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Evaluation of the Mixing System for the West Valley Melter Feed Hold Tank

**C. L. Fow
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B. A. Pulsipher**

January 1989

**Prepared for the U.S. Department of Energy
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SUMMARY

High-level radioactive liquid wastes are currently stored in underground tanks at several Department of Energy facilities. The current plan for management of these wastes is to convert them to a durable borosilicate glass, then isolate the glass in a deep geologic repository.

The process for converting the wastes to glass involves mixing the high-level wastes with glass-forming chemicals, then transforming the resulting slurry into glass using a high-temperature furnace known as a slurry-fed ceramic melter. Maintaining the quality of the glass product and proficient melter operation depends on the ability of the waste slurry preparation and feed systems to produce and maintain a homogeneous mixture of waste and glass-former materials.

Pacific Northwest Laboratory (PNL) performed bench-scale tests in FY 1986 to provide a quantitative description of performance for several feed mixing systems in terms of measurable process variables (Peterson, McCarthy and Muhlstein 1986). One of the systems studied was the West Valley melter feed hold tank (MFHT). The bench-scale study used simulated West Valley melter feed to determine the full-scale parameters for sizing the agitator that is used to mix slurry in the MFHT.

Full-scale tests were performed in FY 1987 at West Valley using the MFHT. The objectives of these tests were as follows: 1) quantify the homogeneity of the melter feed slurry in the MFHT, 2) determine the representativeness of the slurry sample obtained by the liquid-sampling system for the MFHT, 3) determine the time required to resuspend and homogenize the melter feed slurry after a simulated unexpected power outage, and 4) verify the scale-up of the results from the bench-scale mixing study. This report discusses the results from the full-scale tests.

Two types of tests (homogeneity/sampling system tests and resuspension tests) were performed using three concentrations of simulated, nonradioactive melter feed slurry. The melter slurry concentrations tested included the reference melter feed, the reference melter feed concentrated to 75 vol% (concentrated feed), and the reference melter feed diluted to 125 vol%

(dilute feed). The homogeneity/sampling system tests were performed to characterize the melter feed system and verify the bench-scale mixing data. The resuspension tests were performed to determine the time required to resuspend and uniformly mix the solids in the MFHT after a simulated, unexpected power outage.

For the homogeneity/sampling system tests, the homogeneity of the slurry was measured by comparing the average weight percent solids and the elemental concentrations of slurry samples taken from six sampling locations within the tank. The representativeness of the sampling system was determined by comparing the average weight percent solids and the elemental concentrations between the samples taken at six tank sampling locations and the sampling system. For the resuspension tests, the homogeneity of the tank was determined by comparing only weight percent solids of the slurry samples from within the tank.

The statistical design of the homogeneity/sampling system tests required 21 samples at each sampling location to attain a 95% confidence level of detecting a difference of greater than 4.5 elemental weight percent. The statistical design of the resuspension tests required six samples at each sampling location. Samples were taken from the tank and analyzed for total solids and elemental concentrations in a random order. All analyses were conducted at West Valley.

Statistical data analyses were performed on as-analyzed and normalized values of the elemental concentrations. Results from these analyses are summarized in Table S.1. Four significant conclusions can be drawn from this data:

- 1) Analysis of normalized values can sometimes mask interactions. It can be deceptive when certain conditions affect some elements but not others, or affect all elemental constituents similarly. This can have significant impact during feed makeup.
- 2) The 15 Hp, single-speed (155 rpm) agitator installed in the MFHT produces a homogeneous mixture for the three concentrations of simulated melter feed slurry used in these tests. Slurries with significantly greater settling rates may produce different results.

TABLE S.1. Results from Statistical Analysis of Elemental Concentration Data

Slurry Concentration	As-Analyzed Elemental Concentration		
	Sampler Bias Range(a)	Tank Inhomogeneity RSD(b) Range(c)	Analytical RSD Range(d)
Concentrated	0.3% - 5.7%	0.0% - 1.3%	1.4% - 7.8%
Reference	0.4% - 12.2%	0.0% - 0.0%	2.4% - 12.0%
Dilute	2.1% - 29.2%	0.0% - 2.3%	2.0% - 20.7%
	Normalized Elemental Concentration		
Concentrated	0.1% - 1.7%	0.0% - 0.7%	2.6% - 5.3%
Reference	0.8% - 6.9%	0.0% - 0.0%	3.2% - 9.6%
Dilute	0.2% - 13.9%	0.0% - 0.8%	4.2% - 16.1%

- (a) Sampler bias is the difference in elemental concentration between the average of the six tank sampling locations and the liquid-sampling system.
- (b) RSD is defined as relative standard deviation.
- (c) Tank inhomogeneity range is the variability between the six sampling locations (see Figure 4.1).
- (d) Analytical range is the variability in the analytical results for a particular sampling location.

- 3) The liquid-sampling system used in these tests obtains samples that are up to 2.6% higher in total solids, and are significantly different in certain elements. This may be because of the flow rate of the slurry in the sampling system and/or the type of sample bottle used.
- 4) The major source affecting accuracy of the results is the variability in replicate analytical measurements.

Other significant conclusions were drawn from analyses of the data.

These conclusions are as follows:

- After 4 days of settling in the MFHT (simulating an unscheduled power outage), less than 2 hours of agitation were required to resuspend and uniformly mix the slurries tested.
- Scale-up of the bench-scale tests was verified qualitatively. It was shown that the mixing system operated in the turbulent flow region and the liquid in the tank was homogeneous. A precise verification of scale-up of bench-scale data was not possible due to dissimilarities between the bench- and full-scale mixing systems.

- The maximum flow rate of the air-displacement slurry (ADS) pump used for the sampling system does not achieve turbulent flow in a 0.5-in.-(inside)-dia. pipe, which may result in a nonrepresentative sample due to solids settling in the lines.
- The mixing Reynolds numbers calculated for the three concentrations of slurry tested were 1 to 2 orders of magnitude greater than the Reynolds number at which fully turbulent mixing is defined. This implies that the speed of the agitator could be reduced without affecting the homogeneity of the feed in the MFHT, if slurry settling rates are similar to the tested slurries.

The following are recommendations for improving the consistency of the melter feed and reducing the number of required analyses per sample.

- A significant portion of the analytical uncertainty was due to long-term effects (e.g., lab conditions, calibration procedures). A statistical quality control program, detailed in Section 6.5, would isolate and eliminate some of the major sources of variation. Reduced analytical uncertainty reduces the number of required analyses per sample, thereby reducing cost.
- Perform characterization tests on the actual liquid-sampling system to determine procedures that will provide a more representative sample. These tests should include an evaluation of sample bottle design, sampling cycle times, minimum flow rates, and flushing requirements.

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1.0 BACKGROUND AND OBJECTIVES

High-level radioactive liquid wastes are stored in underground tanks at several Department of Energy (DOE) facilities in the United States, including the Savannah River Plant (SRP) in Aiken, South Carolina; the West Valley Demonstration Project (WVDP) in West Valley, New York; and the Hanford Site near Richland, Washington. The current plan for managing high-level radioactive wastes is to convert the waste to a durable borosilicate glass, and isolate the glass in a deep geologic repository.

The process for converting the wastes to glass involves mixing the high-level wastes (HLW) with glass-forming chemicals. The resulting slurry is then transferred to a feed tank and processed through a high-temperature furnace known as a slurry-fed ceramic melter (SFCM). Maintaining the quality of the glass product and safe, efficient operation of the melter depends on the ability of the melter slurry preparation and feed systems to produce and maintain a homogeneous mixture of waste and glass-former materials.

As part of the Nuclear Waste Treatment Program (NWTP), Pacific Northwest Laboratory (PNL) conducted bench-scale tests in FY 1986 to provide a quantitative description of the performance of several feed mixing systems in terms of measurable process variables (Peterson, McCarthy and Muhlstein 1986). One of the systems studied was the West Valley melter feed hold tank (MFHT). For this system, the HLW and glass-former mixture is fed from the MFHT into the melter. The bench-scale study used simulated West Valley melter feed to determine the size of the full-scale agitator that is used to mix the slurry in the MFHT.

In FY 1987, PNL was funded by the NWTP to verify the scale-up of the bench-scale studies. A collaboration between PNL and West Valley concluded with verification tests performed at West Valley using their melter feed system. This arrangement met the objectives of the NWTP, and also provided information to West Valley for characterizing their melter feed system.

The objectives of this study, which was conducted by PNL for the NWTP and WVDP, are as follows: 1) quantify the homogeneity of the melter slurry in the MFHT, 2) determine the representativeness of the slurry sample

obtained by the liquid-sampling system in the MFHT, 3) determine the time required to resuspend the solids and homogenize the melter slurry in the MFHT after a simulated, unexpected power outage, and 4) verify the scale-up of the results from the bench-scale mixing study.

2.0 CONCLUSIONS AND RECOMMENDATIONS

Large-scale mixing tests were performed at West Valley to determine whether the agitator in the MFHT adequately mixes the slurry, and whether the liquid-sampling system installed in the tank provides a representative sample of the slurry. In addition, the tests were performed to verify scale-up of data from bench-scale mixing tests performed by PNL in FY 1986. The objective of the bench-scale tests was to calculate full-scale mixing parameters based on bench-scale data.

Section 2.1 presents the conclusions from the full-scale tests. Section 2.2 lists recommendations for improving the quality of the glass product and reducing the number of required analyses per sample.

2.1 CONCLUSIONS

The following is a list of significant conclusions drawn from the large-scale mixing tests:

- Analysis of normalized values can sometimes mask interactions. It can be deceptive when certain conditions affect some elements but not others, or affect all elemental constituents similarly. This can have significant impact during feed makeup.
- The major source affecting accuracy of the results is the variability in replicate analytical measurements.
- The 15 Hp, single-speed (155 rpm) agitator installed in the MFHT produces a homogeneous mixture for the three concentrations of simulated melter feed slurry used in these tests. Slurries with significantly greater settling rates may produce different results.
- The liquid-sampling system installed in the MFHT obtains samples that are up to 2.6% higher in total solids. This may be due to the flow rate of the slurry and/or the type of sample bottle used.
- After 4 days of settling in the MFHT (simulation of an unscheduled power outage), less than 2 hours of agitation were required to resuspend and uniformly mix the slurries tested.
- The maximum flow rate of the air-displacement slurry (ADS) pump used for the sampling system does not achieve turbulent flow in a 0.5-in.-(inside)-dia. pipe, which may result in a nonrepresentative sample due to solids settling in the lines.

- Scale-up of the bench-scale tests was qualitatively verified by showing that the mixing system operated in the turbulent flow region and the liquid in the tank was homogeneous. A precise verification of scale-up of bench-scale data was not possible due to dissimilarities between the two mixing systems.
- The mixing Reynolds numbers calculated for the three slurry concentrations that were tested were 1 to 2 orders of magnitude greater than the Reynolds number at which fully turbulent mixing is defined. This difference implies that the speed of the agitator could probably be reduced without affecting the homogeneity of the slurry in the MFHT. A reduction of agitator speed would 1) prevent vortexing, which could decrease the accuracy of the liquid-level detector; 2) decrease the erosion rate of the impeller blades; 3) decrease the chance of splashing slurry on internal equipment (which could decrease corrosion); and 4) increase the efficiency of the ADS pump at low liquid levels (air entrainment affects ADS pump operation).

2.2 RECOMMENDATIONS

The following are recommendations for improving the consistency of the melter feed and reducing the number of required analyses per sample.

- Statistical analysis of all inductively coupled plasma (ICP) data that are available from these tests should be completed. In this study, data from only the seven major soluble and insoluble constituents were analyzed to determine homogeneity. Analysis of the other constituents would provide relative standard deviation measurements that are important for waste form qualification and for increasing the accuracy of the cold chemical addition recipe.
- Characterization tests on the actual liquid-sampling system should be performed to determine procedures that will provide a more representative sample. These tests should include an evaluation of sample bottle design, sampling cycle times, minimum flow rates, and flushing requirements.
- A significant portion of the analytical uncertainty was due to long-term effects (e.g., lab conditions, calibration procedures). A statistical quality control program as detailed in Section 6.5 would isolate and eliminate some of the major sources of variation. Reduced analytical uncertainty reduces the number of required analyses per sample, thereby reducing cost.

3.0 MIXING THEORY

The procedures used to design the slurry mixing processes are based on classical rheological models for homogeneous and heterogeneous flow systems. (Rheology is the field of study concerned with the deformation and flow of materials.) The models were used to classify the West Valley melter feed slurry, and verify the scale-up of the bench-scale data. The theoretical models used to define the non-Newtonian behavior of the melter feed slurry are described in Section 3.1. The design of solid-liquid mixing systems is discussed in Section 3.2. Section 3.3 presents the method used to scale-up the bench-scale data.

