant to Hanford Waste*. The range of particle sizes in the simulant was smaller than the particle size distribution for the 95% confidence limit for 95% of the population (1% is 2 microns, 50% is 22 microns, 95% is 460 microns, and 99% is 700 microns) used in the waste feed transfer system analysis used in the WTP design basis, RPP-9805, *Values of Particle Size, Particle Density, and Slurry Viscosity to Use in Waste Feed Delivery Transfer System Analysis*.

This test plan is the third of three test plan documents prepared to address DNFSB 2010-2 Sub-Recommendation Commitment 5.5.3.6, "Test Plan to establish Tank Farm performance capability". This test plan identifies and describes the test objectives, test requirements, and test methods for the SSMD Solids Accumulation test activities. This work is follow-on work to the

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solids accumulation scouting studies performed at the Savannah River National Laboratory where measurement techniques and parameter sensitivity were first investigated (RPP-PLAN-52005). The testing approach is guided by this previous work as well as by input from internal subject matter experts and external consultants familiar with the objectives of the test program. The original discussions held to develop the testing approach are described in WRPS-1105293, *Small-Scale Mixing Demonstration Optimization Workshop Meeting Minutes* and are refined in WRPS-1201374-OS, *One System DNFSB 2010-2 Sub-Recommendation 5 Test Plan Summit Meeting Minutes*. The current scope addresses the buildup of solids in the tanks after multiple tank refills and the changes to the composition and spatial distribution of the solids in the piles over time. The current scope will not address any operational improvement options that evaluate how to re-suspend the dead zones. The current scope will also not address reduced pump performance or how an extended outage may cause the rheology of the waste to change over time. Operational improvements to minimize solids accumulation and re-suspend the dead zones are planned for Fiscal Year 2013. Future testing to evaluate rheology changes remains a consideration for future testing activities.

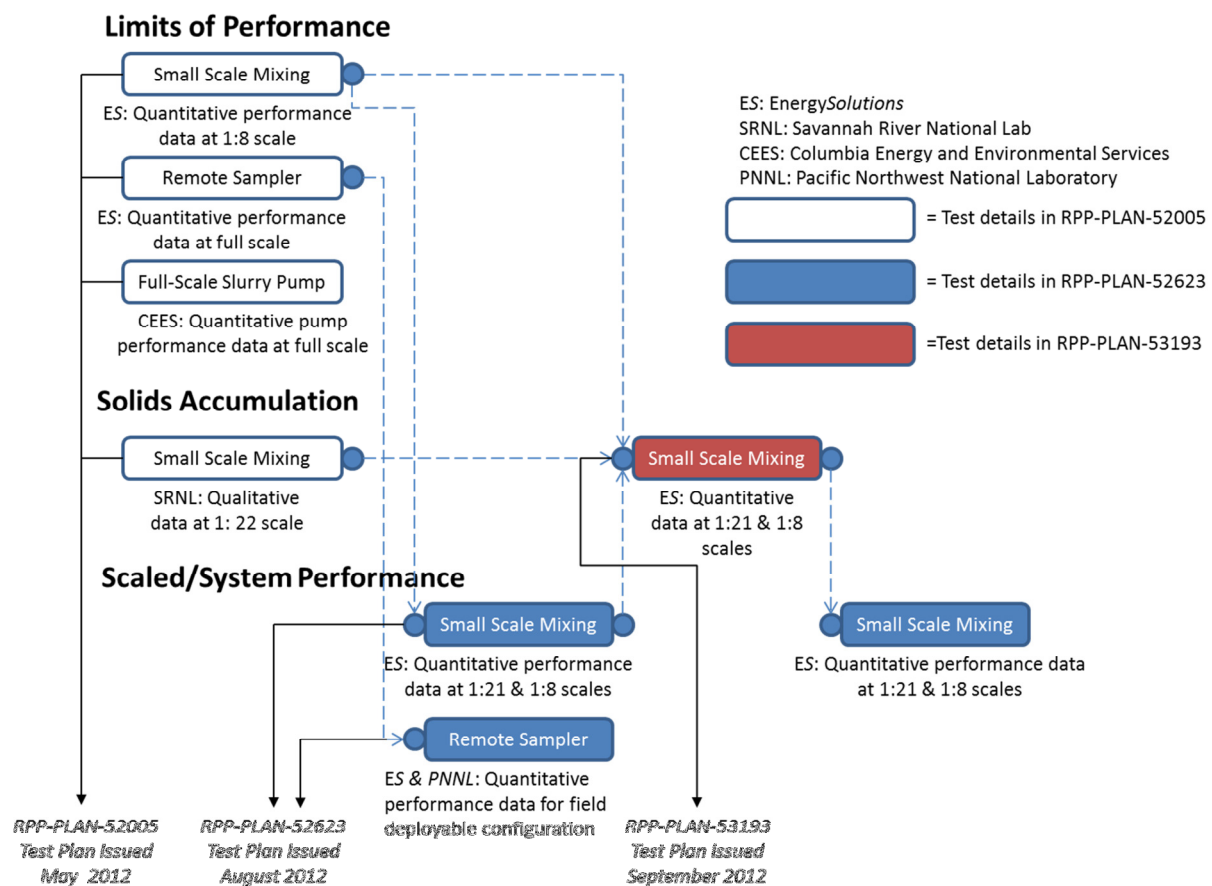


Figure 1-1. WFD Mixing and Sampling Program Test Sequence

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2.0 SCOPE

DNFSB Recommendation 2010-2 has raised WTP safety issues related to tank farms ability to mix, sample, and transfer solids. In response, DOE developed an implementation plan to resolve these issues (DOE Rev. 0 2010-2). The SSMD solids accumulation testing will explore the propensity of the feed staging system to accumulate fast settling solids over the duration of the waste feed delivery mission. As discussed in Section 1.0, this test plan is one of multiple test plan documents that have been prepared to address Commitment 5.5.3.6 of the Implementation Plan.

To ensure that tank farms and WTP mixing and sampling systems are integrated and compatible (i.e., execution of the One System approach) and that the uncertainties identified to date (WRPS-1105293) are addressed, solids accumulation testing will explore the potential for fast settling particles to concentrate in the waste feed staging tanks during the multiple fill, mix, and transfer operations expected to occur over the life of the mission.

The propensity of the Tank Farm's WFD system to accumulate solids will be characterized using tank waste simulants that have typical physical properties that are important to mixing, sampling and transfer (solid particulates sizes and densities, and supernatant density and viscosity), and may not be properties that will be directly measured and compared to WAC requirements. Although Hanford feed staging tanks may exhibit non-Newtonian behavior, some of the Hanford single-shell tank waste has non-Newtonian characteristics, this initial exploratory work to understand how piles of fast settling solids accumulate is limited to studies with Newtonian suspending fluids. Slurry samples will be collected during each batch transfer operation and analyzed for chemical composition to determine the amount of material that is transferred from the tank. By mass balance accounting (mass in minus mass out) the running inventory in the tank will be determined. Additionally, the volume of residual solids deposited as mounds in the tank will be estimated to determine if solids accumulation occurs over multiple fill and empty cycles. Solid samples collected from the mound will be analyzed to determine the chemical content of the mounds. The chemical content of the solid samples will be mapped according to the sample locations to determine how fast settling solids, including spike particles, are spatially distributed in the mounds. In addition, solid samples collected after subsequent cycles from adjacent locations will be compared to determine whether or not the concentration of fast settling solids in the mounds change. Increasing concentrations of fast settling solids in the mounds is indicative of accumulation.

Testing will be performed using the SSMD test platform (see Figure 2-1). Testing will continue to be performed at two scales in accordance the recommendations developed at the initial planning workshop, which provided guidance that a decision regarding a third scale will be held until after performance at the smaller scales is demonstrated (Section 4.2 of RPT-1741-0001, *Tank Farm Mixing Demonstration Planning Workshop*). Testing at each scale will also be performed at two nozzle velocities. Nozzle velocities at each scale will be selected that result in similarly proportioned piles (footprint and depth relative to the difference in scale). Solids accumulation in the similarly proportioned piles will be compared across the two scales. If solids accumulation in both scales is similar, than it can be inferred that solids accumulation in a full-scale tank with similarly proportioned piles will also be similar. At each scale two nozzle velocities will be evaluated. Different nozzle velocities will result in different sized piles and

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may affect solids accumulation attributes that affect the conclusions made about a full-scale system.

Testing will be performed with Hanford waste simulants that are typical for the wide range of characterized waste with respect to ICD-19 WAC in terms of bulk density, solids loading, and slurry viscosity. Testing will be performed with slurries containing dense particles (8 g/cm^3) having particles sizes exceeding 100 microns that are spiked with plutonium oxide surrogates for assessing the potential to concentrate fissile material in the tank. In addition, the spike particles will be used to determine the capability of the system to transfer the fast settling particles for comparisons to ICD-19 requirements with action limits for uranium (U) and plutonium (Pu) and to requirements for waste treatment processability; (e.g., Pu and U unwashed solids concentration).

The test objectives for the SSMD solids accumulation performance evaluation are summarized in Table 2-1.

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Table 2-1. Solids Accumulation Test Objectives

Objective	Success Criteria
<p>Use fast settling base particulate and spike solids in a Newtonian supernatant to determine how fast settling solids are spatially distributed in mounds left in a waste feed staging tank after the feed has been delivered.</p>	<p>Mixing and transfer tests are performed with Hanford tank waste simulant slurries. The slurry contains moderately sized (approximately 100 microns), dense particles (~8 g/cm³ and 9.6 g/cm³) to represent hard-to-transfer waste particles in the Hanford tank waste. These particles are distinguishable in collected samples by a physical or chemical property that can be exploited for separation and subsequent quantification.</p> <p>Solid samples are collected from the tank mounds and analyzed for chemical content. Using the known location of the samples together with the analytical results and approximate shape of the mound, the spatial distribution of fast settling solids in the mounds is mapped.</p> <p>Two campaigns of ten fill and empty cycles are performed at each scale. Each campaign uses a different nozzle velocity for evaluating solids accumulation.</p>
<p>Evaluate how fast settling solids could be spatially distributed in a full-scale DST.</p>	<p>The 1:8-scale and 1:21-scale mixing and transfer systems in the SSMD platform are filled with the same simulant combination and operated at nozzle velocities that result in the formation of similarly proportioned piles relative to the tank scale. The spatial distribution of fast settling solids in the mounds in each scaled system are mapped and compared.</p>
<p>Explore if fast settling spike particles can be concentrated at the bottom of full-scale double shell tank.</p>	<p>Ten waste feed staging fill and empty cycles are performed under similar test conditions (simulant composition of added feed, nozzle velocity, rotational rate, fill volume, equipment configuration) in each scale. Heel samples are collected after the first transfer cycle and the spatial distribution of fast settling solids is mapped.</p> <p>After the tank mounds have formed, a fast settling spike particle, a surrogate for plutonium oxide, is introduced into the tank. After the 5th and 10th complete cycle, heel samples are collected and analyzed for chemical content. Heel samples are collected from mound locations adjacent to previously collected samples. Coarse vertical discretization of the heel samples is performed. The spatial distribution of fast settling solids in the mounds is mapped. The presence of the fast settling spike particles at the bottom of the mound is or is not confirmed. The change in the distribution of the fast settling solids in the three spatial distribution maps is evaluated.</p> <p>Conclusions about the changes in the spatial distribution of the fast settling solids in the mounds of a full-scale DST are made by comparing the results from the two smaller scales.</p>
<p>Evaluate the reliability of the collected data for predicting full-scale performance when the scaling relationship is uncertain.</p>	<p>The solids accumulation studies performed to evaluate how fast settling solids are distributed in the mounds of a waste feed staging tank are repeated at a second nozzle velocity.</p> <p>Conclusions about the spatial distribution of the fast settling solids in the mounds of a full-scale DST are made and then the results are compared to the previous work to determine if the different operating velocity changed the conclusions.</p>

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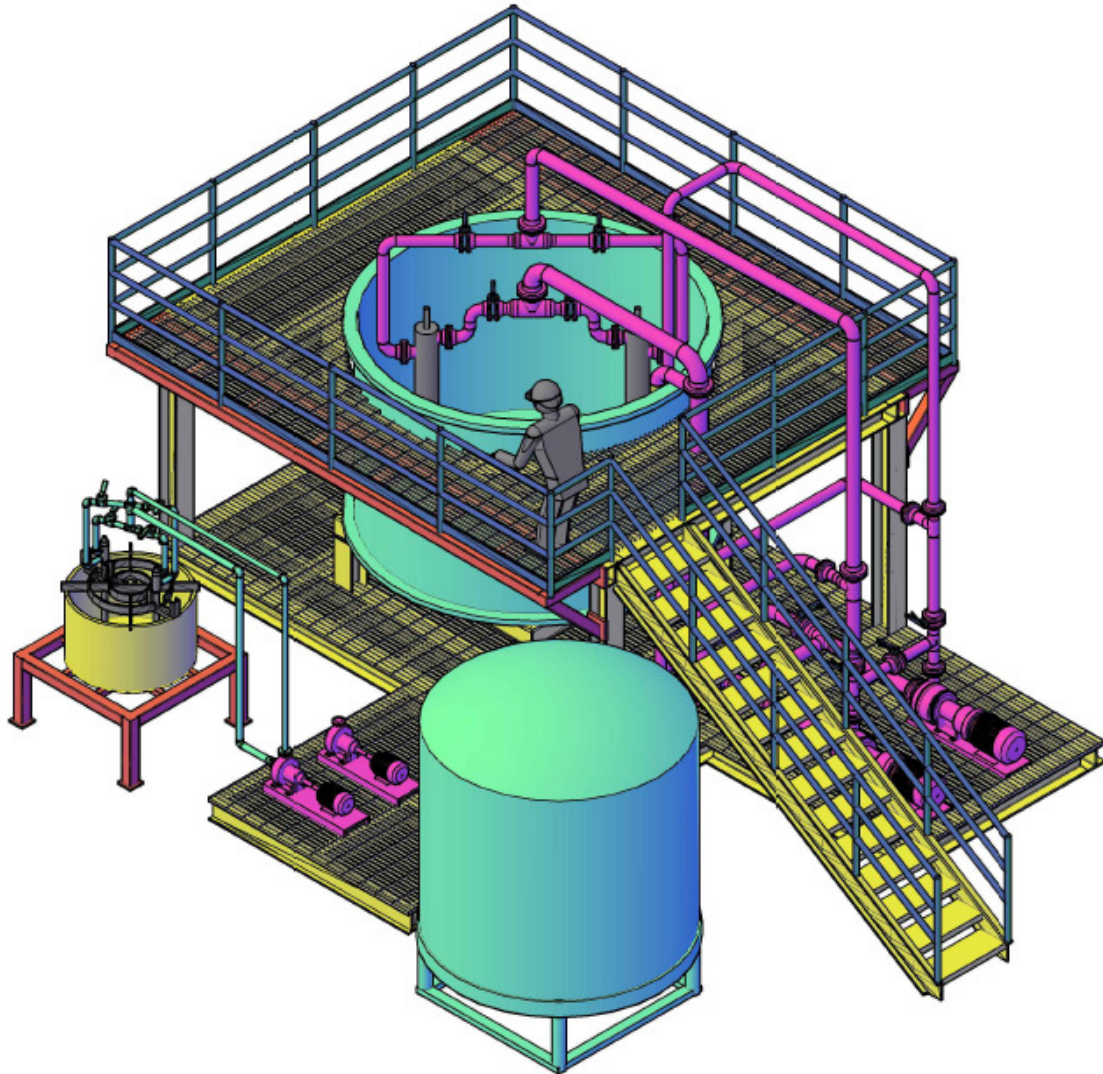


Figure 2-1. Schematic of Small-Scale Mixing Demonstration Test Platform

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3.0 TEST REQUIREMENTS

Test requirements and test guidance have been developed to meet the SSMD solids accumulation performance evaluation test objectives identified in Section 2.0. However, the WFD Mixing and Sampling Program testing falls outside the scope of TFC-PLAN-26, *Test Program Plan*, which defines additional requirements for oversight, development, and the conduct of factory acceptance, construction acceptance, and operational acceptance tests for demonstrating the operability and integrity of new or modified tank farm facilities and systems. The WFD Mixing and Sampling Program testing is evaluating the feasibility of a baseline design for collecting representative samples from the waste feed staging tanks. Testing is developmental and is not evaluating a field deployable design against specific functional characteristics and performance requirements. Testing is performed in accordance with Phase I testing described in TFC-PLAN-90, *Technology Development Management Plan*. Phase I development testing addresses a TOC technology need when existing processes are inadequate, inefficient, or not proven for the intended application. During Phase I testing functional criteria and performance requirements for the promising technology are defined, a prototype working model is constructed, and the prototype is evaluated against the performance criteria. Phase I development implements a graded application of the quality assurance program requirements. Phase I testing generally applies a commercial quality assurance program because there is no implied guarantee that the technology will be adopted by the TOC. Upon successful completion of Phase I testing, which may be an iterative process, additional development (Phase II) may be pursued. Phase II development and testing is performed to a higher quality assurance standard and invokes TOC approved procedures and quality assurance requirements for design control, including design verification, and qualification testing. The WFD Mixing and Sampling Program test planning, test review, test control, and test results reporting requirements are communicated through this test plan and are guided by testing principles described in TFC-ENG-DESIGN-C-18, *Testing Practices*.

In addition to this test plan, the testing contractor will develop operational procedures that include or reference the test configuration, test objectives, test requirements, and provisions for assuring that prerequisites and suitable environmental conditions are met, adequate instrumentation is available and operational, and that necessary monitoring is performed.

The SSMD solids accumulation test activities are performed by EnergySolutions for WRPS.

3.1 TEST SIMULANTS

The capability gap between the TOC and the WTP is defined by the TOC's capability to mix, sample, and transfer large and dense particles, and the WTP's capability to process these particles. Therefore, integral with defining the gap in capabilities is the selection of appropriately complex simulants, integrated with WTP simulant selection, and supported by accurate analytical techniques to characterize the material of interest. The Hanford waste simulants for DNFSB 2010-2 testing are developed and described in RPP-PLAN-51625. Particle size and density are expected to be the most important solids properties for evaluating the propensity of the waste feed staging system to accumulate fast settling solids. Liquid density and

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viscosity are expected to be important liquid phase properties as these properties directly affect particle settling and mobilization from the tank.

The slurry simulant used for SSMD solids accumulation test activities is consistent with simulant development described in RPP-PLAN-51625 and used in recent TOC testing activities. Simulant selection considers parameters (e.g., particle size, density, and viscosity) important to mixing, sampling, and transfer performance because solids accumulation is directly affected by the capability of the system to transfer the particles from the tank. Simulant properties such as hardness and abrasiveness, which are important to evaluating erosion and wear of the tank and pipe walls and the mixing and transfer equipment, are not primary considerations for understanding the capability of the system to mix, sample, and transfer slurries characteristic of Hanford tank waste. However, simulant selection does favor materials that result in less wear on the test equipment when alternatives that match the critical characteristics are available.

Although SSMD solids accumulation testing is Phase I technology development and generally performed to the subcontractors own quality assurance procedures, simulant procurement, preparation, and simulant property data collection are performed to enhanced quality assurance standards as defined in TFC-ESHQ-Q_ADM-C-01, *Graded Quality Assurance*. The enhanced quality assurance standard applied is American Society of Mechanical Engineers (ASME) NQA-1-2004, including addenda, or a later version. As such, additional level of controls beyond the providers published or stated attributes of the item, service, or process are needed to verify critical attributes of the simulants. Simulant materials procured as commercial grade items shall be prepared and qualified to match the critical characteristics of the simulants.

Simulant batches of base material and a Newtonian supernatant are prepared according to prepared recipes. By specifying the mass fraction of each solids component, the density of each solids component, the density of the supernatant, the solids loading, and the batch volume, the required amounts of each solids component are fully defined. Supernatant recipes were determined during previous test activities and will be confirmed using test batches prepared to match the critical characteristics. The base simulant, spike particles, and supernatant simulant used during SSMD solids accumulation testing are described below.

3.1.1 Base Simulant

As discussed in RPP-PLAN-51625, during simulant development for DNFSB 2010-2 test activities metrics that are relevant to mixing and sampling were selected, calculated, and compared between the developed simulants and the Hanford tank waste. The calculated values for the metrics are not used to set operating conditions for testing; metric comparisons are only used to demonstrate that the developed simulants are similar to the Hanford tank waste. Therefore, this test plan does not develop simulants, rather it selects simulants from those previously developed.

3.1.1.1 Base Simulant Description

The base simulant is the mixture of solid particles in the slurry representing the Hanford tank waste. Report RPP-PLAN-51625 recommends three mixtures of particles as the base simulants for WFD Mixing and Sampling Program test activities, low conceptual, typical conceptual, and high conceptual (see Table 3-1). The low conceptual simulant was excluded from consideration

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because it does not contain any fast settling solids. Only the typical and high conceptual simulants were considered for solids accumulation tests. Both the typical and high conceptual simulants contain fast settling solids (stainless steel powder with a density of approximately 8 g/cm³). To represent the fast settling fissile material in the tank waste, the base material will be spiked with a tungsten alloy powder having a density of approximately 9.6 g/cm³. Simulant spikes are discussed in Section 3.1.3.

Table 3-1: Base Particulate Simulant Characteristics

Base Particulates					
Compound	Solid Density (g/cm ³)	Median Particle Size (micron)	Mass Fraction		
			Low	Typical	High
Small Gibbsite	2.42	1.3	1.00	0.27	0
Large Gibbsite	2.42	10	0	0.44	0.03
Small Sand	2.65	57	0	0	0.35
Medium Sand	2.65	148	0	0.13	0
Large Sand	2.65	382	0	0	0.21
Zirconium Oxide	5.7	6	0	0.10	0.08
Stainless Steel	8.0	112	0	0.06	0.33

The stainless steel particles in the base material are a fast settling solid. The free settling velocity, V_t , in the typical supernatant (see Section 3.1.2) can be calculated for the stainless steel particles with specified sizes and densities using Equations 3-1 and 3-2 (from *Handbook of Industrial Mixing: Science and Practice*, Equations 10-1, 10-2 and Table 10-1). Equation 3-1a is for the Stokes Law regime and applies when the particle Reynolds number is less than 0.3. Equation 3-1b is for the Intermediate Law regime and applies when the particle Reynolds number is between 0.3 and 1000. The free settling velocities for stainless steel particle sizes in Table 3-2 result in particle Reynolds numbers, Re_p , (Equation 3-2) in the Intermediate Law regime.

$$V_t = \left(\frac{4gd(\rho_s - \rho_l)}{3\rho_l \left(\frac{24}{Re_p} \right)} \right)^{0.5} \quad (3-1a)$$

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$$V_t = \left(\frac{4gd(\rho_s - \rho_l)}{3\rho_l \left(\frac{18.5}{Re_p^{0.6}} \right)} \right)^{0.5} \quad (3-1b)$$

$$Re_p = \frac{\rho_l V_t d}{\mu} \quad (3-2)$$

Where ρ_s is the particle density, ρ_l is the liquid density, g is the acceleration of gravity, d is the particle diameter, and μ is the dynamic viscosity of the liquid. Table 3-2 tabulates the result of the calculation for the upper percentiles of the stainless steel procured for SSMD scaled performance testing (RPP-PLAN-52623); SSMD solids accumulation testing will use similar material.

Table 3-2: Stainless Steel Settling Velocities

Stainless steel cumulative volume fraction	Approximate stainless steel particle size (microns)	Stainless steel settling velocity (ft/s)
0.90	116	0.064 ($Re_p=0.8$)
0.95	150	0.085 ($Re_p=1.4$)
0.99	229	0.14 ($Re_p=3.4$)

The selected simulant will be used exclusively for all tests. Although using the same simulant composition is not characteristic of expected conditions during the feed delivery mission, it is preferred to keep simulant additions consistent throughout the test. This ensures that the accumulation of the fast settling solids is attributed to system performance and is not due to fluctuations in the simulant content. Furthermore, because the fast settling solids in the typical and high conceptual simulants originate from the same material, stainless steel, it would not be possible to determine whether the accumulated solids originated from either simulant type.

The typical and high conceptual simulants contain the same principle components, gibbsite, zirconium oxide, sand, and stainless steel. The differences between the two simulants are the amounts of each component in the mixture and the size distributions for gibbsite and sand. The typical conceptual simulant was developed in RPP-PLAN-51625 to have mixing and transfer behavior that are consistent with most of the Hanford tank waste; the high conceptual simulant was developed to have performance metrics that are consistent with the most challenging Hanford tank waste. Because solids accumulation will investigate repeated fills and emptying of a waste feed staging tank over the feed delivery mission, it was considered more appropriate to use

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a typical simulant rather than a simulant that was more challenging than most of the characterized Hanford tank waste. This decision process is consistent with the process used to select the simulant used in the solids accumulation scouting studies performed by SRNL. Both the SRNL work and recent SSMD testing (in process) with the typical base simulant indicate that mounds will form in the tank so that the accumulation of fast settling solids can be evaluated using the typical base simulant. Solids accumulation testing will use the typical conceptual simulant exclusively.

The solids loading is initially set to 13 weight percent (wt%). The resulting loading yields 180 g/l for a homogeneously mixed system, which is 10% lower than the action level specified in ICD-19. The actual solids concentration in the transfer line will vary from this loading because the tanks are not homogeneously mixed. The solids loading is higher than that tested in initial scouting studies at SRNL (approximately 100 g/l or 8.4 wt%), which was based on the calculated solids loading for each transfer batch from DST 241-AW-105 to the WTP (SVF-2111, , *TRANSFERS_4MINTIMESTEP(6MELTERS)-MMR-11-031-6.5-8.3R1-2011-03-18-AT-01-31-58_MI.XLSM*). The solids loading was selected to be consistent with SSMD scaled performance testing (RPP-PLAN-52623) and to also ensure that sufficient material is added to the tank to promote solids accumulation in the tank. If the stabilized size of the heel mounds are determined by the operation of the mixer jets and the properties of the simulant (i.e. the properties that affect the effective clearing radius), the mass loading would only be expected to influence the number of cycles needed for the mound to grow to the stable size. Because the mass loading in this testing is higher than the previous work at SRNL, the number of cycles needed to achieve a stable mound size may be encountered sooner than in the previous work. Additional tests with the same simulant are planned during SSMD scaled performance testing. The initial mass loading may be lowered based on the observed mound sizes in the SSMD scaled performance work. Any change will be reflected in the approved run sheets for the solids accumulation work.

3.1.1.2 Base Simulant Qualification

The critical characteristics for the base simulant materials are the particle size distribution and density of the materials. As described in PNNL-20637 and used in RPP-PLAN-51625, particle size distributions, particle density, and mass fractions of the components in the composite simulant can be used to determine the distributions of Archimedes numbers (see Equation 3-3) and jet velocities needed to achieve complete solids suspension for the composite simulant (see Equation 3-4) (Kale and Patwardhan 2005).

$$Ar = \frac{\left(\frac{\rho_s}{\rho_L} - 1\right) g d^3}{\nu^2} \quad (3-3)$$

$$U_n = \frac{\nu}{d} \left[0.13X^{0.22} Ar^{0.38} \left(\frac{D}{d_j}\right)^2 \left(1 + 0.25 \left(\frac{z}{d_j}\right)\right)^{-0.25} \left(1 + 0.75 \left(\frac{z}{D}\right)\right) \right] \quad (3-4)$$

Where U_n is the jet velocity, ν is the kinematic viscosity of the fluid, d is the diameter of the particle, X is the mass ratio of solids to liquids, Ar is the Archimedes number and is defined in

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Equation 3-3, D is the tank diameter, d_j is the jet nozzle diameter, z is the nozzle clearance above the tank bottom, ρ_s is the density of the solid, and ρ_L is the fluid density.

As discussed in PNNL-20637, the Archimedes number is closely related to the settling velocity and is also a parameter in other mixing and transfer metrics such as pump intake, jet suspension velocity, critical shear stress for erosion, critical suspension velocity, suspended particle cloud height, and pipeline critical velocity. The semi-empirical model of the jet velocity needed to achieve complete solids suspension (Equation 3-4) correlates the particle size and density to the jet velocity of a radial wall jet needed to suspend solids in a tank. Base simulant qualification is performed by comparing the distribution of Archimedes numbers and jet velocities needed to achieve complete solids suspension calculated for the procured simulants to the distributions for the recommended simulants documented in Figures 8-1 and 8-2 in RPP-PLAN-51625. To provide comparable results, performance metrics are calculated using the same assumptions used to calculate the metrics for the three conceptual simulants. Metrics are calculated using particle densities and particle size distributions obtained on samples from each procured lot. Because there is no expectation that procured material lots will not be mixed when testing is performed, particle size distributions from multiple lots of similar material may be averaged for the qualification comparisons. For commercial grade material, the particle size distribution provided by the vendor is not adequate for simulant qualification and a particle size analysis of each procured lot shall be performed. Appendix C of RPP-PLAN-51625 includes additional performance metrics, such as the settling velocity, the critical shear stress for erosion of non-cohesive particles, the just suspended impeller speed, and the pipeline critical transport velocity. The procured material will also be compared to the conceptual simulants using these metrics.

The metrics calculated for the conceptual simulants in RPP-PLAN-51625 include typical distributions for some of the components. Therefore, the calculated values represent target values and deviations from the conceptual simulants are anticipated. The appropriateness of candidate material will be evaluated before simulant procurement. For procurement purposes, in absence of samples from actual lots, vendor supplied information (e.g., particle size distributions and particle density) and targeted mass fractions can be used to calculate the performance metrics for comparison to the conceptual simulants. For simulant qualification, calculations will be based on laboratory analysis of samples taken from the procured material and actual weight measurements recorded during testing.

3.1.2 Supernatant Simulant

Developing the Newtonian supernatant composition for DNFSB 2010-2 test activities is informed from modeling Hanford waste processes. Hanford waste process modeling includes tank inventory, accounts for retrieval technologies, waste volume reduction (i.e., evaporation), and includes inventory blending during multiple tank-to-tank transfers. Therefore, an estimate for the chemical composition of each feed batch is calculated and the results are used to select a suitable supernatant density and viscosity for DNFSB 2010-2 test activities.

3.1.2.1 Supernatant Simulant Description

The supernatant simulant is the liquid phase of the simulant slurry. For WFD Mixing and Sampling Program test activities, RPP-PLAN-51625 defines the density and viscosity range for

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the supernatant simulants. These simulants are characterized by liquid density and liquid viscosity as described in Table 6-1 in RPP-PLAN-51625. Solids accumulation test activities will reproduce tank waste staging for feed to the WTP using a consistent supernatant formulation. Using the same supernatant formulation from cycle to cycle ensures that the accumulation behavior is due entirely to the mixing process and not the simulant composition. Different simulant compositions are expected to change the mixing behavior in the tank (e.g., the effective clearing radius of a jet is a function of the supernatant density and viscosity); therefore, the accumulation of solids in the tank is also expected to change with changes in the supernatant composition. In this initial work to understand the propensity to accumulate fast settling solids, a better understanding of the accumulation behavior is expected by eliminating the additional complication of changing the simulant between cycles.

Exploring solids accumulation with a supernatant that has the bounding supernatant properties provided in Table 6-1 in RPP-PLAN-51625 is not representative of the waste feed delivery mission. The bounding supernatants are limiting supernatants and were developed for testing activities that attempt to mobilize large and dense particles during limits of performance testing. Using a bounding simulant that can mobilize large and dense particles is counterproductive for studying the accumulation behavior of fast settling solids.

The typical supernatant listed in Table 3-3 is the preferred simulant for SSMD solids accumulation testing. Similar to the reason for selecting the typical base supernatant, the typical simulant was selected because testing will investigate repeated filling and emptying of a waste feed staging tank over the feed delivery mission so it was considered more appropriate to use a typical supernatant rather than a supernatant that was more or less challenging than most of the characterized Hanford tank waste. This decision process is consistent with the process used to select the supernatant used in the solids accumulation scouting studies performed by SRNL. However, SRNL solids accumulation testing also used available material with similar density and viscosity that had been prepared for other related work.