3.1 PSEUDOHOMOGENEOUS, NON-NEWTONIAN SLURRIES

Viscous, single-phase fluids and pseudohomogeneous, multiphase fluid mixtures (like melter feed) are classified according to their response to shearing stresses. A shear stress is defined as:

$$\tau = \frac{F}{A} \quad (3.1)$$

where τ = shear stress

F = force

A = area.

For Newtonian fluids in laminar flow:

$$\tau = \mu \left(\frac{dv}{dy} \right) \quad (3.2)$$

where μ = viscosity

dv/dy = shear rate in y direction

The viscosity, μ , is a proportionality constant that is independent of shear rate and affected only by pressure and temperature for a Newtonian fluid. A plot of shear stress versus rate of shear in the laminar flow regime is known as a rheogram (Figure 3.1). The slope of the curve is constant for a Newtonian fluid. All fluids that display rheograms that are not linear

FIGURE 3.1. Rheograms for Time-Independent Fluids

through the origin are considered to be non-Newtonian. Non-Newtonian fluids are usually classified as time-independent, time-dependent, or viscoelastic fluids. Since most melter feed slurries are considered time-independent fluids, only that type of fluid will be discussed in this report.

Figure 3.1 presents rheograms for classical time-independent, non-Newtonian fluids. The relationship between apparent viscosity and shear rate for these fluids is described by Figure 3.2. The apparent viscosity is a function of shear rate for non-Newtonian fluids and is analogous to the Newtonian viscosity for Newtonian fluids.

Non-Newtonian fluids are typically classified as fluids with yield stresses and fluids without yield stresses. These classifications are further defined as fluids that decrease in viscosity with increasing applied shear rate (pseudoplastic or yield-pseudoplastic, if the slurry has a yield stress), and fluids that increase in viscosity with increasing applied shear rate (dilatant or yield-dilatant).

FIGURE 3.2. Viscosity Versus Shear Rate for Time-Independent Fluids

The rheological behavior of a non-Newtonian fluid can be represented by the following equation:

$$\tau = \tau_y + K\left(\frac{dv}{dy}\right)^n \quad (3.3)$$

where τ = shear stress, N/m^2 (Pascal)

τ_y = yield stress, N/m^2

K = consistency index, Ns/m^2

dv/dy = shear rate in y direction, sec^{-1}

n = flow behavior index.

The yield-power law model defined by Equation (3.3) reduces to the Bingham Plastic model when $n = 1$, the power law model when $\tau_y = 0$, and the Newtonian model when $\tau_y = 0$ and $n = 1$.

Since the bulk of the available mixing data is based on Newtonian fluids, it is convenient to define an apparent viscosity as follows:

$$\mu_a = \frac{\tau}{(\frac{dv}{dy})} = \frac{\tau_y + K(\frac{dv}{dy})^n}{(\frac{dv}{dy})} \quad (3.4)$$

The apparent viscosity for non-Newtonian fluids is a function of the shear rate. The average shear rate in a mixing system is a function of the level of agitation and is defined as (Metzner and Otto 1957):

$$(\frac{dv}{dy})_{\text{average}} = kN \quad (3.5)$$

Values of the proportionality constant for the mixing shear rate, k , have been established for common impeller configurations. The average value of k for single and double impeller configurations is approximately 11.5 (Peterson, McCarthy and Muhlstein 1986). This number was used to determine the shear rate in the bench-scale and full-scale mixing tanks.

3.2 SOLID-LIQUID MIXING

Although many variables are involved in fluid mixing, a relatively small number of dimensionless groups (Table 3.1) and corresponding functional relationships can be derived by using dimensional analysis. The following functional relationship can be obtained from consideration of a mass and momentum balance around a mixing vessel for a constant density Newtonian fluid (Bird et al. 1960, Dickey and Fenic 1976, Bates et al. 1966).

$$N_p = f [Re, Fr, \frac{T}{D}, \frac{L}{D}, \frac{C}{D}, \frac{p}{D}, \frac{W}{D}, \frac{B}{B_0}] \quad (3.6)$$

$$\text{where } N_p = \text{power number} = \frac{P}{\rho N^3 D^5} \quad (3.7)$$

$$Re = \text{Reynolds number} = \frac{\rho N D^2}{\mu} \quad (3.8)$$

$$Fr = \text{Froude number} = \frac{N^2 D}{g} \quad (3.9)$$

B = number of blades
 B_0 = reference number of blades
 C = clearance between the impeller and tank bottom, m
 D = impeller diameter, m
 L = liquid level in tank, m
 p = impeller pitch, m
 P = power applied to fluid, W
 N = impeller rotational speed, rps
 W = impeller blade width, m
 T = diameter of tank, m
 μ = fluid viscosity, Pa $\cdot$ s
 ρ = fluid density, kg/m³
 g = gravitational constant, m/s²

For mixing systems that are geometrically similar, the functional relationship can be reduced to:

$$N_p = f [Re, Fr] \quad (3.10)$$

The Froude number is a significant variable in mixing systems only where a free liquid surface exists and the flow pattern is affected by gravity. This can be significant in unbaffled tanks when vortexing is occurring. In baffled tanks such as the MFHT, vortexing generally does not occur and the

TABLE 3.1. Dimensionless Groups for Fluid Mixing Applications

<u>Name</u>	<u>Formula</u>	<u>Relationship</u>
Froude number	$Fr = DN^2/g$	inertial/gravitational forces
Power number	$N_p = Pg/\rho N^3 D^5$	imposed/inertial forces
Pumping number	$N_Q = Q/ND^3$	turnovers
Reynolds number	$Re = D^2 N \rho / \mu$	inertial/viscous forces
where Q = volumetric flow rate		

Froude number is not important. Equation (3.10) can be simplified to:

$$N_p = f [Re] \quad (3.11)$$

Figure 3.3 presents an example of this functional relationship for a bench-scale mixing system that was intended to be geometrically similar to the MFHT (Peterson, McCarthy and Muhlstein 1986). There are three regions of interest on the curve--the viscous, transition, and turbulent regions. The viscous or laminar region occurs below a mixing Reynolds number, Re , of about 20. The fully turbulent region, where inertial forces dominate, typically begin at an Re of 1,000 to 10,000, depending on the impeller type. In the turbulent region the power number becomes independent of the Re . The region between laminar and turbulent is the transition region.

Another important dimensionless number is the flow number, N_Q . This dimensionless quantity is defined by the following equations:

FIGURE 3.3. Power Number Versus Reynolds Number Relationship for Bench-Scale Mixing Configuration (Peterson, McCarthy and Muhlstein 1986)

$$N = \frac{Q}{ND^3} \quad (3.12)$$

$$Q = v_b A \quad (3.13)$$

where Q = volumetric flow rate, L/min

N = rotational speed, rpm

D = impeller diameter, cm

N_Q = pumping number

v_b = bulk velocity, cm/s

A = cross sectional area of mixing tank, cm^2

The flow number provides an indication of the pumping capacity of an agitator and the turnover rate of the tank contents.

In considering the extent of solids suspension in an agitated tank, it is useful to define a scale of agitation that ranges from one to ten as shown in Table 3.2 (Gates, Morton and Fondy 1976). An increasing scale number indicates an increasing level of solids suspension and mixing homogeneity. Bulk liquid velocities are related to the scale of agitation, N_I , by Equation 3.14 (Hicks, Morton and Fenic 1976):

$$N_I = \frac{v_b}{3.05} \quad \text{The units on } v_b \text{ are cm/s.} \quad (3.14)$$

Some numerical values for the bulk velocity are shown in Table 3.2. Since a nearly homogeneous melter feed is important for producing a high-quality glass waste form, a scale of agitation of 8 to 10 is necessary for the melter feed slurry application.

The high-efficiency turbine blades installed in the West Valley mixing system produce a primarily axial flow pattern (Figure 3.4). This flow pattern is useful in solids suspension applications since it directs the flow of liquid downward, which tends to sweep solids from the bottom of the tank and prevent solids deposition.

TABLE 3.2. Scale of Agitation for Solids Suspension

Scale of Agitation	Bulk Velocity cm/s (fpm)	Description
1	3.0 (6)	Agitation levels 1-2 characterize applications requiring minimal solids-suspension levels to achieve the process result.
2	6.1 (12)	
		Agitators capable of scale level 1 will:
		<ul style="list-style-type: none"> • Produce motion of all of the solids of the design-settling velocity in the vessel. • Permit moving fillets of solids on the tank bottom, which are periodically suspended.
3	9.1 (18)	Agitation levels 3-5 characterize solids-suspension applications for most chemical-process industries. This scale range is typically used for dissolving solids.
4	12.2 (24)	
5	15.2 (30)	
		Agitators capable of scale level 3 will:
		<ul style="list-style-type: none"> • Suspend all of the solids of design-settling velocity completely off the vessel bottom. • Provide slurry uniformity to at least one-third of fluid-batch height. • Be suitable for slurry drawoff at low exit-nozzle elevations.
6	18.3 (36)	Agitation levels 6-8 characterize applications where the solids-suspension level approaches uniformity.
7	21.3 (42)	
8	24.4 (48)	
		Agitators capable of scale level 6 will:
		<ul style="list-style-type: none"> • Provide concentration uniformity of solids to 96% of the fluid-batch height. • Be suitable for slurry drawoff up to 80% of fluid batch height.
9	27.4 (54)	Agitation levels 9-10 characterize applications where the solids-suspension uniformity is the maximum practical.
10	30.5 (60)	
		Agitators capable of scale level 9 will:
		<ul style="list-style-type: none"> • Provide slurry uniformity of solids to 98% of the fluid-batch height. • Be suitable for slurry drawoff by means of overflow.

Source: Gates, Morton and Fondy 1976.

FIGURE 3.4. Flow Pattern for Dual Axial-Flow Turbines

3.3 SCALE-UP OF BENCH-SCALE DATA

Bench-scale systems are designed to be as geometrically, kinematically, and dynamically similar to full-scale equipment as practical. Geometric similarity requires that the ratio of corresponding dimensions be identical in bench- and full-scale equipment. Emphasis is placed on maintaining certain ratios, including liquid level/tank diameter (L/T), blade diameter/tank diameter (D/T), distance between bottom blade and tank floor/blade diameter (C_1/D), blade width/blade diameter (W/D), and width of the baffles/tank diameter (B_w/T).

Kinematic similarity is normally defined in terms of ratios of corresponding velocities. This definition is comparable to a dimensionless velocity distribution, which is a ratio with respect to a reference velocity such as impeller tip speed. It has been shown by fundamental and experimental analyses that the dimensionless velocity distribution becomes constant in turbulent agitation. A constant dimensionless velocity distribution results

in fixed ratios between corresponding velocities. As a result, kinematic similarity occurs with geometric similarity in turbulent agitation (Rautzen, Corpstein and Dickey 1976).

Dynamic similarity requires that fixed ratios of corresponding forces (inertial, viscous, and gravitational) be held constant. The mixing parameters that relate to these forces are listed in Table 3.1. All of these parameters are a function of both impeller speed, N , and blade diameter, D . It is not possible to hold both N and D constant during scale-up. In addition, the size distribution of particles in solid-liquid agitation systems will not change as the size of the agitation system is scaled up or scaled down.

An empirical approach must be taken to scale-up the bench-scale mixing results. Bench-scale tanks are fabricated to assure dimensional similarity. The range of impeller rotation speeds evaluated is chosen to induce tip speeds that bracket the speeds often used in full-scale equipment. Finally, a scale-up rule is chosen that accounts for the dynamic dissimilarity that will exist between the bench-scale and full-scale equipment.

Geometric similarity fixes the value of D for each of the dynamic similarity parameters listed in Table 3.1. As a result, the scale-up problem becomes one of determining the appropriate large-scale impeller speed. The selection of the appropriate scale-up rule to establish this rotational speed is based on documentation (Rautzen, Corpstein and Dickey 1976) that identifies a scale ratio, R , as:

$$R = \left(\frac{V_2}{V_1}\right)^{1/3} \quad (3.15)$$

where the subscript 2 refers to large-scale equipment and the subscript 1 refers to bench-scale equipment.

The scale ratio is used to fix the physical dimensions of the full-scale equipment as follows:

$$R = \frac{L_2}{L_1} = \frac{T_2}{T_1} = \frac{D_2}{D_1} = \frac{W_2}{W_1} = \frac{B_{w2}}{B_{w1}} \text{ etc.} \quad (3.16)$$

The large-scale impeller speed is then related to bench-scale impeller speed. This relationship adjusts the scale ratio, R , by raising it to an exponent, m :

$$\frac{N_2}{N_1} = \left(\frac{1}{R}\right)^m \quad (3.17)$$

The selection of the exponent, m , is based on a knowledge of the controlling mixing class in the agitation system and on experience. The levels of m associated with common mixing applications are indicated in Table 3.3. Detailed information on a scale-up procedure applied to the MFHT can be found in Peterson, McCarthy and Muhlstein 1986.

TABLE 3.3. Scale-Up Exponents for Agitation

<u>Mixing Application</u>	<u>Dominant Impeller Effect</u>	<u>Description</u>	<u>Exponent, m</u>
Equal blending time	--	Reaction time is controlling	0
Equal surface motion	Shear rate (Fr , tip speed)	Vortex formation	1/2
Equal mass transfer	Shear rate/flow (power/volume, tip speed)	Liquid motion Turbulence Dispersion of solids	2/3
Equal solids suspension	Flow (N_Q)	Suspension of solids	3/4
Equal liquid motion	Flow (torque/volume)	Corresponding Velocities are equal	1

4.0 TEST APPROACH

Two types of tests were performed at West Valley using three concentrations of simulated, nonradioactive melter feed slurry. The tests are identified as the homogeneity/sampling system test and the resuspension test and are described in more detail in Sections 4.2 and 4.3, respectively. The melter slurry concentrations that were tested included the reference melter feed, the reference melter feed concentrated to 75 vol% (concentrated feed), and the reference melter feed diluted to 125 vol% (dilute feed). The physical properties of the slurry are presented in Table 6.1.

The tests were statistically designed by PNL to determine whether an adequately homogeneous slurry is obtained in the MFHT, and whether the liquid-sampling system provides a representative sample of the melter feed. The homogeneity of the slurry was measured by comparing the average weight percent solids and the elemental concentrations of slurry samples taken from six sampling locations within the tank, as shown in Figure 4.1. All sampling locations are at about the same radial distance from the agitator. As a result, homogeneity of the slurry as a function of radial distance cannot be determined. However, bench-scale test data indicated that homogeneity of the slurry did not vary with radial distance. The full-scale system is expected to yield the same results.

The ability of the sampling system to obtain a representative sample from the tank was determined by comparing the average weight percent solids and the elemental concentrations from the samples obtained at the six tank sampling locations to those from the sampling system. Section 4.1 discusses the assumptions made to determine the number of required samples at each of the sampling locations.

Samples were taken from the tank and analyzed in random order for weight percent solids and elemental concentrations (ICP analyses) at West Valley. The elemental concentrations were used to calculate the weight percent oxide. An additional sample was taken from each location during each of the three homogeneity/sampling system tests. These additional samples were sent to PNL for rheological and physical evaluations. The rheological and physical

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FIGURE 4.1. Sample Point Locations

characteristics data were used to verify the scale-up of the bench-scale mixing data. Slurry characterization data are presented in Section 6.1, and verification of bench-scale data is discussed in Section 6.6.

4.1 STATISTICAL EXPERIMENTAL DESIGN

The tests were statistically designed to provide a high level of confidence regarding the accuracy of the test conclusions. The number of samples required to achieve a high level of confidence is dependent upon several factors, including the size of the standard deviation of replicate samples and analyses, the probability of making erroneous conclusions from statistical tests concerning the homogeneity of the slurry, and the desired detectable differences between the weight percent solids averages (or elemental concentrations) at different locations within the MFHT.

4.1.1 Standard Deviation of Replicate Samples and Analyses

The standard deviation is a measurement of the variability between replicate determinations of the tank contents. The tank contents were determined by measuring the weight percent total solids and elemental weight percents (ICP analyses). A potential exists for random variations between replicate samples obtained from the same location/slurry combination. Additionally, variations exist between replicate analyses of the same sample due to the imprecision of the instrumentation and sample preparation techniques.

The standard deviation of replicate determinations of the tank contents must be determined before examining the required number of replicates at each location. Previously conducted work at PNL on factors affecting homogeneity in a small-scale feed tank provided an estimate of the standard deviation of weight percent solids. It was determined that the variability between replicate samples was the major source of variation, and the variability between replicate analyses on the same sample was nearly negligible. For the full-scale tests, the standard deviation of weight percent total solids was assumed to be equal to the standard deviation obtained from the bench-scale study: 0.9 wt% (Peterson, McCarthy and Muhlstein 1986).

Standard deviation estimates for the ICP elemental concentration measurements were obtained from a variability study conducted for West Valley at

PNL by the Materials Characterization Center (MCC). The study suggested that the major source of sample-to-sample variability was due to the imprecision of the ICP analysis process. The relative standard deviation (RSD; the standard deviation represented as a percent of the wt% average) for seven of the major elements appeared to be less than 4% in all cases. Although these estimates were obtained from experiments conducted using PNL analytical instruments and personnel, they were assumed to be representative of West Valley data.

4.1.2 Probability of Erroneous Conclusions

Any test of a hypothesis that involves sampling poses the chance of making an erroneous conclusion. Consider an examination of homogeneity in a tank. If it were concluded that no differences exist between average wt% solids at different locations, homogeneity is suggested. If it were concluded that differences exist between the averages, lack of homogeneity is suggested. The difference that forms the basis of the conclusion is found by comparing the average weight percent total solids (or elemental weight percent) in a sample for two location/slurry combinations. Drawing the correct conclusion depends on correctly determining whether any differences are statistically significant. Drawing an incorrect conclusion could result in unnecessary process downtime and additional costs. The acceptable probability of not making a false positive or false negative conclusion was chosen by West Valley at 95 percent.

4.1.3 Desired Detectable Differences

Because the homogeneity of the solution within the tank was determined by examining the differences between average weight percents, the size of the differences that could adversely affect glass quality was determined. These differences, which should have a high probability of detection, become the desired detectable differences. For this project, the desired detectable differences were identified as approximately 2 to 5 wt% total solids.

4.1.4 Number of Samples per Location/Slurry Combination

The required number of samples was determined using the assumptions described above. For the wt% total solids determinations, a 0.9 wt% standard

deviation was used. Six samples at each of the 18 location/slurry combinations (six sampling locations times three slurry concentrations) results in at least a 95% chance of detecting a true difference of at least 2.1 wt% total solids between two of the means.

An RSD of 4% was used for the elemental weight percent determinations (ICP determinations). Twenty-one samples at each of the 18 location/slurry combinations results in a 95% chance of detecting a true difference between two of the means of at least 4.5% of the elemental weight percent.

Therefore, 21 samples were taken at each of the sampling locations for the homogeneity/sampling tests because both weight percent total solids and elemental concentrations were used to determine homogeneity. Six samples at each of the sampling locations were taken for the resuspension tests where wt% solids was the only criteria for determining homogeneity.

4.2 HOMOGENEITY/SAMPLING SYSTEM TESTS

The homogeneity/sampling system test was performed to characterize the melter feed system at West Valley and to verify the scale-up of the bench-scale mixing studies performed at PNL. The characterization of the melter feed system included quantifying the homogeneity of the melter feed and determining if the liquid-sampling system obtains representative samples of the slurry. The liquid-sampling system consists of a pulsating air displacement pump that transfers slurry from near the bottom of the tank to an in-line sampler approximately 20 ft away.

The homogeneity of the melter feed slurry was quantified from measurements of the wt% solids and selected elemental concentrations of slurry samples taken from six locations within the MFHT (see Figure 4.1). The representativeness of the sample from the liquid-sampling system was determined by comparing the wt% solids and selected elemental concentrations of this sample to the samples drawn from the six locations. The samples were taken from the tank in a random order. The sampling order is summarized in Tables A.1 through A.3 of Appendix A. Descriptions of the liquid-sampling system and the device used to take the samples from the tank are provided in Sections 5.2 and 5.3, respectively.

The statistical experimental design for the homogeneity/sampling system tests determined that 21 samples were required from each of the six locations in the tank and from the liquid-sampling system. A large number of samples were required because of the imprecision of the instrumentation and sample preparation techniques for the ICP analyses.

Three concentrations of melter feed slurry were tested. The concentrated melter feed slurry was tested first. The simulant was formed in the chemical makeup tank and pumped through a grinder to the MFHT. The grinder theoretically reduces the size of particles in the simulant to less than 200 mesh. The slurry in the MFHT was agitated for several weeks before sampling began. For this test, a total of 154 samples were taken from the tank in a random order--21 samples at each of the 7 sampling locations (as determined by the statistical analysis) and 1 sample from each sampling location, which were combined for rheological and physical analyses. Total sampling time for the first test was 1.5 days. Sampling times decreased with successive tests as the operators gained more experience.

At the end of the homogeneity/sampling system test with the concentrated melter feed slurry, the resuspension test (described in Section 4.3) was started. After the resuspension test, the slurry was diluted to the concentration of the reference melter feed and the homogeneity/sampling system test was repeated, followed by the resuspension test. The slurry was again diluted to 125 vol% of the reference slurry. Samples were taken for the homogeneity/sampling system test, followed by another resuspension test.

4.3 RESUSPENSION TESTS

The resuspension test was performed to determine the time required to resuspend the solids and homogenize the melter slurry in the MFHT after an unexpected power outage. For this test, the agitator was turned off for approximately 4 days. Samples were taken from the tank at the six specified locations approximately 2, 6, and 24 hours after the agitator was turned back on. The homogeneity of the tank was determined by comparing wt% total solids only.

The statistical experimental design for the resuspension test required six samples from each of the six sampling locations. The samples were drawn from the tank in a random order using the tank sampler (see Section 5.3 for a description of the tank sampler). The sampling order is summarized in Tables A.4 through A.6 of Appendix A. The resuspension tests were performed after completion of the homogeneity/sampling system tests for each slurry concentration. It took approximately 1 hour to take 36 samples.

5.0 TEST EQUIPMENT

The equipment used included the MFHT at West Valley, a mechanical liquid-sampling system, a bottle-and-rod tank sampler, a Haake viscometer, and a bench-scale mixing system. This section describes the major equipment used for the mixing studies.

5.1 WEST VALLEY MELTER FEED HOLD TANK

The MFHT is used to feed the radioactive liquid waste and glass formers slurry (melter feed) to the ceramic melter. A schematic of the tank is depicted in Figure 5.1.

The stainless steel tank is 10 ft in diameter and approximately 10 ft high. Its capacity is 5,000 gallons. The MFHT is equipped with an agitator, four equally spaced standard baffles, heating coils, and a cooling jacket. The heating coils are positioned inside the tank near the tank wall.

The agitator mixes the slurry in the MFHT to assure a homogeneous mixture is fed to the ceramic melter. In addition, the agitator must resuspend the melter feed that has settled to the bottom of the tank during an unscheduled power failure. The agitator motor is 15 Hp, single speed (155 rpm), with a 460 volt, 3-phase DC drive. The agitator is equipped with two 36-in.-dia., high-efficiency turbine impellers. Each impeller has three blades (see Figure 5.1).

5.2 MECHANICAL LIQUID-SAMPLING SYSTEM

The purpose of the liquid-sampling system is to obtain a representative sample of melter feed from the MFHT. During actual melter operations, the sample will be used to qualify the glass for disposal in a repository. The liquid-sampling system consists of two major pieces of equipment: 1) the ADS pump, and 2) the Hydragard Series 2000[®] liquid sampler.

[®] Registered tradename of Hinds International, Inc., Hillsboro, Oregon.

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FIGURE 5.1. Schematic of West Valley Melter Feed Hold Tank

For the tests described here, the piping between the ADS pump and liquid sampler did not exactly duplicate the arrangement that will be used in the actual system. Half-inch tubing with a 1/16-in. wall thickness was used for 4/5 the distance between the ADS pump and the Hydragard sampler. Approximately 5 ft before the sampler, the tubing widened to the 3/4-in. tubing (approximately 0.5-in. inside dia.) specified by the sampler manufacturer.

Line sizes and fittings were not replicated, and the sample bottles used with the Hydragard sampler were considerably different from those that are planned for use in the actual system. Therefore, results from the sampling system tests described in this report may not be representative of results from the actual sampling system.

5.2.1 Air-Displacement Slurry Pump

The basic operating sequence for the ADS pump is portrayed in Figure 5.2. The entire operating cycle can be described by a series of four steps. The first step positions the valve open, while the second step allows the chamber to fill with the liquid slurry. The third step positions the

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FIGURE 5.2. Operating Cycles of ADS Pump

valve closed. The fourth step is the discharge step, where compressed air is valved into the chamber, thus forcing the slurry out the discharge line. The air can be described as a reciprocating piston. By controlling the air pressure and cycle time, the flow rate can be controlled. This cycle creates a pulsating flow.

The slurry pump should be capable of pumping the melter feed in the turbulent flow regime, which should prevent solids from settling in the piping and either plugging the line and/or not providing a representative sample to the in-line liquid sampler. The ADS pump is capable of pumping the melter feed slurry at flow rates between 50 and 250 L/hr. The cycle times for these flow rates are 100 and 25 seconds, respectively. For the tests in this study, the cycle time and air pressure were adjusted for an average flow rate of 250 L/hr. This flow rate corresponds to an average velocity of 1.8 ft/s for a pipe with a 0.5-in. inside diameter.

5.2.2 Hydragard Series 2000 Liquid Sampler

The Hydragard sampler (Figure 5.3) was developed for the Savannah River Laboratory and is designed to provide positive extraction and collection of samples from in-line process flows that are toxic, radioactive, or chemically severe. The sampler is a manually activated, closed loop system for use with robotic systems.