The liquid density for the typical supernatant is the median density from the unfiltered dataset used to derive the low and high density values in RPP-PLAN-51625. The dataset is the liquid density of the feed batches to the WTP calculated using the Hanford Tank Waste Operations Simulator model (RPP-RPT-48681, *Hanford Tank Waste Operations Simulator Model Data Package for the River Protection Project System Plan Rev. 6 Cases*). The unfiltered dataset does not exclude the low activity waste transfers or the high density HLW feed batches after 2040. Excluding these values, the typical supernatant has a density nearer the 85th-percentile. The typical supernatant is characterized as having a liquid density of about 1.29 g/ml and an estimated liquid viscosity of 3.3 cP. The viscosity of the supernatant simulant is determined by the salt(s) used to attain the desired density, and is comparable to the value determined using the relationship in Figure 6-2 of RPP-PLAN-51625. An aqueous solution of 31.5 wt % anhydrous sodium thiosulfate will produce a supernatant with properties similar to the targeted simulant.

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Table 3-3: Newtonian Liquid Supernatant Simulant Characteristics

Supernatant (density/viscosity)	Target Simulant Properties @ 20°C		Simulant Properties @ 20°C		Simulant Composition
	Density (g/ml)	Viscosity (cP)	Density (g/ml)	Viscosity (cP)	
Typical/Typical	1.29	3.3	1.284	3.60	31.5 wt% sodium thiosulfate

3.1.2.2 Supernatant Simulant Qualification

For the supernatant, the critical characteristics are the liquid density and liquid viscosity. To qualify the supernatant for use, the critical characteristics will be measured when the simulant batches are prepared. The simulant recipe for the supernatant simulant was developed in the laboratory, but will need to be scaled to the volume needed for each test. SSMD scaled performance testing (see RPP-PLAN-52623) uses the same supernatant and may identify an updated recipe to meet targeted conditions with the procured material. The liquid density and liquid viscosity will be measured at testing temperatures to confirm that the prepared batch is within the required range for simulant density and viscosity. Dissolution of anhydrous sodium thiosulfate is exothermic so that the temperature of the liquid increases as it is prepared. The viscosity of the supernatant decreases nearly linearly as the temperature increases from 15°C to 25°C; over this range the viscosity change is about 0.5 cP. The supernatant must be prepared to minimize viscosity variations due to significant changes in supernatant temperatures. Steps to control supernatant viscosity include temperature control, allow sufficient preparation time for ambient cooling, or mix hydrated sodium thiosulfate with anhydrous sodium thiosulfate. The dissolution of hydrated sodium thiosulfate is endothermic and results in some cooling. Preparing consistent simulant batches from test to test will facilitate the analysis of the data between tests and is expected to be more important for the data analysis than performing tests at specific conditions.

Therefore, for the typical density and typical viscosity fluids, 1.284 g/ml and 3.60 cP, respectively, the acceptable range of liquid densities and viscosities is $\pm 5\%$ and ± 0.25 cP, respectively. The supernatant will be attained using sodium thiosulfate. The two properties cannot be adjusted independently using the single component; if the two properties cannot be attained within the tolerances specified with the procured material, the supernatant will be prepared to match the target density rather than the target viscosity which was selected from a density-viscosity relationship.

The liquid property measurements will be measured on-site with the appropriate instrumentation (e.g., hydrometer, viscometer, and rheometer) calibrated, controlled, and maintained in accordance with ASME NQA-1-2004, Requirement 12 including addenda, or a later version. Supernatant viscosity will be determined using a set program that controls the shear rate to generate the rheogram. The program will include a pre-shear period and two evolutions over the shear rate range. The viscosity shall be determined on the second down curve used to generate the rheogram. Functional checks with reference standards covering the expected range of solutions used during testing shall be performed daily to ensure that the instrument is being properly maintained. Corrective actions, commensurate with the significance of an out-of-

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calibration condition, shall be performed. Appropriate instrumentation for measuring liquid viscosity of the Newtonian fluid is a programmable rheometer capable of taking controlled shear rate and controlled shear stress measurements. The rheometer shall also have the capability to control sample temperatures. Data collection shall be performed in accordance with ASME NQA-1-2004, Requirement 11, including addenda, or a later version. To ensure that the prepared simulant is appropriate for use, liquid properties will be measured prior to adding base simulant solids and therefore will be performed at the start of testing and as each new batch of simulant is prepared. In addition, viscosity will also be measured at the completion of testing, and during testing if necessary, to assess changes that may occur during the course of testing. The base solids in the samples collected during and after testing will be removed by filtering prior to collecting viscosity and density measurements.

3.1.3 Spike Particulates

A spike particulate will be included in the solids accumulation testing as a plutonium oxide surrogate. RPP-RPT-50941, *Review of Plutonium Oxide Receipts into Hanford Tank Farms*, indicates that the practical upper limit particle size for the PuO₂ and Pu metal in the transferable Hanford tank waste is 100 microns. RPP-RPT-50941 also indicates that the amount of PuO₂ and Pu metal in all of the tank waste is on the order of 10s of kilograms and is likely to be primarily PuO₂ because Pu metal fines are not thermodynamically stable in tank waste and may not have survived the extended storage time. For this reason, solids accumulation testing will include a PuO₂ surrogate and will not include a Pu metal surrogate.

The surrogate considered is a larger size of the tungsten alloy the is planned to be used as a plutonium oxide surrogate in WTP testing. The tungsten alloy has a density of 9.6 g/cm³ and particle size characteristics shown in Table 3-4. The targeted particle size distributions for the spike is a d₅₀ of 40 microns with additional particles up to 100 microns. For comparison, the WTP design basis particle size for plutonium oxide is 10 microns. The tungsten alloy is subject to the same simulant qualification process as the base simulant (see Section 3.1.1.2). The spike will replace 1 weight percent of the solids added to the tank, replacing an equivalent mass of stainless steel. Using the model of Kale and Patwardhan (2005), the jet velocity needed to suspend the tungsten alloy particles (Equation 3-4) can be used to determine the size of plutonium oxide particle that would be suspended at the same jet velocity. For two components of different densities (ρ_{S1} and ρ_{S2}), Equation 3-4 can be used to determine the sizes (d_1 and d_2) of the particles that have the same jet velocity needed to suspend the particles in the same suspending fluid and jet mixed tank. The resulting relationship is shown in Equation 3-5 and the equivalent size particles in the typical supernatant ($\rho_L = 1.284$ g/ml) are presented in Table 3-4.

$$d_1^{0.14}(\rho_{S1} - \rho_L)^{0.38} = d_2^{0.14}(\rho_{S2} - \rho_L)^{0.38} \quad (3-5)$$

A similar analysis can be performed using the free settling velocity in Equations 3-1 and 3-2. The results show that the free settling velocity of the spike particle is equivalent to the free settling velocity of a PuO₂ particle that is 90% of its own size and a Pu particle that is 67% of its own size. Similarly, the jet velocity needed to suspend the spike particle will also suspend a PuO₂ particle that is 62% of its own size and a Pu particle that is 13% of its own size. With the understanding that the fast settling particles do not need to be suspended by the jets in order to

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accumulate in the tanks, these results suggest that the spike particle with a density of 9.6 g/cm^3 is not an acceptable surrogate for the accumulation of Pu metal particles but may be acceptable as a surrogate for PuO_2 particles.

Table 3-4: Spike Particle Equivalent Settling Velocities of PuO_2 and Pu Metal

Spike particle cumulative volume fraction	Approximate spike particle size (microns)	Size of PuO_2 with equivalent velocity (microns) ^a		Size of Pu with equivalent velocity (microns) ^a	
		U_n	V_t	U_n	V_t
0.05	10	6.2	9.2	1.3	6.9
0.50	40	25	37	5.1	27
0.99	100	62	90	13	62

^a The density of spike particle used in the calculation is 9.6 g/cm^3 . The density of PuO_2 used in the calculation is 11 g/cm^3 . The density of Pu metal used in the calculation is 19 g/cm^3 . The supernatant density used in the calculation is 1.284 g/ml and the viscosity is 3.6 cP .

3.1.4 Flow Regime

When considering different scales, the flow regime among the scales must be consistent. A discussion of the flow regime for the full-scaled and SSMD tanks was presented in Section 3.1.4 of RPP-PLAN-52623. The flow regime at the inlet of the transfer pump and within the transfer lines was determined to be turbulent for all scales using the typical supernatant.

3.2 TEST EQUIPMENT AND INSTRUMENTATION

The SSMD solids accumulation activities described in this test plan will use the 1:21- and 1:8-scale tanks of the SSMD test platform (Figure 2-1) located at Monarch Machine & Tool Company, Inc. in Pasco, WA to evaluate the propensity for fast settling solids to accumulate in the feed staging tanks over the course of the waste feed delivery mission. The SSMD test platform has been used for previous test activities and will continue to be used to address uncertainties in the WFD Mixing and Sampling Program. The SSMD test platform was constructed to perform mixer jet pump testing at two different scales, approximately 1:21 (43.2-inch diameter tank) and 1:8 (120-inch diameter tank). Both tanks will be used for solids accumulation testing.

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The SSMD test platform was constructed according to scale from 241-AY-102. According to ORP-11242 Rev. 6, *River Protection Project System Plan*, tanks with riser geometries similar to 241-AW-105 will account for about 72% of the total waste volume that will be transferred to the WTP from the 13 feed staging tanks (SVF-2111). Therefore, waste loads from DST 241-AW-105 have been selected as the model tank for investigating solids accumulation. The plan view of DST 241-AW-105 is shown in Figure 3-1 (from Sheet 5 of H-14-010502, *Dome Penetration Schedules (WST/WSTA) Tank 241-AW-105*, Rev 0). The mixer jet pump locations will be maintained under the 241-AY-102 configuration but the air lift circulators will be removed. Tanks similar to 241-AW-105 do not have air lift circulators and removing these obstructions would facilitate heel volume estimations. The mixer jet pump locations in 241-AW-105 are different than 241-AY-102, the pumps are two feet closer to the center of the tank and one is offset by 5°. A comparison of the mixer jet pump and transfer pump locations between 241-AY-102 and 241-AW-105 is shown in Figure 3-2. Because the mixer jet pump locations are further away from where the mounds will form (along the perimeter of the tank at 0° and 180° in Figure 3-2), the mound size in the SSMD tanks is expected to be larger than would be observed if the mixer jet pump locations were moved to the configuration in 241-AW-105. A preliminary geometry evaluation showed that the area cleared by the mixer jet pumps differed by less than 4% over a wide clearing radius range; compared to 241-AW-105 the geometry for 241-AY-102 cleared less area for the same effective clearing radii. Based on this preliminary geometrical analysis as well as risks to cost and schedule, the construction effort required to move the mixer jet pumps was not considered warranted for the solids accumulation testing. The scaled tanks will not be modified to move the mixer jet pump locations closer to the center of the tank. The properties of the DSTs used to geometrically scale the test tanks and the scaled properties of the two-scaled tanks are provided in Table 3-5.

The main components of the test platform include: a 3,000-gallon flush tank, a 160-gallon (43.2-inch diameter) clear acrylic test tank (TK-201), a 3,900-gallon (120-inch diameter) clear acrylic test tank (TK-301), dual rotating mixer jet pump assemblies, and the slurry transfer pumps for both TK-201 and TK-301. Flow from the tanks enters the two mixer jet pump suction inlets on the bottom of the mixer jet pump, and is combined into one flow stream as it is routed through the pump driving the system. The pump discharge is split with half of the flow returning to each mixer jet pump. As each mixer jet pump is rotating, the flow is discharged back into the tank through two opposing jet nozzles located on the side of the mixer jet pump just above the pump suction inlet. Between scales, the mixer jet pump assemblies and transfer pumps for each tank are independent. The slurry transfer pumps are not submersible pumps, they are progressive cavity pumps located outside of the test tanks; the inlets of the pump are connected to 3/8-inch inner diameter suction lines that are placed within the tanks. The end of the suction lines inside each tank is fitted with a machined orifice matching the requirements in Table 3-5. The transfer pump suction inlet shall be placed consistent with the location of Riser-012. The scaled height of the pump suction inlet shall be equivalent to the height of the transfer pump inlet in the full-scale DST transfer system, which is 0.8 inches from the tank bottom in TK-301 and 0.28 inches from the tank bottom in TK-201 (see Table 3-5). Ancillary equipment, such as the support structure, the control system, video monitoring, and simulated piping to transfer and sample the material from the tank are also part of the test platform. For solids accumulation work, auxiliary mixing tanks and transfer systems are necessary to prepare fresh simulant batches that will be mixed and pumped into the tank in between each fill and empty cycle. The auxiliary tanks have a coned bottomed with a bottom discharge and are equipped with a single shaft mixer with dual

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impellers. Note that the SSMD test platform will be modified from previous tests to remove the simulated air lift circulators; DST 241-AW-105 does not have air lift circulators.

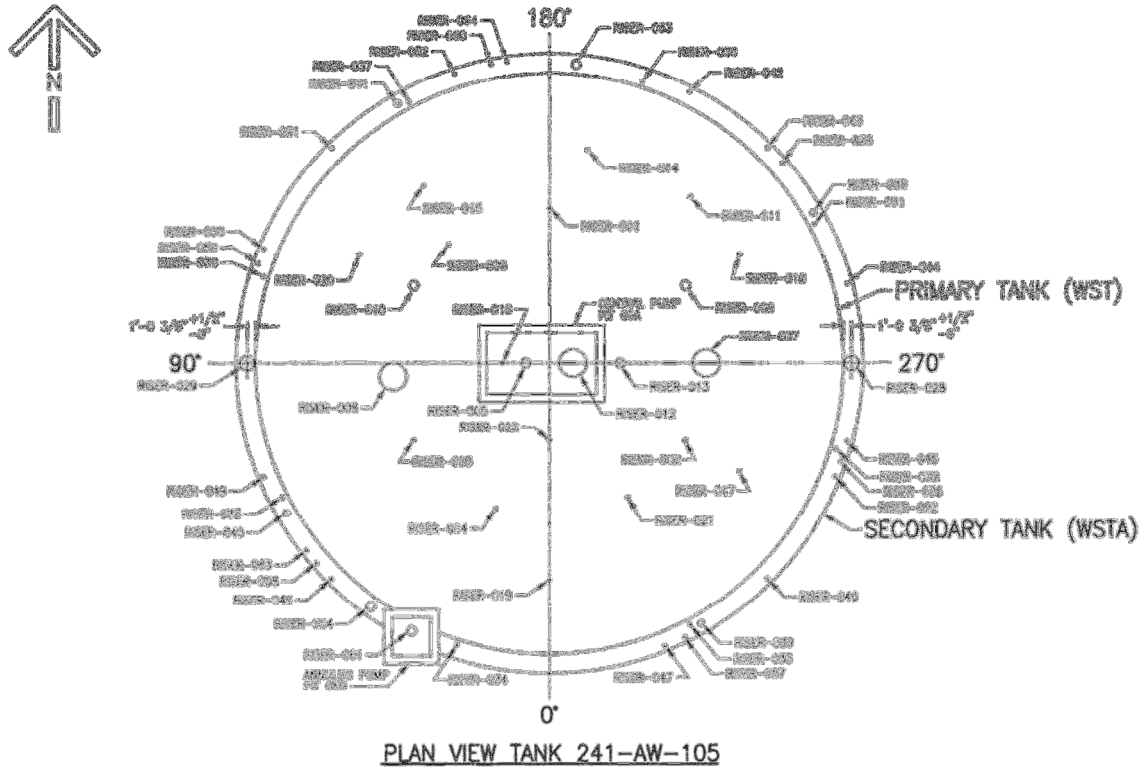
The transfer system piping, valving, and instrumentation (e.g., in-line Coriolis meters, and magnetic flow meters) will replicate the transfer system from previous SSMD testing reported in RPP-49740, *Small-Scale Mixing Demonstration Sampling and Batch Transfers Results Report*. The test configuration includes a closed recirculation loop from the tank. The recirculation loop accommodates sample collection. Flow control is automated using programmable logic controllers connected to a human-machine interface. System data, including date and time, slurry temperature, mixer jet pump rates and position, slurry flow rates, tank level, and specific gravity measurements in the transfer pump discharge, will be monitored and recorded using a data acquisition system.

The internal passageways of the mixer jet pumps driving pump and the slurry transfer pump are larger than the transfer lines; therefore, particles with a high settling velocity (e.g. stainless steel powder in the base simulant) may settle in the pump because the velocity through the pump may be reduced below the critical velocity of the particles. Modifications to the transfer system to minimize the collection of particles have been implemented and will be retained for SSMD solids accumulation unless improvements are identified. The extent that particles can collect in the transfer pump was evaluated in developmental testing for SSMD scaled performance testing so that this condition can be captured as a source of error. In addition, the slurry lines shall be purged in between campaigns to reduce the potential that settled solids from one campaign contaminate the results of a subsequent campaign. The transfer lines do not need to be purged between cycles of the same campaign because the accumulation of solids over the entire campaign is being evaluated.

When operating in a recycle mode to stabilize the mixing tank prior to performing batch transfers, the transfer line shall be discharged back into the tank. During batch transfer operations the transfer line shall be discharged for sample collection or waste collection.

All measuring and test equipment, including gauges and instrumentation, used for testing activities shall be controlled, calibrated under conditions typical of the test environment, adjusted, and maintained to required accuracy limits. The condition and the reported accuracy of each instrument shall be documented in a test log.

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Note: Mixer jet pumps will be in Riser-007 (270°) and Riser-008 (90°). Transfer pump will be in Riser-012 (270°)

Figure 3-1. Plan View Tank 241-AW-105

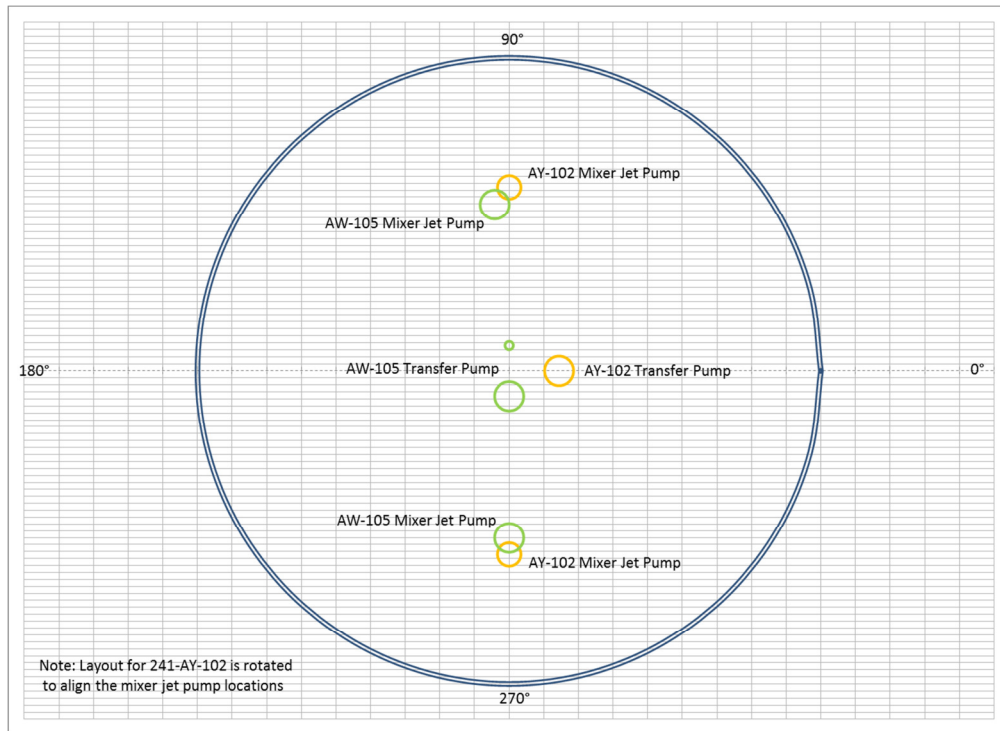


Figure 3-2: Comparison of Equipment Layout for 241-AY-102 and 241-AW-105

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Table 3-5: Small-Scale Mixing Demonstration Tank Geometrically Scaled Properties

Property	Full-Scale DST (AW-105)	1:8 Scale	1:21 Scale
Diameter (in)	900 (75 ft)	120 (10 ft)	43.2 (3.6 ft)
Scale Factor	1	0.1333	0.048
Fill Height (in)	416 (34.7 ft)	55.5 (4.63 ft)	20.0 (1.67 ft)
Transfer Batch Volume (gallons)	145,000	344	16
Bottom Geometry	Flat w/12-inch corner radius	Flat w/1.6-inch corner radius	Flat w/0.6-inch corner radius
Fill Volume ¹ (gallons)	~1,140,000	~2,700	~126
Mixer Jet Pump 1 Location ²	Riser-007 270°, 20 feet	270°, 2.9 feet	270°, 0.96 feet (12.7 in as-built)
Mixer Jet Pump 2 Location ²	Riser-003 85°, 20 feet	90°, 2.9 feet	90°, 0.96 feet (12.7 in as-built)
Mixer Jet Pump Suction Elevation ³ (in)	5±1	0.67±0.13	0.24±0.05
Mixer Jet Pump Suction Diameter (in)	11	1.47	0.53
Mixer Jet Pump Nozzle Diameter (in)	6	0.80	0.28
Mixer Jet Pump Nozzle Elevation ³ (in)	18	2.4	0.86
Mixer Jet Pump Operating Rate ⁵	10,400 gpm/MJP 59 ft/s/nozzle	95-120 gpm/MJP 30.3-38.3 ft/s/nozzle	8.3-11.8 gpm/MJP 21.6-30.8 ft/s/nozzle
Mixer Jet Rotation Rate (rpm)	0.2	See Eq. 3-6	See Eq. 3-6
Transfer Pump Location ²	Riser-012 270°, 3 feet	270°, 0.4 feet	270°, 0.14 feet
Transfer Pump Suction Inlet Diameter (in) ⁴	2.25-2.40	0.32	0.25
Transfer Pump Suction Inlet Height (in) ³	6	0.8	0.28
Transfer Line Diameter (in)	3.07 (3-inch Schedule 40)	½"-poly tubing (0.375-inch inner diameter)	½"-poly tubing (0.375-inch inner diameter)
Tank Obstructions	None	None	None

¹ Fill volume is determined by linear scaling of the tank diameter and sludge volume height.

² The reference point for DST locations presented in this table defines 0° as the bottom of 241-AW-105 in a plan view drawing of the tank. Provided distances are design distances from the center of the riser to the center of the tank. The locations of the mixer jet pumps in the scaled tanks were originally constructed to match DST 241-AY-102 and are not modified for these tests.

³ Elevation is relative to the tank bottom.

⁴ The pump suction inlet diameter of the full-scale transfer pump is underdevelopment and the tabulated value is based on similar transfer pumps used on the Hanford site to convey waste. The inlet size on the 1:21 scale tank is not geometrically scaled. The resulting inlet size was too small to accommodate the particle sizes targeted.

⁵ The mixer jet operating rates for the two scaled systems are typical operating rates used during testing. The full-scale equivalent is being investigated and is expected to be within this range.

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3.3 OPERATING PARAMETERS AND TEST METHODS

The operating conditions for the SSMD solids accumulation testing will be consistent with previous SSMD performance testing. The mixer jets shall rotate continuously clockwise with no rotational offset between mixer jet pumps; the streams will be synchronized to meet in the center of the tank. The rotational speed of the mixer jet pump (ω_{tank}) shall be set according to the mixer jet pump nozzle velocity (U_{jet}) and tank diameter (d_{tank}) in accordance with Equation 3-6, which is consistent with the relationship for scaled performance testing (RPP-PLAN-52623).

$$\omega_{\text{tank}} = \frac{(0.2 \text{ rpm})U_{\text{jet}}}{\left(\frac{d_{\text{tank}}}{900 \text{ inches}}\right) 59 \text{ ft/s}} \quad (3-6)$$

The constant values included in the Equation 3-6 are the full-scale parameters for the rotational rate (0.2 rpm), tank diameter (900 inches), and mixer jet pump nozzle velocity (59 ft/s).

Solids accumulation testing will be performed using two nozzle velocities at each scale. Each nozzle velocity will be maintained during each campaign that consists of ten fill and empty cycles. Previous solids accumulation work (SRNL-STI-2012-00508 (in process)) at SRNL tested nozzle velocities using a scale factor exponent equal to 0.33 (22.4 ft/s) and 0.29 (23.5 ft/s). The latter was determined at run time as a velocity that resulted in dead zones so that solids accumulation could be evaluated; higher velocities did not accumulate solids using simulants similar to those specified for SSMD solids accumulation testing. The appropriate nozzle velocities to use during the SSMD solids accumulation testing must result in “dead zones” within the tank. If the jet nozzle velocity is high enough to prevent build-up in the tank, then the accumulation of solids will not be adequately quantified. Similar to SRNL studies, the nozzle velocity for the first campaign in the 1:21-scale tank is selected using the equal power-per-volume scale up relationship. Based on a nozzle velocity of 59 ft/s at full scale, a tank diameter ratio of 20.8 and a scale factor exponent of 1/3, the nozzle velocity for the first campaign in the 1:21-scale system is 21.4 ft/s. Based on the 0.28-inch diameter nozzle, the flow rate per mixer jet pump is 8.25 gallons per minute (16.5 gallons per minute supplied to both pumps). The system, including simulant, will be operated at this velocity for a minimum of 30 tank rotations to ensure that a suitable mound for quantification is formed. If a suitable mound is not formed the starting nozzle velocity will need to be lowered for the first campaign. Based on SRNL testing a suitable mound is approximately 1-inch high at a peak, 15-inches long (edge to edge), and 4-inches wide in the radial direction. Larger mounds are also suitable for testing. If the mound size is adequate at 21.4 ft/s, then the effective clearing radius at this velocity will be measured and the test campaign will be conducted. If the mound size is too small, the nozzle velocity will be decreased until a suitable size mound is attained and the campaign will be run at the final setting. The nozzle velocity for the first campaign in the 1:8-scale system will be set so that the effective clearing radius is scaled proportionally from the measured value from the 1:21-scale test. If the effective clearing radius measured in the 1:21-scale test is 80% of the maximum value needed to clear the tank bottom, then the jet velocity for the 1:8-scale test would also be set so that the effective clearing radius is 80% of the maximum value. The effective clearing radius

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comparison attempts to equalize the relative dimensions of the mounds (i.e., similar performance for mound accumulation).

The second nozzle velocity will be evaluated at the time of testing to ensure that accumulation data can be collected. If the mound size from the first campaign was approximately equal to the suitable size, then the jet velocity for the second campaign would be lower than the first to ensure that a quantifiable mound is attained during testing. If the mound was greater than the suitable size then the velocity would be increased until a mound similar to the suitable size is attained. Similar to the first campaign, the effective clearing radius at the second campaign nozzle velocity in the 1:21-scale tank will be measured and used to establish the nozzle velocity for the second campaign in the 1:8-scale tank. If the mound size was close to the suitable size and the repeated volume estimates of the mound suggests that a smaller mound could also be estimated using the technique, the second campaign could target a nozzle velocity that results in a smaller mound.

Each tank in the SSMD test platform will be operated in a recirculation mode until a stable state mixing condition is established. The stable state is indicated by consistent mass flow rate and specific gravity readings from the Coriolis meter, after adjusting for cyclical variations caused by the rotating jets. Previous operating experience indicates that approximately 20-30 rotations of the mixer jet pumps are sufficient to result in a stabilized state. Once the tank reaches the stable state, the first of 6.5 batch transfers will be initiated. The batch volume for the 1:8-scale tank is 344 gallons and is the scaled volume for a 145,000 gallon transfer. Similarly, the batch volume for the 1:21-scale tank is 16 gallons. The batch volume will either be diverted to a sample collection basin (see Section 3.4) or pumped to the waste collection.

The mixer jet pump flow rate and rotational rate shall be maintained during each batch transfer but stopped for at least 20 minutes in between transfers to allow the suspended solids time to settle. Turning of the mixer jets in between transfers is consistent with the expected operation during the feed delivery mission. Developmental testing at SRNL concluded for the 1:22-scale system that the shut down duration did not significantly change the amount of material transferred when the shut down duration was extended from 20 minutes to four days. After the specified holding time, subsequent batch transfers will be initiated, repeating the holding time in between each complete transfer. During the hold time in between batches, the slurry will be recirculated through the transfer system to prevent line plugging. After each tank volume transfer (equals 6.5 batches) is completed, the tank will not be empty; a residual slurry will be left in the tank. In the full-scale tank the residual volume is equivalent to 72-inches of slurry, which is maintained to avoid cavitation when the mixer jet pumps are operating at full speed. Operation of the scaled tanks mimics the volume residual. After a full tank transfer volume is removed from the tank, the tank will contain solid mounds that are outside the area of influence of the mixer jet pumps as well as solids that were suspended in the slurry that was not removed from the tank. The residual slurry containing the suspendable solids will be pumped from the tank to expose the solid mounds after each tank volume transfer. Scouting studies at SRNL noted that the deposition of the less dense solids (i.e. gibbsite and zirconium oxide) made it difficult to delineate the mounds in the photographs. Scouting studies minimized the deposition of the suspendable solids by agitating the tank contents as the liquid was removed to expose the mounds. The mixer jets were directed towards the center of the tank, away from the mounds, and turned down to a flow rate that was sufficient to maintain a suspension of the small and less

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dense particles but not visually disturb the piles. The removed slurry will be pumped back into the tank before the next cycle. Because the inlet of the transfer pump is positioned above the tank bottom, a supplemental pump may be necessary to drain all of the free liquid from the tank to completely expose the mounds. A description and quantification of the solids remaining in tank, including a photographic or video record, will be prepared after each tank volume transfer (see Section 3.4.5). Solid samples shall be collected (see Section 3.4.4) from one of the solid mounds left in the tank after the 1st, 5th, and 10th tank volume transfers. Collecting solids samples in between tank volume transfers allows for an assessment of fast settling particle accumulation and spike particle migration into the mound as subsequent tank volume transfers are performed. Solid samples shall only be collected from the second mound after the 10th tank volume transfer. Collecting samples from the second mound only after the last cycle ensures that the solid content of the mound is not influenced by collecting the physical samples. Solid samples shall be collected with minimal disturbance to the mounds.

After information for determining the volume of the solid mounds is collected (see Section 3.4.5), the slurry removed to expose the mounds will be added back to the tank. Then a fresh batch of simulant shall be added to the tank. The volume of new simulant added to the tank returns the tank to the fill height identified in Table 3-5 and is equal to the 6.5 batch transfer volumes just removed from the tank. The fresh batch of simulant will be prepared in an auxiliary mixing tank(s) so that it can be well mixed prior to and during the transfer into the test tank. During refilling care shall be taken to prevent or minimize any disturbance of mounds left behind after the previous transfer. The transfer from an auxiliary mixing tank into the mixing tank will be similar to the DST process that is expected to add the new slurry to the center of the tank. Testing at SRNL used the fastest fill rate that did not appear to disturb the piles.

A series of transfer and refill operations shall be performed and the solids left in the tank shall be characterized prior to the start of the next tank fill (see Section 3.4.5). Solids characterization can include length, depth, and width measurements of the mounds coupled with photographs that show the mound topography. Additionally, qualitative descriptions of the residual solids will be documented to augment the photographic records. Ten successive transfer and refill operations will be performed to evaluate whether or not the mounds left in the tank continues to increase after each tank volume transfer. Preliminary results from the SRNL solids accumulation scouting studies suggest that solids may cease to accumulate after seven cycles (SRNL-STI-2012-00508 (in process)). Ten tank volume transfers represent one-half of the number of tank volume transfers that will originate from DST 241-AW-105, the tank with the greatest number of planned transfers to the WTP. Other feed staging tanks used more than a few times (>3) will participate in 7 to 13 tank volume transfers (SVF-2111).