The sample bottles allow flow to recirculate in the bottle. Figure 5.4 shows two sample bottle designs. The bottle on the right was used in the tests discussed in this report, and the bottle on the left is the design currently planned for actual operations. When the bottle is removed from the concentric needle filler subassembly [item (3) on Figure 5.3], a double-layered septum in the cap cleans the dip tubes. This procedure prevents radioactive waste from dripping onto the sample shroud.

5.3 BOTTLE-AND-ROD TANK SAMPLER

The tank sampler (Figure 5.5) was used to obtain slurry samples at six locations in the MFHT. This type of sampler is called the bottle-and-rod sampling system. Three 60-ml polyethylene bottles were secured to the long pole at locations corresponding to the specified sampling depths within the

- (1) Sampler Subassembly - Extracts sample from process line on operator or robot command.
- (2) Limit Switch Subassembly - Provides system status verification signals for the valve "open" and "closed" positions.
- (3) Concentric Needle Filler Subassembly - Transfers the sample to the sample container and returns excess sample to the process flow.
- (4) Sample Container Shroud Subassembly - Positions and protects the sample container in the sampling system, and provides a backup containment seal in the event of failure of the primary seal between the concentric needle filler and the sample container. Standard material of construction is optically clear polycarbonate.

FIGURE 5.3. Hydragard Series 2000 Liquid-Sampler

FIGURE 5.4. Sample Bottles for Hydragard Liquid-Sampler

tank. The bottles were capped with a cork that had a rod imbedded in it, and then lowered into the tank. The bottles were then uncorked in a predetermined, random order. After about 10 seconds, the bottles were recorked.

This tank sampler design was chosen because it is simple and fundamentally different from the liquid-sampling system. The liquid-sampling system consists of a pipeline from the ADS pump to the sampler, and solids could settle in that line and/or in the sample bottle. The tank sampler collects samples directly from the tank. Comparing data from the two sampling systems provided information on possible deficiencies associated with the ADS pump/Hydragard sampling system. Section 6.3 discusses the results from the sampling system study.

FIGURE 5.5. Schematic of Bottle-and-Rod Tank Sampler

5.4 HAAKE VISCOMETER

Laboratory rheological data were obtained with a Haake Rotovisco[®] viscometer equipped with an M150 measuring/drive head and the NV sensor system. To measure rheological properties using this instrument, the sample must be placed into the gap between two coaxial cylinders. A motor drives the inner cylinder. A viscosity-related torque, caused by the resistance of the sample to shearing, acts on the inner cylinder. This torque deflects a measuring spring placed between the motor and the inner cylinder. The magnitude of the spring deflection correlates linearly with the torque. The spring deflection is transformed into an electrical signal. The torque and tachometer signals are recorded.

The speed signal required for determining shear rate does not come from the rotor directly but from the tacho-generator that sits above the motor. The tacho-generator is mechanically linked to the motor. For slurries that do not exhibit yield stresses, the speeds of the tacho-generator, motor, and rotor are identical so that the speed signal also defines the rotor speed. A slurry with a yield stress will clamp the rotor to the cup until the applied torque is greater than the yield stress.

While the rotor is still clamped and at a standstill, the motor rotation will cause some spring deflection and consequently a torque signal on the ordinate. For a slurry with a yield stress, a straight line, leaning off the vertical with an angle (β), is recorded initially (Figure 5.6). When the shear stress surpasses the yield value, the rotor breaks loose from its clamp and picks up speed, accelerating almost instantaneously to the signal speed. The change in the behavior of the sample becomes apparent in the rapid turn of the flow curve. The ordinate value of this turning point defines the yield value.

[®] Registered tradename of Haake Buchler Instruments, Inc., Saddlebrook, New Jersey.

FIGURE 5.6. Flow Curve (Rheogram) for a Yield-Pseudoplastic Fluid

5.5 BENCH-SCALE MIXING SYSTEM

The bench-scale mixing system was used by PNL in 1986 to examine the effects of significant independent variables on the distribution of solids in several types of mixing systems, and provide a rational basis for the design of full-scale mixing systems (Peterson, McCarthy and Muhlstein 1986). One of the mixing systems evaluated was the West Valley MFHT.

The bench-scale system (Figure 5.7) was comprised of a 15-in.-dia., 11-gal, clear plastic tank equipped with removable baffles, heating coils, and impellers. The agitator was driven by a 0.25 Hp, 0-1100 rpm, AC motor. The assembly was supported on "frictionless" bearings to facilitate the measurement of the torque applied to the slurry in the tank. A load cell connected to a 10-in. lever arm measured this applied torque.

The West Valley MFHT mixing system was not designed at the time the bench-scale tests were conducted. Therefore, it was assumed that the West Valley system would have two impellers--a 4-blade flat turbine for the lower

FIGURE 5.7. Bench-Scale Mixing System

impeller and a high efficiency turbine for the upper impeller. The impellers are shown in Figure 5.8. A comparison of the geometric similarity between the two systems is presented in Table 6.10.

FIGURE 5.8. Bench-Scale Impellers

6.0 EXPERIMENTAL RESULTS AND DISCUSSION

The homogeneity/sampling system and resuspension tests were designed to permit a sound statistical analysis of the data. The objectives of the statistical design were to determine whether an adequately homogeneous slurry is obtained in the MFHT, and whether the liquid-sampling system provides a representative sample of the melter feed.

Homogeneity of the feed slurry was quantified by comparing the size of the differences between the average weight percent total solids, or elemental weight percents, across the six sample locations within the tank. If a significant difference existed between any of the six averages, the melter feed slurry was considered nonhomogeneous. The liquid-sampling system was determined to provide a representative sample if an insignificant difference existed between the average weight percent solids, or elemental weight percents, of its sample and the average of those samples obtained from the six sample locations.

Waste samples were sent to PNL for physical and rheological characterization. Results from those analyses are presented in Section 6.1. The statistical analysis of the data received from West Valley is described in Section 6.2. Results of the data analyses for the homogeneity/sampling system tests and the resuspension tests are presented in Sections 6.3 and 6.4, respectively. Graphs of the results from the statistical analyses are presented in Appendix B. During analysis of the data, effects from the analytical instrumentation and procedures were observed. These effects are explained in Section 6.5. In addition, Section 6.5 discusses ways to decrease the error associated with these effects. Section 6.6 discusses the verification of the bench-scale mixing studies.

6.1 WASTE CHARACTERIZATION

An extra sample from each sampling location was taken during the homogeneity/sampling system tests and sent to PNL. The samples from each slurry concentration were combined, and physical and rheological analyses were performed on the combined samples. These evaluations provided the background

information needed to verify the scale-up of the bench-scale data, and determine if the slurry was pumped through the liquid-sampling system in the turbulent flow regime.

6.1.1 Physical Properties

The density, weight percent total solids, and elemental weight percents (ICP analysis) of the combined samples were measured at PNL. Table 6.1 presents a summary of the results. The numbers resulting from the analyses conducted at West Valley are included in the table for comparison, as are the properties of the slurry used in the bench-scale mixing study performed at PNL in FY 1986.

The density of the combined reference slurry was similar to the density of the slurry used in the bench-scale studies (1.29 as compared to 1.26 gr/ml for the bench-scale work). The PNL measurements of weight percent solids were lower than the measurements obtained at West Valley and higher than the measurements of the slurry used for the bench-scale tests. The weight percent solids of the bench-scale slurry was 36.5 wt%, the combined reference sample was 41.8 wt%, and the average of all the reference melter feed slurry samples was 44.4 wt%.

6.1.2 Rheological Properties

Laboratory evaluations using the Haake viscometer were conducted to determine the effects of shear rate on the apparent viscosity of the slurries

TABLE 6.1. Physical Properties of Melter Feed Slurries

Property	Melter Feed Slurry Concentration						
	Concentrated		Reference			Dilute	
	PNL (a)	WV (b)	PNL	B-S (c)	WV	PNL	WV
Specific gravity	1.40	NE (d)	1.29	1.26	NE	1.25	NE
Total solids, wt%	46.0	51.1	41.8	36.5	44.4	33.9	36.1

(a) Properties of the combined samples measured at PNL.

(b) Averages of all the samples analyzed at West Valley.

(c) Properties of simulated West Valley melter feed used in the bench-scale mixing study performed at PNL (Peterson, McCarthy and Muhlstein 1986).

(d) NE = not evaluated.

tested. The rheogram (plot of shear stress versus shear rate) for each slurry is presented in Figures 6.1 through 6.3. These plots indicate that the reference and dilute melter slurries are Newtonian fluids and the concentrated melter slurry is a non-Newtonian, yield-pseudoplastic fluid. Viscosity of the reference and dilute slurries is about 3.1 and 2.7 cP,

38807138.1M

FIGURE 6.1. Rheogram of Combined Sample of the Reference Melter Feed Slurry

38807138.2M

FIGURE 6.2. Rheogram of Combined Sample of the Dilute Melter Feed Slurry

38807138.3M

FIGURE 6.3. Rheogram of Combined Sample of the Concentrated Melter Feed Slurry

respectively. The viscosity of the concentrated melter slurry is a function of the shear rate. For a shear rate of 30 s⁻¹ [the average shear rate in the mixing tank (Section 3.1)], the viscosity of the concentrated melter feed slurry is 22 cP.

The rheological data discussed above was used to verify the bench-scale data and determine if the slurries were pumped through the sampling system in the turbulent flow regime. Verification of bench-scale data is discussed in Section 6.6. Rheological data, as it pertains to pumping fluid in a pipe, is discussed in this section.

A Newtonian analysis was used to determine the transport properties of the reference and dilute slurries, and a non-Newtonian model (Hanks 1978) was used to analyze the concentrated slurry. Results from those calculations are summarized in Table 6.2. Reynolds numbers of 2900 and 3225 were calculated for the reference and dilute melter feed slurries, respectively, for a

TABLE 6.2. Rheological and Transport Properties of Combined Melter Feed Samples for Slurry Sampling System

Property	Melter Feed Slurry Concentration		
	Concentrated	Reference	Dilute
Classification	Yield-Pseudoplastic	Newtonian	Newtonian
Critical Reynolds No.	2600	2100	2100
Reynolds No. at 1.8 ft/s	1550	2900	3225
Critical velocity, ft/s	2.9	1.3	1.2
Design velocity, ft/s ^(a)	3.9	2.5	2.3
Actual velocity, ft/s	1.8	1.8	1.8
Flow regime ^(b)	Laminar	Transitional	Transitional

- (a) Design velocity for non-Newtonian slurries is defined as the critical velocity + 1 ft/s (Ande et al. 1971). For Newtonian slurries, the design velocity is the velocity that corresponds to a Reynolds number of 4000.
- (b) Turbulent flow regime begins at design velocity. If actual velocity is less than design velocity, flow is either laminar or transitional. If velocity is below design velocity and Reynolds number is below critical Reynolds number, then flow is laminar.

0.5-in.-(inside)-dia. pipe and an average velocity of 1.8 ft/s. These values are slightly above the Newtonian critical Reynolds number for circular pipes (i.e., 2100).

Results from the non-Newtonian analysis of the concentrated melter feed slurry indicated a critical Reynolds numbers of 2600 and a critical velocity of 2.9 ft/s in a 0.5-in.-(inside) dia.-pipe. The concentrated melter feed slurry was pumped at 1.8 ft/s. This velocity is well below the critical velocity, indicating that the slurry was likely in the laminar flow regime. General practice for designing pipelines for non-Newtonian slurry transport suggests pumping the slurry at velocities exceeding the critical velocity by at least 1 ft/s to prevent solids from settling (Ande et al. 1971).

The combined sample of the reference melter feed slurry was slightly different rheologically from the melter feed slurry used in the bench-scale tests. The bench-scale slurry was considered a yield-pseudoplastic fluid with a yield stress of 0.22 Pa, a consistency index of 0.0051 Pa-s, and a flow behavior index of 0.979 (Peterson, McCarthy and Muhlstein 1986). As

stated earlier, the combined reference melter slurry was considered a Newtonian fluid. The viscosity of the bench-scale and reference melter feed slurries is approximately 8 and 3.2 cP, respectively. The bench-scale melter feed slurry exhibited behavior similar to the concentrated melter feed slurry. The scale-up of bench-scale data was evaluated for all three slurry concentrations.

6.2 STATISTICAL ANALYSIS OF THE DATA

The sample and analysis orders were completely random, with two exceptions. The sample and analysis order over slurry concentrations for both types of tests was not random. All samples of the concentrated slurry were obtained and analyzed first, followed by the dilute samples and finally the reference samples. This restriction causes any comparison between the slurry concentrations to be confounded with a comparison across time. However, because the comparisons between slurries are not of major interest, confounding slurry effects with time does not have a significant impact.

Sample and analysis order for the resuspension tests was not completely randomized over the slurry and sampling times (referred to as wait-time). The analysis order was randomized between the 2- and 6-hr wait-time samples; however, the 24-hr wait-time samples were all analyzed after the 2- and 6-hr samples. The effect of these restrictions will be discussed in greater detail in Section 6.4. The sampling order for each test is presented in Tables A.1 through A.6 in Appendix A, and the analysis order for each sample is presented in Tables A.7 through A.12.

Both as-analyzed elemental weight percents and normalized elemental oxide weight percents were available from West Valley for the samples obtained from the homogeneity/sampling system tests. The normalized elemental oxide weight percents were determined by first assuming an oxide form for each element, then dividing the oxide weight percent of one elemental oxide by the sum of all the elemental oxide weight percents. As a result, the normalized weight percents sum to 100%.

$$C_n = \frac{C_i}{\sum C_i} \quad (6.1)$$

where C_n = normalized elemental oxide weight percent

C_i = assumed elemental oxide weight percent

$\sum C_i$ = sum of the elemental oxide weight percents

The sum of the oxide weight percents, $\sum C_i$, is a random variable that is dependent on each elemental as-analyzed weight percent.

A preliminary analysis showed that the effects of the sampling system and location differences for some of the elements were masked by the "noise" added to the as-analyzed values through normalization. Therefore, the as-analyzed values were used in the statistical analyses to determine the statistical significance of those effects.

Although the as-analyzed results show that some effects are statistically significant, practical significance is probably best determined by examining the effect on the normalized oxide results. This is because the acceptance criteria for the feed composition will probably be in terms of the normalized oxide weight percents (i.e., silicon dioxide must be within $\pm 8\%$ of the target normalized oxide weight percent). Practical significance versus statistical significance will be discussed further.

In the discussion that follows, a 95% confidence level was assumed for defining statistical significance. A marginal significance was noted when effects were significant at the 90% confidence level. All statistical analyses and manipulations were conducted using SAS[®], a statistical software program. The results of the statistical analyses of the homogeneity/sampling system test data are discussed in Section 6.3. The results of the resuspension tests are discussed in Section 6.4.

6.3 HOMOGENEITY/SAMPLING SYSTEM TESTS

The homogeneity/sampling system tests were performed to characterize the melter feed system at West Valley and verify the scale-up of the bench-scale

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mixing studies performed at PNL. Results of scale-up verification are presented in Section 6.6. The characterization of the melter feed system included quantifying the homogeneity of the melter feed and determining if the liquid-sampling system obtains representative samples of the slurry.

Elemental weight percent determinations were available for 25 elements, in addition to the determinations of weight percent total solids. Seven of the 25 elements were used in the statistical analyses (Al, B, Ca, Fe, Li, Na, Si). These oxides were chosen because they are the major soluble and insoluble constituents in the melter feed slurry.

Preliminary analyses showed that the analytical uncertainty for several of the elemental determinations was not the same across the three concentrations of slurry. Discussions with West Valley analytical staff indicated that the dilute feed was "sticky" and more difficult to suspend. Eventually, a stirring rod was used to break precipitates off the sample container wall before shaking the container to obtain a homogeneous, representative aliquot. The concentrated and reference melter feed slurries did not require the use of a stirring rod. In addition, it appears that the analytical laboratory methods or instrumentation may have provided sources of uncertainty that affect the long-term accuracy of the measurement. This long-term analytical effect is discussed in greater detail in Section 6.5. Because the replicate variations are different for each slurry concentration, a separate statistical analysis was conducted for each concentration.

Several outlying observations were observed through graphical portrayals and deleted from the statistical analysis. The graphs of the weight percent total solids and each element versus analytical order are presented in Appendix B. The outliers that were deleted are listed in Table 6.3. Several samples appeared to be outliers for nearly all of the elemental constituents, while others were outliers for only one of the constituents. Other outliers (mostly typographical errors) were detected, corrected, and incorporated into the statistical analysis.

The statistical analysis technique of Analysis of Variance (ANOVA) was used to examine the homogeneity of the MFHT and the representativeness of the samples obtained using the mechanical liquid-sampling system. Because there

TABLE 6.3. Outliers That Were Deleted from All Statistical Analyses

<u>As-Analyzed</u>	<u>Elements Deleted</u>	<u>Normalized</u>	<u>Elements Deleted</u>
041-CON-021 ^(a)	Ca	022-CON-153	Na
086-CON-124	ALL	041-CON-021	Ca
016-REF-078	Na	100-CON-147	ALL
027-REF-154	Ca	106-CON-029	ALL
076-REF-097	ALL, TOTAL SOLIDS	115-CON-158	Na
086-REF-127	ALL	140-CON-146	ALL
116-REF-082	Na	142-CON-156	Na
022-DIL-090	TOTAL SOLIDS	145-CON-162	Na
027-DIL-148	TOTAL SOLIDS	148-CON-159	Na
040-DIL-107	TOTAL SOLIDS	027-REF-154	Ca
073-DIL-136	ALL, TOTAL SOLIDS	074-REF-097	ALL
094-DIL-109	ALL	116-REF-082	Al
129-DIL-132	ALL	XXX-DIL-<007 ^(b)	Ca
		017-DIL-008	Ca
		062-DIL-078	Al
		073-DIL-136	ALL
		083-DIL-077	Al
		094-DIL-109	ALL
		100-DIL-079	Al
		114-DIL-046	ALL
		129-DIL-132	Li
		130-DIL-084	Fe
		140-DIL-047	ALL

(a) The first three numbers indicate the order the sample was taken, the next three letters indicate the concentration of the slurry (REF = reference, CON = concentrated, and DIL = dilute melter feed slurry), and the final three numbers indicate the order the sample was analyzed.

(b) Calcium data from the first seven dilute samples that were analyzed were deleted from all statistical analyses.

were several measured responses per sample (several elemental constituents and a total solids measurement), a multivariate ANOVA plus the usual univariate ANOVAs was used. The ANOVA computational technique has been fully described in Box, Hunter, and Hunter (1978). The multivariate technique accounts for the correlative structure that exists between the elemental determinations, and performs a simultaneous examination of the seven constituents.

The results of the statistical analyses for the homogeneity/sampling system tests are summarized in Table 6.4. The average elemental weight percents and total solids at the different locations in the MFHT were not significantly different from one another, as indicated in Table 6.4 by statistically insignificant values for the nonhomogeneous standard deviations. Thus, the MFHT appears to be homogeneous for the three concentrations of slurry tested. The analytical variation appears to be the major source of uncertainty. Efforts to reduce the variability of the apparent tank contents should focus on improving the analytical precision rather than improving tank homogeneity.

Elemental weight percent analyses of the concentrated melter feed samples from the liquid-sampling system showed a significant difference from the other samples for Al, B, Fe, and possibly Si. The sampler bias is quantified in Table 6.5. The bias is defined as the difference between the mean of the liquid-sampling system (sampler) samples and the tank samples. The relative bias (Column 2) is defined as the bias divided by the average weight percents.

The sampler bias was not statistically significant for the Ca, Li, Na, and total solids determinations for the concentrated slurry. (The calcium data appeared to be erroneous for the concentrated slurry, as shown by the plot of calcium determinations by analytical order in Figure B.3 of Appendix B. Thus, the calcium data is considered unreliable only for the concentrated slurry.) For the dilute and reference slurries, the sampler bias was statistically significant for Al, B, Ca, Fe, Si, and total solids. However, there was no significant sampler bias for the Li or Na results. The sampler

TABLE 6.4. Estimates of the Inhomogeneity and Analytical Variations Using the As-Analyzed Data

Element	Concentrated Slurry					Reference					Dilute				
	Mean, (a) wt%	Inhomo- geneity, (b) Std. Dev.	Ana- (c) lytical, Std. Dev.	Inhomo- geneity, RSD, %	Ana- lytical, RSD, %	Mean, wt%	Inhomo- geneity, Std. Dev.	Ana- lytical, Std. Dev.	Inhomo- geneity, RSD, %	Ana- lytical, RSD, %	Mean, wt%	Inhomo- geneity, Std. Dev.	Ana- lytical, Std. Dev.	Inhomo- geneity, RSD, %	Ana- lytical, RSD, %
Total Solids	51.1	0	0.71	0	1.4	44.4	0	1.08	0	2.4	36.1	0	0.73	0	2.0
Al	3.1	0.04 ^(d)	0.17	1.3	5.6	2.4	0	0.14	0	6.0	1.8	0.04 ^(c)	0.16	2.2	8.9
B	4.5	0	0.23	0	5.1	3.5	0	0.41	0	12.0	2.5	0	0.15	0	6.2
Ca	0.16 ^(e)	0.005 ^(d)	0.04	3.1	22.1	0.17	0	0.01	0	7.1	0.16	0.001 ^(c)	0.02	0.6	10.9
Fe	5.1	0.03 ^(d)	0.31	0.6	6.1	4.2	0	0.23	0	5.6	3.2	0.02 ^(c)	0.21	0.6	6.5
Li	1.2	0	0.07	0	6.2	1.0	0	0.08	0	8.1	0.77	0	0.11	0	14.3
Na	4.1	0.04 ^(d)	0.32	1.0	7.8	3.4	0	0.37	0	10.7	2.5	0	0.52	0	20.7
Si	18.4	0.17 ^(d)	1.19	0.9	6.5	15.3	0	1.17	0	7.6	11.5	0.26 ^(c)	1.26	2.3	10.9

(a) The mean is calculated excluding the ADS samples.

(b) Standard deviation due to inhomogeneity is the estimated standard deviation between the average weight percents at the six tank sample locations.

(c) The analytical standard deviation is calculated from the replicate samples taken within each location.

(d) Not statistically significantly greater than zero.

(e) Calcium data appear to reflect some nonrandom behavior as seen in the residual plots in Appendix B.

TABLE 6.5. Sampler System Bias and Relative Bias for As-Analyzed Data

Element	Concentrated			Reference			Dilute		
	Bias	Rel. Bias ^(a)	Sign. ^(b)	Bias	Rel. Bias	Sign.	Bias	Rel. Bias	Sign.
Total Solids	0.16	0.3%	N	0.59	1.6%	Y	0.93	2.1%	Y
Al	0.11	3.6%	Y	0.12	5.1%	Y	0.18	10.1%	Y
B	0.16	3.7%	Y	0.39	11.5%	Y	0.18	7.3%	Y
Ca	0.01	5.7%	N	0.02	12.2%	Y	0.05	29.1%	Y
Fe	0.24	4.8%	Y	0.14	3.6%	Y	0.31	10.1%	Y
Li	0.03	2.7%	N	-0.003	0.4%	N	0.04	5.8%	N
Na	0.09	2.3%	N	-0.04	1.0%	N	0.24	9.8%	N
Si	0.57	3.1%	Y	1.16	7.7%	Y	1.53	13.5%	Y

(a) The relative bias is defined as the bias divided by the average total as-analyzed wt%.

(b) Sign. refers to statistically significant, (Y)es or (N)o.

bias is greatest when the feed slurry is more dilute. The concentrated slurry, although pumped in the laminar flow regime, has a yield stress that may be preventing the solids from settling.

Although the sampler bias is statistically significant, it is necessary to determine whether the size of the observed bias is practically significant. As discussed in Section 6.2, practical significance is best defined by examining the effect of the bias on the normalized weight percents because the waste acceptance criteria will most likely be in terms of the normalized determinations.

A statistical analysis similar to that presented above was performed on the normalized oxide weight percents. Table 6.6 presents the results of those analyses. The outliers that were detected and deleted from the statistical analyses are shown in Table 6.3. Again, it should be noted that the analytical variation is the dominant source of uncertainty and that the MFHT appears to be homogeneous.

For the concentrated feed, the effect of sampler bias is not statistically significant for the seven constituents. For the dilute feed, the

TABLE 6.6. Estimates of the Inhomogeneity and Analytical Variations Using the Normalized Data

Element	Concentrated Slurry					Reference					Dilute				
	Mean, (a) wt%	Inhomo- ^(b) geneity, Std. Dev.	Ana- ^(c) lytical, Std. Dev.	Inhomo- geneity, RSD, %	Ana- lytical, RSD, %	Mean, wt%	Inhomo- geneity, Std. Dev.	Ana- lytical, Std. Dev.	Inhomo- geneity, RSD, %	Ana- lytical, RSD, %	Mean, wt%	Inhomo- geneity, Std. Dev.	Ana- lytical, Std. Dev.	Inhomo- geneity, RSD, %	Ana- lytical, RSD, %
Al	7.4	0.015 ^(d)	0.29	0.2	3.9	6.9	0	0.26	0	3.8	7.1	0	0.30	0	4.2
B	10.7	0.012 ^(d)	0.36	0.1	3.4	10.0	0	0.96	0	9.6	9.7	0	0.60	0	6.2
Ca	0.4	0.011 ^(d)	0.07	2.8	18.7	0.5	0	0.03	0	6.8	0.6	0	0.05	0	8.3
Fe	12.1	0	0.33	0	2.7	12.1	0	0.47	0	3.9	12.3	0	0.57	0	4.7
Li	2.8	0.018 ^(d)	0.15	0.1	5.3	2.9	0	0.20	0	6.7	3.0	0	0.32	0	10.6
Na	9.8	0.072	0.40	0.7	4.1	9.9	0	0.84	0	8.4	9.7	0.073 ^(d)	1.55	0.8	16.1
Si	44.1	0.167 ^(d)	1.16	0.4	2.6	44.4	0	1.40	0	3.2	44.5	0.208 ^(d)	2.07	0.5	4.7

(a) The mean is calculated excluding the ADS samples.