3.4 SAMPLE COLLECTION AND CHEMICAL ANALYSIS

3.4.1 Simulant Qualification

Prior to the performing the first test of each campaign and subsequent cycles within a campaign, the simulants will be prepared and qualified. The solid particulates are qualified for use prior to testing in accordance with Section 3.1.1.2. The supernatant will be qualified on-site in accordance with requirements in Section 3.1.2.2. The first batch of simulant can be prepared in the mixing tank but subsequent batches within a campaign shall be prepared in an auxiliary tank

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so that the critical properties can be confirmed prior to mixing the new material with residual material in the tank. Once the critical properties of the supernatant are confirmed the base solids can be added. For supernatant batches prepared in the auxiliary tank, the base solids will be added to the supernatant before it is transferred to the mixing tank but after the critical properties of the supernatant are confirmed. Adding the base material to the supernatant in the auxiliary tank ensures that adding the solids to the slurry does not adversely affect the accumulation of material in the tank.

3.4.2 Pre-Transfer Samples

Prior to conducting the first batch transfer, the tank contents are mixed at the operating conditions until mixing in the tank has stabilized. During tank stabilization, the transfer pump is engaged so that the specific gravity of the transferrable slurry can be monitored by a Coriolis meter located downstream of the transfer pump. During tank stabilization the transfer pump discharge is re-circulated back into the tank. Monitoring the mass flow rate and slurry specific gravity will allow an assessment of the systems capability to mix and convey the complex simulant. Once the system has stabilized, two pre-transfer samples have historically been collected to provide a basis that subsequent transfer batches have content similar to the pre-transfer samples that are used to certify the batch for delivery. For solids accumulation activities pre-transfer samples will not be collected. Scaled performance testing performed according to RPP-PLAN-52623 evaluates the adequacy of the pre-transfer sample to characterize each transfer batch.

3.4.3 Batch Transfer Samples

Prior to conducting the first batch transfer, the tank contents are mixed at the operating conditions until mixing in the tank has stabilized. Once the tank contents are stabilized, batch transfers are initiated and slurry samples for each transfer batch, including each half-batch transfer, are collected for chemical analysis. Similar to previous work, batch transfer samples will be diversion samples through sample ports whose valves are programmatically controlled and correlated to the position of the mixer jet nozzles using encoders. Samples shall be collected downstream of the transfer pump. Batch transfer samples shall be collected in a manner that avoids bias. To avoid bias introduced by flow dynamics around the sample port, the full stream will be diverted to collect the samples. To avoid bias caused by the cyclical nature of the mixing system that directs the jet directly at the transfer pump twice per revolution, the transfer samples shall be collected for an integer value of full rotations of the mixer jets. Samples for the 1:21-scale tank shall collect the entire volume of the transfer batch (16 gallons) and this volume shall be sub-sampled for chemical analysis (see Section 3.4.6). For the 1:8-scale system, only part of the transfer batch will be collected for sampling. For the 1:8-scale system, the slurry will be diverted into a single collection basin during four regularly spaced intervals during each transfer. The four slurry samples are combined to form a representative sample for the entire transfer batch that will subsequently be subsampled. The duration for collecting the four diversion samples will be equivalent and will be equal to the duration for an integer value of mixer jet full rotations. For the half batch transfer, the interval between collections is shorter, but the collected volume is the same. Because the mixer jet pumps rotate at different speeds for the two different nozzle velocities considered, the sample duration and hence volume of material collected during sampling varies between tests. The total volume of the slurry sample collected during a transfer

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for the 1:8-scale system will be similar to the full transfer batch volume for the 1:21-scale system (16 gallons). The collected sample will be approximately 4.7% of the 344 gallon transfer batch. The mass and volume of the collected material for the batch transfer samples shall be measured and recorded. The collected volume is then sub-sampled for chemical analysis (see Section 3.4.6).

3.4.4 Heel Samples

After the 6.5 batches have been withdrawn from the tank, the tank will contain residual material composed of the solid mounds in the dead zones as well as residual slurry that is not pumped from the tank. The suspended material in the slurry will settle throughout the tank if sufficient time is provided to clarify the fluid. After the 1st, 5th and 10th tank volume transfer in a campaign, core samples will be collected from the residual tank solids. Scouting studies at SRNL developed a core sampling technique that was successful at removing cores if the free liquid in the tank was removed prior to sampling the mounds. A schematic of the core sampler is provided in Appendix A. A similar device and one modified for the increased height of the 1:8-scale tank and expected greater depth of the heel mounds, will be used to collect the core samples. After the first tank volume transfer, core samples shall be collected from the largest of the two mounds. After the fifth tank volume transfer, core samples shall be collected from the same mound sampled after the first tank volume transfer. Because holes left in the mounds are filled with solids deposited after each cycle, samples collected from the mounds in subsequent cycles (i.e., the fifth and tenth) must not overlap previous sample locations. The number and locations of the samples collected for the first and fifth cycles must account for the need to sample the mounds in subsequent cycles. In addition, the number of samples collected after the first and fifth cycles must not remove more than five percent of the mound. Because there is no need to keep the mound intact after the tenth cycle, the largest number of samples will be collected after the tenth cycle. Sample locations can be marked on the bottom of the transparent tank when the core sampler is inserted to collect each core. All core sample location coordinates must be recorded with each sample identification number so that a map of the fast settling solids can be prepared from the sample results. After the final tank volume transfer, core samples shall be collected from both mounds. After each campaign (i.e., ten tank volume transfers), the core location markings shall be removed from the tank bottom.

The number of samples collected from each mound depends on the size of the mound. Core sample locations will include locations to characterize the center of the mounds. SRNL scouting studies anticipated that heel growth would occur by the deposition of fast settling solids on the edges of the mounds but found that the greatest concentration of fast settling solids occurs in the center of the mounds. Core samples shall be withdrawn from the mounds without disturbing the neighboring material. SRNL demonstrated that the mounds could be core sampled without disrupting the integrity of the mounds if the liquid level was lowered to expose the mounds. Core samples will be collected in a pattern that resembles the mound (e.g. triangular). It is expected that 3 to 10 samples will be sufficient to assess how the fast settling solids are distributed throughout the mounds (evenly distributed versus concentrated at the center or the edges). For the smallest mounds two to three samples shall be collected from the mound near the tank wall and one shall be collected from the mound towards the center of the tank. For larger mounds this pattern will be followed expanding the number of tank wall samples to three or four depending on the size of the mound. At the end of the campaign, two samples from the interior

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of the tank shall also be collected to characterize the suspended material that settles in the tank once the final batch transfer is completed and the mixer jet and transfer pumps are turned off.

The collected samples may be subdivided into segments. Collecting analytical data from different segments of the core allows for some vertical discretization of the heel. At a minimum each core taken from the 1:21-scale tank that is longer than 0.5 inches in length will be subdivided so that the presence of the spike component in the bottom half inch of the mounds can be determined. Similarly, each core taken from the 1:8-scale tank that is longer than 1 inch in length will be subdivided so that the presence of the spike component in the bottom inch of the mounds can be determined. With the exception of the bottom segment, which is 0.5 inches long in the 1:21-scale tank and 1-inch long in the 1:8-scale tank, the length of any additional discretization will be based on visual observation of layering in the sample cores. To reduce the number of analytical samples submitted to the laboratory, core samples will only be divided into more than two segments if layering is evident. An example of layering is shown in Figure A-3 in Appendix A. The minimum length of any segment is determined by the analytical laboratories sample volume requirements and the depth of the mounds. The length of the segment, or entire core if segments are not collected, will be recorded so that coarse vertical partitioning of the fast settling solids can be mapped. The segments will be placed into separate containers, individually labeled, and shipped off-site for chemical analysis in accordance with requirements in Section 3.4.6.

3.4.5 Heel Volume Measurement

Scouting studies at SRNL successfully demonstrated two techniques for estimating the heel volume (SRNL-STI-2012-00508 (in process)). Both techniques required that the liquid level in the tank be lowered to expose the solids. One method successfully demonstrated used an automated positioning system and laser depth finder to measure the depth from a known reference elevation to the surface of the mound. The x-, y- positioning was computer controlled. The height of the mound at each position was determined by subtracting the distance measurement to the surface of the mound from the reference elevation used to establish the distance. The x,y,z measurements were plotted in MS Excel to create three dimensional maps of the mounds. An area was computed for each measurement location. Areas closer to tank wall were larger than more central areas. Each measurement of mound height was multiplied by its associated area to give an increment of volume. Increments of volume were summed to obtain the mound volume. The measurement uncertainty for this technique is (preliminarily) estimated to be 7%.

A photographic technique was also demonstrated at SRNL. For the photographic technique a camera was setup at a stationary point ten feet above the tank. In addition, a hand held camera was available. Enough agitation was applied to suspend most of the gibbsite but not enough to disturb sand and stainless steel. Most of the gibbsite suspension was pumped from the tank and then the remainder was drained before any measurements were taken. The goal was to limit the deposition of gibbsite on the mounds. Arrow shaped boards marked with N and S and a dial indicator initially indicating zero tank level were placed in the tank to identify the north and south mounds and the fact that the tank was nominally empty. The entire tank was photographed using the overhead camera and then the handheld camera was used to obtain a closer image of each mound. Then the liquid level in the tank was increased in increments by adding the withdrawn fluid back into the tank. At each new tank level the dial indicators were reset and

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photographs were taken. This process was repeated until both mounds were submerged. Later, the photographs were analyzed to determine the shoreline of the mounds and the area within the shoreline for each tank level. The shapes were superimposed using marker devices placed into the tank for alignment and a topographic map was formed. Mound volume was calculated by integrating area (square inches) with height (inches) to give volume (cubic inches). The initial method used to perform the integration was Simpson's Rule. However, that method requires that all level increments be equal. Later, the Trapezoidal Method was used which is less accurate but more flexible. The measurement uncertainty for this technique is (preliminarily) estimated to be 20% for large mounds and more for smaller mounds. Volume estimates between the laser measurement technique and the photographic technique were within 20% for the larger observed pile but were not as accurate for the smaller pile because the liquid height increment used to establish the shore line approximated the height of the mound and therefore good resolution of the mounds could not be established.

SRNL scouting studies demonstrated the viability of each technique, although the laser measurement technique was reported to be more accurate. For the larger of the two mounds, SRNL reported that the accuracy of the laser technique was $\pm 7\%$ compared to $\pm 20\%$ for the photographic technique. In addition, the photographic technique required much more labor and analysis after the information was collected. Adoption of one or both of these techniques for SSMD solids accumulation testing will consider the most efficient use of resources (budget and schedule).

If only the photographic technique is adopted to estimate the heel, a check on the accuracy of the technique can be performed if the volume of fluid added back into the tank to raise the liquid level is measured and recorded and the resulting liquid level is also measured and recorded. The volume of both mounds can be determined from the difference between the expected liquid level increase for the volume of fluid added to an empty tank and the observed liquid level increase. If the mounds were fully submerged at the end of the last transfer and the free liquid was drained from the tank, it will be assumed that the pores remained saturated when calculating the volume displaced by the solids.

After the final transfer of each campaign in the 1:21-scale tank, the information necessary to characterize the volume of the mound will be collected as described above and then the entire contents of each mound will be removed from the tank and weighed. The contents or a representative sample of the contents will then be rinsed to remove the supernatant, dried, and weighed to determine the total solids content in the mounds. The mound boundaries will be obscured by the suspended solids that settled when the mixer jet pumps were turned off. The criteria used to delineate the mound boundaries (e.g., edge height that is equivalent to the height of the settled solids in the center of the tank) must be consistent across scales and campaigns. The dried contents will be homogenized and two samples will be collected to characterize the component speciation of the mounds (see Section 3.4.6). Subsequently, the remaining mass in the tanks will be removed and the dried mass of rinsed solids determined. Because of the much larger anticipated size of the piles in the 1:8-scale system, it may not be practical to replicate this process in its entirety for the larger tank. Performing a total tank solids characterization after the final transfer of the campaign for the 1:8-scale tank will be reevaluated when the total volume of the mounds left in the tank is understood.

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3.4.6 Chemical Analysis

Prior to the start of testing, analytical method development shall be performed to determine the sample preparation error associated with measuring the base material content in the presence of the supernatant rheology modifiers. This will be done for SSMD scaled performance testing and is applicable to SSMD solids accumulation testing that uses similar simulants. The analytical method is considered acceptable if it produces an unbiased result with a relative standard deviation of less than 10%.

The collected volume from each batch transfer sample will exceed the amount practical for laboratory analysis and will be subsampled at the test platform. For batch transfers, the collected volume representing each transfer batch will be settled in a large volume container. In previous testing, the collected material was clarified for 24 hours in a mixer barrel prior to decanting the liquid. This method will be refined during SSMD scaled performance testing to ensure that the subsamples can be collected in a reasonable amount of time and be representative of the content of the composited material. The mass and volume of the slurry will be recorded. The liquid will be decanted. If solids are present in the decanted liquid, the decanted liquid will be homogenized and sampled. The collected sample will be weighed and filtered to remove the collected solids. The solids will be rinsed to remove any supernatant residue, dried, and weighed to determine the weight percent solids decanted with the liquid. For mass balance purposes the solids captured in the decanted liquid will be assumed to slow settling small gibbsite. The weight percent solids in the decant will be multiplied by the mass of the decanted liquid to determine the mass of decanted solids in the slurry sample.

After decanting, the wetted solids will be mixed in a rotating mixer barrel prior to sub-sampling. Four representative and an equal number of archive samples will be collected randomly from the solids. The four wet solid subsamples of the batch transfer samples and core sampler segments (i.e., heel samples) will be shipped off-site for laboratory analysis; the four archive samples for the batch transfer will be retained on-site in a managed area to prevent a loss of sample integrity. Archive samples will be analyzed if the analytical samples become lost or damaged or if additional analysis is determined to be necessary. The samples will be analyzed for the weight percent of dry solids and the weight percent of each primary constituent (gibbsite, zirconium oxide, silica sand, and stainless steel) in the dry solids. The analytical laboratory will receive the samples, weigh the samples, filter the solids from the liquid, rinse sodium thiosulfate from the filtrate, dry the solids, weigh the dried solids, and then subsample the material for analysis. Portions of the subsample(s) will be digested using EPA Method 3052, *Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices*. Sample content of aluminum, chromium, iron, nickel, and zirconium will be determined using EPA Method 6010C, *Inductively Coupled Plasma – Atomic Emission Spectrometry*. The mass concentration of gibbsite in each subsample is determined from aluminum results; the mass concentration of stainless steel is determined from the chromium, iron, and nickel results; the mass concentration of zirconium oxide is determined from the zirconium results. Portions of the subsample(s) will also be totally digested using the fusion procedure of ASTM D4698, *Standard Practice for Total Digestion of Sediment Samples for Chemical Analysis of Various Metals*. Silicon content for quantifying the sands present in the digested samples will be determined using EPA Method 6010C. In preliminary work reported by the laboratory, ASTM D4658 yielded better results for sand than EPA Method 3052. A compatible method to analyze the spike component is under development.

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Off-site analytical services will be performed by a laboratory that operates under a Quality Assurance program that has been evaluated against quality requirements in ASME NQA-1-2004 including addenda, or a later version. Analytical data is required to be enhanced quality so that all sample collection, sample analysis, sample handling, and data reporting shall be traceable to the test performed. The sample results shall be reported in a Microsoft Excel¹ compatible format.

The sample results, including weight percent dry solids and weight percent of each simulant component in the dried solids will be used along with mass balance expressions to evaluate the mass balance on the tank system (see Section 3.4.7 and Appendix B).

3.4.7 Mass Balance

The mass balance equations for the solids in tank, expressed in terms of the data that will be collected during the test is describe in Appendix B and summarized here. The mass of each component added to the tank is recorded as it is added to the tank. The mass flow rate, volumetric flow rate and specific gravity of the material withdrawn from the tank during each transfer is also recorded in one second increments during each transfer. The mass flow rate data or volumetric flow rate data and specific gravity data can be integrated to determine the total mass transferred from the tank. Adjusting for the supernatant density, the mass of solids transferred from the tank can be determined. The mass of each component transferred from the tank in each transfer batch can be estimated from the chemical composition data for each transfer batch once the mass of transferred solids in each batch is determined. Compiling all the batches yields an estimate for the mass of each component withdrawn from the tank. The difference between the amount of each component added during a campaign and the amount withdrawn from the tank during each campaign yields an estimate for the amount of material left in the tank.

After each campaign, the mass of residual solids in the mounds in the 1:21-scale tank will be dried and then measured once it is emptied from the tank. The rest of the material from the tank will be added to the dried material and it can be subsampled for analysis (Section 3.4.6) to determine the mass of each component left in the tank. However, for the 1:8-scale tank the mass in the tank could be between 2000 and 4000 pounds. Accurately drying, weighing, and homogenizing such a large volume of material to collect a representative sample of the solids may not be practical to close the mass balance. Therefore, testing in the 1:8-scale tank will rely on the difference between added material and removed material to calculate the material remaining in the tank. The error in the estimate for the mass of each component transferred may only yield a gross approximation for the residual mass of each component left in the tank. The error in the mass of the material removed is derived from integrating the mass flow rate readings reported every second from the coriolis meter. The uncertainty for the mass flow rate reading from the coriolis meter is $\pm 1\%$ and is largely attributed to the uncertainty in the data acquisition system reporting the values. Calculating the speciation of the mass transferred introduces additional errors. The analytical measurements for each transfer batch have analytical uncertainties on the order of $\pm 10\%$, which must be propagated for the 70 sequential transfers (six full transfers and one half transfer for each of ten cycles). Propagating the uncertainty results in a speciation uncertainty of about 83%. There is additional unknown uncertainty pertaining to

¹ MS Excel® is a registered trademark of the Microsoft Corporation, Redmond, WA.

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how well the analyzed samples represent the entire transfer batch. Therefore, in order to provide as much insight into the content of the mounds, more core samples will be taken after the tenth cycle than after the first or fifth cycles (see Section 3.4.4). In addition, qualitative descriptions of the mounds will be made after the tenth cycle is completed. The mound will be sliced radially several times to expose the interior of the mound. At a minimum, the radial slices will divide the mound into four sections, including a slice down the apparent center of the mound. Observations of layering or an uneven distribution of solids in the mound will be documented and captured in still photographs. Photographic records of horizontal slices of the mounds, also in several inch increments, will also be taken and compared.

3.4.8 Other Performance Data

In addition to collecting slurry samples for chemical analysis, other performance data will be collected. Each system in the SSMD test platform has the capability to record operational parameters such as test time, slurry temperature, mixer jet pump flow rate, mixer jet angular position, mixer jet pump rotational rate, tank level, slurry transfer rate, and slurry specific gravity. This data is recorded by a data acquisition system and shall record data for the entire test duration. In addition, performance data shall also be recorded in the test log during testing. Performance data describing the dimensions of any accumulated material in the tank shall be collected throughout the test, noting specifically when changes in tank stability occur due to a change or process interruption. In addition, cloud height and effective clearing radius measurements shall also be recorded in the test log. The effective clearing radius can be determined while the mixer jets are running by measuring the distance from the edge of the mixer jet pump nozzle to the edge of the pile of solids that has stabilized on the sides of the tank. Multiple measurements shall be collected in each test to determine an average effective clearing radius. Measurements shall be collected for each batch transfer to support an evaluation of changes in the system as the tank level is lowered.

3.4.9 Solids Accumulation Analysis

Once the analytical data for all of the collected samples is analyzed the performance of the system will be evaluated. The mass of each component transferred from the tank will be calculated and compared to the amount of material added to the tank. After the campaign is completed, the difference will be the estimate for the material that is left in the tank at the end of the campaign. In the 1:21-scale system, this estimate will be compared to the heel solid measurements collected at the end of the campaign. The distribution of mass using this heel solids estimate will be compared to the initial distribution of mass in the simulant to compare how effectively fast settling solids are removed from the tank relative to slow settling solids. In addition, the change in the amounts of each component transferred from batch to batch will be evaluated for changes between cycles. If the mass of a particular component transferred in sequential cycles is constant and equal to the amount added for each cycle, the solid is not being continuously accumulated in the tank.

Additionally, the volume of the solids mounds will be calculated and plotted as a function of transfer cycle to determine if and when the volume of the mound stabilizes. This analysis will assume that the pore volume in the mounds is stable. The point at which the mounds appear to stabilize will be compared across scales as well as nozzle velocities. If the mounds stabilize similarly among the two scales, it can be inferred that a full-scale DST operated under similar

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conditions would stabilize similarly. Without a scale up relationship for mound accumulation, there will be no basis to state that the planned operating conditions of the full-scale system will stabilize similarly. However, if the performance at two different nozzle velocities is similar, it can be inferred that the mound stabilization behavior may not be strongly dependent on the operating conditions and hence the scale-up relationship.

Also, the mass fraction of each fast settling solids (i.e., stainless steel, sand, and tungsten alloy) in the core samples will be mapped according to the x,y sample location in the mound and segment height if a coarse vertical discretization of the cores samples was obtained. Plotting the fraction of fast settling solids in collected core samples as a function of its location and overlaid with a mound profile would show how the fast settling solids are spatially distributed in the mounds. Solid content collected from adjacent sample locations but from different cycles of the same campaign will be compared to determine if the fraction of fast settling solids in the heel increases as subsequent cycles are performed. Increasing content of the fast settling solids after subsequent cycles is indicative of solids accumulation but is only indicative of a change if the size of the cores that are compared were the same. The conclusions on solids accumulation will be compared across scales and nozzle velocities. If similar accumulation behavior is observed, then it can be inferred that a full-scale DST operated under similar conditions would accumulate solids similarly. Without a scale up relationship for mound accumulation, there will be no basis to state that the planned operating conditions of the full-scale system will accumulate solids similarly. However, if the performance at two different nozzle velocities is similar, it can be inferred that the accumulation behavior may not be strongly dependent on the operating conditions and hence the scale-up relationship.

Finally, the mass fraction of the spike particle (i.e., tungsten alloy) in the bottom segment of the core samples will be mapped according to the x,y sample location if a coarse vertical discretization of the cores samples was obtained. The tungsten alloy was added after the initial cycle so that a mound of fast settling solids was already present in the tank when the tungsten alloy was added; therefore, the tungsten alloy could not be at the bottom of the mound as a result of initial deposition. The presence of the tungsten alloy at the bottom of a mound would indicate that the most dense particle added to the tank migrates to the bottom of the mound over the course of multiple fill and empty cycles and could become concentrated at the bottom of the waste feed staging tank. The absence of the spike particle at the bottom of the mound would suggest that the fast settling solids are deposited in the pile and mixing under similar conditions is inadequate to disturb the center of the piles enough to allow concentration of particles added to the tank in subsequent cycles. The mass content of the other components in the sample segment would need to be taken into consideration to ensure that, if present at the bottom center of the mound, the spike particle was not deposited into an open core hole from the previous cycle. The conclusions on spike particle migration through the mound will be compared across scales and nozzle velocities. If similar migration behavior is observed, then it can be inferred that a full-scale DST operated under similar conditions would concentrate solids similarly. Without a scale up relationship for mound accumulation, there will be no basis to state that the planned operating conditions of the full-scale system will concentrate solids similarly. However, if the performance at two different nozzle velocities is similar, it can be inferred that the concentration behavior may not be strongly dependent on the operating conditions and hence the scale-up relationship.

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4.0 TEST COORDINATION

All testing equipment operations are performed by trained and qualified subcontracted personnel under the supervision of a Test Director. An operations plan, including test run sheets, will be prepared that describes the precautions and limitations, the testing sequences, testing prerequisites, startup conditions, and test procedures in stepwise detail. The TOC technical representative(s) must concur with the operations plan. The Test Director coordinates testing activities including ensuring that all test conditions required for the startup of testing have been performed and all test records (e.g., Test Log, Test Deficiency Reports, Test Change Requests, etc.) are maintained. The Test Director is also responsible for coordinating test activities with the Quality Assurance representative to ensure testing is performed in accordance with the approved quality assurance plan. While tests are conducted, the Test Director will also determine which changes do not adversely affect the acceptance criteria and/or methods by which the acceptance criteria are to be accomplished and are considered “inconsequential” or “minor” and approve these test changes. All other changes require concurrence with the TOC technical representative(s) before the change(s) is/are implemented.

4.1 PRECAUTIONS AND LIMITATIONS

The Job Hazards Analysis is the process for identifying, evaluating, controlling, and communicating potential hazards associated with the work being performed, including modifications to test facilities and test equipment. SSMD solids accumulation testing is being performed in a test facility constructed to perform the testing. The test facility is governed by a facility specific Job Hazards Analysis documented in a Job Hazards Analysis checklist or equivalent document. Changing conditions that modify the test facility or equipment to accommodate testing will be evaluated in a revision to the Job Hazards Analysis before the modifications to the facility or equipment are performed. Workers performing work in the test facility governed by the Job Hazards Analysis shall review the documented hazards and acknowledge that they understand the hazards associated with the work being performed and will abide by controls (e.g., don required personal protective equipment, obey posted signs and placards) put in place to mitigate or eliminate the hazards.

Any special precautions that must be taken or test limitations will be documented in the operations plan specifically prepared for each activity and will be communicated to workers before the start of work during a Pre-Job briefing.

4.2 SEQUENCE OF TESTING, PLANT CONDITIONS, SPECIAL EQUIPMENT

Any special requirements for the testing sequence, plant conditions (including connecting to site utilities and site restoration), or special equipment that are not identified in Section 3.0 will be documented in the operations plan specifically prepared for each activity.

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5.0 DATA COLLECTION AND TEST RESULTS REPORTING

Testing shall be conducted in accordance with an approved operations plan. The approved SSMD data collection and accuracy plan that was updated for all DNFSB 2010-2 work scope (PL-SSMD-EG-0003, *Waste Feed Delivery Small Scale Mixing Demonstration Data Collection and Accuracy Plan Rev. 2*) is applicable for solids accumulation work at the SSMD test platform. The data collection and accuracy plan shall be updated as necessary if on-going analytical development work indicates that the analytical uncertainty information previously provided is out of date or if additional instrumentation is necessary to perform tasks identified in this test plan. All test activities shall be performed according to test run sheets. All major testing activities shall be documented in a test log. Test deficiencies shall be reported in a Test Deficiency record.

Test data identified in Section 3.0 , including test durations and test conditions, shall be recorded in the test log. Applicable data not recorded by a data acquisition system shall be recorded on the run sheet or recorded in the test log. All electronic data collected by a data acquisition system shall be content reviewed for error and anomalies. Electronic records shall be submitted to the TOC for evaluation.

All laboratory analysis results shall be accompanied by a chain of custody report that was prepared when the samples were collected. The chain of custody shall identify the samples by a unique name, describe the sample type and list the analyses to be performed. The chain of custody shall also document the preparers name and shall acknowledge receipt at the analytical laboratory. All laboratory analysis results shall be submitted to the TOC technical representative in an MS Excel compatible format.

A test result report shall be prepared this test activity. SSMD solids accumulation test activities shall be documented in a test data package that is submitted to the TOC by *EnergySolutions*. The TOC shall perform the required analysis and document the findings in a test report that is reviewed by *EnergySolutions*.

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APPENDIX A. SRNL SOLIDS ACCUMULATION STUDIES CORE SAMPLER

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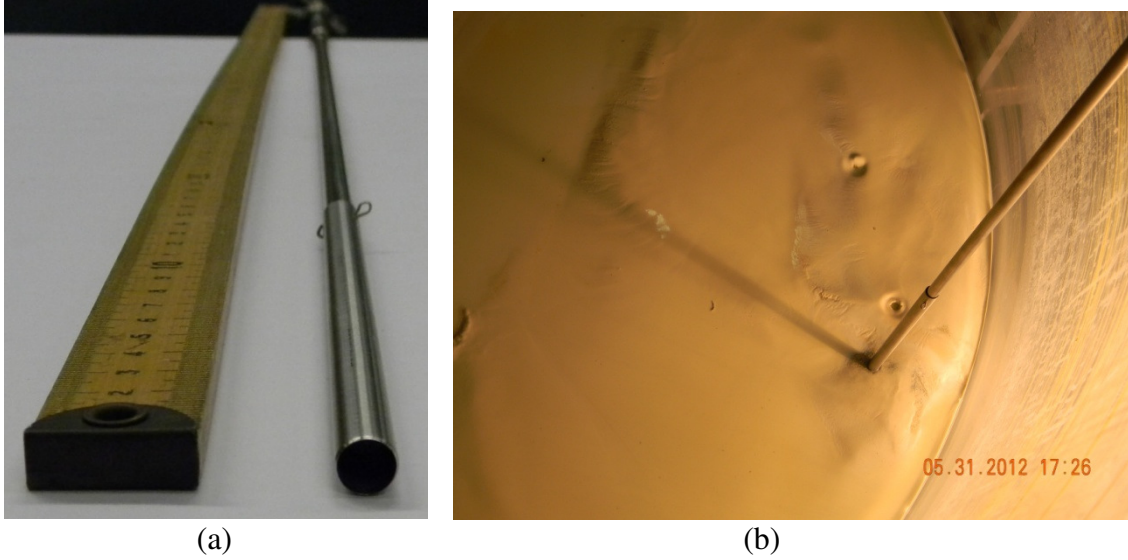


Figure A-2. Core Sampling: (a) The core end of the sampler, (b) a core being extracted during Cycle 1 of Campaign 1



Figure A-3. Campaign 2, core sample 20-2 taken from the North mound after Cycle 10

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Appendix B. **MASS BALANCE**

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This following derivation describes the mass balance for the Solids Accumulation task.

Nomenclature:

M, m: mass ρ : density V: volume X: mass fraction Q: Volumetric flow rate

\dot{M} : Mass Flow Rate

Superscripts:

IN: Mass initially added to the mixing tank

OUT: Mass transferred from the mixing tank

HEEL: Mass that is not transferred from the mixing tank after transfers are completed

SLURRY: The slurry that is transferred in a batch

DIVERSION: A transfer sample is collected by diverting the flow into a collection basin. This is the diversion sample.

DECANT: The diversion sample is decanted and then subsampled. Decant refers to the decanted fluid after it is poured from the collection basin.

WS: (shortened for wet solids) The diversion sample is decanted and then subsampled. Wet solids refers to the residual in the collection basin after it is decanted.

Subscripts:

i: component (gibbsite, silica sand, stainless steel, or zirconium oxide)

L: shortened for liquid/supernatant

S: shortened for solids

Mass balance on initial tank contents:

The initial mass of material added to the tank is the sum of the mass of the supernatant and the mass of each dried component (gibbsite, silica sand, stainless steel, or zirconium oxide). The mass of the supernatant is determined by the measured density and fill volume. The fill volume is determined by the tank radius, r_{TANK} , and the fill height, h_L^{IN} . The mass of each component added to the tank, M_i^{IN} , is measured before it is added to the tank.

$$M^{IN} = M_L^{IN} + M_S^{IN} = \rho_L V_L^{IN} + \sum M_i^{IN} = \rho_L (\pi r_{TANK}^2 h_L^{IN}) + \sum M_i^{IN} \quad B-1$$

Mass balance on transferred slurry in one batch:

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In the following discussion, multiple samples will be taken and averaged to represent the sampled quantity. For simplicity, the discussion is presented as if only a single sample is collected. Slurry will be transferred from the tank in successive batches. The mass fraction of each component in a dried solid sample collected during the batch transfer will be determined by an analytical laboratory. In addition, the mass fraction of solids and liquid in the transferred slurry will also be determined from a sample collected during the batch transfer. The mass fractions will be applied to the entire transfer to determine the amount of each component transferred in the batch.

The transferred slurry will contain supernatant and solids. The mass of the transferred slurry is the sum of the mass of supernatant and the solids.

$$M^{OUT} = M_L^{OUT} + M_S^{OUT} \quad \text{B-2}$$

The mass of the transferred slurry, M^{OUT} , will be measured. In the 1:21-scale system the mass of the slurry transferred is weighed directly. In the 1:8-scale system the mass of the slurry transferred is determined using the average specific gravity of the transferred slurry, $\bar{\rho}_{SLURRY}$, and the volumetric flow rate, Q_{SLURRY}^{OUT} , which are determined from the data reported in one second increments, Δt , by the Coriolis meter. The mass flow rate, \dot{M}_{SLURRY}^{OUT} , could also be used to determine the mass transferred.

$$M^{OUT} = \sum \bar{\rho}_{SLURRY} Q_{SLURRY}^{OUT} \Delta t = \sum \dot{M}_{SLURRY}^{OUT} \Delta t \quad \text{B-3}$$

The transferred slurry is collected for characterization by diverting the flow to a collection basin. For the 1:21-scale system, 100% the transferred material is collected in the diversion sample. For the 1:8-scale system, a similar volume to the 1:21-scale transfer batch is diverted to a collection basin during the transfer. The volume is approximately 4.5% of the full 1:8-scale transfer batch. One-fourth of the required volume is collected for each of four evenly spaced intervals during the transfer. The diversion sample is weighed, $M^{DIVERSION}$. The ratio of the mass of the full transfer batch and the diversion sample is the diversion ratio, $f_{DIVERSION}$.

$$f_{DIVERSION} = \frac{M^{OUT}}{M^{DIVERSION}} \quad \text{B-4}$$

For the 1:21-scale system the diversion ratio is 1 (i.e., the full transfer batch is collected as the diversion sample) and for the 1:8-scale system the diversion ratio is about 22.

The diversion sample is too wet and too large for analysis and is clarified and then the liquid is decanted. Both the decanted liquid and settled solids are weighed, homogenized and subsampled. In order to determine the mass fraction of each component transferred, the subsamples are sent to the analytical laboratory for characterization. The decanted liquid is

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weighed, M^{DECANT} . The mass of the wet solids, M^{WS} , is measured or determined as the difference between the mass of the collected volume, $M^{DIVERSION}$, and the mass of the decanted liquid, M^{DECANT} . It is expected that both the decanted solution and wet solids will both contain solids and supernatant. The solids are exclusively the target components, gibbsite, silica sand, stainless steel, or zirconium oxide. Dissolved components added to attain the correct supernatant density and viscosity dissolve and are rinsed from the solid samples prior to weighing the solids. Additionally, the target analytes are insoluble and do not dissolve in the liquid.

$$M^{DIVERSION} = M^{DECANT} + M^{WS} = M_S^{DECANT} + M_L^{DECANT} + M_S^{WS} + M_L^{WS} \quad B-5$$

In order to determine the mass of solids in the decant of the diversion sample, M_S^{DECANT} , the decanted solution is homogenized and a subsample is collected and weighed, m^{DECANT} . Note the lower case 'm' is used to denote a mass quantity for a subsample. The weighed subsample is filtered to collect the solids. The filtrate is rinsed (to remove any precipitated sodium thiosulfate) and dried in order to determine the mass of solids in the subsample of the decant sample, m_S^{DECANT} . The mass fraction of solids in the subsample (and decant solution if the solution was homogeneously mixed when the subsample was collected) is the ratio of mass of dried solids in the subsample and the wet weight of the subsample.

$$x_S^{DECANT} = \frac{m_S^{DECANT}}{m^{DECANT}} \quad B-6$$

The mass of solids in the decanted solution of the diversion sample is the product of the mass fraction of solids in the decant subsample and the mass of the decanted solution.

$$M_S^{DECANT} = x_S^{DECANT} M^{DECANT} \quad B-7$$

If adequate time is allowed for settling and a good decanting technique is applied, M_S^{DECANT} can be assumed to be very slow settling, small gibbsite and may be negligible. If not negligible the solids in the decant is assumed to be all gibbsite, so that the fraction of component i in the decant solution, x_i^{DECANT} , is one for gibbsite and zero for each of the other components.

$$M_i^{DECANT} = x_i^{DECANT} M_S^{DECANT} = x_i^{DECANT} x_S^{DECANT} M^{DECANT} \quad B-8$$

In order to determine the solids content in the wet solids of the diversion sample after the clarified solution has been decanted, a subsample of the homogenized wet solids is collected and weighed, m^{WS} . The wet subsample is then rinsed to remove sodium thiosulfate, dried and weighed, m_S^{WS} . The solids content of the wet solids, x_S^{WS} , is the ratio of the two measurements.

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$$x_S^{WS} = \frac{m_S^{WS}}{m^{WS}} \quad \text{B-9}$$

The collected subsample of the wet solids from the diversion sample is analyzed for chemical content. The mass fraction of each component i in the solids, x_i^{WS} , is determined by the analytical laboratory.

The mass of solids transferred and retained in the wet solids of the diversion sample is the product of the solids content of the wet solids, determined using the subsample, and the mass of wet solids in the diversion sample.

$$M_S^{WS} = x_S^{WS} M^{WS} \quad \text{B-10}$$

The mass of each solid component in the solids transferred and retained in the wet solids is the product of the mass of solids retained in the wet solids and the mass fraction of each component in the solids.

$$M_i^{WS} = x_i^{WS} M_S^{WS} = x_i^{WS} x_S^{WS} M^{WS} \quad \text{B-11}$$

The mass of each component i in the diversion sample is the sum of the mass of each component in the wet solids and the decanted solution.

$$M_i^{DIVERSION} = M_i^{DECANT} + M_i^{WS} = x_i^{DECANT} x_S^{DECANT} M^{DECANT} + x_i^{WS} x_S^{WS} M^{WS} \quad \text{B-12}$$

The mass of each component i transferred from the tank in the batch is the product of the diversion ratio and the mass of each component in the diversion sample.

$$M_i^{OUT} = f_{DIVERSION} M_i^{DIVERSION} \quad \text{B-13}$$

Mass balance on subsequent transfers in a cycle:

The process is repeated for each transfer batch in a cycle. The transferred masses are added together to get the entire mass transferred during a cycle.

$$M_{CYCLE,i}^{OUT} = \sum_{Batch=1}^{6.5} M_i^{OUT} \quad \text{B-14}$$

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Mass balance on subsequent additions for additional cycles in a campaign of ten cycles:

For subsequent cycles mass additions are made. Equation B-1 applies except that the volume of supernatant added, V_L^{IN} , is determined by direct measurement as it is added to the tank. The total amount of each component added during a campaign is the sum of each amount added during each cycle of the campaign.

$$M_{TOTAL,i}^{IN} = \sum_{Cycle=1}^{10} M_i^{IN} \quad B-15$$

Mass balance on subsequent subtractions for additional cycles in a campaign of ten cycles:

For subsequent cycles mass transfers are made. Equation B-14 applies and is additive. The total amount of each component transferred during a campaign is the sum of each amount transferred during each cycle of the campaign.

$$M_{TOTAL,i}^{OUT} = \sum_{Cycle=1}^{10} M_{CYCLE,i}^{OUT} \quad B-16$$

Mass Balance on tank heel contents:

After the last transfer is completed, the tank is not empty. The last transfer does not remove all of the liquid from the tank, a certain heel amount, equal to 72 inches of slurry in the full-scale double shell tank, is not removed from the tank. The slurry will contain suspended solids while the mixer jets are running. The suspended solids will settle, coating the bottom of the tank and any mounds of solids that are not influenced by the mixer jets. Together these solids comprise the heel solids left in the tank. Because there is no chemical reactions occurring for any of the analytes, the theoretical mass of each component left in the heel is the difference between the total mass of the component added during the campaign and the amount transferred out of the tank.

$$M_i^{HEEL} = M_{TOTAL,i}^{IN} - M_{TOTAL,i}^{OUT} \quad B-17$$

Core samples will be collected from the heel mounds to characterize the solid content of the mounds. However, earlier work suggests that the composition of the heel mounds is not uniform, the center of the mounds contain more fast settling particles than the edges. Therefore, it is not expected that the core samples will be adequate to estimate the heel content and close the mass balance.



FROM THE DESK OF

Raymond J. Skwarek
Manager, One System IPT

Date: September 24, 2012 WRPS-1203839-OS

To: L. M. Peurrung, Chair
Large-Scale Integrated Mixing System Expert Review Team

Subject: ONE SYSTEM TECHNICAL TEAM RESPONSE TO REVIEW OF WASTE
FEED DELIVERY MIXING AND SAMPLING PROGRAM SOLIDS
ACCUMULATION TEST PLAN (ERT-20)

The One System Technical team appreciates the Large-Scale Integrated Mixing System Expert Review Team (ERT) review (Enclosure 1) of the subject document. We also appreciate the opportunity the One System Technical team had to address the ERT questions prior to issuing the ERT formal review letter. This helped put this phase of testing in the proper perspective which we believe you have appropriately characterized in your review letter, “... *the stated test objective – to “evaluate the propensity” of solids to accumulate – is relatively qualitative and exploratory. The intent seems to be to “get a feel for” the maximum jet nozzle velocities that leave some solids unmixed at each scale, how quickly the mounds build up, how much of each fast-settling solid component they contain, and whether that mound composition seems to be changing from cycle to cycle.*” We have modified the test plan to clarify this perspective.

This response letter addresses the three specific technical subjects identified by the ERT, followed by the One System response.

1. *“The ERT recommends that the One System team first establish the scaling behavior of effective clearing radius, either from the scaled/system performance test data or as a precursor to this test. Since mounds accumulate where clearing does not occur, accumulation behavior likely scales in roughly the same way. The ERT recommends basing the velocity selections at the two scales on this scaling relationship – or, alternatively, trying to match the dimensionless footprint of the mounds at the two scales. In any case, the ERT recommends that the document better describe how the results of testing will be used to predict or evaluate full-scale behavior.”*

The One System Technical team acknowledges that the scaling relationship for solids accumulation has not been explored or established. The Savannah River National Laboratory scouting studies represent the first data points from testing at only one scale. We agree with your recommendation that setting the initial jet velocities on a performance measure that can be observed and set to be equivalent in both tanks is an appropriate method. The test plan has been modified to set the initial velocities based on cleaning radius observations collected prior to the start of formal testing. Selecting velocities that result in similar cleaning behavior is expected to

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result in similar accumulation behavior between scales. If test results show similar accumulation behavior, then confidence in predicting full-scale accumulation behavior is improved.

- 2. "If mounds form quickly, then estimating their size only after 1, 5, and 10 cycles is insufficient. The ERT recommends taking enough data to resolve the heel mound growth curve and determine when mounds reach their maximum size. Based on the scouting studies, additional measurement(s) would be needed between the 1st and 5th cycles. Data could be taken after each cycle until the test director determines that growth has stopped."*

The One System Technical team agrees with your recommendation that heel mound growth should be measured after each pump-down cycle. This will allow more precise determination of when the mound sizes stabilize. The test plan has been modified to include mound size measurement after each pump-down cycle.

- 3. "If the simulant components are indistinguishable in each cycle, it is impossible to determine whether the solids that accumulated in the mounds are fixed in place once settled or whether there is intermixing, settling, or enrichment from the heavier solids from cycle to cycle. If layers are not apparent in the core samples, the ERT recommends that One System consider changing the color of the simulant sand (for example) as a way of qualitatively assessing whether solids in mounds are mobile or immobile."*

While the original objectives of the solids accumulation testing did not include the level of experimental detail or analytical rigor necessary to track simulant components based on the batch they were originally introduced to the tank, the One System Technical team agrees this would provide additional useful information. The ERT recommendation to introduce colored simulant based on its sequential introduction batch is a novel concept but one that we feel presents significant results analysis challenges because of the large volumes of multi-component simulants that will be collected. We believe tracking simulants by batch introduction is a candidate for a simpler scouting study that should be considered for future work as part of the discussions during the upcoming results workshop. However, we also believe that a simplified concept of tracking migration of fast settling particles over time can be included in the upcoming tests by introducing a unique, fast-settling spike in the third and later batches of introduced simulant. Our initial consideration for a spike is to use a material similar in characteristics to the Tungsten alloy simulant component used by the WTP as a fissile material surrogate during Computational Fluid Dynamics modeling Verification and Validation testing. Final acceptance of the selected spike will be dependent on analytic methods capability confirmation. Analysis of different vertical sections of the core samples should show the tendency of this spike to distribute within the mounds over time. While this approach does not capture the full intent of the ERT's recommendation, it does allow for an initial exploration of the concept within the scope, cost, and schedule constraints of the planned solids accumulation testing.

In addition to the specific responses highlighted above, the One System Technical team has reviewed the ERT document suggestions provided on a separate document review record and modified the DNFSB commitment document. The updated draft document (Enclosure 2) incorporating comments received from all reviewers, and the disposition of the ERT individual review comments (Enclosure 3) are included for your information. Please note that we have

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added the additional ERT comments provided via e-mail on September 18, 2012, to the bottom of the ERT consolidated comment list (Enclosure 3).

Please feel free to contact me at 372-9117, or Mike Thien at 372-3665 if you have any further questions regarding our response to the ERT review.

Sincerely,



R. J. Skwarek, Manager
One System Integrated Project Team

MGT:MEH

- Enclosure(s):
1. ERT-20 Review Letter (3 pages)
 2. RPP-PLAN-53193, Rev. C, Draft, "One System Waste Feed Delivery Mixing and Sampling Program Solids Accumulation Test Plan" (52 pages)
 3. LSIMS ERT Document Review Record (11 pages)

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Enclosure 1

ERT-20 Feed Test Plan 3

Large-Scale Integrated Mixing System Expert Review Team

(L. Peurrung, Chair; R. Calabrese, R. Grenville, E. Hansen, R. Hemrajani)

To: Tom Fletcher, Tank Farms Federal Project Director; Michael D. Johnson, WRPS President and Project Manager, Tank Operations Contract

Cc: Ray Skwarek, One System IPT Manager; Rick Kacich, One System IPT Deputy Manager; Mike Thien, WRPS; ERT Members

Subject: One System Waste Feed Delivery Mixing and Sampling Program Solids Accumulation Test Plan (ERT-20)

Date: September 4, 2012

The Large-Scale Integrated Mixing System Expert Review Team (ERT) was asked to review “One System Waste Feed Delivery Mixing and Sampling Program Solids Accumulation Test Plan” (RPP-PLAN-53193, Rev A). This document is the third of three meant to satisfy Commitment 5.5.3.6 in the Implementation Plan for DNFSB Recommendation 2010-2, “Test Plan to establish Tank Farm performance capability.” Per the commitment, WRPS will “conduct testing to determine the range of waste physical properties that can be retrieved and transferred to WTP and determine the capability of tank farm staging tank sampling systems to provide samples that will characterize waste and determine compliance with the [Waste Acceptance Criteria]. This work will include development of a test plan.” This third test plan focuses specifically on solids accumulation testing using the 1:21 and 1:8 Small Scale Mixing Demonstration (SSMD) test platforms. The objective of testing, per the document, is “to evaluate the propensity for the waste feed delivery baseline design to accumulate fast settling solids in the feed staging tanks.” To aid its review, the ERT had the benefit of the draft (Rev D) of report SRNL-STI-2012-00508 providing the results of scouting studies of the same nature performed by Savannah River National Laboratory. The ERT appreciates access to this draft information.

As in the review of the first test plan document, the lines of inquiry for the ERT’s review were:

- Are the major points of the document communicated well to the intended audience?
- Does the document provide a clear set of test objectives and requirements?
- Are the proposed approaches to testing sufficiently defined and technically defensible?
- Is simulant selection appropriate? Does the document meet its intent of “qualifying” the simulants proposed?

The ERT first observes that measurement uncertainties in the techniques for sample analysis and heel volume estimation (about 10%) are such that it will be difficult to make firm conclusions about mass balance and whether and when solid particles are no longer

ERT-20 Feed Test Plan 3

accumulating in the test vessel. If the heels are not to be fully characterized but are only sampled to indicate relative amounts of solid constituents, then it will also be difficult to quantify the amount of fast settling solids that remain in the feed tank after ten cycles. As such, results will be qualitative or semi-quantitative rather than fully quantitative. That said, the stated test objective – to “evaluate the propensity” of solids to accumulate – is relatively qualitative and exploratory. The intent seems to be to “get a feel for” the maximum jet nozzle velocities that leave some solids unmixed at each scale, how quickly the mounds build up, how much of each fast-settling solid component they contain, and whether that mound composition seems to be changing from cycle to cycle. Performing these tests at two scales likewise gives a feel for whether any of those aspects change with scale. However, we don’t actually know yet how accumulation scales with vessel size, making selection of jet nozzle velocities at the two scales somewhat arbitrary and calling into question whether comparisons of mounds at the two scales are truly “apples to apples”. The ERT recommends that the OneSystem team first establish the scaling behavior of effective clearing radius, either from the scaled/system performance test data or as a precursor to this test. Since mounds accumulate where clearing does not occur, accumulation behavior likely scales in roughly the same way. The ERT recommends basing the velocity selections at the two scales on this scaling relationship – or, alternatively, trying to match the dimensionless footprint of the mounds at the two scales. In any case, the ERT recommends that the document better describe how the results of testing will be used to predict or evaluate full-scale behavior.

The ERT observes that gaining insight into the mechanisms of mound formation and evolution from cycle to cycle would be an important outcome from these tests. In the SRNL scouting studies, the mounds seemed to form rather quickly (at least for some conditions) and then stay the same size. The core samples show bottom segments rich in stainless steel particles with lighter solids toward the top. It is difficult to tell from the SRNL data whether the overall stainless steel fraction in the heel increases from cycle to cycle. The core sample photos could not be used to determine if layering is apparent in the cores. The ERT recommends the following for assessing the mound:

- If mounds form quickly, then estimating their size only after 1, 5, and 10 cycles is insufficient. The ERT recommends taking enough data to resolve the heel mound growth curve and determine when mounds reach their maximum size. Based on the scouting studies, additional measurement(s) would be needed between the 1st and 5th cycles. Data could be taken after each cycle until the test director determines that growth has stopped.
- If the simulant components are indistinguishable in each cycle, it is impossible to determine whether the solids that accumulated in the mounds are fixed in place once settled or whether there is intermixing, settling, or enrichment from the heavier solids from cycle to cycle. If layers are not apparent in the core samples, the ERT recommends that OneSystem consider changing the color of the simulant sand (for example) as a way of qualitatively assessing whether solids in mounds are mobile or immobile.

Comments from individual ERT members are attached. The ERT hopes you find this review helpful, and we look forward to your response per the ERT Charter.

ERT-20 Feed Test Plan 3

Review Participants:

August 27, 2012: Loni Peurrung, Richard Grenville, Erich Hansen, Ramesh Hemrajani

August 30, 2012: Loni Peurrung, Rich Calabrese, Richard Grenville, Erich Hansen, Ramesh Hemrajani, Mike Thien, Pat Lee

August 31, 2012: Loni Peurrung, Rich Calabrese, Erich Hansen, Ramesh Hemrajani

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Enclosure 2

RPP-PLAN-53193, Rev. C

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One System Waste Feed Delivery Mixing and Sampling Program Solids Accumulation Test Plan

KP Lee
Washington River Protections Solutions, LLC

Richland, WA 99352
U.S. Department of Energy Contract DE-AC27-08RV14800

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Key Words: One System, Tank Farm Mixing and Sampling, Waste Feed Delivery, DNFSB Recommendation 2010-2, Solids Accumulation, Small Scale Mixing Demonstration

Abstract: This plan addresses the technical approach and test requirements for the Small-Scale Mixing Demonstration Solids Accumulation test activity being performed under the Mixing and Sampling Program to support waste feed delivery to the Hanford Waste Treatment and Immobilization Plant. Using a simulant that is typical of Hanford tank waste, testing will evaluate the propensity for fast settling solids to accumulate in the waste feed staging tanks as multiple fill and empty cycles deliver feed to the Hanford waste treatment plant.

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Release Approval

Date

Release Stamp

Approved For Public Release

RPP-PLAN-53193, Rev. C

EXECUTIVE SUMMARY

The primary purpose of the Tank Operations Contractor Mixing and Sampling Program is to mitigate the technical risks associated with the ability of the tank farms waste feed delivery systems to mix and sample High-Level Waste feed adequately to meet the Hanford Waste Treatment and Immobilization Plant Waste Acceptance Criteria. In a series of testing activities the Tank Operations Contractor will conduct tests to determine the range of waste physical properties that can be retrieved and transferred. Using two geometrically scaled tanks, testing and analysis will determine the propensity for the waste feed delivery mixing and transfer system to accumulate fast settling solids in the feed staging tanks. This test plan is the third of three test plan documents that are being prepared to address Defense Nuclear Facilities Safety Board DNFSB 2010-2, Sub-Recommendation 5, Commitment 5.5.3.6, "Test Plan to establish Tank Farm performance capability" and addresses the technical approach and test requirements for the Small-Scale Mixing Demonstration solids accumulation test activities being performed to support waste feed delivery. The solids accumulation tests are patterned after the duty cycle for double shell tank 241-AW-105, which is planned to have the greatest number of transfers to the Hanford waste treatment plant (ORP-11242 Rev. 6, *River Protection Project System Plan*).

The tests being conducted to define the capabilities of the mixing, sampling, and transfer system are focused on three areas: limits of performance, solids accumulation, and scaled/system performance. Solids accumulation testing at two scales is described in this test plan. Solids accumulation testing is exploratory and is being conducted to understand the potential to concentrate dense fissile material in a waste feed staging tank that is subjected to repeated waste feed fill and empty cycles. Solids accumulation work will demonstrate mixing, sampling, and transfer performance using simulants representing a typical Hanford waste. Testing will be performed with base particulate solids in a Newtonian suspending fluid that are characteristic of Hanford waste in terms of bulk particle density, particle size, solids loading, supernatant density, supernatant viscosity, and slurry density. The slurry will contain dense particles (8 g/cm^3) having particle sizes exceeding 100-microns for assessing the propensity to accumulate fast settling solids in the waste feed staging tanks. A tungsten alloy powder with a particle density of approximately 9.6 g/cm^3 will be included in the simulant beginning with the third fill and empty cycle. The potential to concentrate fissile material in the tank will be evaluated with this spike particle. Core samples will be taken from the mounds to determine if the spike component migrates to the bottom of the mounds during subsequent fill and empty cycles. In addition, the spike particles will also be used to determine the capability of the system to transfer fast settling spike particles for comparisons to waste feed characterization requirements for uranium (U) and plutonium (Pu) and to requirements for waste treatment processability; (e.g., Pu and U unwashed solids concentration). These tests will use the Small-Scale Mixing Demonstration test platforms used in previous Waste Feed Delivery Mixing and Sampling Program test activities; however, the operating conditions and simulants tested will be expanded to collect additional performance data.

For the test activity covered in this test plan, the test objectives along with success criteria are identified. The necessary equipment to conduct the tests and collect the necessary data is identified and described. This work is follow-on work to the solids accumulation scouting

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studies performed at the Savannah River National Laboratory where measurement techniques and parameter sensitivity were first investigated (RPP-PLAN-52005). The simulants that are appropriate for testing are identified and qualified in accordance with the recommendations in RPP-PLAN-51625, *Waste Feed Delivery Mixing and Sampling Program Simulant Definition for Tank Farm Performance Testing*. The most important properties identified for solids accumulation work include variations to: mixer jet nozzle velocity and the sequential fill and empty cycles that simulate the multiple uses of the waste feed staging tanks during the waste feed delivery mission.

Small-Scale Mixing Demonstration solids accumulation testing will be conducted to:

- Use fast settling base particulate and spike solids in a Newtonian supernatant to determine at small scale how fast settling solids are spatially distributed in mounds left in a waste feed staging tank after the feed has been delivered.
- Evaluate how fast settling solids could be spatially distributed in a full-scale double-shell tank.
- Explore if fast settling spike particles can be concentrated at the bottom of full-scale double-shell tank.
- Evaluate the reliability of the collected data for predicting full-scale performance when the scaling relationship is uncertain.

Mixing, transfer, and heel accumulation data at two scales will be collected and analyzed to determine if the fast settling solids accumulate in the tank after ten fill and empty cycles are performed. The first fill cycle fills an empty tank with the waste simulant. The first transfer cycle uses the mixer jet pumps to mix the tank contents at one of two nozzle velocities and a pump to transfer material from the tank in 6.5 sequential transfer batches. Batch transfer samples are collected to quantify the amount of material transferred. After the final transfer of the first cycle, it is expected that there will be mounds of solids that accumulate along the perimeter and on the bottom of the tank in the area that is outside the area of influence of the two mixer jet pumps. Heel samples will be collected from these solids and the volume of solids in the mounds will be estimated. The tank is then filled to volume with additional, fresh simulant made to the same composition as the first cycle. Care will be taken when refilling the tank with fresh simulant so that the solid piles that accumulated in the tank are not disturbed. The process is repeated, estimating the volume of the solid mounds after each tank volume transfer (i.e., 6.5 transfer batches). Beginning with the third fill cycle a fraction of the fast settling solids will be replaced with a higher density spike solid that is chemically different from the other simulant components. The fill and empty cycles are repeated until ten cycles are completed. Heel samples are collected from the mounds after the first, fifth and tenth tank volume transfer. The spatial distribution of fast settling solids in the heel is determined by comparing component concentrations in the mound from the known sample locations. In the deepest parts of the mounds, the collected samples will be segmented to capture coarse vertical partitioning. The results will be mapped to show where the fast settling solids tend to accumulate in the tank. In addition, the potential to concentrate dense fissile material on the bottom of a mound will be evaluated by noting whether the spike particulate, which is added after the initial mounds are

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formed, is found at the bottom of a mound. Two campaigns of ten fill and empty cycles will be performed at each scale. The composition of the simulant used will be the same in all tests but different nozzle velocities will be set for each campaign. For the first campaign in the 1:21-scale tank the nozzle velocity will be set at the equal power per volume scaling condition. For the first campaign in the 1:8-scale tank the nozzle velocity will be set so that similarly proportioned mounds are attained. The nozzle velocity for the second campaign in the 1:21-scale system will be determined based on the performance in the first campaign. Similar to the first campaign, the nozzle velocity for the second campaign in the 1:8-scale tank will be set to match the mound proportions from the second campaign in the 1:21-scale tank. Therefore, twenty tests will be conducted in the 1:21 and 1:8 scale mixing tanks in the Small-Scale Mixing Demonstration test platform.

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TERMS

Abbreviations and Acronyms

ASME	American Society of Mechanical Engineers
BNI	Bechtel National, Inc.
DOE	U.S. Department of Energy
DNFSB	Defense Nuclear Facilities Safety Board
DST	double-shell tank
DQO	data quality objective
HLW	high-level waste
ICD	Interface Control Document
MDT	SRNL mixing demonstration tank
ORP	Office of River Protection
Pu	plutonium
PNNL	Pacific Northwest National Laboratory
RPP	River Protection Project
RSD	Remote sampler Demonstration
SRNL	Savannah River National Laboratory
SSMD	Small-Scale Mixing Demonstration
TOC	Tank Operations Contractor
U	uranium
WAC	waste acceptance criteria
WFD	Waste Feed Delivery
WRPS	Washington River Protection Solutions, LLC
WTP	Hanford Waste Treatment and Immobilization Plant

Units

°C	degrees Celsius
cP	centipoise
ft	feet
in	inch
g	gram
gpm	gallons per minute
l	liter
Hz	hertz
MHz	megahertz
ml	milliliter
Pa	Pascal
s	second

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1.0 INTRODUCTION

The Office of River Protection (ORP) has defined the interface between the two prime River Protection Project (RPP) contractors, Bechtel National, Inc. (BNI) and Washington River Protection Solutions (WRPS), in a series of interface control documents (ICDs). The primary waste interface document is 24590-WTP-ICD-MG-01-019, *ICD-19-Interface Control Document for Waste Feed* (also known as ICD-19). Section 2.3 of ICD-19 states, that the Tank Operations Contractor (TOC) baseline sampling plans and capabilities are not currently compatible with WTP sample and analysis requirements.

The primary purpose of the TOC Waste Feed Delivery (WFD) Mixing and Sampling Program is to mitigate the technical risks associated with the ability of the tank farms feed delivery systems to adequately mix and sample High Level Waste (HLW) feed to meet the Hanford Waste Treatment and Immobilization Plant (WTP) Waste Acceptance Criteria (WAC). Initial work for the WFD Mixing and Sampling Program demonstrated that the concept functionality for the first feed tank to deliver consistent feed delivery batches was viable. However, uncertainties related to scale-up, simulant representativeness, data uncertainty, optimizing system performance, applicability to all feed tanks, feed conditioning, and understanding emerging WTP solids handling risks still need to be addressed. The TOC has identified two critical risks TOC-12-64 and TOC-12-65 per the TFC-PLAN-39, Rev. G, *Risk Management Plan*, which address sampling methods and emerging changes to WAC requirements. The root of the mixing and sampling risk is the ability to collect samples that are characteristic of the tank waste, including the rapidly settling solids in the HLW for the purpose of demonstrating compliance with the WTP waste acceptance requirements. In addition, in November 2011, the U.S. Department of Energy (DOE) issued the implementation plan for the Defense Nuclear Facility Safety Board (DNFSB) Recommendation 2010-2 (DOE Rec. 2010-2, Rev. 0, *Implementation Plan for Defense Nuclear Safety Board Recommendation 2010-2*), which addresses safety concerns associated with the ability of the WTP to mix, sample, and transfer fast settling particles. DOE developed the implementation plan to resolve these issues and a related TOC issue concerning the capability of the mixing and transfer system to adequately mix the tanks to minimize the buildup of waste solids in the waste feed staging tanks that are re-used during the feed delivery mission.

Through multiple test activities, the TOC will determine the range of waste physical properties that can be retrieved and transferred to WTP, and determine the capability of tank farm staging tank sampling systems to provide samples that will characterize the tank waste to determine compliance with the WAC. These tests will reduce the technical risk associated with the overall mixing, sampling, and transferring of HLW feed to WTP so that all WAC requirements are met. Report RPP-PLAN-41807, *Waste Feed Delivery Mixing and Sampling Program Plan and Test Requirements* defined the three test requirements for continued WFD Mixing and Sampling Program testing to address DNFSB concerns. In accordance with DNFSB 2010-2 Sub-Recommendation Commitment 5.5.3.6, "Test Plan to establish Tank Farm performance capability", test plans are prepared to further refine testing requirements as follows:

- Limits of performance - determine the range of waste physical properties that can be mixed, sampled, and transported under varying modes of operation. These tests will use both the Remote Sampler Demonstration (RSD) platform and the Small-Scale Mixing

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Demonstration (SSMD) platform. In addition, a test using a full-scale slurry transfer pump will be performed. Specific test requirements and additional details for the limits of performance testing activities are documented in RPP-PLAN-52005, *One System Waste Feed Delivery Mixing and Sampling Program Limits of Performance and Solids Accumulation Scouting Studies Test Plan*.

- Solids accumulation - perform scaled testing to understand the accumulation and spatial distribution of the remaining solids in a double-shell tank (DST) during multiple fill, mix, and transfer operations that are typical of the HLW feed delivery mission. These tests include scouting activities at the Savannah River National Laboratory (SRNL) Mixing Demonstration Tank (MDT) and the SSMD platform. Specific test requirements and additional details for the SRNL solids accumulation testing activities are documented in RPP-PLAN-52005. Draft SRNL test results and recommendations used to develop this test plan are documented in SRNL-STI-2012-00508, *Solids Accumulation Scouting Studies* (in process). Specific test requirements and additional details for the SSMD solids accumulation testing activities are documented in this test plan.
- Scaled/system performance - demonstrate mixing, sampling, and transfer performance using a realistic simulant representing a broad spectrum of Hanford waste to meet WTP WAC Data Quality Objectives (DQO) sampling confidence requirements. These tests will use both the SSMD and the RSD platforms. The RSD platform is full scale; therefore, RSD system performance testing activities will collect additional system performance data at full scale. Specific test requirements and additional details for the SSMD scaled performance and RSD system performance testing activities are documented in RPP-PLAN-52623, *One System Waste Feed Delivery Mixing and Sampling Program System Performance Test Plan*.