(b) Standard deviation due to inhomogeneity is the estimated standard deviation between the average weight percents at the six tank sample locations.

(c) The analytical standard deviation is calculated from the replicate samples taken within each location.

(d) Not statistically significantly greater than zero.

sampler bias is significant for only Ca. However, for the reference feed, there is a sampler bias for Ca, Li, and Na, and possibly for B and Si. The bias is positive for Ca, B, and Si but negative for Li and Na.

An explanation for the normalized sampler bias results can be found by examining the as-analyzed relative biases shown in Table 6.5. When the sampler relative bias is somewhat consistent across all constituents, as is the case for the concentrated slurry, the effect of normalizing cancels any bias. However, when the relative bias is not consistent across all constituents, as is the case for the reference and dilute slurries, normalizing underestimates the elemental concentration for those constituents with little or no sampler bias in the as-analyzed results. However, normalizing does not entirely eliminate the bias for those elements that had a large relative bias on the as-analyzed data.

The above discussion illustrates the problem of determining the effects of various conditions by examining the normalized values. Although normalizing may be appropriate in certain applications, it can be deceptive when certain conditions affect some elements but not others or affect all elemental constituents similarly. The effect of those changing conditions may be hidden by the normalizing technique. Thus, careful use of normalized values is recommended.

The size of the sampler biases for the normalized data is shown in Table 6.7. Because the magnitude of the absolute biases for the reference material is large with respect to the estimated standard deviations due to random analytical variations, a decrease in the bias would have a practical, significant influence on the accuracy and precision of sample determinations obtained from the MFHT. Thus, it would prove beneficial to improve the ability of the sampler system to obtain more representative samples from the MFHT. Additionally, because there were no significant differences between the average elemental concentrations or total solids at each of the six tank sampling locations, the slurry within the MFHT appears to be homogeneous under the conditions of this experiment.

It is likely that the sample bottle used in these experiments (see Figure 5.4b) allowed solids to settle in the bottom of the bottle, which could

TABLE 6.7. Sampler System Bias and Relative Bias for Normalized Data

<u>Element</u>	<u>Concentrated</u>			<u>Reference</u>			<u>Dilute</u>		
	<u>Bias</u>	<u>Rel. Bias(a)</u>	<u>Sign.(b)</u>	<u>Bias</u>	<u>Rel. Bias</u>	<u>Sign.</u>	<u>Bias</u>	<u>Rel. Bias</u>	<u>Sign.</u>
Al	0.01	0.1%	N	-0.06	0.8%	N	-0.01	0.2%	N
B	0.06	0.6%	N	0.57	5.8%	Mar	-0.27	2.7%	N
Ca	0.01	1.7%	N	0.03	6.9%	Y	0.09	13.9%	Y
Fe	0.18	1.5%	N	-0.26	2.1%	N	0.05	0.4%	N
Li	-0.02	0.5%	N	-0.14	5.8%	Y	-0.17	4.6%	N
Na	0.07	0.8%	N	-0.57	5.7%	Y	-0.13	1.4%	N
Si	0.05	0.1%	N	0.79	1.8%	Mar	0.85	1.9%	N

(a) The relative bias is defined as the bias divided by the average total normalized weight percents.

(b) Sign. refers to statistically significant: (Y)es, (N)o or (Mar)ginal.

account for the higher weight percent solid determinations obtained from these samples. In addition, rheological evaluations of the slurries (Section 6.1) indicated that the reference and dilute concentrations were pumped in the transitional flow regime and the concentrated slurry was pumped in the laminar flow regime. Solids may be momentarily settling in the line during low flow, then resuspending when the next pulse of flow is delivered by the ADS pump.

In summary, results from the homogeneity/sampling system tests indicated that the agitator in the MFHT provides a homogeneous mixture of the three concentrations of slurry tested. In addition, the ADS pump/Hydragard sampling system appears to obtain a sample that is higher in solids.

6.4 RESUSPENSION TESTS

The resuspension tests were designed to determine the length of time required by the agitation system to obtain a homogeneous slurry after a simulated, unscheduled power outage. The samples taken for these tests were analyzed only for weight percent total solids. The tank was considered homogeneous if there were no significant differences (less than 2.1 wt%) between

the determinations of average weight percent total solids from the samples analyzed. The samples were taken at six sampling locations within the MFHT (see Figure 4.1).

A three-factor analysis of variance was conducted to determine the significance of the wait-time, tank depth, and access nozzle effects, and test whether the individual effects of each of the three factors were consistent when compared to other factors (two-factor and three-factor interactions). For example, there may be no difference between the total solids at the top, bottom, and middle of the tank after 6 hrs of mixing, but there may be a difference between total solids at those tank levels after 2 hrs of mixing. This example represents a two-factor interaction between tank depth and wait-time.

Although the data contained a few outliers, the conclusions derived from the statistical analyses were not affected by including or excluding the outliers. Therefore, all data were included in the statistical analyses. The results of the analyses are shown in Table 6.8.

TABLE 6.8. Results of Statistical Analysis of Resuspension Data

<u>Effect</u>	<u>Concentrated</u>	<u>Reference</u>	<u>Dilute</u>
Wait-Time	N ^(a)	Y	Y
Tank Depth	N	Y	N
Nozzle	N	Mar.	N
Interactions	N	N	N
Std. Dev. (σ)	1.20	0.87	0.49
Overall Average	49.9 wt%	44.0 wt%	36.0 wt%
RSD	2.4	2.0	1.4

(a) "N" signifies no significant effect (differences less than 2.1 wt% between the average weight percent solids measurements), "Y" signifies a significant effect, and "Mar." is a marginal significant effect (differences less than 5 wt%).

A preliminary analysis showed that the results obtained from the resuspension test using the concentrated melter feed were more variable than the results obtained using the reference and concentrated melter feed. The results from the test using the dilute melter feed were the least variable. Thus, a separate statistical analysis was conducted for each slurry concentration.

6.4.1 Concentrated Melter Feed Slurry

For the concentrated slurry, there were no significant effects or interactions as a result of wait-time, tank depth, or access nozzle. Thus, for a similar concentrated slurry, homogeneity is reached after only 2 hrs of mixing.

It should be noted that the average weight percent total solids is 49.9 for the samples taken during the resuspension tests, and 51.1 for those taken during the homogeneity tests. The apparent difference resulted from the analytical variations over time. To aid in the quality control efforts for these experiments, a control sample was obtained from the tank during the homogeneity/sampling system test using the concentrated melter feed slurry. This sample was divided into numerous analytical samples that were submitted to the analytical laboratory to be analyzed with the other samples.

The control samples were analyzed every sixteenth sample. The sample was submitted throughout the concentrated, dilute, and reference sample analyses. Figure 6.4 plots the total solids determination by analytical order for the control sample. During the analysis of the concentrated resuspension samples, three of the control sample analyses were less than 49 wt%, whereas the results obtained during the homogeneity tests were mostly clustered around 51 wt%. Thus, it appears that long-term analytical variations account for the difference between the weight percent solids analyzed for the homogeneity/sampling system and resuspension tests. The magnitude of the long-term analytical variations will affect the number of analyses required during actual operations. Methods to reduce this effect are discussed in Section 6.5.

FIGURE 6.4. Total Solids Versus Analytical Order for the Control Samples

6.4.2 Reference Melter Feed Slurry

The results of the statistical analysis on the reference slurry resuspension tests are somewhat ambiguous. A significant wait-time effect caused the average total solids concentration to decrease with mixing time, contrary to expected results. The averages are shown in Table 6.9. At this time there is no reasonable explanation for the decrease in total solids as the tank continues mixing. It should be noted that all the 2-hr samples were obtained first, followed by the 6-hr samples then the 24-hr samples. Because of this restriction on randomization, the effect of wait-time is completely confounded with sampling order.

There also is a significant tank depth effect on the reference resuspension tests. The average total solids at the bottom and middle of the tank appear to be less than the total solids at the top of the tank. Again, this result is unexpected. Finally, a marginally significant difference exists between the nozzle C and D total solids. No significant interactions were manifested between the three factors of interest.

TABLE 6.9. Averages Representing the Significant Effects
Shown in Table 6.8

Reference Slurry:

<u>Wait-Time Effect</u>	<u>Average Total Solids</u>	<u>Sign.(a)</u>
2 hours	44.53 wt%	A
6 hours	44.03 wt%	B
24 hours	43.44 wt%	C
<u>Tank Depth Effect</u>	<u>Average Total Solids</u>	<u>Sign.</u>
Bottom	43.85 wt%	A
Middle	43.79 wt%	A
Top	44.37 wt%	B
<u>Nozzle Effect</u>	<u>Average Total Solids</u>	<u>Sign.</u>
C	44.16 wt%	A
D	43.84 wt%	B

Dilute Slurry:

<u>Wait-time Effect</u>	<u>Average Total Solids</u>	<u>Sign.</u>
2 hours	35.93 wt%	A
6 hours	35.79 wt%	A
24 hours	36.33 wt%	B

(a) Averages with the same letter under the "Sign." heading are not statistically different from each other. Averages with different letters are significantly different.

6.4.3. Dilute Melter Feed Slurry

For the dilute slurry, the only significant effect was a difference between the total solids for the three wait-times (see Table 6.8). There was no significant difference between the 2- and 6-hr average total solids. However, the average total solids after 24 hrs of mixing is significantly greater than the 2- and 6-hr averages. Although this suggests that more than 6 hrs are needed to suspend the solids, a more extensive examination reveals that the observed difference between the 2- and 24-hr samples is probably due to analytical variation only. The average weight percent solids for each wait-time are shown in Table 6.9.

As discussed in Section 6.2, the 2- and 6-hr samples were analyzed before the 24-hr samples. Figure 6.4 shows that the control samples analyzed near the end of the dilute analyses appear to have much higher total solids than those analyzed with the first part of the resuspension dilute samples. Those high control samples correspond with the 24-hr samples such that the difference between the 24-hr and the 2- and 6-hr results are completely confounded with a change in the analytical laboratory. Thus, it appears that the dilute slurry in the MFHT is probably homogeneous after 2 hr of mixing.

In summary, the concentrated and dilute slurry resuspension tests show that homogeneity is reached in the MFHT after only 2 hr of agitation. The results of the reference slurry resuspension tests are not as expected, although they are statistically significant. Assuming that some other unknown factor contributed to the reference slurry resuspension results, an agitation of 2 hr after agitator shutdown is recommended to obtain a homogeneous slurry.

6.5 ANALYTICAL UNCERTAINTIES

As the statistical analyses progressed, it became apparent that the analytical variations were not entirely random. In the test plan, the analytical order was completely randomized within each slurry concentration for the homogeneity tests and the resuspension tests (with the exception of the 24-hr resuspension samples, which were always analyzed last within a slurry concentration). Plots of the total solids or elemental weight percents after removing the effects of the factors studied in the experiment (residual plots) should resemble a random band of values when plotted by analytical order. However, as seen in the residual plots shown in Appendix B (Figures B.1 through B.24), there are definite trends across analytical order. These long-term effects account for a sizable portion of the total analytical variation.

In addition to the long-term trends, several plots show clusters of data that are out of line with the rest of the data (e.g., Figures B.6, B.12, and

B.13). Because these outlying values occur in a cluster over time, it is apparent that the contributing factor can be attributed to the analytical laboratory.

Plots of the analytical determinations for the control samples are shown in Appendix B (Figures B.25 through B.31). These plots also reveal the long-term analytical variation. The magnitude of the analytical uncertainty will directly affect the number of analyses that will be required during actual "hot" operations. If the analytical uncertainties can be reduced, the required number of feed analyses for characterization and waste form qualification purposes can be reduced. Moreover, if the analytical uncertainty is reduced, the cold chemical addition recipe will be more accurate, thereby improving the ability to produce a glass composition that is nearer the optimal target composition.

Reducing analytical uncertainty should begin with statistical quality control charting using control standards that are representative of the feed slurry. The standards should represent the full spectrum of the elemental matrixes found in the feed slurry, and should include all of the analytical preparation steps. The analytical process must first be brought into statistical control by identifying and eliminating the special causes of variation (i.e., those variations that present themselves in unpredictable ways). A special-cause variation produces erratic outliers, a cluster of outliers, or a sudden shift in the analytical results. When a method has been implemented to assure statistical control, a program could be undertaken to reduce the "common cause" variations by eliminating or reducing long-term variations. Such a program would involve careful study of the control charts, statistical evaluation of any correlative behavior of data available on other variables, and statistical design and analysis of experiments examining the hypotheses proposed to identify the sources of long-term uncertainty.