A TOC simulant plan, RPP-PLAN-51625, *Waste Feed Delivery Mixing and Sampling Program Simulant Definition for Tank Farm Performance Testing*, was developed to define the simulant objectives for this testing. Simulants were developed to represent the complete range of physical properties for the broader spectrum of Hanford waste tanks, and to address specific testing requirements summarized above. This represents a broadening of objectives from earlier SSMD and RSD testing. The simulants and operating conditions in this earlier testing were intended to simulate the particle size, density distribution, and operating configuration of Hanford DST 241-AY-102, the first tank waste to be delivered to WTP. The particle size distribution for the SSMD simulant for DST 241-AY-102 (1% is 0.39 microns, 50% is 13.2 microns, 95% is 200 microns, and 99% is 394 microns) is documented in PNNL-20637, *Comparison of Waste Feed Delivery Small-Scale Mixing Demonstration Simulant to Hanford Waste*. The range of particle sizes in the simulant was smaller than the particle size distribution for the 95% confidence limit for 95% of the population (1% is 2 microns, 50% is 22 microns, 95% is 460 microns, and 99% is 700 microns) used in the waste feed transfer system analysis used in the WTP design basis, RPP-9805, *Values of Particle Size, Particle Density, and Slurry Viscosity to Use in Waste Feed Delivery Transfer System Analysis*.

This test plan is the third of three test plan documents prepared to address DNFSB 2010-2 Sub-Recommendation Commitment 5.5.3.6, “Test Plan to establish Tank Farm performance capability”. This test plan identifies and describes the test objectives, test requirements, and test methods for the SSMD Solids Accumulation test activities. This work is follow-on work to the

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solids accumulation scouting studies performed at the Savannah River National Laboratory where measurement techniques and parameter sensitivity were first investigated (RPP-PLAN-52005). The testing approach is guided by this previous work as well as by input from internal subject matter experts and external consultants familiar with the objectives of the test program. The original discussions held to develop the testing approach are described in WRPS-1105293, *Small-Scale Mixing Demonstration Optimization Workshop Meeting Minutes* and are refined in WRPS-1201374-OS, *One System DNFSB 2010-2 Sub-Recommendation 5 Test Plan Summit Meeting Minutes*. The current scope addresses the buildup of solids in the tanks after multiple tank refills and the changes to the composition and spatial distribution of the solids in the piles over time. The current scope will not address any operational improvement options that evaluate how to re-suspend the dead zones. The current scope will also not address reduced pump performance or how an extended outage may cause the rheology of the waste to change over time. Operational improvements to minimize solids accumulation and re-suspend the dead zones are planned for Fiscal Year 2013. Future testing to evaluate rheology changes remains a consideration for future testing activities.

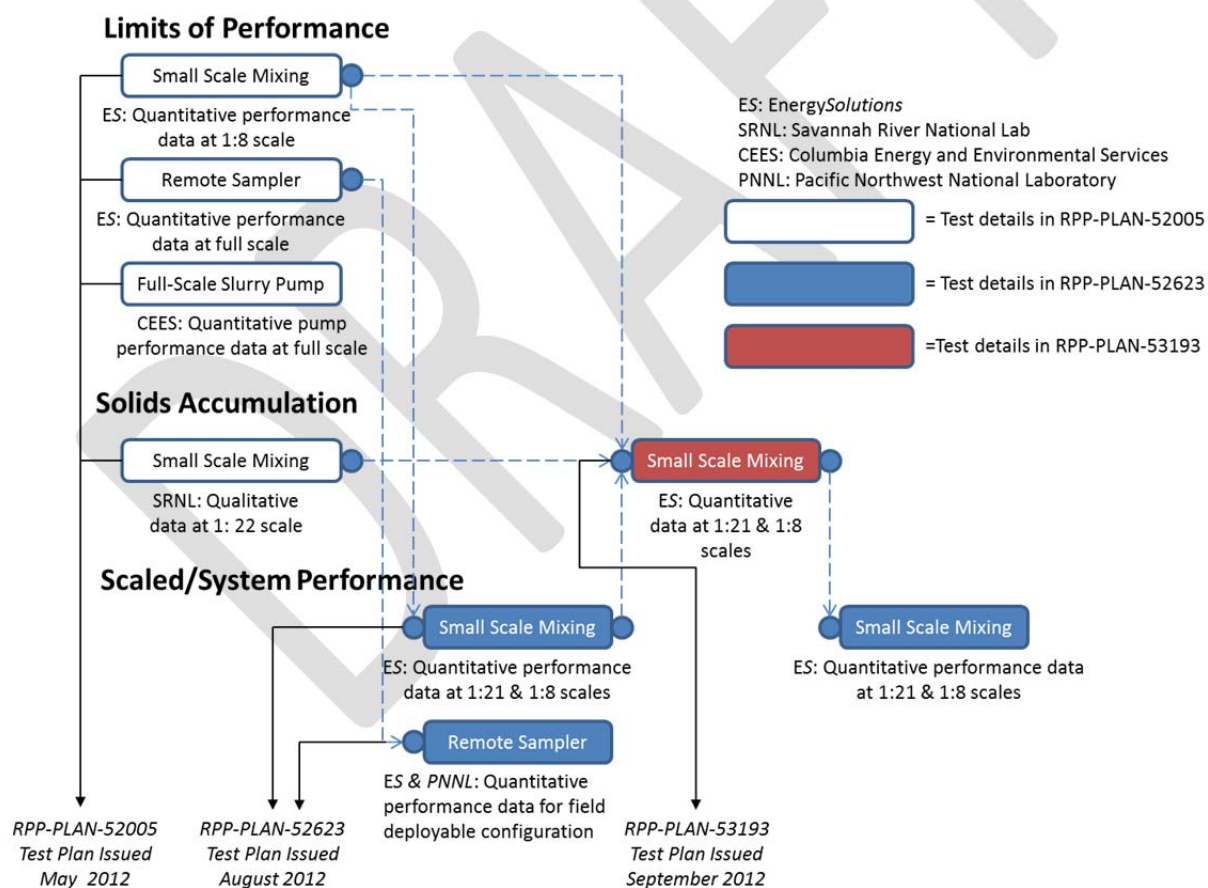


Figure 1-1. WFD Mixing and Sampling Program Test Sequence

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2.0 SCOPE

DNFSB Recommendation 2010-2 has raised WTP safety issues related to tank farms ability to mix, sample, and transfer solids. In response, DOE developed an implementation plan to resolve these issues (DOE Rev. 0 2010-2). The SSMD solids accumulation testing will explore the propensity of the feed staging system to accumulate fast settling solids over the duration of the waste feed delivery mission. As discussed in Section 1.0, this test plan is one of multiple test plan documents that have been prepared to address Commitment 5.5.3.6 of the Implementation Plan.

To ensure that tank farms and WTP mixing and sampling systems are integrated and compatible (i.e., execution of the One System approach) and that the uncertainties identified to date (WRPS-1105293) are addressed, solids accumulation testing will explore the potential for fast settling particles to concentrate in the waste feed staging tanks during the multiple fill, mix, and transfer operations expected to occur over the life of the mission.

The propensity of the Tank Farm's WFD system to accumulate solids will be characterized using tank waste simulants that have typical physical properties that are important to mixing, sampling and transfer (solid particulates sizes and densities, and supernatant density and viscosity), and may not be properties that will be directly measured and compared to WAC requirements. Slurry samples will be collected during each batch transfer operation and analyzed for chemical composition to determine the amount of material that is transferred from the tank. By mass balance accounting (mass in minus mass out) the running inventory in the tank will be determined. Additionally, the volume of residual solids deposited as mounds in the tank will be estimated to determine if solids accumulation occurs over multiple fill and empty cycles. Solid samples collected from the mound will be analyzed to determine the chemical content of the mounds. The chemical content of the solid samples will be mapped according to the sample locations to determine how fast settling solids, including spike particles, are spatially distributed in the mounds. In addition, solid samples collected after subsequent cycles from adjacent locations will be compared to determine whether or not the concentration of fast settling solids in the mounds change. Increasing concentrations of fast settling solids in the mounds is indicative of accumulation.

Testing will continue to be performed at two scales in accordance the recommendations developed at the initial planning workshop, which provided guidance that a decision regarding a third scale will be held until after performance at the smaller scales is demonstrated (Section 4.2 of RPT-1741-0001, *Tank Farm Mixing Demonstration Planning Workshop*). Testing at each scale will also be performed at two nozzle velocities. Nozzle velocities at each scale will be selected that result in similarly proportioned piles (footprint and depth relative to the difference in scale). Solids accumulation in the similarly proportioned piles will be compared across the two scales. If solids accumulation in both scales is similar, than it can be inferred that solids accumulation in a full-scale tank with similarly proportioned piles will also be similar. At each scale two nozzle velocities will be evaluated. Different nozzle velocities will result in different sized piles and may affect solids accumulation attributes that affect the conclusions made about a full-scale system.

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Testing will be performed with Hanford waste simulants that are typical for the wide range of characterized waste with respect to ICD-19 WAC in terms of bulk density, solids loading, and slurry viscosity. Testing will be performed with slurries containing dense particles (8 g/cm^3) having particles sizes exceeding 100 microns that are spiked with plutonium oxide surrogates for assessing the potential to concentrate fissile material in the tank. In addition, the spike particles will be used to determine the capability of the system to transfer the fast settling particles for comparisons to ICD-19 requirements with action limits for U and Pu and to requirements for waste treatment processability; (e.g., Pu and U unwashed solids concentration).

The test objectives for the SSMD solids accumulation performance evaluation are summarized in

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Table 2-1.

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Table 2-1. Solids Accumulation Test Objectives

Objective	Success Criteria
<p>Use fast settling base particulate and spike solids in a Newtonian supernatant to determine how fast settling solids are spatially distributed in mounds left in a waste feed staging tank after the feed has been delivered.</p>	<p>Mixing and transfer tests are performed with Hanford tank waste simulant slurries. The slurry contains moderately sized (approximately 100 microns), dense particles (~8 g/cm³ and 9.6 g/cm³) to represent hard-to-transfer waste particles in the Hanford tank waste. These particles are distinguishable in collected samples by a physical or chemical property that can be exploited for separation and subsequent quantification.</p> <p>Solid samples are collected from the tank mounds and analyzed for chemical content. Using the known location of the samples together with the analytical results and approximate shape of the mound, the spatial distribution of fast settling solids in the mounds is mapped.</p> <p>Two campaigns of ten fill and empty cycles are performed at each scale. Each campaign uses a different nozzle velocity for evaluating solids accumulation.</p>
<p>Evaluate how fast settling solids could be spatially distributed in a full-scale DST.</p>	<p>The 1:8-scale and 1:21-scale mixing and transfer systems in the SSMD platform are filled with the same simulant combination and operated at nozzle velocities that result in the formation of similarly proportioned piles relative to the tank scale. The spatial distribution of fast settling solids in the mounds in each scaled system are mapped and compared.</p>
<p>Explore if fast settling spike particles can be concentrated at the bottom of full-scale double shell tank.</p>	<p>Ten waste feed staging fill and empty cycles are performed under similar test conditions (simulant composition of added feed, nozzle velocity, rotational rate, fill volume, equipment configuration) in each scale. Heel samples are collected after the first transfer cycle and the spatial distribution of fast settling solids is mapped.</p> <p>After the tank mounds have formed, a fast settling spike particle, a surrogate for plutonium oxide, is introduced into the tank. After the 5th and 10th complete cycle, heel samples are collected and analyzed for chemical content. Heel samples are collected from mound locations adjacent to previously collected samples. Coarse vertical discretization of the heel samples is performed. The spatial distribution of fast settling solids in the mounds is mapped. The presence of the fast settling spike particles at the bottom of the mound is or is not confirmed. The change in the distribution of the fast settling solids in the three spatial distribution maps is evaluated.</p> <p>Conclusions about the changes in the spatial distribution of the fast settling solids in the mounds of a full-scale DST are made by comparing the results from the two smaller scales.</p>
<p>Evaluate the reliability of the collected data for predicting full-scale performance when the scaling relationship is uncertain.</p>	<p>The solids accumulation studies performed to evaluate how fast settling solids are distributed in the mounds of a waste feed staging tank are repeated at a second nozzle velocity.</p> <p>Conclusions about the spatial distribution of the fast settling solids in the mounds of a full-scale DST are made and then the results are compared to the previous work to determine if the different operating velocity changed the conclusions.</p>

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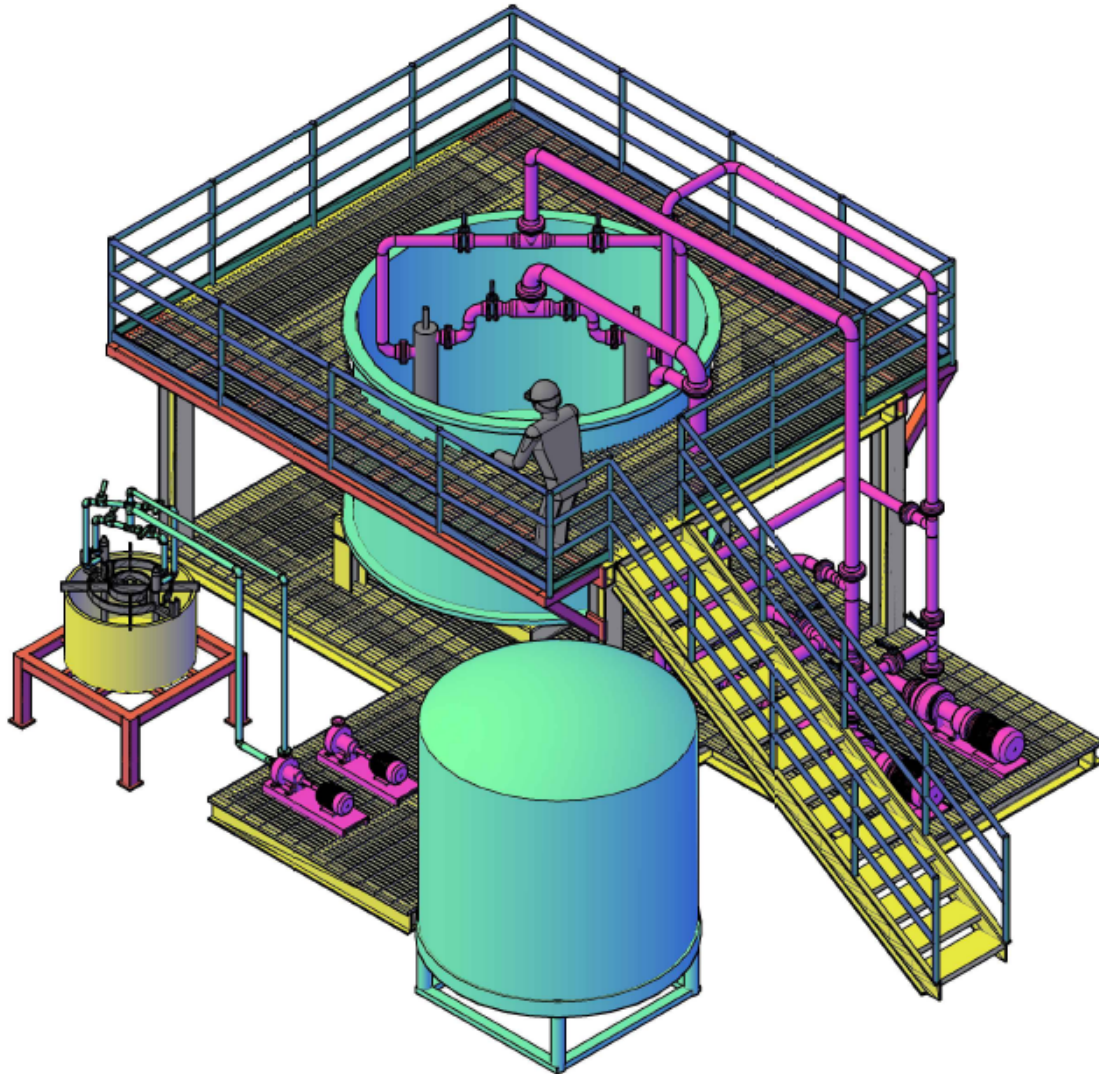


Figure 2-1. Schematic of Small-Scale Mixing Demonstration Test Platform

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3.0 TEST REQUIREMENTS

Test requirements and test guidance have been developed to meet the SSMD solids accumulation performance evaluation test objectives identified in Section 2.0. However, the WFD Mixing and Sampling Program testing falls outside the scope of TFC-PLAN-26, *Test Program Plan*, which defines additional requirements for oversight, development, and the conduct of factory acceptance, construction acceptance, and operational acceptance tests for demonstrating the operability and integrity of new or modified tank farm facilities and systems. The WFD Mixing and Sampling Program testing is evaluating the feasibility of a baseline design for collecting representative samples from the waste feed staging tanks. Testing is developmental and is not evaluating a field deployable design against specific functional characteristics and performance requirements. Testing is performed in accordance with Phase I testing described in TFC-PLAN-90, *Technology Development Management Plan*. Phase I development testing addresses a TOC technology need when existing processes are inadequate, inefficient, or not proven for the intended application. During Phase I testing functional criteria and performance requirements for the promising technology are defined, a prototype working model is constructed, and the prototype is evaluated against the performance criteria. Phase I development implements a graded application of the quality assurance program requirements. Phase I testing generally applies a commercial quality assurance program because there is no implied guarantee that the technology will be adopted by the TOC. Upon successful completion of Phase I testing, which may be an iterative process, additional development (Phase II) may be pursued. Phase II development and testing is performed to a higher quality assurance standard and invokes TOC approved procedures and quality assurance requirements for design control, including design verification, and qualification testing. The WFD Mixing and Sampling Program test planning, test review, test control, and test results reporting requirements are communicated through this test plan and are guided by testing principles described in TFC-ENG-DESIGN-C-18, *Testing Practices*.

In addition to this test plan, the testing contractor will develop operational procedures that include or reference the test configuration, test objectives, test requirements, and provisions for assuring that prerequisites and suitable environmental conditions are met, adequate instrumentation is available and operational, and that necessary monitoring is performed.

The SSMD solids accumulation test activities are performed by EnergySolutions for WRPS.

3.1 TEST SIMULANTS

The capability gap between the TOC and the WTP is defined by the TOC's capability to mix, sample, and transfer large and dense particles, and the WTP's capability to process these particles. Therefore, integral with defining the gap in capabilities is the selection of appropriately complex simulants, integrated with WTP simulant selection, and supported by accurate analytical techniques to characterize the material of interest. The Hanford waste simulants for DNFSB 2010-2 testing are developed and described in RPP-PLAN-51625. Particle size and density are expected to be the most important solids properties for evaluating the propensity of the waste feed staging system to accumulate fast settling solids. Liquid density and

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viscosity are expected to be important liquid phase properties as these properties directly affect particle settling and mobilization from the tank.

The slurry simulant used for SSMD solids accumulation test activities is consistent with simulant development described in RPP-PLAN-51625 and used in recent TOC testing activities. Simulant selection considers parameters (e.g., particle size, density, and viscosity) important to mixing, sampling, and transfer performance because solids accumulation is directly affected by the capability of the system to transfer the particles from the tank. Simulant properties such as hardness and abrasiveness, which are important to evaluating erosion and wear of the tank and pipe walls and the mixing and transfer equipment, are not primary considerations for understanding the capability of the system to mix, sample, and transfer slurries characteristic of Hanford tank waste. However, simulant selection does favor materials that result in less wear on the test equipment when alternatives that match the critical characteristics are available.

Although SSMD solids accumulation testing is Phase I technology development and generally performed to the subcontractors own quality assurance procedures, simulant procurement, preparation, and simulant property data collection are performed to enhanced quality assurance standards as defined in TFC-ESHQ-Q_ADM-C-01, *Graded Quality Assurance*. The enhanced quality assurance standard applied is ASME NQA-1-2004, including addenda, or a later version. As such, additional level of controls beyond the providers published or stated attributes of the item, service, or process are needed to verify critical attributes of the simulants. Simulant materials procured as commercial grade items shall be prepared and qualified to match the critical characteristics of the simulants.

Simulant batches of base material and a Newtonian supernatant are prepared according to prepared recipes. By specifying the mass fraction of each solids component, the density of each solids component, the density of the supernatant, the solids loading, and the batch volume, the required amounts of each solids component are fully defined. Supernatant recipes were determined during previous test activities and will be confirmed using test batches prepared to match the critical characteristics. The base simulant, spike particles, and supernatant simulant used during SSMD solids accumulation testing are described below.

3.1.1 Base Simulant

As discussed in RPP-PLAN-51625, during simulant development for DNFSB 2010-2 test activities metrics that are relevant to mixing and sampling were selected, calculated, and compared between the developed simulants and the Hanford tank waste. The calculated values for the metrics are not used to set operating conditions for testing; metric comparisons are only used to demonstrate that the developed simulants are similar to the Hanford tank waste. Therefore, this test plan does not develop simulants, rather it selects simulants from those previously developed.

3.1.1.1 Base Simulant Description

The base simulant is the mixture of solid particles in the slurry representing the Hanford tank waste. Report RPP-PLAN-51625 recommends three mixtures of particles as the base simulants for WFD Mixing and Sampling Program test activities, low conceptual, typical conceptual, and high conceptual (see Table 3-1). The low conceptual simulant was excluded from consideration

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because it does not contain any fast settling solids. Only the typical and high conceptual simulants were considered for solids accumulation tests. Both the typical and high conceptual simulants contain fast settling solids (stainless steel powder with a density of approximately 8 g/cm³). To represent the fast settling fissile material in the tank waste, the base material will be spiked with a tungsten alloy powder having a density of approximately 9.6 g/cm³. Simulant spikes are discussed in Section 3.1.3.

Table 3-1: Base Particulate Simulant Characteristics

Base Particulates					
Compound	Solid Density (g/cm ³)	Median Particle Size (micron)	Mass Fraction		
			Low	Typical	High
Small Gibbsite	2.42	1.3	1.00	0.27	0
Large Gibbsite	2.42	10	0	0.44	0.03
Small Sand	2.65	57	0	0	0.35
Medium Sand	2.65	148	0	0.13	0
Large Sand	2.65	382	0	0	0.21
Zirconium Oxide	5.7	6	0	0.10	0.08
Stainless Steel	8.0	112	0	0.06	0.33

The stainless steel particles in the base material are a fast settling solid. The free settling velocity, V_t , in the typical supernatant (see Section 3.1.2) can be calculated for the stainless steel particles with specified sizes and densities using Equations 3-1 and 3-2 (from *Handbook of Industrial Mixing: Science and Practice*, Equations 10-1, 10-2 and Table 10-1). Equation 3-1a is for the Stokes Law regime and applies when the particle Reynolds number is less than 0.3. Equation 3-1b is for the Intermediate Law regime and applies when the particle Reynolds number is between 0.3 and 1000. The free settling velocities for stainless steel particle sizes in Table 3-2 result in particle Reynolds numbers, Re_p , (Equation 3-2) in the Intermediate Law regime.

$$V_t = \left(\frac{4gd(\rho_s - \rho_l)}{3\rho_l \left(\frac{24}{Re_p} \right)} \right)^{0.5} \quad (3-1a)$$

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$$V_t = \left(\frac{4gd(\rho_s - \rho_l)}{3\rho_l \left(\frac{18.5}{Re_p^{0.6}} \right)} \right)^{0.5} \quad (3-1b)$$

$$Re_p = \frac{\rho_l V_t d}{\mu} \quad (3-2)$$

Where ρ_s is the particle density, ρ_l is the liquid density, g is the acceleration of gravity, d is the particle diameter, and μ is the dynamic viscosity of the liquid. Table 3-2 tabulates the result of the calculation for the upper percentiles of the stainless steel procured for SSMD scaled performance testing (RPP-PLAN-52623); SSMD solids accumulation testing will use similar material.

Table 3-2: Stainless Steel Settling Velocities

Stainless steel cumulative volume fraction	Approximate stainless steel particle size (microns)	Stainless steel settling velocity (ft/s)
0.90	116	0.064 ($Re_p=0.8$)
0.95	150	0.085 ($Re_p=1.4$)
0.99	229	0.14 ($Re_p=3.4$)

The selected simulant will be used exclusively for all tests. Although using the same simulant composition is not characteristic of expected conditions during the feed delivery mission, it is preferred to keep simulant additions consistent throughout the test. This ensures that the accumulation of the fast settling solids is attributed to system performance and is not due to fluctuations in the simulant content. Furthermore, because the fast settling solids in the typical and high conceptual simulants originate from the same material, stainless steel, it would not be possible to determine whether the accumulated solids originated from either simulant type.

The typical and high conceptual simulants contain the same principle components, gibbsite, zirconium oxide, sand, and stainless steel. The differences between the two simulants are the amounts of each component in the mixture and the size distributions for gibbsite and sand. The typical conceptual simulant was developed in RPP-PLAN-51625 to have mixing and transfer behavior that are consistent with most of the Hanford tank waste; the high conceptual simulant was developed to have performance metrics that are consistent with the most challenging Hanford tank waste. Because solids accumulation will investigate repeated fills and emptying of a waste feed staging tank over the feed delivery mission, it was considered more appropriate to use

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a typical simulant rather than a simulant that was more challenging than most of the characterized Hanford tank waste. This decision process is consistent with the process used to select the simulant used in the solids accumulation scoping studies performed by SRNL. Both the SRNL work and recent SSMD testing (in process) with the typical base simulant indicate that mounds will form in the tank so that the accumulation of fast settling solids can be evaluated using the typical base simulant. Solids accumulation testing will use the typical conceptual simulant exclusively.

The solids loading is initially set to 13 weight percent (wt%). The resulting loading yields 180 g/l for a homogeneously mixed system, which is 10% lower than the action level specified in ICD-19. The actual solids concentration in the transfer line will vary from this loading because the tanks are not homogeneously mixed. The solids loading is higher than that tested in initial scoping studies at SRNL (approximately 100 g/l or 8.4 wt%), which was based on the calculated solids loading for each transfer batch from DST 241-AW-105 to the WTP (SVF-2111, , *TRANSFERS_4MINTIMESTEP(6MELTERS)-MMR-11-031-6.5-8.3R1-2011-03-18-AT-01-31-58_MI.XLSM*). The solids loading was selected to be consistent with SSMD scaled performance testing (RPP-PLAN-52623) and to also ensure that sufficient material is added to the tank to promote solids accumulation in the tank. If the stabilized size of the heel mounds are determined by the operation of the mixer jets and the properties of the simulant (i.e. the properties that affect the effective clearing radius), the mass loading would only be expected to influence the number of cycles needed for the mound to grow to the stable size. Because the mass loading in this testing is higher than the previous work at SRNL, the number of cycles needed to achieve a stable mound size may be encountered sooner than in the previous work. Additional tests with the same simulant are planned during SSMD scaled performance testing. The initial mass loading may be lowered based on the observed mound sizes in the SSMD scaled performance work. Any change will be reflected in the approved run sheets for the solids accumulation work.

3.1.1.2 Base Simulant Qualification

The critical characteristics for the base simulant materials are the particle size distribution and density of the materials. As described in PNNL-20637 and used in RPP-PLAN-51625, particle size distributions, particle density, and mass fractions of the components in the composite simulant can be used to determine the distributions of Archimedes numbers (see Equation 3-3) and jet velocities needed to achieve complete solids suspension for the composite simulant (see Equation 3-4) (Kale and Patwardhan 2005).

$$Ar = \frac{\left(\frac{\rho_s}{\rho_L} - 1\right) g d^3}{\nu^2} \quad (3-3)$$

$$U_n = \frac{\nu}{d} \left[0.13X^{0.22} Ar^{0.38} \left(\frac{D}{d_j}\right)^2 \left(1 + 0.25 \left(\frac{z}{d_j}\right)\right)^{-0.25} \left(1 + 0.75 \left(\frac{z}{D}\right)\right) \right] \quad (3-4)$$

Where U_n is the jet velocity, ν is the kinematic viscosity of the fluid, d is the diameter of the particle, X is the mass ratio of solids to liquids, Ar is the Archimedes number and is defined in

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Equation 3-4, D is the tank diameter, d_j is the jet nozzle diameter, z is the nozzle clearance above the tank bottom, ρ_s is the density of the solid and ρ_L is the fluid density.

As discussed in PNNL-20637 the Archimedes number is closely related to the settling velocity and is also a parameter in other mixing and transfer metrics such as pump intake, jet suspension velocity, critical shear stress for erosion, critical suspension velocity, suspended particle cloud height, and pipeline critical velocity. The semi-empirical model of the jet velocity needed to achieve complete solids suspension (Equation 3-4) correlates the particle size and density to the jet velocity of a radial wall jet needed to suspend solids in a tank. Base simulant qualification is performed by comparing the distribution of Archimedes numbers and jet velocities needed to achieve complete solids suspension calculated for the procured simulants to the distributions for the recommended simulants documented in Figures 8-1 and 8-2 in RPP-PLAN-51625. To provide comparable results, performance metrics are calculated using the same assumptions used to calculate the metrics for the three conceptual simulants. Metrics are calculated using particle densities and particle size distributions obtained on samples from each procured lot. Because there is no expectation that procured material lots will not be mixed when testing is performed, particle size distributions from multiple lots of similar material may be averaged for the qualification comparisons. For commercial grade material, the particle size distribution provided by the vendor is not adequate for simulant qualification and a particle size analysis of each procured lot shall be performed. Appendix C of RPP-PLAN-51625 includes additional performance metrics, such as the critical shear stress for erosion of non-cohesive particles, just suspended impeller speed, pulse jet mixer critical suspension velocity for non-cohesive solids, pulse jet mixer cloud height for non-cohesive solids, and pipeline critical transport velocity. The procured material will also be compared to the conceptual simulants using these metrics.

The metrics calculated for the conceptual simulants in RPP-PLAN-51625 include typical distributions for some of the components. Therefore, the calculated values represent target values and deviations from the conceptual simulants are anticipated. The appropriateness of candidate material will be evaluated before simulant procurement. For procurement purposes, in absence of samples from actual lots, vendor supplied information (e.g., particle size distributions and particle density) and targeted mass fractions can be used to calculate the performance metrics for comparison to the conceptual simulants. For simulant qualification, calculations will be based on laboratory analysis of samples taken from the procured material and actual weight measurements recorded during testing.

3.1.2 Supernatant Simulant

Developing the Newtonian supernatant composition for DNFSB 2010-2 test activities is informed from modeling Hanford waste processes. Hanford waste process modeling includes tank inventory, accounts for retrieval technologies, waste volume reduction (i.e., evaporation), and includes inventory blending during multiple tank-to-tank transfers. Therefore, an estimate for the chemical composition of each feed batch is calculated and the results are used to select a suitable supernatant density and viscosity for DNFSB 2010-2 test activities.

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3.1.2.1 Supernatant Simulant Description

The supernatant simulant is the liquid phase of the simulant slurry. For WFD Mixing and Sampling Program test activities, RPP-PLAN-51625 defines the density and viscosity range for the supernatant simulants. These simulants are characterized by liquid density and liquid viscosity as described in Table 6-1 in RPP-PLAN-51625. Solids accumulation test activities will reproduce tank waste staging for feed to the WTP using a consistent supernatant formulation. Using the same supernatant formulation from cycle to cycle ensures that the accumulation behavior is due entirely to the mixing process and not the simulant composition. Different simulant compositions are expected to change the mixing behavior in the tank (e.g., the effective clearing radius of a jet is a function of the supernatant density and viscosity); therefore, the accumulation of solids in the tank is also expected to change with changes in the supernatant composition. In this initial work to understand the propensity to accumulate fast settling solids, a better understanding of the accumulation behavior is expected by eliminating the additional complication of changing the simulant between cycles.

Exploring solids accumulation with a supernatant that has the bounding supernatant properties provided in Table 6-1 in RPP-PLAN-51625 is not representative of the waste feed delivery mission. The bounding supernatants are limiting supernatants and were developed for testing activities that attempt to mobilize large and dense particles during limits of performance testing. Using a bounding simulant that can mobilize large and dense particles is counterproductive for studying the accumulation behavior of fast settling solids.

The typical supernatant listed in Table 3-3 is the preferred simulant for SSMD solids accumulation testing. Similar to the reason for selecting the typical base supernatant, the typical simulant was selected because testing will investigate repeated filling and emptying of a waste feed staging tank over the feed delivery mission so it was considered more appropriate to use a typical supernatant rather than a supernatant that was more or less challenging than most of the characterized Hanford tank waste. This decision process is consistent with the process used to select the supernatant used in the solids accumulation scouting studies performed by SRNL. However, SRNL solids accumulation testing also used available material with similar density and viscosity that had been prepared for other related work.

The liquid density for the typical supernatant is the median density from the unfiltered dataset used to derive the low and high density values in RPP-PLAN-51625. The dataset is the liquid density of the feed batches to the WTP calculated using the Hanford Tank Waste Operations Simulator model (RPP-RPT-48681, *Hanford Tank Waste Operations Simulator Model Data Package for the River Protection Project System Plan Rev. 6 Cases*). The unfiltered dataset does not exclude the low activity waste transfers or the high density HLW feed batches after 2040. Excluding these values, the typical supernatant has a density nearer the 85th-percentile. The typical supernatant is characterized as having a liquid density of about 1.29 g/ml and an estimated liquid viscosity of 3.3 cP. The viscosity of the supernatant simulant is determined by the salt(s) used to attain the desired density, and is comparable to the value determined using the relationship in Figure 6-2 of RPP-PLAN-51625. An aqueous solution of 31.5 wt % anhydrous sodium thiosulfate will produce a supernatant with properties similar to the targeted simulant.

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Table 3-3: Newtonian Liquid Supernatant Simulant Characteristics

Supernatant (density/viscosity)	Target Simulant Properties @ 20°C		Simulant Properties @ 20°C		Simulant Composition
	Density (g/ml)	Viscosity (cP)	Density (g/ml)	Viscosity (cP)	
Typical/Typical	1.29	3.3	1.284	3.60	31.5 wt% sodium thiosulfate

3.1.2.2 Supernatant Simulant Qualification

For the supernatant, the critical characteristics are the liquid density and liquid viscosity. To qualify the supernatant for use, the critical characteristics will be measured when the simulant batches are prepared. The simulant recipe for the supernatant simulant was developed in the laboratory, but will need to be scaled to the volume needed for each test. SSMD scaled performance testing (see RPP-PLAN-52623) uses the same supernatant and may identify an updated recipe to meet targeted conditions with the procured material. The liquid density and liquid viscosity will be measured at testing temperatures to confirm that the prepared batch is within the required range for simulant density and viscosity. Dissolution of anhydrous sodium thiosulfate is exothermic so that the temperature of the liquid increases as it is prepared. The viscosity of the supernatant decreases nearly linearly as the temperature increases from 15°C to 25°C; over this range the viscosity change is about 0.5 cP. The supernatant must be prepared to minimize viscosity variations due to significant changes in supernatant temperatures. Steps to control supernatant viscosity include temperature control, allow sufficient preparation time for ambient cooling, or mix hydrated sodium thiosulfate with anhydrous sodium thiosulfate. The dissolution of hydrated sodium thiosulfate is endothermic and results in some cooling. Preparing consistent simulant batches from test to test will facilitate the analysis of the data between tests and is expected to be more important for the data analysis than performing tests at specific conditions.

Therefore, for the typical density and typical viscosity fluids, 1.284 g/ml and 3.60 cP, respectively, the acceptable range of liquid densities and viscosities is $\pm 5\%$ and ± 0.25 cP, respectively. The supernatant will be attained using sodium thiosulfate. The two properties cannot be adjusted independently using the single component; if the two properties cannot be attained within the tolerances specified with the procured material, the supernatant will be prepared to match the target density rather than the target viscosity which was selected from a density-viscosity relationship.

The liquid property measurements will be measured on-site with the appropriate instrumentation (e.g., hydrometer, viscometer, and rheometer) calibrated, controlled, and maintained in accordance with ASME NQA-1-2004, Requirement 12 including addenda, or a later version. Supernatant viscosity will be determined using a set program that controls the shear rate to generate the rheogram. The program will include a pre-shear period and two evolutions over the shear rate range. The viscosity shall be determined on the second down curve used to generate the rheogram. Functional checks with reference standards covering the expected range of solutions used during testing shall be performed daily to ensure that the instrument is being properly maintained. Corrective actions, commensurate with the significance of an out-of-

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calibration condition, shall be performed. Appropriate instrumentation for measuring liquid viscosity of the Newtonian fluid is a programmable rheometer capable of taking controlled shear rate and controlled shear stress measurements. The rheometer shall also have the capability to control sample temperatures. Data collection shall be performed in accordance with ASME NQA-1-2004, Requirement 11, including addenda, or a later version. To ensure that the prepared simulant is appropriate for use, liquid properties will be measured prior to adding base simulant solids and therefore will be performed at the start of testing and as each new batch of simulant is prepared. In addition, viscosity will also be measured at the completion of testing, and during testing if necessary, to assess changes that may occur during the course of testing. The base solids in the samples collected during and after testing will be removed by filtering prior to collecting viscosity and density measurements.

3.1.3 Spike Particulates

A spike particulate will be included in the solids accumulation testing as a plutonium oxide surrogate. RPP-RPT-50941, *Review of Plutonium Oxide Receipts into Hanford Tank Farms*, indicates that the practical upper limit particle size for the PuO₂ and Pu metal in the transferable Hanford tank waste is 100 microns. RPP-RPT-50941 also indicates that the amount of PuO₂ and Pu metal in all of the tank waste is on the order of 10s of kilograms and is likely to be primarily PuO₂ because Pu metal fines are not thermodynamically stable in tank waste and may not have survived the extended storage time. For this reason, solids accumulation testing will include a PuO₂ surrogate and will not include a Pu metal surrogate.

The surrogate considered is a larger size of the tungsten alloy used as a plutonium oxide surrogate in WTP testing. The tungsten alloy has a density of 9.6 g/cm³ and particle size characteristics shown in Table 3-4. The targeted particle size distributions for the spike is a d₅₀ of 40 microns with additional particles up to 100 microns. For comparison, the WTP design basis particle size for plutonium oxide is 10 microns. The tungsten alloy is subject to the same simulant qualification process as the base simulant (see Section 3.1.1.2). The spike will replace 1 weight percent of the solids added to the tank, replacing an equivalent mass of stainless steel. Using the model of the model of Kale and Patwardhan (2005), the jet velocity needed to suspend the tungsten alloy particles (Equation 3-4) can be used to determine the size of plutonium oxide particle that would be suspended at the same jet velocity. For two components of different densities (ρ_{S1} and ρ_{S2}), Equation 3-4 can be used to determine the sizes (d_1 and d_2) of the particles that have the same jet velocity needed to suspend the particles in the same suspending fluid and jet mixed tank. The resulting relationship is shown in Equation 3-5 and the equivalent size particles in the typical supernatant ($\rho_L = 1.284$ g/ml) are presented in Table 3-4.

$$d_1^{0.14}(\rho_{S1} - \rho_L)^{0.38} = d_2^{0.14}(\rho_{S2} - \rho_L)^{0.38} \quad (3-5)$$

A similar analysis can be performed using the free settling velocity in Equations 3-1 and 3-2. The results show that the free settling velocity of the spike particle is equivalent to the free settling velocity of a PuO₂ particle that is 90% of its own size and a Pu particle that is 67% of its own size. Similarly, the jet velocity needed to suspend the spike particle will also suspend a PuO₂ particle that is 62% of its own size and a Pu particle that is 13% of its own size. With the understanding that the fast settling particles do not need to be suspended by the jets in order to

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accumulate in the tanks, these results suggest that the spike particle with a density of 9.6 g/cm^3 is not an acceptable surrogate for the accumulation of Pu metal particles but may be acceptable as a surrogate for PuO_2 particles.

Table 3-4: Spike Particle Equivalent Settling Velocities of PuO_2 and Pu Metal

Spike particle cumulative volume fraction	Approximate spike particle size (microns)	Size of PuO_2 with equivalent velocity (microns) ^a		Size of Pu with equivalent velocity (microns) ^a	
		U_n	V_t	U_n	V_t
0.05	10	6.2	9.2	1.3	6.9
0.50	40	25	37	5.1	27
0.99	100	62	90	13	62

^a The density of spike particle used in the calculation is 9.6 g/cm^3 . The density of PuO_2 used in the calculation is 11 g/cm^3 . The density of Pu metal used in the calculation is 19 g/cm^3 . The supernatant density used in the calculation is 1.284 g/ml and the viscosity is 3.6 cP .

3.1.4 Flow Regime

When considering different scales, the flow regime among the scales must be consistent. A discussion of the flow regime for the full-scaled and SSMD tanks was presented in Section 3.1.4 of RPP-PLAN-52623. The flow regime at the inlet of the transfer pump and within the transfer lines was determined to be turbulent for all scales using the typical supernatant.

3.2 TEST EQUIPMENT AND INSTRUMENTATION

The SSMD solids accumulation activities described in this test plan will use the 1:21- and 1:8-scale tanks of the SSMD test platform (Figure 2-1) located at Monarch Machine & Tool Company, Inc. in Pasco, WA to evaluate the propensity for fast settling solids to accumulate in the feed staging tanks over the course of the waste feed delivery mission. The SSMD test platform has been used for previous test activities and will continue to be used to address uncertainties in the WFD Mixing and Sampling Program. The SSMD test platform was constructed to perform mixer jet pump testing at two different scales, approximately 1:21 (43.2-inch diameter tank) and 1:8 (120-inch diameter tank). Both tanks will be used for solids accumulation testing.

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The SSMD test platform was constructed according to scale from 241-AY-102. According to ORP-11242 Rev. 6, *River Protection Project System Plan*, tanks with riser geometries similar to 241-AW-105 will account for about 72% of the total waste volume that will be transferred to the WTP from the 13 feed staging tanks (SVF-2111). Therefore, waste loads from DST 241-AW-105 have been selected as the model tank for investigating solids accumulation. The plan view of DST 241-AW-105 is shown in Figure 3-1 (from H-14-010502, Sheet 5, Rev 0). The mixer jet pump locations will be maintained under the 241-AY-102 configuration but the air lift circulators will be removed. Tanks similar to 241-AW-105 do not have air lift circulators and removing these obstructions would facilitate heel volume estimations. The mixer jet pump locations in 241-AW-105 are different than 241-AY-102, the pumps are two feet closer to the center of the tank and one is offset by 5°. A comparison of the mixer jet pump and transfer pump locations between 241-AY-102 and 241-AW-105 is shown in Figure 3-2. Because the mixer jet pump locations are further away from where the mounds will form (along the perimeter of the tank at 0° and 180° in Figure 3-2), the mound size in the SSMD tanks is expected to be larger than would be observed if the mixer jet pump locations were moved to the configuration in 241-AW-105. A preliminary geometry evaluation showed that the area cleared by the mixer jet pumps differed by less than 4% over a wide clearing radius range; compared to 241-AW-105 the geometry for 241-AY-102 cleared less area for the same effective clearing radii. Based on this preliminary geometrical analysis as well as risks to cost and schedule, the construction effort required to move the mixer jet pumps was not considered warranted for the solids accumulation testing. The scaled tanks will not be modified to move the mixer jet pump locations closer to the center of the tank. The properties of the DSTs used to geometrically scale the test tanks and the scaled properties of the two-scaled tanks are provided in Table 3-5.

The main components of the test platform include: a 3,000-gallon flush tank, a 120-gallon (43.2-inch diameter) clear acrylic test tank (TK-201), a 2,358-gallon (120-inch diameter) clear acrylic test tank (TK-301), dual rotating mixer jet pump assemblies, and the slurry transfer pumps for both TK-201 and TK-301. Flow from the tanks enters the two mixer jet pump suction inlets on the bottom of the mixer jet pump, and is combined into one flow stream as it is routed through the pump driving the system. The pump discharge is split with half of the flow returning to each mixer jet pump. As each mixer jet pump is rotating, the flow is discharged back into the tank through two opposing jet nozzles located on the side of the mixer jet pump just above the pump suction inlet. Between scales, the mixer jet pump assemblies and transfer pumps for each tank are independent. The slurry transfer pumps are not submersible pumps, they are progressive cavity pumps located outside of the test tanks; the inlets of the pump are connected to 3/8-inch inner diameter suction lines that are placed within the tanks. The end of the suction lines inside each tank is fitted with a machined orifice matching the requirements in Table 3-5. The transfer pump suction inlet shall be placed consistent with the location of Riser-012. The scaled height of the pump suction inlet shall be equivalent to the height of the transfer pump inlet in the full-scale DST transfer system, which is 0.8 inches from the tank bottom in TK-301 and 0.28 inches from the tank bottom in TK-201 (see Table 3-5). Ancillary equipment, such as the support structure, the control system, video monitoring, and simulated piping to transfer and sample the material from the tank are also part of the test platform. For solids accumulation work, auxiliary mixing tanks and transfer systems are necessary to prepare fresh simulant batches that will be mixed and pumped into the tank in between each fill and empty cycle. The auxiliary tanks have a coned bottomed with a bottom discharge and are equipped with a single shaft mixer with dual

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impellers. Note that the SSMD test platform will be modified from previous tests to remove the simulated air lift circulators; DST 241-AW-105 does not have air lift circulators.

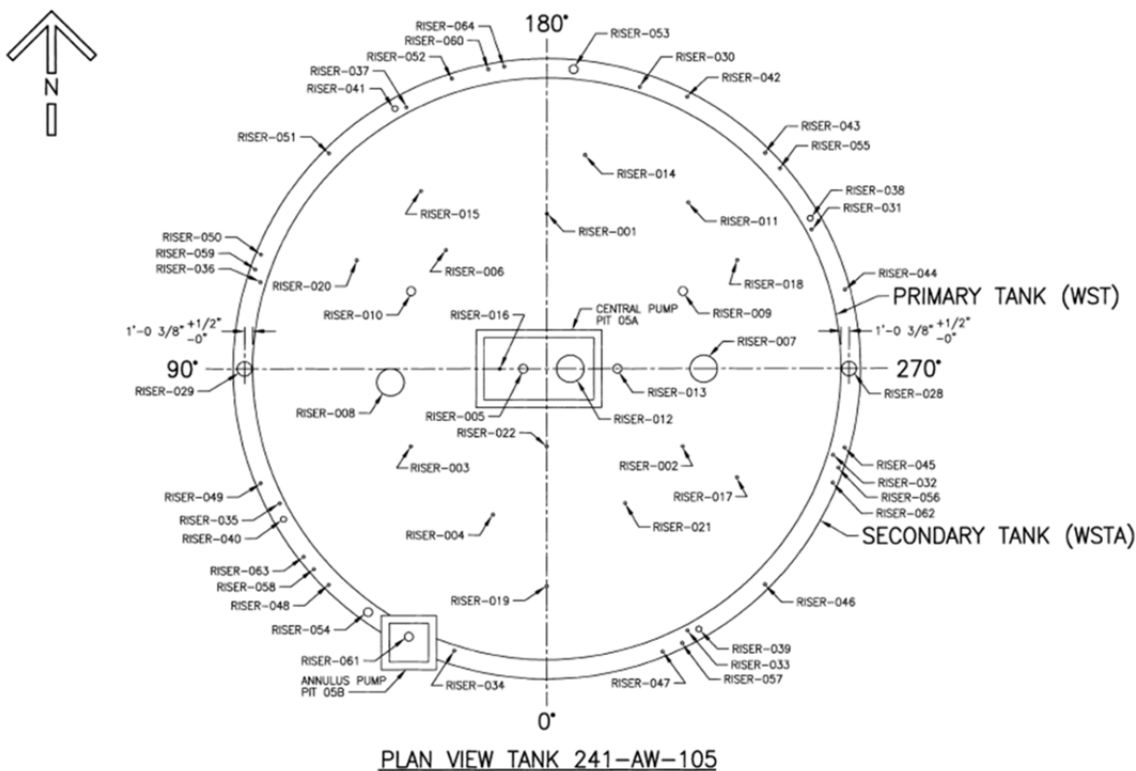
The transfer system piping, valving, and instrumentation (e.g., in-line Coriolis meters, and magnetic flow meters) will replicate the transfer system from previous SSMD testing reported in RPP-49740, *Small-Scale Mixing Demonstration Sampling and Batch Transfers Results Report*. The test configuration includes a closed recirculation loop from the tank. The recirculation loop accommodates sample collection. Flow control is automated using programmable logic controllers connected to a human-machine interface. System data, including date and time, slurry temperature, mixer jet pump rates and position, slurry flow rates, tank level, and specific gravity measurements in the transfer pump discharge, will be monitored and recorded using a data acquisition system.

The internal passageways of the mixer jet pumps driving pump and the slurry transfer pump are larger than the transfer lines; therefore, particles with a high settling velocity (e.g. stainless steel powder in the base simulant) may settle in the pump because the velocity through the pump may be reduced below the critical velocity of the particles. Modifications to the transfer system to minimize the collection of particles have been implemented and will be retained for SSMD solids accumulation unless improvements are identified. The extent that particles can collect in the transfer pump was evaluated in developmental testing for SSMD scaled performance testing so that this condition can be captured as a source of error. In addition, the slurry lines shall be purged in between campaigns to reduce the potential that settled solids from one campaign contaminate the results of a subsequent campaign. The transfer lines do not need to be purged between cycles of the same campaign because the accumulation of solids over the entire campaign is being evaluated.

When operating in a recycle mode to stabilize the mixing tank prior to performing batch transfers, the transfer line shall be discharged back into the tank. During batch transfer operations the transfer line shall be discharged for sample collection or waste collection.

All measuring and test equipment, including gauges and instrumentation, used for testing activities shall be controlled, calibrated under conditions typical of the test environment, adjusted, and maintained to required accuracy limits. The condition and the reported accuracy of each instrument shall be documented in a test log.

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Note: Mixer jet pumps will be in Riser-007 (270°) and Riser-008 (90°). Transfer pump will be in Riser-012 (270°)

Figure 3-1. Plan View Tank 241-AW-105



Figure 3-2: Comparison of Equipment Layout for 241-AY-102 and 241-AW-105

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Table 3-5: Small-Scale Mixing Demonstration Tank Geometrically Scaled Properties

Property	Full-Scale DST (AW-105)	1:8 Scale	1:21 Scale
Diameter (in)	900 (75 ft)	120 (10 ft)	43.2 (3.6 ft)
Scale Factor	1	0.1333	0.048
Fill Height (in)	416 (34.7 ft)	55.5 (4.63 ft)	20.0 (1.67 ft)
Transfer Batch Volume (gallons)	145,000	344	16
Bottom Geometry	Flat w/12-inch corner radius	Flat w/1.6-inch corner radius	Flat w/0.6-inch corner radius
Fill Volume ¹ (gallons)	~1,140,000	~2,700	~126
Mixer Jet Pump 1 Location ²	Riser-007 270°, 20 feet	270°, 2.9 feet	270°, 0.96 feet (12.7 in as-built)
Mixer Jet Pump 2 Location ²	Riser-003 85°, 20 feet	90°, 2.9 feet	90°, 0.96 feet (12.7 in as-built)
Mixer Jet Pump Suction Elevation ³ (in)	5±1	0.67±0.13	0.24±0.05
Mixer Jet Pump Suction Diameter (in)	11	1.47	0.53
Mixer Jet Pump Nozzle Diameter (in)	6	0.80	0.28
Mixer Jet Pump Nozzle Elevation ³ (in)	18	2.4	0.86
Mixer Jet Pump Operating Rate ⁵	10,400 gpm/MJP 59 ft/s/nozzle	95-120 gpm/MJP 30.3-38.3 ft/s/nozzle	8.3-11.8 21.6-30.8 ft/s/nozzle
Mixer Jet Rotation Rate (rpm)	0.2	See Eq. 3-1	See Eq. 3-1
Transfer Pump Location ²	Riser-012 270°, 3 feet	270°, 0.4 feet	270°, 0.14 feet
Transfer Pump Suction Inlet Diameter (in) ⁴	2.25-2.40	0.32	0.25
Transfer Pump Suction Inlet Height (in) ⁴	6	0.8	0.28
Transfer Line Diameter (in)	3.07 (3-inch Schedule 40)	½"-poly tubing (0.375-inch inner diameter)	½"-poly tubing (0.375-inch inner diameter)
Tank Obstructions	None	None	None

¹ Fill volume is determined by linear scaling of the tank diameter and sludge volume height.

² The reference point for DST locations presented in this table defines 0° as the bottom of 241-AW-105 in a plan view drawing of the tank. Provided distances are design distances from the center of the riser to the center of the tank. The locations of the mixer jet pumps in the scaled tanks were originally constructed to match DST 241-AY-102 and are not modified for these tests.

³ Elevation is relative to the tank bottom.

⁴ The pump suction inlet diameter of the Full-Scale Transfer Pump is underdevelopment and the tabulated value is based on similar transfer pumps used on the Hanford site to convey waste. The inlet size on the 1:21 scale tank is not geometrically scaled. The resulting inlet size was too small to accommodate the particle sizes targeted.

⁵ The mixer jet operating rates for the two scaled systems are typical operating rates used during testing. The full-scale equivalent is being investigated and is expected to be within this range.

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3.3 OPERATING PARAMETERS AND TEST METHODS

The operating conditions for the SSMD solids accumulation testing will be consistent with previous SSMD performance testing. The mixer jets shall rotate continuously clockwise with no rotational offset between mixer jet pumps; the streams will be synchronized to meet in the center of the tank. The rotational speed of the mixer jet pump (ω_{tank}) shall be set according to the mixer jet pump nozzle velocity (U_{jet}) and tank diameter (d_{tank}) in accordance with Equation 3-6, which is consistent with the relationship for scaled performance testing (RPP-PLAN-52623).

$$\omega_{\text{tank}} = \frac{(0.2 \text{ rpm})U_{\text{jet}}}{\left(\frac{d_{\text{tank}}}{900 \text{ inches}}\right) 59 \text{ ft/s}} \quad (3-6)$$

The constant values included in the Equation 3-6 are the full-scale parameters for the rotational rate (0.2 rpm), tank diameter (900 inches), and mixer jet pump nozzle velocity (59 ft/s).

Solids accumulation testing will be performed using two nozzle velocities at each scale. Each nozzle velocity will be maintained during each campaign that consists of ten fill and empty cycles. Previous solids accumulation work (SRNL-STI-2012-00508 (in process)) at SRNL tested nozzle velocities using a scale factor exponent equal to 0.33 (22.4 ft/s) and 0.29 (23.5 ft/s). The latter was determined at run time as a velocity that resulted in dead zones so that solids accumulation could be evaluated; higher velocities did not accumulate solids using simulants similar to those specified for SSMD solids accumulation testing. The appropriate nozzle velocities to use during the SSMD solids accumulation testing must result in “dead zones” within the tank. If the jet nozzle velocity is high enough to prevent build-up in the tank, then the accumulation of solids will not be adequately quantified. Similar to SRNL studies, the nozzle velocity for the first campaign in the 1:21-scale tank is selected using the equal power-per-volume scale up relationship. Based on a nozzle velocity of 59 ft/s at full scale, a tank diameter ratio of 20.8 and a scale factor exponent of 1/3, the nozzle velocity for the first campaign in the 1:21-scale system is 21.4 ft/s. Based on the 0.28-inch diameter nozzle, the flow rate per mixer jet pump is 8.25 gallons per minute (16.5 gallons per minute supplied to both pumps). The system, including simulant, will be operated at this velocity for a minimum of 30 tank rotations to ensure that a suitable mound for quantification is formed. If a suitable mound is not formed the starting nozzle velocity will need to be lowered for the first campaign. Based on SRNL testing a suitable mound is approximately 1-inch high at a peak, 15-inches long (edge to edge), and 4-inches wide in the radial direction. Larger mounds are also suitable for testing. If the mound size is adequate at 21.4 ft/s, then the effective clearing radius at this velocity will be measured and the test campaign will be conducted. If the mound size is too small, the nozzle velocity will be decreased until a suitable size mound is attained and the campaign will be run at the final setting. The nozzle velocity for the first campaign in the 1:8-scale system will be set so that the effective clearing radius is scaled proportionally from the measured value from the 1:21-scale test. If the effective clearing radius measured in the 1:21-scale test is 80% of the maximum value needed to clear the tank bottom, then the jet velocity for the 1:8-scale test would also be set so that the effective clearing radius is 80% of the maximum value. The effective clearing radius

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comparison attempts to equalize the relative dimensions of the mounds (i.e., similar performance for mound accumulation).

The second nozzle velocity will be evaluated at the time of testing to ensure that accumulation data can be collected. If the mound size from the first campaign was approximately equal to the suitable size, then the jet velocity for the second campaign would be lower than the first to ensure that a quantifiable mound is attained during testing. If the mound was greater than the suitable size then the velocity would be increased until a mound similar to the suitable size is attained. Similar to the first campaign, the effective clearing radius at the second campaign nozzle velocity in the 1:21-scale tank will be measured and used to establish the nozzle velocity for the second campaign in the 1:8-scale tank. If the mound size was close to the suitable size and the repeated volume estimates of the mound suggests that a smaller mound could also be estimated using the technique, the second campaign could target a nozzle velocity that results in a smaller mound.

Each tank in the SSMD test platform will be operated in a recirculation mode until a stable state mixing condition is established. The stable state is indicated by consistent mass flow rate and specific gravity readings from the Coriolis meter, after adjusting for cyclical variations caused by the rotating jets. Previous operating experience indicates that approximately 20-30 rotations of the mixer jet pumps are sufficient to result in a stabilized state. Once the tank reaches the stable state, the first of 6.5 batch transfers will be initiated. The batch volume for the 1:8-scale tank is 344 gallons and is the scaled volume for a 145,000 gallon transfer. Similarly, the batch volume for the 1:21-scale tank is 16 gallons. The batch volume will either be diverted to a sample collection basin (see Section 3.4) or pumped to the waste collection.

The mixer jet pump flow rate and rotational rate shall be maintained during each batch transfer but stopped for at least 20 minutes in between transfers to allow the suspended solids time to settle. Turning of the mixer jets in between transfers is consistent with the expected operation during the feed delivery mission. Developmental testing at SRNL concluded for the 1:22-scale system that the shut down duration did not significantly change the amount of material transferred when the shut down duration was extended from 20 minutes to four days. After the specified holding time, subsequent batch transfers will be initiated, repeating the holding time in between each complete transfer. During the hold time in between batches, the slurry will be recirculated through the transfer system to prevent line plugging. After each tank volume transfer (equals 6.5 batches) is completed, the tank will not be empty; a residual slurry will be left in the tank. In the full-scale tank the residual volume is equivalent to 72-inches of slurry, which is maintained to avoid cavitation when the mixer jet pumps are operating at full speed. Operation of the scaled tanks mimics the volume residual. After a full tank transfer volume is removed from the tank, the tank will contain solid mounds that are outside the area of influence of the mixer jet pumps as well as solids that were suspended in the slurry that was not removed from the tank. The residual slurry containing the suspendable solids will be pumped from the tank to expose the solid mounds after each tank volume transfer. Scouting studies at SRNL noted that the deposition of the less dense solids (i.e. gibbsite and zirconium oxide) made it difficult to delineate the mounds in the photographs. Scouting studies minimized the deposition of the suspendable solids by agitating the tank contents as the liquid was removed to expose the mounds. The mixer jets were directed towards the center of the tank, away from the mounds, and turned down to a flow rate that was sufficient to maintain a suspension of the small and less

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dense particles but not visually disturb the piles. The removed slurry will be pumped back into the tank before the next cycle. Because the inlet of the transfer pump is positioned above the tank bottom, a supplemental pump may be necessary to drain all of the free liquid from the tank to completely expose the mounds. A description and quantification of the solids remaining in tank, including a photographic or video record, will be prepared after each tank volume transfer. Solid samples shall be collected (see Section 3.4.4) from one of the solid mounds left in the tank after the 1st, 5th, and 10th tank volume transfers. Collecting solids samples in between tank volume transfers allows for an assessment of fast settling particle accumulation and spike particle migration into the mound as subsequent tank volume transfers are performed. Solid samples shall only be collected from the second mound after the 10th tank volume transfer. Collecting samples from the second mound only after the last cycle ensures that the solid content of the mound is not influenced by collecting the physical samples. Solid samples shall be collected with minimal disturbance to the mounds.

After information for determining the volume of the solid mounds is collected (see Section 3.4.5), the slurry removed to expose the mounds will be added back to the tank. Then a fresh batch of simulant shall be added to the tank. The volume of new simulant added to the tank returns the tank to the fill height identified in Table 3-5 and is equal to the 6.5 batch transfer volumes just removed from the tank. The fresh batch of simulant will be prepared in an auxiliary mixing tank(s) so that it can be well mixed prior to and during the transfer into the test tank. During refilling care shall be taken to prevent or minimize any disturbance of solids left behind after the previous transfer. The transfer from an auxiliary mixing tank into the mixing tank will be similar to the DST process that is expected to add the new slurry to the center of the tank. Testing at SRNL used the fastest fill rate that did not appear to disturb the piles.

A series of transfer and refill operations shall be performed and the solids left in the tank shall be characterized prior to the start of the next tank fill (see Section 3.4.5). Solids characterization can include length, depth, and width measurements of the mounds coupled with photographs that show the mound topography. Additionally, qualitative descriptions of the residual solids will be documented to augment the photographic records. Ten successive transfer and refill operations will be performed to evaluate whether or not the mounds left in the tank continues to increase after each tank volume transfer. Preliminary results from the SRNL solids accumulation scouting studies suggest that solids may cease to accumulate after seven cycles (SRNL-STI-2012-00508 (in process)). Ten tank volume transfers represent about one-half of the number of tank volume transfers that will originate from DST 241-AW-105, the tank with the greatest number of planned transfers to the WTP.

3.4 SAMPLE COLLECTION AND CHEMICAL ANALYSIS

3.4.1 Simulant Qualification

Prior to the performing the first test of each campaign and subsequent cycles within a campaign, the simulants will be prepared and qualified. The solid particulates are qualified for use prior to testing in accordance with Section 3.1.1.2. The supernatant will be qualified on-site in accordance with requirements in Section 3.1.2.2. The first batch of simulant can be prepared in the mixing tank but subsequent batches within a cycle shall be prepared in an auxiliary tank so that the critical properties can be confirmed prior to mixing the new material with residual

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material in the tank. Once the critical properties of the supernatant are confirmed the base solids can be added. For supernatant batches prepared in the auxiliary tank, the base solids will be added to the supernatant before it is transferred to the mixing tank but after the critical properties of the supernatant are confirmed. Adding the base material to the supernatant in the auxiliary tank ensures that adding the solids to the slurry does not adversely affect the accumulation of material in the tank.

3.4.2 Pre-Transfer Samples

Prior to conducting the first batch transfer, the tank contents are mixed at the operating conditions until mixing in the tank has stabilized. During tank stabilization, the transfer pump is engaged so that the specific gravity of the transferrable slurry can be monitored by a Coriolis meter located downstream of the transfer pump. During tank stabilization the transfer pump discharge is re-circulated back into the tank. Monitoring the mass flow rate and slurry specific gravity will allow an assessment of the systems capability to mix and convey the complex simulant. Once the system has stabilized, two pre-transfer samples have historically been collected to provide a basis that subsequent transfer batches have content similar to the pre-transfer samples that are used to certify the batch for delivery. For solids accumulation activities pre-transfer samples will not be collected. Scaled performance testing performed according to RPP-PLAN-52623 evaluates the adequacy of the pre-transfer sample to characterize each transfer batch.