Because long-term analytical uncertainty is a major portion of the total analytical uncertainty, and a reduction in the analytical uncertainty is highly desirable because of the reduced cost and improved quality of the

product, it is recommended that a statistical quality control program as described above be implemented in the analytical laboratory.

6.6 SCALE-UP VERIFICATION

Bench-scale mixing tests were performed in 1986 to provide a rational basis for the design of full-scale mixing systems (Peterson, McCarthy and Muhlstein 1986). One of the mixing systems evaluated was the MFHT. However, the MFHT was not designed at the time the bench-scale tests were conducted, and assumptions were made about the system design. The full-scale system is not geometrically, kinematically, or dynamically similar to the bench-scale system. Therefore, an exact verification of the scale-up procedure was not possible. Although the scale-up procedure used in Peterson, McCarthy and Muhlstein (1986) could not be verified, it is concluded that the design of the MFHT mixing system is suitable for homogenizing the tank contents.

6.6.1 Geometric Similarities

The dimensions of the bench-scale and the MFHT mixing systems are shown in Table 6.10. As shown in this table, the two systems are not geometrically similar. The greatest difference between the two mixing systems can be attributed to the different impellers. This difference is highlighted by the pumping numbers, which are much lower for the high-efficiency turbines used in the MFHT. The tank geometric ratios are also different in some cases, although this difference is not as significant as the differences in the impellers.

6.6.2 Kinematic and Dynamic Similarities

The Reynolds number and power number were determined for the MFHT and are compared to the parameters estimated from a scale-up procedure (Peterson, McCarthy and Muhlstein 1986) (Table 6.11 and Figure 6.5). The relatively large Reynolds numbers for the MFHT mixing system indicate that the system was operating in the turbulent flow region (i.e., $Re > 100,000$). The large variation in the Reynolds numbers is primarily a result of the variation in the apparent viscosity of the slurries. The power number for the MFHT mixing system is much lower than that for the bench-scale mixing system because different types of impellers were used. The large variation in viscosity has

TABLE 6.10. Comparison of Geometric Mixing System Parameters

Parameters	Bench-Scale Mixing System	Full-Scale Mixing System
<u>Top Impeller</u>		
Number of blades	3	3
Type of blade	High-efficiency turbine	High-efficiency turbine
Pumping number, N_Q	0.78	0.47
<u>Bottom Impeller</u>		
Number of blades	4	3
Type of blade	Flat-blade turbine	High-efficiency turbine
Pumping number, N_Q	0.8	0.47
<u>Dimensions</u>		
Tank dia. (T), in.	15	120
Impeller dia. (D), in.	4.5	36
Liquid level (L), in.	15	102
Clearance between impeller and tank bottom (C_1), in.	0.9	11.6
Distance between impellers (C_2), in.	4.5	56
Baffle width (B_w), in.	1.2	10
Impeller rotational speed (N), rpm	30-1000	155
<u>Geometric Ratios</u>		
T/D	3.3	3.3
L/D	3.3	2.8
L/T	1	0.85
C_1/D	0.2	0.32
C_2/D	1	1.5
B_w/D	0.27	0.28

very little impact on the power number because the inertial forces are much larger than the viscous forces in the turbulent flow region. The power delivered to the fluid is primarily a function of the density of the fluid and is nearly independent of the viscosity. Even though there were significant differences between the bench-scale and the MFHT mixing systems,

TABLE 6.11. Comparison of Kinematic and Dynamic Mixing System Parameters

Slurry Concentration	Apparent Viscosity (cP)	Reynolds, Number, Re	Power Number, N_p	Estimated Power to Fluid (Hp)
Concentrated	22.1	140,000	0.64	13.7
Reference	3.1	900,000	0.69	13.5
Dilute	2.7	1,000,000	0.70	13.3
Full-scale estimate	19	74,800	5.0	14.8

38807138.4M

FIGURE 6.5. Power Number Versus Reynolds Number for the Bench-Scale and Full-Scale Mixing Systems

the actual estimated power delivered to the fluid was only slightly smaller than the full-scale estimate based on bench-scale testing.

The data in Table 6.11 for the MFHT mixing test was calculated using the equations presented in Section 3 and rheological data presented in Section 6.1.2. The apparent viscosities for the slurries tested in the MFHT were estimated from Equation 3.4 using the rheological data from Figures 6.1, 6.2, and 6.3. The Reynolds numbers were calculated using Equation 3.8 and

the apparent viscosities. The power number was calculated using Equation 3.7, the fluid properties, the mixing system parameters in Table 6.10, and an estimate of the power delivered to the fluid.

The scale of agitation was calculated for the MFHT using the volumetric flow rate (29,700 gpm) supplied by the agitator manufacturer. Results from these calculations are presented in Table 6.12. Using the manufacturer-supplied flow rate and the cross sectional area of the MFHT, the bulk velocity was estimated using Equation 3.13. The scale of agitation was calculated using Equation 3.14. The calculated scale of agitation for the MFHT compares well with the scale of agitation recommended by Peterson, McCarthy and Muhlstein (1986).

The scale of agitation should be the same for the three slurries tested since the mixing system was operated in the turbulent flow region. This is shown by Equation 6.1, which is obtained by combining Equations 3.12, 3.13, and 3.14.

$$N_I = \frac{N_Q ND^3}{3.05A} \quad (6.1)$$

In the MFHT mixing system, the rotational speed (N), the impeller diameter (D), and the tank area (A) are fixed. Since the pumping number (N_Q) is a constant in the turbulent flow region where the MFHT mixing system operates, the scale of agitation is also constant.

TABLE 6.12. Scale of Agitation

Pumping capacity (supplied by manufacturer)	29,700 gpm
Calculated bulk velocity	50.6 ft/min
Calculated scale of agitation (all slurries tested)	8.4
Recommended scale of agitation (Peterson et al. 1986)	8.0
Pumping number (both turbines), N_Q	0.94

6.6.3 Conclusions

The scale-up from the bench-scale mixing system was qualitatively verified because the testing and statistical analysis showed that the tank was homogeneous for the three slurries tested. As shown in Section 6.3, Table 6.4, there were no significant differences between the six different sampling locations. This result is expected since the MFHT was tested at very high Reynolds numbers, indicating a high level of turbulence and mixing. The actual power requirement for a scale of agitation of 8 in the MFHT was within 10% of the power requirement estimated from the bench-scale mixing experiments.

Since the system operates at such a high Reynolds number, there is significant flexibility for handling feeds with a wide range of properties. As shown in the Table 6.11, the apparent viscosity can change by nearly an order of magnitude and mixing will remain in the turbulent flow region. If the mixing system is operated in the turbulent flow region, the scale of agitation should not change significantly and the feed mixture should be homogenous.

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APPENDIX A

SAMPLE AND ANALYSIS ORDER

APPENDIX A

SAMPLE AND ANALYSIS ORDER

Two types of tests were performed at West Valley using three concentrations of simulated, nonradioactive melter feed slurry. These two tests are called the homogeneity/sampling system test and the resuspension test. The homogeneity/sampling system test was performed to characterize the melter feed system at West Valley and to verify the scale-up of the bench-scale mixing studies performed at PNL. The resuspension test was performed to determine the time required to resuspend the solids and homogenize the melter slurry in the melter feed hold tank (MFHT) after an unexpected power outage.

The tests were statistically designed by PNL to determine whether an adequately homogeneous solution is obtained in the MFHT, and whether the liquid-sampling system provides a representative sample of the melter feed. The statistical design for the homogeneity and sampling system test determined that 21 samples at each of the seven sampling locations [six in the tank (Figure 4.1), plus the liquid-sampling system] were required to provide a 95% confidence that the correct conclusions were drawn. Only six samples at the six tank sampling locations were required for the resuspension tests.

The samples were taken from the tank and analyzed in a predetermined, random order. The sampling order for the homogeneity and sampling system tests is summarized in Tables A.1 through A.3. For the resuspension tests, the sampling order is summarized in Tables A.4 through A.6. The analysis order for the homogeneity and sampling system tests and the resuspension tests are summarized in Tables A.7 through A.9 and Tables A.10 through A.12, respectively.

TABLE A.1. Sampling Order for the Homogeneity and Sampling System Test Using the Concentrated Melter Feed Slurry

Order	Location ^(a)		Order	Location		Order	Location		Order	Location	
1	TOP	NOZZLE C	43	MIDDLE	NOZZLE C	85	SAMPLER		127	MIDDLE	NOZZLE C
2	BOTTOM	NOZZLE D	44	SAMPLER		86	BOTTOM	NOZZLE C	128	TOP	NOZZLE D
3	MIDDLE	NOZZLE C	45	MIDDLE	NOZZLE D	87	TOP	NOZZLE D	129	SAMPLER	
4	MIDDLE	NOZZLE C	46	MIDDLE	NOZZLE C	88	BOTTOM	NOZZLE C	130	TOP	NOZZLE C
5	MIDDLE	NOZZLE D	47	TOP	NOZZLE D	89	SAMPLER		131	TOP	NOZZLE D
6	TOP	NOZZLE C	48	SAMPLER		90	BOTTOM	NOZZLE D	132	TOP	NOZZLE C
7	TOP	NOZZLE D	49	BOTTOM	NOZZLE D	91	MIDDLE	NOZZLE D	133	BOTTOM	NOZZLE D
8	TOP	NOZZLE C	50	SAMPLER		92	MIDDLE	NOZZLE D	134	SAMPLER	
9	SAMPLER ^(b)		51	MIDDLE	NOZZLE C	93	TOP	NOZZLE C	135	BOTTOM	NOZZLE C
10	MIDDLE	NOZZLE D	52	TOP	NOZZLE C	94	SAMPLER		136	TOP	NOZZLE D
11	MIDDLE	NOZZLE C	53	MIDDLE	NOZZLE D	95	MIDDLE	NOZZLE D	137	TOP	NOZZLE D
12	SAMPLER		54	MIDDLE	NOZZLE C	96	TOP	NOZZLE C	138	MIDDLE	NOZZLE C
13	TOP	NOZZLE D	55	SAMPLER		97	BOTTOM	NOZZLE C	139	TOP	NOZZLE D
14	SAMPLER		56	TOP	NOZZLE D	98	SAMPLER		140	BOTTOM	NOZZLE D
15	BOTTOM	NOZZLE C	57	BOTTOM	NOZZLE C	99	TOP	NOZZLE D	141	MIDDLE	NOZZLE D
16	BOTTOM	NOZZLE D	58	MIDDLE	NOZZLE C	100	TOP	NOZZLE C	142	BOTTOM	NOZZLE D
17	BOTTOM	NOZZLE D	59	BOTTOM	NOZZLE C	101	TOP	NOZZLE C	143	BOTTOM	NOZZLE D
18	TOP	NOZZLE D	60	SAMPLER		102	SAMPLER		144	BOTTOM	NOZZLE C
19	BOTTOM	NOZZLE C	61	BOTTOM	NOZZLE D	103	MIDDLE	NOZZLE D	145	MIDDLE	NOZZLE D
20	BOTTOM	NOZZLE D	62	MIDDLE	NOZZLE D	104	TOP	NOZZLE C	146	MIDDLE	NOZZLE C
21	BOTTOM	NOZZLE D	63	MIDDLE	NOZZLE C	105	BOTTOM	NOZZLE C	147	BOTTOM	NOZZLE D
22	MIDDLE	NOZZLE D	64	TOP	NOZZLE D	106	TOP	NOZZLE C	148	MIDDLE	NOZZLE D
23	BOTTOM	NOZZLE D	65	MIDDLE	NOZZLE C	107	TOP	NOZZLE D	149	BOTTOM	NOZZLE D
24	MIDDLE	NOZZLE C	66	MIDDLE	NOZZLE C	108	MIDDLE	NOZZLE C	150	SAMPLER	
25	TOP	NOZZLE D	67	TOP	NOZZLE D	109	TOP	NOZZLE C	151	BOTTOM	NOZZLE C
26	SAMPLER		68	MIDDLE	NOZZLE D	110	BOTTOM	NOZZLE C	152	TOP	NOZZLE C
27	MIDDLE	NOZZLE C	69	BOTTOM	NOZZLE D	111	TOP	NOZZLE C	153	MIDDLE	NOZZLE D
28	TOP	NOZZLE D	70	MIDDLE	NOZZLE D	112	SAMPLER		154	BOTTOM	NOZZLE C
29	BOTTOM	NOZZLE D	71	MIDDLE	NOZZLE D	113	BOTTOM	NOZZLE D			
30	BOTTOM	NOZZLE C	72	TOP	NOZZLE C	114	MIDDLE	NOZZLE D			
31	TOP	NOZZLE D	73	MIDDLE	NOZZLE C	115	SAMPLER				
32	BOTTOM	NOZZLE D	74	MIDDLE	NOZZLE C	116	BOTTOM	NOZZLE C			
33	TOP	NOZZLE D	75	BOTTOM	NOZZLE C	117	MIDDLE	NOZZLE D			
34	BOTTOM	NOZZLE D	76	BOTTOM	NOZZLE D	118	MIDDLE	NOZZLE C			
35	BOTTOM	NOZZLE C	77	BOTTOM	NOZZLE C	119	BOTTOM	NOZZLE D			
36	SAMPLER		78	MIDDLE	NOZZLE C	120	TOP	NOZZLE C			
37	TOP	NOZZLE D	79	TOP	NOZZLE D	121	BOTTOM	NOZZLE D			
38	TOP	NOZZLE C	80	SAMPLER		122	MIDDLE	NOZZLE C			
39	MIDDLE	NOZZLE D	81	TOP	NOZZLE C	123	BOTTOM	NOZZLE C			
40	SAMPLER		82 ^(c)	MIDDLE	NOZZLE D	124	TOP	NOZZLE C			
41	TOP	NOZZLE C	83	MIDDLE	NOZZLE D	125	BOTTOM	NOZZLE C			
42	TOP	NOZZLE C	84	TOP	NOZZLE D	126	BOTTOM	NOZZLE C			

(a) See Figure 4.1.