3.4.3 Batch Transfer Samples

Prior to conducting the first batch transfer, the tank contents are mixed at the operating conditions until mixing in the tank has stabilized. Once the tank contents are stabilized, batch transfers are initiated and slurry samples for each transfer batch, including each half-batch transfer, are collected for chemical analysis. Similar to previous work, batch transfer samples will be diversion samples through sample ports whose valves are programmatically controlled and correlated to the position of the mixer jet nozzles using encoders. Samples shall be collected downstream of the transfer pump. Batch transfer samples shall be collected in a manner that avoids bias. To avoid bias introduced by flow dynamics around the sample port, the full stream will be diverted to collect the samples. To avoid bias caused by the cyclical nature of the mixing system that directs the jet directly at the transfer pump twice per revolution, the transfer samples shall be collected for an integer value of full rotations of the mixer jets. Samples for the 1:21-scale tank shall collect the entire volume of the transfer batch (16 gallons) and this volume shall be sub-sampled for chemical analysis (see Section 3.4.6). For the 1:8-scale system, only part of the transfer batch will be collected for sampling. For the 1:8-scale system, the slurry will be diverted into a single collection basin during four regularly spaced intervals during each transfer. The four slurry samples are combined to form a representative sample for the entire transfer batch that will subsequently be subsampled. The duration for collecting the four diversion samples will be equivalent and will be equal to an integer value of mixer jet full rotations. For the half batch transfer, the interval between collections is shorter, but the collected volume is the same. Because the mixer jet pumps rotate at different speeds for the two different nozzle velocities considered, the sample duration and hence volume of material collected during sampling varies between tests. The total volume of the slurry sample collected during a transfer for the 1:8-scale system will be similar to the full transfer batch volume for the 1:21-scale system

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(16 gallons). The collected sample will be approximately 4.7% of the 344 gallon transfer batch. The mass and volume of the collected material for the batch transfer samples shall be measured and recorded. The collected volume is then sub-sampled for chemical analysis (see Section 3.4.6).

3.4.4 Heel Samples

After the 6.5 batches have been withdrawn from the tank, the tank will contain residual material composed of the solid mounds in the dead zones as well as residual slurry that is not pumped from the tank. The suspended material in the slurry will settle throughout the tank if sufficient time is provided to clarify the fluid. After the 1st, 5th and 10th tank volume transfer in a campaign, core samples will be collected from the residual tank solids. Scouting studies at SRNL developed a core sampling technique that was successful at removing cores if the free liquid in the tank was removed prior to sampling the mounds. A schematic of the core sampler is provided in Attachment A. A similar device and one modified for the increased height of the 1:8-scale tank and expected greater depth of the heel mounds, will be used to collect the core samples. After the first tank volume transfer, core samples shall be collected from the largest of the two mounds. After the fifth tank volume transfer, core samples shall be collected from the same mound sampled after the first tank volume transfer. Because holes left in the mounds are filled with solids deposited after each cycle, samples collected from the mounds in subsequent cycles (i.e., the fifth and tenth) must not overlap previous sample locations. The number and locations of the samples collected for the first and fifth cycles must account for the need to sample the mounds in subsequent cycles. In addition, the number of samples collected after the first and fifth cycles must not remove more than five percent of the mound. Because there is no need to keep the mound intact after the tenth cycle, the largest number of samples will be collected after the tenth cycle. Sample locations can be marked on the bottom of the transparent tank when the core sampler is inserted to collect each core. All core sample location coordinates must be recorded with each sample identification number so that a map of the fast settling solids can be prepared from the sample results. After the final tank volume transfer, core samples shall be collected from both mounds. After each campaign (i.e., ten tank volume transfers), the core location markings shall be removed from the tank bottom.

The number of samples collected from each mound depends on the size of the mound. Core sample locations will include locations to characterize the center of the mounds. SRNL scouting studies anticipated that heel growth would occur by the deposition of fast settling solids on the edges of the mounds but found that the greatest concentration of fast settling solids occurs in the center of the mounds. Core samples shall be withdrawn from the mounds without disturbing the neighboring material. SRNL demonstrated that the mounds could be core sampled without disrupting the integrity of the mounds if the liquid level was lowered to expose the mounds. Core samples will be collected in a pattern that resembles the mound (e.g. triangular). It is expected that 3 to 10 samples will be sufficient to assess how the fast settling solids are distributed throughout the mounds (evenly distributed versus concentrated at the center or the edges). For the smallest mounds two samples shall be collected from the mound near the tank wall and one shall be collected from the mound towards the center of the tank. For larger mounds this pattern will be followed expanding the number of tank wall samples to three or four depending on the size of the mound. At the end of the campaign, two core samples from the interior of the tank shall also be collected to characterize the suspended material that settles in

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the tank once the final batch transfer is completed and the mixer jet and transfer pumps are turned off.

The collected samples may be subdivided into segments. Collecting analytical data from different segments of the core allows for some vertical discretization of the heel. At a minimum each core taken from the 1:21-scale tank that is longer than 0.5 inches in length will be subdivided so that the presence of the spike component in the bottom half inch of the mounds can be determined. Similarly, each core taken from the 1:8-scale tank that is longer than 1 inch in length will be subdivided so that the presence of the spike component in the bottom inch of the mounds can be determined. With the exception of the bottom segment, which is 0.5 inches long in the 1:21-scale tank and 1-inch long in the 1:8-scale tank, the length of any additional discretization will be based on visual observation of layering in the sample cores. To reduce the number of analytical samples submitted to the laboratory, core samples will only be divided into more than two segments if layering is evident. An example of layering is shown in Figure A-3 in Attachment A. . The minimum length of any segment is determined by the analytical laboratories sample volume requirements and the depth of the mounds. The length of the segment, or entire core if segments are not collected, will be recorded so that coarse vertical partitioning of the fast settling solids can be mapped. The segments will be placed into separate containers, individually labeled, and shipped off-site for chemical analysis in accordance with requirements in Section 3.4.5.

3.4.5 Heel Volume Measurement

Scouting studies at SRNL successfully demonstrated two techniques for estimating the heel volume (SRNL-STI-2012-00508 (in process)). Both techniques required that the liquid level in the tank be lowered to expose the solids. One method successfully demonstrated used an automated positioning system and laser depth finder to measure the depth from a known reference elevation to the surface of the mound. The x-, y- positioning was computer controlled. The height of the mound at each position was determined by subtracting the distance measurement to the surface of the mound from the reference elevation used to establish the distance. The x,y,z measurements were plotted in MS Excel to create three dimensional maps of the mounds. An area was computed for each measurement location. Areas closer to tank wall were larger than more central areas. Each measurement of mound height was multiplied by its associated area to give an increment of volume. Increments of volume were summed to obtain the mound volume. The measurement uncertainty for this technique is (preliminarily) estimated to be 7%.

A photographic technique was also demonstrated at SRNL. For the photographic technique a camera was setup at a stationary point ten feet above the tank. In addition, a hand held camera was available. Enough agitation was applied to suspend most of the gibbsite but not enough to disturb sand and stainless steel. Most of the gibbsite suspension was pumped from the tank and then the remainder was drained before any measurements were taken. The goal was to limit the deposition of gibbsite on the mounds. Arrow shaped boards marked with N and S and a dial indicator initially indicating zero tank level were placed in the tank to identify the north and south mounds and the fact that the tank was nominally empty. The entire tank was photographed using the overhead camera and then the handheld camera was used to obtain a closer image of each mound. Then the liquid level in the tank was increased in increments by adding the withdrawn fluid back into the tank. At each new tank level the dial indicators were reset and

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photographs were taken. This process was repeated until both mounds were submerged. Later, the photographs were analyzed to determine the shoreline of the mounds and the area within the shoreline for each tank level. The shapes were superimposed using marker devices placed into the tank for alignment and a topographic map was formed. Mound volume was calculated by integrating area (square inches) with height (inches) to give volume (cubic inches). The initial method used to perform the integration was Simpson's Rule. However, that method requires that all level increments be equal. Later, the Trapezoidal Method was used which is less accurate but more flexible. The measurement uncertainty for this technique is (preliminarily) estimated to be 20% for large mounds and more for smaller mounds. Volume estimates between the laser measurement technique and the photographic technique were within 20% for the larger observed pile but were not as accurate for the smaller pile because the liquid height increment used to establish the shore line approximated the height of the mound and therefore good resolution of the mounds could not be established.

SRNL scouting studies demonstrated the viability of each technique, although the laser measurement technique was reported to be more accurate. For the larger of the two mounds, SRNL reported that the accuracy of the laser technique was $\pm 7\%$ compared to $\pm 20\%$ for the photographic technique. In addition, the photographic technique required much more labor and analysis after the information was collected. Adoption of one or both of these techniques for SSMD solids accumulation testing will consider the most efficient use of resources (budget and schedule).

If only the photographic technique is adopted to estimate the heel, a check on the accuracy of the technique can be performed if the volume of fluid added back into the tank to raise the liquid level is measured and recorded and the resulting liquid level is also measured and recorded. The volume of both mounds can be determined from the difference between the expected liquid level increase for the volume of fluid added to an empty tank and the observed liquid level increase. If the mounds were fully submerged at the end of the last transfer and the free liquid was drained from the tank, it will be assumed that the pores remained saturated when calculating the volume displaced by the solids.

After the final transfer of each campaign in the 1:21-scale tank, the information necessary to characterize the volume of the mound will be collected as described above and then the entire contents of each mound will be removed from the tank and weighed. The contents will then be rinsed to remove the supernatant, dried, and weighed to determine the total solids content in the mounds. The mound boundaries will be obscured by the suspended solids that settled when the mixer jet pumps were turned off. The criteria used to delineate the mound boundaries (e.g., edge height that is equivalent to the height of the settled solids in the center of the tank) must be consistent across scales and campaigns. The dried contents will be homogenized and two samples will be collected to characterize the component speciation of the mounds (see Section 3.4.6). Subsequently, the remaining mass in the tanks will be removed and the dried mass of rinsed solids determined. Because of the much larger anticipated size of the piles in the 1:8-scale system, it may not be practical to replicate this process in its entirety for the larger tank. Performing a total tank solids characterization after the final transfer of the campaign for the 1:8-scale tank will be reevaluated when the total volume of the mounds left in the tank is understood.

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3.4.6 Chemical Analysis

Prior to the start of testing, analytical method development shall be performed to determine the sample preparation error associated with measuring the base material content in the presence of the supernatant rheology modifiers. This will be done for SSMD scaled performance testing and is applicable to SSMD solids accumulation testing that uses similar simulants. The analytical method is considered acceptable if it produces an unbiased result with a relative standard deviation of less than 10%.

The collected volume from each batch transfer sample will exceed the amount practical for laboratory analysis and will be subsampled at the test platform. For batch transfers, the collected volume representing each transfer batch will be settled in a large volume container. In previous testing, the collected material was clarified for 24 hours in a mixer barrel prior to decanting the liquid. This method will be refined during SSMD scaled performance testing to ensure that the subsamples can be collected in a reasonable amount of time and be representative of the content of the composited material. The mass and volume of the slurry will be recorded. The liquid will be decanted. The decanted liquid will be homogenized and sampled. The collected sample will be weighed and filtered to remove any collected solids. The solids will be rinsed to remove any supernatant residue, dried, and weighed to determine the weight percent solids decanted with the liquid. For mass balance purposes the solids captured in the decanted liquid will be assumed to slowly settle small gibbsite. The weight percent solids in the decant will be multiplied by the mass of the decanted liquid to determine the mass of decanted solids in the slurry sample.

After decanting, the wetted solids will be mixed in a rotating mixer barrel prior to sub-sampling. Four representative and an equal number of archive samples will be collected randomly from the solids. The four wet solid subsamples of the batch transfer samples and core sampler segments (i.e., heel samples) will be shipped off-site for laboratory analysis; the four archive samples for the batch transfer will be retained on-site in a managed area to prevent a loss of sample integrity. The samples will be analyzed for the weight percent of dry solids and the weight percent of each primary constituent (gibbsite, zirconium oxide, silica sand, and stainless steel) in the dry solids. The analytical laboratory will receive the samples, weigh the samples, filter the solids from the liquid, rinse sodium thiosulfate from the filtrate, dry the solids, weigh the dried solids, and then subsample eight times. Portions of the eight subsamples will be digested using EPA Method 3052, *Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices*. Sample content of aluminum, chromium, iron, nickel, and zirconium will be determined using EPA Method 6010C, *Inductively Coupled Plasma – Atomic Emission Spectrometry*. The mass concentration of gibbsite in each subsample is determined from aluminum results; the mass concentration of stainless steel is determined from the chromium, iron, and nickel results; the mass concentration of zirconium oxide is determined from the zirconium results. Portions of the eight subsamples will also be totally digested using the fusion procedure of ASTM D4698, *Standard Practice for Total Digestion of Sediment Samples for Chemical Analysis of Various Metals*. Silicon content for quantifying the sands present in the digested samples will be determined using EPA Method 6010C. In preliminary work reported by the laboratory, ASTM D4658 yielded better results for sand than EPA Method 3052.

Archive samples will be analyzed if the analytical samples become lost or damaged or if additional analysis is determined to be necessary. Off-site analytical services will be performed

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by a laboratory that operates under a Quality Assurance program that has been evaluated against quality requirements in ASME NQA-1-2004 including addenda, or a later version. Analytical data is required to be enhanced quality so that all sample collection, sample analysis, sample handling, and data reporting shall be traceable to the test performed. The sample results shall be reported in a Microsoft Excel¹ compatible format.

The sample results, including weight percent dry solids and weight percent of each simulant component in the dried solids will be used along with mass balance expressions to evaluate the mass balance on the tank system (see Section 3.4.7 and Appendix B).

3.4.7 Mass Balance

The mass balance equations for the solids in tank, expressed in terms of the data that will be collected during the test is describe in Appendix B and summarized here. The mass of each component added to the tank is recorded as it is added to the tank. The mass flow rate, volumetric flow rate and specific gravity of the material withdrawn from the tank during each transfer is also recorded in one second increments during each transfer. The mass flow rate data or volumetric flow rate data and specific gravity data can be integrated to determine the total mass transferred from the tank. Adjusting for the supernatant density, the mass of solids transferred from the tank can be determined. The mass of each component transferred from the tank in each transfer batch can be estimated from the chemical composition data for each transfer batch once the mass of transferred solids in each batch is determined. Compiling all the batches yields an estimate for the mass of each component withdrawn from the tank. The difference between the amount of each component added during a campaign and the amount withdrawn from the tank during each campaign yields an estimate for the amount of material left in the tank.

After each campaign, the mass of residual solids in the mounds in the 1:21-scale tank will be dried and then measured once it is emptied from the tank. The rest of the material from the tank will be added to the dried material and it will be subsampled for analysis (Section 3.4.6) to determine the mass of each component left in the tank. However, for the 1:8-scale tank the mass in the tank could be between 2000 and 4000 pounds. Accurately drying, weighing, and homogenizing such a large volume of material to collect a representative sample of the solids may not be practical to close the mass balance. Therefore, testing in the 1:8-scale tank will rely on the difference between added material and removed material to calculate the material remaining in the tank. The error in the mass of the material removed is derived from integrating the mass flow rate readings reported every second from the coriolis meter. The uncertainty for the mass flow rate reading from the coriolis meter is $\pm 1\%$ and is largely attributed to the uncertainty in the data acquisition system reporting the values. Calculating the speciation of the mass transferred introduces additional errors. The analytical measurements for each transfer batch have analytical uncertainties on the order of $\pm 10\%$, which must be propagated for the 70 sequential transfers (six full transfers and one half transfer for each of ten cycles). Propagating the uncertainty through results in a speciation uncertainty of about 83%. There is additional unknown uncertainty pertaining to how well the analyzed samples represent the entire transfer batch. Therefore, the error in the estimate for the mass of each component transferred may only yield a gross approximation for the residual mass of each component left in the tank. In order to

¹ MS Excel® is a registered trademark of the Microsoft Corporation, Redmond, WA.

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provide as much insight into the content of the mounds, more core samples will be taken after the tenth cycle than after the first or fifth cycles (see Section 3.4.4). In addition, qualitative descriptions of the mounds will be made after the tenth cycle is completed. The mound will be sliced radially several times to expose the interior of the mound. At a minimum, the radial slices will divide the mound into four sections, including a slice down the apparent center of the mound. Observations of layering or an uneven distribution of solids in the mound will be documented and captured in still photographs. Photographic records of horizontal slices of the mounds, also in several inch increments, will also be taken and compared.

3.4.8 Other Performance Data

In addition to collecting slurry samples for chemical analysis, other performance data will be collected. Each system in the SSMD test platform has the capability to record operational parameters such as test time, slurry temperature, mixer jet pump flow rate, mixer jet angular position, mixer jet pump rotational rate, tank level, slurry transfer rate and slurry specific gravity. This data is recorded by a data acquisition system and shall record data for the entire test duration. In addition, performance data shall also be recorded in the test log during testing. Performance data describing the dimensions of any accumulated material in the tank shall be collected throughout the test, noting specifically when changes in tank stability occur due to a change or process interruption. In addition, cloud height and effective clearing radius measurements shall also be recorded in the test log. The effective clearing radius can be determined while the mixer jets are running by measuring the distance from the edge of the mixer jet pump nozzle to the edge of the pile of solids that has stabilized on the sides of the tank. Multiple measurements shall be collected in each test to determine an average effective clearing radius. Measurements shall be collected for each batch transfer to support an evaluation of changes in the system as the tank level is lowered.

3.4.9 Solids Accumulation Analysis

Once the analytical data for all of the collected samples is analyzed the performance of the system will be evaluated. The mass of each component transferred from the tank will be calculated and compared to the amount of material added to the tank. After the campaign is completed, the difference will be the estimate for the material that is left in the tank at the end of the campaign. In the 1:21-scale system, this estimate will be compared to the heel solid measurements collected at the end of the campaign. The distribution of mass using this heel solids estimate will be compared to the initial distribution of mass in the simulant to compare how effectively fast settling solids are removed from the tank relative to slow settling solids. In addition, the change in the amounts of each component transferred from batch to batch will be evaluated for changes between cycles. If the mass of a particular component transferred in sequential cycles is constant and equal to the amount added for each cycle, the solid is not being continuously accumulated in the tank.

Additionally, the volume of the solids mounds will be calculated and plotted as a function of transfer cycle to determine if and when the volume of the mound stabilizes. This analysis will assume that the pore volume in the mounds is stable. The point at which the mounds appear to stabilize will be compared across scales as well as nozzle velocities. If the mounds stabilize similarly among the two scales, it can be inferred that a full-scale DST operated under similar conditions would stabilize similarly. Without a scale up relationship for mound accumulation,

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there will be no basis to state that the planned operating conditions of the full-scale system will stabilize similarly. However, if the performance at two different nozzle velocities is similar, it can be inferred that the mound stabilization behavior may not be strongly dependent on the operating conditions and hence the scale-up relationship.

Also, the mass fraction of each fast settling solids (i.e., stainless steel, sand, and tungsten alloy) in the core samples will be mapped according to the x,y sample location in the mound and segment height if a coarse vertical discretization of the cores samples was obtained. Plotting the fraction of fast settling solids in collected core samples as a function of its location and overlaid with a mound profile would show how the fast settling solids are spatially distributed in the mounds. Solid content collected from adjacent sample locations but from different cycles of the same campaign will be compared to determine if the fraction of fast settling solids in the heel increases as subsequent cycles are performed. Increasing content of the fast settling solids after subsequent cycles is indicative of solids accumulation but is only indicative of a change if the size of the cores that are compared were the same. The conclusions on solids accumulation will be compared across scales and nozzle velocities. If similar accumulation behavior is observed, then it can be inferred that a full-scale DST operated under similar conditions would accumulate solids similarly. Without a scale up relationship for mound accumulation, there will be no basis to state that the planned operating conditions of the full-scale system will accumulate solids similarly. However, if the performance at two different nozzle velocities is similar, it can be inferred that the accumulation behavior may not be strongly dependent on the operating conditions and hence the scale-up relationship.

Finally, the mass fraction of the spike particle (i.e., tungsten alloy) in the bottom segment of the core samples will be mapped according to the x,y sample location if a coarse vertical discretization of the cores samples was obtained. The tungsten alloy was added after the initial cycle so that a mound of fast settling solids was already present in the tank when the tungsten alloy was added; therefore, the tungsten alloy could not be at the bottom of the mound as a result of initial deposition. The presence of the tungsten alloy at the bottom of a mound would indicate that the most dense particle added to the tank migrates to the bottom of the mound over the course of multiple fill and empty cycles and could become concentrated at the bottom of the waste feed staging tank. The absence of the spike particle at the bottom of the mound would suggest that the fast settling solids are deposited in the pile and mixing under similar conditions is inadequate to disturb the center of the piles enough to allow concentration of particles added to the tank in subsequent cycles. The mass content of the other components in the sample segment would need to be taken into consideration to ensure that, if present at the bottom center of the mound, the spike particle was not deposited into an open core hole from the previous cycle. The conclusions on spike particle migration through the mound will be compared across scales and nozzle velocities. If similar migration behavior is observed, then it can be inferred that a full-scale DST operated under similar conditions would concentrate solids similarly. Without a scale up relationship for mound accumulation, there will be no basis to state that the planned operating conditions of the full-scale system will concentrate solids similarly. However, if the performance at two different nozzle velocities is similar, it can be inferred that the concentration behavior may not be strongly dependent on the operating conditions and hence the scale-up relationship.

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4.0 TEST COORDINATION

All testing equipment operations are performed by trained and qualified subcontracted personnel under the supervision of a Test Director. An operations plan, including test run sheets, will be prepared that describes the precautions and limitations, the testing sequences, testing prerequisites, startup conditions, and test procedures in stepwise detail. The TOC technical representative(s) must concur with the operations plan. The Test Director coordinates testing activities including ensuring that all test conditions required for the startup of testing have been performed and all test records (e.g., Test Log, Test Deficiency Reports, Test Change Requests, etc.) are maintained. The Test Director is also responsible for coordinating test activities with the Quality Assurance representative to ensure testing is performed in accordance with the approved quality assurance plan. While tests are conducted, the Test Director will also determine which changes do not adversely affect the acceptance criteria and/or methods by which the acceptance criteria are to be accomplished and are considered “inconsequential” or “minor” and approve these test changes. All other changes require concurrence with the TOC technical representative(s) before the change(s) is/are implemented.

4.1 PRECAUTIONS AND LIMITATIONS

The Job Hazards Analysis is the process for identifying, evaluating, controlling, and communicating potential hazards associated with the work being performed, including modifications to test facilities and test equipment. SSMD solids accumulation testing is being performed in a test facility constructed to perform the testing. The test facility is governed by a facility specific Job Hazards Analysis documented in a Job Hazards Analysis checklist or equivalent document. Changing conditions that modify the test facility or equipment to accommodate testing will be evaluated in a revision to the Job Hazards Analysis before the modifications to the facility or equipment are performed. Workers performing work in the test facility governed by the Job Hazards Analysis shall review the document hazards and acknowledge that they understand the hazards associated with the work being performed and will abide by controls (e.g., don required personal protective equipment, obey posted signs and placards) put in place to mitigate or eliminate the hazards.

Any special precautions that must be taken or test limitations will be documented in the operations plan specifically prepared for each activity and will be communicated to workers before the start of work during a Pre-Job briefing.

4.2 SEQUENCE OF TESTING, PLANT CONDITIONS, SPECIAL EQUIPMENT

Any special requirements for the testing sequence, plant conditions (including connecting to site utilities and site restoration), or special equipment that are not identified in Section 3.0 will be documented in the operations plan specifically prepared for each activity.

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5.0 DATA COLLECTION AND TEST RESULTS REPORTING

Testing shall be conducted in accordance with an approved operations plan. The approved SSMD data collection and accuracy plan that was updated for all DNFSB 2010-2 work scope (PL-SSMD-EG-0003, *Waste Feed Delivery Small Scale Mixing Demonstration Data Collection and Accuracy Plan Rev. 2*) is applicable for solids accumulation work at the SSMD test platform. The data collection and accuracy plan shall be updated as necessary if on-going analytical development work indicates that the analytical uncertainty information previously provided is out of date or if additional instrumentation is necessary to perform tasks identified in this test plan. All test activities shall be performed according to test run sheets. All major testing activities shall be documented in a test log. Test deficiencies shall be reported in a Test Deficiency record.

Test data identified in Section 3.0 , including test durations and test conditions, shall be recorded in the test log. Applicable data not recorded by a data acquisition system shall be recorded on the run sheet or recorded in the test log. All electronic data collected by a data acquisition system shall be content reviewed for error and anomalies. Electronic records shall be submitted to the TOC for evaluation.

All laboratory analysis results shall be accompanied by a chain of custody report that was prepared when the samples were collected. The chain of custody shall identify the samples by a unique name, describe the sample type and list the analyses to be performed. The chain of custody shall also document the preparers name and shall acknowledge receipt at the analytical laboratory. All laboratory analysis results shall be submitted to the TOC technical representative in an MS Excel compatible format.

A test result report shall be prepared this test activity. SSMD solids accumulation test activities shall be documented in a test data package that is submitted to the TOC by *EnergySolutions*. The TOC shall perform the required analysis and document the findings in a test report that is reviewed by *EnergySolutions*.

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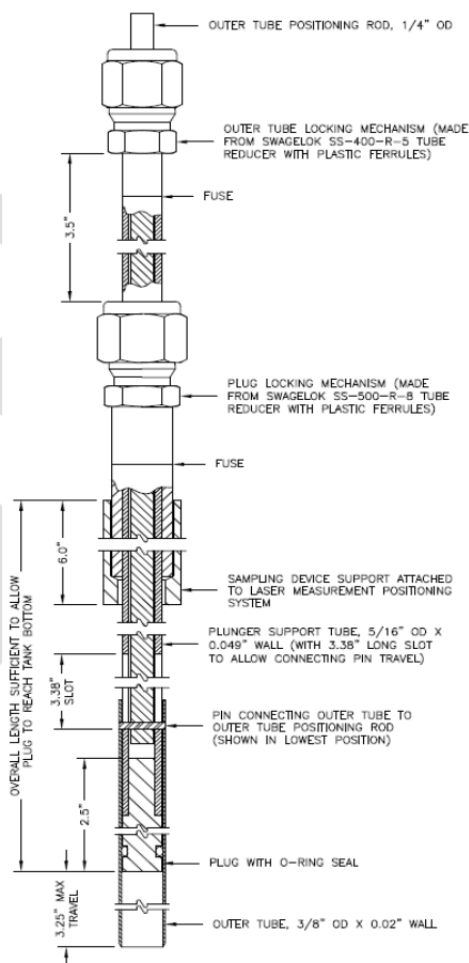
APPENDIX A. SRNL SOLIDS ACCUMULATION SCOUTING STUDIES CORE
SAMPLER

DRAFT

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The prototype core sampler for solids accumulation testing was developed at SRNL during the SRNL Solids Accumulation Scouting Studies. One of the objectives of the SRNL work was to develop applicable techniques to be used during the SSMD solids accumulation testing. The core sampler (See Figures A-1 and A-2) was tested with trial solids mounds and found to extract good cores if the solids were slightly damp and the plug was mildly packed before retracting the sampler. Fig. A-2(a) shows the bottom of the sampler and Fig. A-2(b) shows sampling. The sampler removes most of the solids plug targeted, however, there is always a little bit of the core left behind. The amount left behind was not quantified because of the difficulty obtaining the remnants. As can be seen in Fig. A-2(b), in most cases the overlying solids on the mound, assumed to be mostly gibbsite, back fill the hole as soon as the core sampler removes a plug. However, trial runs indicate better than 95% of the plug is removed by the core sample. Furthermore, when looking from the bottom it was not possible to see where a core was taken.

Figure A-1. Solids Sampler to Extract a Core of Accumulated Solids



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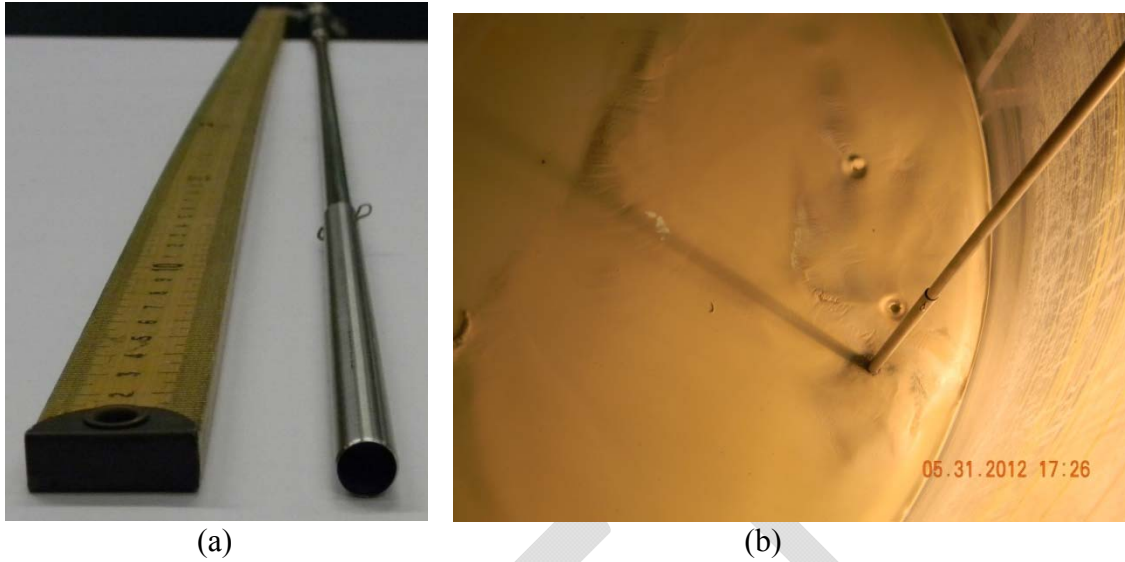


Figure A-2. Core Sampling: (a) The core end of the sampler, (b) a core being extracted during Cycle 1 of Campaign 1



Figure A-3Campaign 2, core sample 20-2 taken from the North mound after Cycle 10,

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Appendix B. **MASS BALANCE**

DRAFT

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This following derivation describes the mass balance for the Solids Accumulation task.

Nomenclature:

M, m: mass ρ: density V: volume X: mass fraction Q: Volumetric flow rate

\dot{M} : Mass Flow Rate

Superscripts:

IN: Mass initially added to the mixing tank

OUT: Mass transferred from the mixing tank

HEEL: Mass that is not transferred from the mixing tank after transfers are completed

SLURRY: The slurry that is transferred in a batch

DIVERSION: A transfer sample is collected by diverting the flow into a collection basin. This is the diversion sample.

DECANT: The diversion sample is decanted and then subsampled. Decant refers to the decanted fluid after it is poured from the collection basin.

WS: (shortened for wet solids) The diversion sample is decanted and then subsampled. Wet solids refers to the residual in the collection basin after it is decanted.

Subscripts:

i: component (gibbsite, silica sand, stainless steel, or zirconium oxide)

L: shortened for liquid/supernatant

S: shortened for solids

Mass Balance on Initial Tank Contents:

The initial mass of material added to the tank is the sum of the mass of the supernatant and the mass of each dried component (gibbsite, silica sand, stainless steel, or zirconium oxide). The mass of the supernatant is determined by the measured density and fill volume. The fill volume is determined by the tank radius, r_{TANK} , and the fill height, h_L^{IN} . The mass of each component added to the tank, M_i^{IN} , is measured before it is added to the tank.

	$M^{IN} = M_L^{IN} + M_S^{IN} = \rho_L V_L^{IN} + \sum M_i^{IN} = \rho_L (\pi r_{TANK}^2 h_L^{IN}) + \sum M_i^{IN}$	B-1
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Mass Balance on Transferred Slurry in One Batch:

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In the following discussion, multiple samples will be taken and averaged to represent the sampled quantity. For simplicity, the discussion is presented as if only a single sample is collected. Slurry will be transferred from the tank in successive batches. The mass fraction of each component in a dried solid sample collected during the batch transfer will be determined by an analytical laboratory. In addition, the mass fraction of solids and liquid in the transferred slurry will also be determined from a sample collected during the batch transfer. The mass fractions will be applied to the entire transfer to determine the amount of each component transferred in the batch.

The transferred slurry will contain supernatant and solids. The mass of the transferred slurry is the sum of the mass of supernatant and the solids.

$$M^{OUT} = M_L^{OUT} + M_S^{OUT} \quad B-2$$

The mass of the transferred slurry, M^{OUT} , will be measured. In the 1:21-scale system the mass of the slurry transferred is weighed directly. In the 1:8-scale system the mass of the slurry transferred is determined using the average specific gravity of the transferred slurry, $\bar{\rho}_{SLURRY}$, and the volumetric flow rate, Q_{SLURRY}^{OUT} , which are determined from the data reported in one second increments, Δt , by the Coriolis meter. The mass flow rate, \dot{M}_{SLURRY}^{OUT} , could also be used to determine the mass transferred.

$$M^{OUT} = \sum \bar{\rho}_{SLURRY} Q_{SLURRY}^{OUT} \Delta t = \sum \dot{M}_{SLURRY}^{OUT} \Delta t \quad B-3$$

The transferred slurry is collected for characterization by diverting the flow to a collection basin. For the 1:21-scale system, 100% the transferred material is collected in the diversion sample. For the 1:8-scale system, a similar volume to the 1:21-scale transfer batch is diverted to a collection basin during the transfer. The volume is approximately 4.5% of the full 1:8-scale transfer batch. One-fourth of the required volume is collected for each of four evenly spaced intervals during the transfer. The diversion sample is weighed, $M^{DIVERSION}$. The ratio of the mass of the full transfer batch and the diversion sample is the diversion ratio, $f_{DIVERSION}$.

$$f_{DIVERSION} = \frac{M^{OUT}}{M^{DIVERSION}} \quad B-4$$

For the 1:21-scale system the diversion ratio is 1 (i.e., the full transfer batch is collected as the diversion sample) and for the 1:8-scale system the diversion ratio is about 22.

The diversion sample is too wet and too large for analysis and is clarified and then the liquid is decanted. Both the decanted liquid and settled solids are weighed, homogenized and subsampled. In order to determine the mass fraction of each component transferred, the subsamples are sent to the analytical laboratory for characterization. The decanted liquid is

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weighed, M^{DECANT} . The mass of the wet solids, M^{WS} , is measured or determined as the difference between the mass of the collected volume, $M^{DIVERSION}$, and the mass of the decanted liquid, M^{DECANT} . It is expected that both the decanted solution and wet solids will both contain solids and supernatant. The solids are exclusively the target components, gibbsite, silica sand, stainless steel, or zirconium oxide. Dissolved components added to attain the correct supernatant density and viscosity dissolve and are rinsed from the solid samples prior to weighing the solids. Additionally, the target analytes are insoluble and do not dissolve in the liquid.

$$M^{DIVERSION} = M^{DECANT} + M^{WS} = M_S^{DECANT} + M_L^{DECANT} + M_S^{WS} + M_L^{WS} \quad B-5$$

In order to determine the mass of solids in the decant of the diversion sample, M_S^{DECANT} , the decanted solution is homogenized and a subsample is collected and weighed, m^{DECANT} . Note the lower case 'm' is used to denote a mass quantity for a subsample. The weighed subsample is filtered to collect the solids. The filtrate is rinsed (to remove any precipitated sodium thiosulfate) and dried in order to determine the mass of solids in the subsample of the decant sample, m_S^{DECANT} . The mass fraction of solids in the subsample (and decant solution if the solution was homogeneously mixed when the subsample was collected) is the ratio of mass of dried solids in the subsample and the wet weight of the subsample.

$$x_S^{DECANT} = \frac{m_S^{DECANT}}{m^{DECANT}} \quad B-6$$

The mass of solids in the decanted solution of the diversion sample is the product of the mass fraction of solids in the decant subsample and the mass of the decanted solution.

$$M_S^{DECANT} = x_S^{DECANT} M^{DECANT} \quad B-7$$

If adequate time is allowed for settling and a good decanting technique is applied, M_S^{DECANT} can be assumed to be very slow settling, small gibbsite and may be negligible. If not negligible the solids in the decant is assumed to be all gibbsite, so that the fraction of component i in the decant solution, x_i^{DECANT} , is one for gibbsite and zero for each of the other components.

$$M_i^{DECANT} = x_i^{DECANT} M_S^{DECANT} = x_i^{DECANT} x_S^{DECANT} M^{DECANT} \quad B-8$$

In order to determine the solids content in the wet solids of the diversion sample after the clarified solution has been decanted, a subsample of the homogenized wet solids is collected and weighed, m^{WS} . The wet subsample is then rinsed to remove sodium thiosulfate, dried and weighed, m_S^{WS} . The solids content of the wet solids, x_S^{WS} , is the ratio of the two measurements.

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$$x_S^{WS} = \frac{m_S^{WS}}{m^{WS}} \quad \text{B-9}$$

The collected subsample of the wet solids from the diversion sample is analyzed for chemical content. The mass fraction of each component i in the solids, x_i^{WS} , is determined by the analytical laboratory.

The mass of solids transferred and retained in the wet solids of the diversion sample is the product of the solids content of the wet solids, determined using the subsample, and the mass of wet solids in the diversion sample.

$$M_S^{WS} = x_S^{WS} M^{WS} \quad \text{B-10}$$

The mass of each solid component in the solids transferred and retained in the wet solids is the product of the mass of solids retained in the wet solids and the mass fraction of each component in the solids.

$$M_i^{WS} = x_i^{WS} M_S^{WS} = x_i^{WS} x_S^{WS} M^{WS} \quad \text{B-11}$$

The mass of each component i in the diversion sample is the sum of the mass of each component in the wet solids and the decanted solution.

$$M_i^{DIVERSION} = M_i^{DECANT} + M_i^{WS} = x_i^{DECANT} x_S^{DECANT} M^{DECANT} + x_i^{WS} x_S^{WS} M^{WS} \quad \text{B-12}$$

The mass of each component i transferred from the tank in the batch is the product of the diversion ratio and the mass of each component in the diversion sample.

$$M_i^{OUT} = f_{DIVERSION} M_i^{DIVERSION} \quad \text{B-13}$$

Mass balance on subsequent transfers in a cycle:

The process is repeated for each transfer batch in a cycle. The transferred masses are added together to get the entire mass transferred during a cycle.

$$M_{CYCLE,i}^{OUT} = \sum_{Batch=1}^{6.5} M_i^{OUT} \quad \text{B-14}$$

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Mass balance on subsequent additions for additional cycles in a campaign of ten cycles:

For subsequent cycles mass additions are made. Equation B-1 applies except that the volume of supernatant added, V_L^{IN} , is determined by direct measurement as it is added to the tank. The total amount of each component added during a campaign is the sum of each amount added during each cycle of the campaign.

$$M_{TOTAL,i}^{IN} = \sum_{Cycle=1}^{10} M_i^{IN} \quad B-15$$

Mass balance on subsequent subtractions for additional cycles in a campaign of ten cycles:

For subsequent cycles mass transfers are made. Equation B-14 applies and is additive. The total amount of each component transferred during a campaign is the sum of each amount transferred during each cycle of the campaign.

$$M_{TOTAL,i}^{OUT} = \sum_{Cycle=1}^{10} M_{CYCLE,i}^{OUT} \quad B-16$$

Mass Balance on Tank Heel Contents:

After the last transfer is completed, the tank is not empty. The last transfer does not remove all of the liquid from the tank, a certain heel amount, equal to 72 inches of slurry in the full-scale double shell tank, is not removed from the tank. The slurry will contain suspended solids while the mixer jets are running. The suspended solids will settle, coating the bottom of the tank and any mounds of solids that are not influenced by the mixer jets. Together these solids comprise the heel solids left in the tank. Because there is no chemical reactions occurring for any of the analytes, the theoretical mass of each component left in the heel is the difference between the total mass of the component added during the campaign and the amount transferred out of the tank.

$$M_i^{HEEL} = M_{TOTAL,i}^{IN} - M_{TOTAL,i}^{OUT} \quad B-17$$

Core samples will be collected from the heel mounds to characterize the solid content of the mounds. However, earlier work suggests that the composition of the heel mounds is not uniform, the center of the mounds contain more fast settling particles than the edges. Therefore, it is not expected that the core samples will be adequate to estimate the heel content and close the mass balance.

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Enclosure 3

LSIMS ERT DOCUMENT REVIEW RECORD			REVIEW NUMBER:	ERT-20 Feed Test Plan 3
			DOCUMENT NUMBER:	RPP-PLAN-53193 Rev A
			DOCUMENT TITLE:	One System Waste Feed Delivery Mixing and Sampling Program Solids Accumulation Test Plan
Comment			Comments and Recommendations:	Resolution:
Number	Reviewer	Type*		
1	LMP	M	Test plan proposes measuring heel accumulation after first, fifth, and tenth fill. Preliminary data (SRNL memo, Table 6) suggest that mounds are essentially the same after five and ten fills. Should plan be altered to look at third fill, for example, instead?	Heel volume will be measured after every cycle. The option to perform fewer than 10 cycles in a campaign has been removed. Until more tests are performed, there is no definitive information on solids accumulation to say with certainty that the 1:8-scale will behave the same. Also, in real time it will not be known if the tank mounds have stabilized in volume or whether the newly added spike particle is migrating during subsequent cycles.
2	LMP	O	“Evaluate the propensity” is a fuzzy test objective. “Determine if fast settling solids will accumulate” is also somewhat so absent more detail on how results at two scales will be analyzed and interpreted at full scale.	Agree, this was intentionally “fuzzy” to highlight that these are the first exploratory tests at multiple scales. Additional words have been added to make this concept more prominent and apparent to the reader. Test objectives wording has also been modified.
3	LMP	O	Page 3-19: Whether 10-20% error is acceptable depends on the magnitude of the effect you’re trying to resolve, i.e., does mound volume increase by more than 10% from start to finish of campaign? Suggest including this in the discussion rather than just pointing to test director discretion.	Test director discretion has been removed. Method really depends on cost and schedule and 20% may be improved upon if additional care is applied and more frequent measurements are taken (i.e., after each cycle).
4	LMP	M	Section 5.0: No detail on analysis of data. What will be reported to meet objective/success criterion “to determine if fast settling solids will accumulate”?	Details of data collection for mass balance calculations added in App B.
5	LMP	E	Page 1-3, Figure 1-1: Instead of saying “follow up test plan” in legend, say “this test plan.”	Accepted.
6	LMP	O	Table 2-1: “Up to ten stagings”; what will be basis for cutting testing short?	See response to comment #1.
7	LMP	E	Page 3-6, bottom: Rather than as worded “use significantly more dense material” (which sounds like you’re using “denser material”), say “a larger amount of dense material.”	Text has been edited for clarity.
8	LMP	E	Page 3-7: Should be “principal” not “principle.”	No change.

*Type: **E** – Editorial, addresses word processing errors that do not adversely impact the integrity of the document.
O – Optional, comment resolution would provide clarification, but does not impact the integrity of the document
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9	LMP	O	Very end of Section 3.3: “if it is demonstrated”; how? If you only measure volume of solids after first and fifth batch, how will you know that the volume has “stabilized” such that you can stop?	See response to comment #1.
10	RRH	O	<p>GENERAL</p> <ul style="list-style-type: none"> With a soft objective of “Assessing the propensity to accumulate fast settling solids in the waste feed staging tanks,” this test plan appears to be good; although there are several issues that are not addressed adequately in this document. However it is suggested that while a significant effort is planned, some additional sub-objectives could enhance the value to this tank farm project. These sub-objectives can be adding limited tests with one or two more jet velocities to establish correlations for ECR and cloud height. Also additional jet velocities can be selected based on ‘high’ solids accumulation to ‘no’ solids accumulation. Conducting such tests on two scales would also provide important scaling factors. It is important to establish if the contents of solids mounds are replaced from cycle to cycle by new solids addition; or once mounds are formed they become fixed in their contents and all solids fed in subsequent cycles leave the vessel during transfers. This determination can have important implication on the design and operating strategy of the mixing vessel. How confident are we on the selected simulant representing properties of waste material to be processed? 	<p>See response to Comment #2.</p> <p>A new component will be added beginning in the third cycle. Core samples will determine whether or not this component replaces mound material at the bottom of the pile. If it is there than pile migration is confirmed. If it is not there, our current expectation that the piles don’t really move and distribute themselves is supported by the test.</p> <p>The simulant plan developed the simulant to have characteristics important to mixing and transfer that are similar to the characterized tank waste.</p>
11	RRH	E	Page 2-1: ‘Yield Stress’ has been mentioned in the document at three locations. I understand that only Newtonian simulant will be used in this test program. Therefore yield stress should not be mentioned.	Yield stress has been removed from the document.
12	RRH	O	Results of SRNL study in 1:22 scale are used as focal point for this test program. It should	SRNL work was developmental and we will learn from it. Clearly the small pile

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			be recognized that SRNL study used only two jet velocities 23.9 & 22.9 ft/s, which are not much different. In addition the jet velocities of the two pump jet mixers are differed by 1 ft/s. Therefore the results cannot provide any effect of jet velocity on the mixer performance.	at SRNL was a quantification concern. We will chose two velocities that result in good sized piles but it is expected that velocity range in the smaller tank will be smaller than the velocity range in the bigger tank.
13	RRH	O	In Table 3-1 there are 7 compounds; but chemically they are only 4: Gibbsite, Sand, ZrO and SS. Is there value to reducing these characteristics to 4 compounds. Also if this plan will use only 'Typical' simulant, 2 out of 7 compounds will not be added anyway.	A fifth spike compiound is being added to the simulant. There is no performance benefit to using four vs five components. Any benefit would be related to lower analyticial interference and lower analytical costs.
14	RRH	O	In 3.1.2.1, it is stated that 'Typical' supernatant will be used. It should be recognized that this is not most conservative because for suspension performance low density/high viscosity represents conservative combination.	Solids accumulation is seeking a more realistic simulant, not a conservative one. Initial testing with the typical/typical shows that this simulant combination will result in mounds so that accumulation can be evaluated.
15	RRH	E	In Table 3-4, it would help to <ul style="list-style-type: none"> • add a row for Pump Mixer Jet Nozzle Velocity – 59 ft/s for FS and projected ranges for the other two scales • use the unit of 'ft' for vessel diameter and height • for mixer jet rotation rate it refers to Equation 3-5, which is actually Equation 3-1. 	Changed as requested.
16	RRH		Equation 3-1 on page 3-15 is fine. However it assumes that X/T is constant on all scales. X/T is okay for 1:21 scale, but slightly different for 1:8 scale.	Acknowledged.
17	RRH		Page 3-17, 3.4.2: Protocol for sub-sampling is not described, e.g., how will solids be mixed.	Transferred slurry (~16 gallons from each scale tank) will be decanted and the solids will be mixed in a rotating cement mixer prior to subsampling (added to 3.4.6) and shipping to the lab.
18	RRH		Page 3-19, middle of first paragraph: It is indicated that accounting for the saturated pores of the wetted solids should be taken into account. It does not mention how.	The technique that requires pore volume estimation is not being considered. Instead the pore space in the settled mounds will be assumed constant and the volume of the mounds will be determined using the photographic technique. The mass of the mounds will be determined afer the final cycle of each campaign.

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19	RRH		3.4.6 Chemical Analysis: Chemistry of this technique is not defined or if it has been tested for accuracy.	Analysis discussion has been added. Analytical accuracy work is in process.
20	RRH		It is not clear that what will be used to compare the volume of the mounds with – initial volume of the batch fed, total volume processed in 10 cycles, or any other.	Mass balance discussion added in Appendix B. Also mass of mounds will be weighed.
21	RRH		In my judgement the volume of mounds may be minimized if the two pump mixer jets are out of phase from each other. It is suggested to demonstrate this perhaps during initial shake-down tests.	This is planned work for FY13.
22	EKH		THIS DOES NOT INCLUDE THE GENERAL COMMENTS MADE IN THE LETTER.	Acknowledged.
23	EKH	E	Page i, sentence starting with “Testing will also be performed with slurries...”: This sentence and the previous sentence seem to indicate you are testing more than one type of simulant. Recommend you reword.	Editorial correction made.
24	EKH	O	Page i, same sentence as above “... and the capability of sampling fissile material for comparison to requirements with action limits for U and Pu and to requirements for waste treatment processability...”: Not sure if this is correct; you are not testing the sampler system that will be used in the plant and what are the action limits that the data you’re collecting will be compared against? (Page 2-1, second to last paragraph has same wording.)	Editorial correction made.
25	EKH	E	General: Density of solids are typically reported as g/cm ³ not g/ml.	Editorial correction made.
26	EKH	E	Page 1-2, “Solids Accumulation”: I assume that” propensity” and “understanding” are the same? In paragraph, there seems to be a broader grasp in determining what is accumulating rather than just the fast settling solids as described in Page i. Do not care for the use of propensity.....	See response to Comment #2.
27	EKH	E	Page 1-3, last sentence: Recommend that you list what the experts recommended for this activity; seems you might not have captured all of their thoughts.	We will clarify with Eric. Followed up on the [hone, and then added discussion in Section 1.0. This work will not address operational optimaization, as recommended in the workshops. This is to be investigated at a later time.

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28	EKH	E	Page 2-1, third paragraph: You are using a Newtonian simulant; not sure if it has a yield stress or are you stating the mounds of solids have a yield stress. Either remove the term or be clear on how it will be characterized in this task. I believe yield stress is used throughout this document.	Yield stress has been removed from the document.
29	EKH	M	Page 2-1, third paragraph, second and third sentences: What errors are associated with the “chemical” analysis and what resulting error will you have in the mass balance? Is this acceptable?	Preliminary laboratory evaluation of chemical analysis suggest analytical errors on the order of 10% can be expected. Since this test work is focused on determine initial gross responses of the scaled DST systems, 10% errors are considered acceptable.
30	EKH	M	Page 2-1, third paragraph: Not clear to me that you’re satisfying what is written on Page 1-2, “Solids Accumulation”. It states that you want to understand the accumulation and distribution. My interpretation of distribution in this case would be particle size. Not sure if this is the authors’.	Clarified to “spatial distribution”. We are interested in how the fast settling solids are distributed in the heel (in the center, on the bottom, at the edges). Additional discussion added to the text.
31	EKH	M	Page 2-1, third paragraph: Question: is core sampling a method that is defensible in nature such that you can assess fast settling solids are accumulating? Did the SRNL tests show this to be the case where they could assess fast settling solids are accumulating using the core method? Note ERT general comment on accumulation.	The core sampling performed during SRNL test demonstrated the ability to quantify the fast settling solids in the core samples. While the SRNL work did not attempt to calculate concentration of different simulant the SSMD analytical laboratory method development has demonstrated the ability to determine concentrations of each solid simulant component. Increasing concentration over time of fast settling solids will indicate accumulation.
32	EKH	M	Page 2-1, second to last paragraph: Is one simulant at a specific concentration of fast settling solids sufficient to quantify the ability of the fast settling solids (FSS) to settle in the mounds or to accumulate? Or to make it more clear, if I started with a 0.1 wt% of FSS in the incoming slurry, would the same mass fraction of FSS settle in the mounds as that of 6 wt% FSS? Does concentration in the feed make a difference? Can a simple ratio of the 6 wt% FSS between what was transferred	The test plan has been revised to clarify that this work is being conducted to further the understanding of how solids in the tanks will behave. It is recognized that this work is done with only a single simulant and that other simulants could be have differently. This work is being conducted to identify the potential magnitude of the accumulation problem.

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			and what as calculated to be in the mound the same for any wt% FSS?	
33	EKH	O	Page 3-5, second paragraph: Remove “The recommended particles are irregularly shaped particles.” The simulant document guided the decision on what is used.	Accepted.
34	EKH	E	Equation 3-1 and 3-2: Provide reference for these equations.	Accepted. “...from Handbook of Industrial Mixing: Science and Practice, Equations 10-1, 10-2 and Table 10-1.”
35	EKH	E	Page 3-15, third paragraph, seventh sentence: Note that the during the SRNL tests, the difference between the 22.4 and 23.5 ft/s jet velocities resulted in approximately 1.4% additional SS solids (compared to the SS used) in the 22.4 ft/s test. Not sure these two velocities are significantly different in performance.	See response to Comment #12.
36	EKH	M	Any “Should” has to be changed to “must or shall” or these actions removed.	A review has been conducted and the changes have been made.
37	EKH	E	Page 3-16, second paragraph: Question: is there a vessel at the test facility that can prepared feed for the 1/8 th scale? If not, how will this task be performed?	There are two 450 gallon mixing tanks that will be used to prepare the simulant. Discussion added.
38	EKH	O	Page 3-18, first and second paragraphs: Will the method used by SRNL to minimize the fines that collect on the surface of the mounds be used prior to core sampling and obtaining surface measurements (fines seem to impact this a lot and were removed via reduced PJM velocity, etc.)? Will pumpdown be performed as done in the SRNL test? This report states that pumpdown was done by SRNL, but does not state that such will be performed. This is also applicable to Section 3.4.5	Expanded discussion. The same technique will be used with the exception of the drain hole that was added to the SRNL tank. The liquids will be pumped down.
39	EKH	M	Page 3-18, second paragraph: It is expected that 3 to10 samples will be sufficient to characterize the contents of each mound. Not sure this is a true statement, given SRNL data and the thousands of cores pulled by Hanford tank farms that such an assessment can be made. If such can be done, how would the	After looking at the SRNL data it is clear that collecting only a few the cores will not be able to representatively sample the entire mound because the spatial distribution of FSS in the mounds is not uniform. Therefore the core samples will only be used to identify how FSSs are spatially distributed in the mounds

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			data be used and what are the errors?	(heavy in the center, light on the edges, all at the bottom, etc.) the cores will also be used to see if the spike migrates to the bottom of established piles.
40	EKH	O	Page 3-18, third paragraph: See comment 30 about PSD?	Clarification on distribution added throughout text.
41	EKH	M	Page 3-19, first paragraph: What is saturated pore volumes? Clarify. Most likely there will be additional questions, given the response.	See response to Comment #18.
42	EKH	M	Page 3-20, first paragraph, last sentence: How are the analytical methods determined to be “unbiased”? Please provide explanation.	The lab is currently performing a series of analytical accuracy measurements (testing eight subsamples on redundant samples in each simulant formulation) to quantify the uncertainty in the analytical method. The results will be evaluated for bias using statistical software.
43	EKH	O	Page 3-20, Section 3.4.7, second paragraph: Will the 1:21 scale heel be analyzed? If so, state it clearly. This section is not clear on that.	Yes. On-going discussions with the subcontractor are being conducted to see if a workable solution for charactering the mass in the 1:8-scale tank can be found.
44	RVC	O	Page 3-5: The sentence “ <i>The recommended particles are irregularly shaped particles.</i> ” should be removed. It only serves to generate difficult questions.	Removed.
45	RVC	E	Table 2-2: Same density given in footnote a for Pu and PuO ₂ .	Corrected.
46	RVC	O	Table 3-3: Why do the viscosities of the Low/High and High/High simulants differ by a factor of 2? Why are they not the same?	During simulant plan development it was observed that finding a simple recipe for a low viscosity / high density supernatant using readily available non-hazardous materials in water was not practical. Because the Low/High was not presented as a bounding simulant, it was not pursued further.
47	RVC	O	Section 3.2, page 3-13: When operating in recycle mode, how do you set the location of the point where the slurry is returned back into the tank? Does the location affect/influence the settling pattern and mound buildup prior to the beginning of pump out?	The recycle is discharged near to top of the liquid level so that it drains down the tank wall in the area closest to a mixer jet pump. When the jet is directed at the wall, the upward flow helps to disperse the returned solids throughout the tank similar to if it were discharged towards the wall from the jet.
48	RVC	O	Section 3.3, pages 3-15 and 3-16: You should not fix sample collection to the 1 st , 5 th	A new scheme has been devised. A spike component will be added during

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			and 10 th tank volume transfer. This constrains your ability to measure the rate of solids buildup. You should be more flexible to establish an accurate growth rate curve. You are willing to collect for less than 10 transfers but there is no provision for more than 10. You should also devise a scheme to determine if new solids are exchanged for older ones after a steady mound volume has been attained. Your current procedure does not permit this.	the third fill. The bulk of the mound should be established at this time, certainly the bottom center will be established. Core samples will be collected after the 5 th fill and will look for the spike in the mounds. Core samples will also look for the spike after the 10 th cycle. There is no longer a provision to reduce the number of cycles. Budget and schedule constraints limit the number of tests to 10 in each campaign. An exception to the test plan is always a possibility when warranted
49	RVC	O	Section 3.3, page 3-16 forward: You state “ <i>The new slurry should be well mixed prior to and during the transfer.</i> ” Yet there is no discussion of the mix preparation tank in this report. Can you assure us that the solids will be completely (not uniformly) suspended so none will be left behind? What impeller is used? Tank/impeller size? Baffles? A discussion of the mix preparation and transfer tank should be included.	Added limited discussion.
50	RVC	O	Section 3.4.2, page 3-17: It is stated: “ <i>Prior to conducting the first batch transfer, the tank contents are mixed at the operating conditions until mixing in the tank has stabilized.</i> ” What does stabilized mean with respect to solids suspension and accumulation? Is it possible for the mounds will grow to their ultimate volume during the stabilization phase rather than between transfers 5 to 10?	Added discussion that stabilized is determined by the recycled slurry specific gravity similar to the two previous test plans. Stabilized is only indirectly tied to mound stabilization. If the SpG of the transferable slurry is reasonably stable then the solids in the tank are assumed to be reasonably stable which means that the mounds are not undergoing rapid changes in size or content. Past experience indicates that the mounds do not change significantly when the SpG has stabilized and pile dynamics has always been an informal check on tank stability. SRNL shows that the piles develop quickly.

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51	RVC	O	Section 3.4.4, pages 3-17 and 3-18: How do you analyze the core samples to extract vertical information? What are you looking for and what will you do with that information? Do you always core all the way to the bottom of the mound/settled layer?	If the core has a good consistency it can be removed from the sampler intact and then divided into segments. Knowing where the core was taken an approximate height of the core segment, the information can be used to tell if and where fast settling solids concentrate. In addition analyzing lower core segments for the spike particulate will let us know if newly added material can end up in the bottom of a pile. This would suggest that the piles are dynamically changing rather than forming a permanent composition.
52	RVC	O	Section 3.4.5, pages 3-18 and 3-19: It would be useful to provide a reference for the different heel volume measurement techniques.	The heel volume measurement technique is from SRNL work. The draft final test report has been added as a reference.
53	RVC	O	Section 3.4.6, pages 3-19 and 3-20: Specifically, what chemical analysis techniques will be used? What is the expected accuracy? There is no discussion or reference.	Added discussion of method. The lab is in the process of conducting analytical uncertainty measurements.
54	RVC	M	Section 3.4.7, page 3-20: I am still not comfortable with not closing the mass balance. Every attempt should be made to demonstrate, based on the collected data, that this is acceptable. The uncertainty should be analyzed. I would like to see this addressed in the data and test results reports.	The heel in the small test vessel will be measured and sampled to close the mass balance. We will pursue better closure for the larger tank by exploring practical ways to dry, weigh, and homogenize the heel for sampling.
	RKG		All of Dr. Grenville's comments were incorporated in the review letter.	Acknowledged.
Supp-1			The chemical analysis section (3.4.6) doesn't indicate the approach for analysis of the tungsten particles. Is this readily determined by EPA Method 6010C etc?	As it stands now, the spike particle still needs refining. I have been unable to track down a material that meets three necessary criteria, has the right size (40-100+ microns), has the right density (9.6-11.4 g/cc) and is compatible with the remaining simulants considering both reactivity and analytical interference. The tungsten alloy used in LSIT testing is

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				tracked using Cr, which we use to track SS. This complicates the analysis. Further, the lab we use has some experience with digesting tungsten products and their initial concern is that one or more precipitates would form with the other metals and interfere with the quantification of those components. Discussions with the lab and material suppliers continue. We are investigating a tungsten/copper alloy, as well as Molybdenum powders, but the best product has not been identified yet
Supp-2			It looks like you're planning to use a tungsten particle with d50 of 40 microns (Section 3.1.3), with a settling velocity equivalent to 37-micron PuO2 (Table 3-4). The steel particles are d50 = 112 microns and settle at ~0.1 ft/s (Table 3-2). I can't really find a direct comparison in the document between the settling velocity of the steel and the tungsten, but if tungsten is only slightly more dense than steel (9.6 vs. 8.0) but one third the size, I'm not sure it's really fair to call the tungsten particles "very fast settling" compared to steel. (Weren't both selected to mimic settling of the same sort of largish plutonium oxide particle?)	Very fast settling particles was used to distinguish the spike particles from the base. I agree that the settling velocities will not be significantly different and I have removed the use of very fast settling particles and instead use spike particles when specifically talking about the spike particles and just fast settling solids when referring to SS and spikes.
Supp-3			If tungsten particles are small compared to sand and steel particles, will they be sucked down into the pores of the settled bed when the fluid in the test vessel is drained completely for mound volume measurements? Since the actual vessels always retain a heel, that would be somewhat unphysical. It might be worth doing a small mock-up in the lab to see what happens.	This artifact can be evaluated at bench scale during developmental testing.
Supp-4			These appear to be a step in the direction of quantitative results and predictions of full-	The objectives have been revised to be more consistent with the discussion

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		<p>scale behavior compared to the original document. However, experimental uncertainties in chemical analysis and mound volume are still large, +/- 10%. Note that one of Erich's detailed comments was about total uncertainty in mass balance, not just the analytical uncertainty of 10%. Moreover, is 10% acceptable for chemical analysis? We recommend the addition of a paragraph that explains why 10% is acceptable and how it preserves the ability to make quantitative conclusions. In short, we're not sure you've thought through the error bars you'll have when you're trying to draw the conclusions that are in the revised success criteria. If you have, the document would benefit from that discussion.</p>	<p>added in Section 3.4.9, which includes certain caveats about predictions to full-scale. Reworded objective:</p> <ul style="list-style-type: none"> ▪ Evaluate how fast settling solids could be spatially distributed in a full-scale double-shell tank. ▪ Explore if fast settling spike particles can be concentrated at the bottom of full-scale double-shell tank. <p>Also we have thought about the error bars on the mass predictions and feel that the largest error will be in the specification of the mass of each component left behind. The reported uncertainty in the mass flow rate readings that will be used to estimate the mass transferred from the tank is ~1% and the scale used to add material to the tank is 0.1%. Speciation of the mass is subject to the largest error of 10%. This is propagated over several transfer batches. The propagated error in the speciation after the 70th transfer would be ~83%. We have revised the text to state that speciation in the heel will be a gross estimate.</p>	

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ERT-20 Feed Test Plan 3

Large-Scale Integrated Mixing System Expert Review Team

(L. Peurrung, Chair; R. Calabrese, R. Grenville, E. Hansen, R. Hemrajani)

To: Ray Skwarek, One System IPT Manager

From: Loni Peurrung, Chair, Large-Scale Integrated Mixing System Expert Review Team

Subject: Concurrence on ***One System Waste Feed Delivery Mixing and Sampling Program Solids Accumulation Test Plan*** (ERT-20 Feed Test Plan 3)

Date: September 24, 2012

Dear Mr. Skwarek:

The Large-Scale Integrated Mixing System Expert Review Team (ERT) concurs with the disposition of ERT comments documented in ERT-20 Feed Test Plan 3 as described in your response WRPS-1203839-OS dated September 24, 2012.

This letter closes review ERT-20 Feed Test Plan 3.