(b) "Sampler" refers to Liquid Sampling System.

(c) A 250 ml sample was drawn. This sample is referred to as the "CONTROL" sample.

TABLE A.2. Sampling Order for the Homogeneity and Sampling System Test Using the Reference Melter Feed Slurry

Order	Location ^(a)		Order	Location		Order	Location		Order	Location	
1	TOP	NOZZLE C	43	BOTTOM	NOZZLE C	85	MIDDLE	NOZZLE C	127	BOTTOM	NOZZLE D
2	TOP	NOZZLE D	44	TOP	NOZZLE C	86	TOP	NOZZLE C	128	SAMPLER	
3	MIDDLE	NOZZLE D	45	BOTTOM	NOZZLE D	87	MIDDLE	NOZZLE C	129	TOP	NOZZLE C
4	SAMPLER		46	MIDDLE	NOZZLE C	88	MIDDLE	NOZZLE C	130	MIDDLE	NOZZLE D
5	MIDDLE	NOZZLE C	47	TOP	NOZZLE C	89	MIDDLE	NOZZLE C	131	BOTTOM	NOZZLE D
6	BOTTOM	NOZZLE C	48	TOP	NOZZLE C	90	TOP	NOZZLE C	132	MIDDLE	NOZZLE D
7	TOP	NOZZLE D	49	SAMPLER		91	BOTTOM	NOZZLE C	133	SAMPLER	
8	TOP	NOZZLE D	50	MIDDLE	NOZZLE D	92	BOTTOM	NOZZLE C	134	BOTTOM	NOZZLE C
9	SAMPLER ^(b)		51	TOP	NOZZLE D	93	BOTTOM	NOZZLE C	135	TOP	NOZZLE C
10	BOTTOM	NOZZLE C	52	MIDDLE	NOZZLE D	94	TOP	NOZZLE C	136	TOP	NOZZLE C
11	TOP	NOZZLE C	53	BOTTOM	NOZZLE C	95	SAMPLER		137	TOP	NOZZLE D
12	TOP	NOZZLE D	54	BOTTOM	NOZZLE C	96	BOTTOM	NOZZLE D	138	BOTTOM	NOZZLE C
13	TOP	NOZZLE D	55	SAMPLER		97	TOP	NOZZLE C	139	SAMPLER	
14	MIDDLE	NOZZLE D	56	BOTTOM	NOZZLE D	98	MIDDLE	NOZZLE C	140	MIDDLE	NOZZLE D
15	MIDDLE	NOZZLE D	57	TOP	NOZZLE D	99	SAMPLER		141	BOTTOM	NOZZLE D
16	BOTTOM	NOZZLE C	58	TOP	NOZZLE C	100	BOTTOM	NOZZLE D	142	TOP	NOZZLE D
17	TOP	NOZZLE D	59	SAMPLER		101	TOP	NOZZLE C	143	MIDDLE	NOZZLE D
18	TOP	NOZZLE C	60	BOTTOM	NOZZLE D	102	TOP	NOZZLE C	144	MIDDLE	NOZZLE D
19	BOTTOM	NOZZLE D	61	MIDDLE	NOZZLE D	103	MIDDLE	NOZZLE D	145	SAMPLER	
20	SAMPLER		62	MIDDLE	NOZZLE C	104	TOP	NOZZLE D	146	BOTTOM	NOZZLE C
21	BOTTOM	NOZZLE C	63	SAMPLER		105	TOP	NOZZLE D	147	TOP	NOZZLE C
22	BOTTOM	NOZZLE D	64	TOP	NOZZLE D	106	SAMPLER		148	MIDDLE	NOZZLE C
23	BOTTOM	NOZZLE C	65	BOTTOM	NOZZLE D	107	MIDDLE	NOZZLE C	149	SAMPLER	
24	BOTTOM	NOZZLE C	66	TOP	NOZZLE C	108	MIDDLE	NOZZLE C	150	MIDDLE	NOZZLE D
25	MIDDLE	NOZZLE D	67	MIDDLE	NOZZLE D	109	BOTTOM	NOZZLE D	151	BOTTOM	NOZZLE D
26	SAMPLER		68	BOTTOM	NOZZLE D	110	MIDDLE	NOZZLE D	152	SAMPLER	
27	MIDDLE	NOZZLE C	69	MIDDLE	NOZZLE C	111	TOP	NOZZLE D	153	BOTTOM	NOZZLE C
28	MIDDLE	NOZZLE D	70	BOTTOM	NOZZLE D	112	BOTTOM	NOZZLE D	154	MIDDLE	NOZZLE D
29	MIDDLE	NOZZLE C	71	MIDDLE	NOZZLE C	113	BOTTOM	NOZZLE C			
30	TOP	NOZZLE D	72	MIDDLE	NOZZLE C	114	SAMPLER				
31	MIDDLE	NOZZLE D	73	BOTTOM	NOZZLE C	115	TOP	NOZZLE D			
32	SAMPLER		74	SAMPLER		116	TOP	NOZZLE D			
33	TOP	NOZZLE C	75	TOP	NOZZLE D	117	MIDDLE	NOZZLE C			
34	BOTTOM	NOZZLE C	76	BOTTOM	NOZZLE D	118	TOP	NOZZLE D			
35	MIDDLE	NOZZLE C	77	TOP	NOZZLE D	119	BOTTOM	NOZZLE D			
36	MIDDLE	NOZZLE C	78	MIDDLE	NOZZLE D	120	TOP	NOZZLE D			
37	MIDDLE	NOZZLE C	79	TOP	NOZZLE C	121	BOTTOM	NOZZLE C			
38	BOTTOM	NOZZLE C	80	TOP	NOZZLE D	122	MIDDLE	NOZZLE D			
39	MIDDLE	NOZZLE C	81	MIDDLE	NOZZLE C	123	MIDDLE	NOZZLE D			
40	TOP	NOZZLE C	82	SAMPLER		124	BOTTOM	NOZZLE D			
41	SAMPLER		83	BOTTOM	NOZZLE D	125	BOTTOM	NOZZLE C			
42	BOTTOM	NOZZLE D	84	BOTTOM	NOZZLE D	126	TOP	NOZZLE C			

(a) See Figure 4.1.

(b) "Sampler" refers to Liquid Sampling System.

TABLE A.3. Sampling Order for the Homogeneity and Sampling System Test Using the Dilute Melter Feed Slurry

Order	Location ^(a)		Order	Location		Order	Location		Order	Location	
1	TOP	NOZZLE C	43	BOTTOM	NOZZLE D	85	BOTTOM	NOZZLE C	127	TOP	NOZZLE D
2	MIDDLE	NOZZLE C	44	SAMPLER		86	SAMPLER		128	BOTTOM	NOZZLE C
3	BOTTOM	NOZZLE C	45	MIDDLE	NOZZLE C	87	BOTTOM	NOZZLE D	129	TOP	NOZZLE D
4	BOTTOM	NOZZLE C	46	BOTTOM	NOZZLE C	88	TOP	NOZZLE D	130	SAMPLER	
5	BOTTOM	NOZZLE D	47	SAMPLER		89	TOP	NOZZLE C	131	TOP	NOZZLE C
6	TOP	NOZZLE C	48	TOP	NOZZLE D	90	BOTTOM	NOZZLE C	132	TOP	NOZZLE C
7	TOP	NOZZLE C	59	MIDDLE	NOZZLE D	91	BOTTOM	NOZZLE D	133	MIDDLE	NOZZLE C
8	BOTTOM	NOZZLE C	50	TOP	NOZZLE C	92	TOP	NOZZLE C	134	BOTTOM	NOZZLE D
9	TOP	NOZZLE D	51	SAMPLER		93	SAMPLER		135	BOTTOM	NOZZLE D
10	MIDDLE	NOZZLE C	52	BOTTOM	NOZZLE D	94	MIDDLE	NOZZLE D	136	MIDDLE	NOZZLE D
11	BOTTOM	NOZZLE D	53	SAMPLER		95	MIDDLE	NOZZLE D	137	BOTTOM	NOZZLE C
12	BOTTOM	NOZZLE C	54	MIDDLE	NOZZLE C	96	MIDDLE	NOZZLE C	138	SAMPLER	
13	TOP	NOZZLE C	55	TOP	NOZZLE C	97	BOTTOM	NOZZLE D	139	MIDDLE	NOZZLE D
14	MIDDLE	NOZZLE D	56	BOTTOM	NOZZLE D	98	BOTTOM	NOZZLE D	140	TOP	NOZZLE D
15	TOP	NOZZLE D	57	MIDDLE	NOZZLE D	99	BOTTOM	NOZZLE D	141	BOTTOM	NOZZLE C
16	BOTTOM	NOZZLE C	58	BOTTOM	NOZZLE D	100	SAMPLER		142	SAMPLER	
17	TOP	NOZZLE D	59	BOTTOM	NOZZLE D	101	BOTTOM	NOZZLE C	143	TOP	NOZZLE D
18	MIDDLE	NOZZLE D	60	MIDDLE	NOZZLE D	102	MIDDLE	NOZZLE C	144	MIDDLE	NOZZLE C
19	MIDDLE	NOZZLE C	61	SAMPLER		103	MIDDLE	NOZZLE C	145	TOP	NOZZLE C
20	TOP	NOZZLE C	62	MIDDLE	NOZZLE C	104	SAMPLER		146	MIDDLE	NOZZLE D
21	BOTTOM	NOZZLE D	63	BOTTOM	NOZZLE C	105	BOTTOM	NOZZLE D	147	MIDDLE	NOZZLE C
22	SAMPLER ^(b)		64	BOTTOM	NOZZLE C	106	TOP	NOZZLE D	148	MIDDLE	NOZZLE C
23	BOTTOM	NOZZLE C	65	TOP	NOZZLE D	107	TOP	NOZZLE C	149	BOTTOM	NOZZLE C
24	BOTTOM	NOZZLE D	66	TOP	NOZZLE D	108	TOP	NOZZLE C	150	SAMPLER	
25	MIDDLE	NOZZLE C	67	TOP	NOZZLE C	109	MIDDLE	NOZZLE C	151	MIDDLE	NOZZLE D
26	BOTTOM	NOZZLE D	68	SAMPLER		110	TOP	NOZZLE C	152	MIDDLE	NOZZLE D
27	SAMPLER		69	TOP	NOZZLE D	111	TOP	NOZZLE D	153	MIDDLE	NOZZLE D
28	MIDDLE	NOZZLE C	70	TOP	NOZZLE D	112	SAMPLER		154	TOP	NOZZLE D
29	BOTTOM	NOZZLE C	71	BOTTOM	NOZZLE D	113	MIDDLE	NOZZLE C			
30	MIDDLE	NOZZLE C	72	BOTTOM	NOZZLE D	114	BOTTOM	NOZZLE C			
31	MIDDLE	NOZZLE C	73	SAMPLER		115	TOP	NOZZLE C			
32	BOTTOM	NOZZLE D	74	MIDDLE	NOZZLE C	116	MIDDLE	NOZZLE D			
33	BOTTOM	NOZZLE C	75	MIDDLE	NOZZLE D	117	TOP	NOZZLE D			
34	MIDDLE	NOZZLE D	76	BOTTOM	NOZZLE D	118	TOP	NOZZLE C			
35	BOTTOM	NOZZLE C	77	SAMPLER		119	MIDDLE	NOZZLE D			
36	MIDDLE	NOZZLE C	78	MIDDLE	NOZZLE C	120	TOP	NOZZLE D			
37	MIDDLE	NOZZLE D	79	BOTTOM	NOZZLE C	121	MIDDLE	NOZZLE D			
38	TOP	NOZZLE C	80	SAMPLER		122	TOP	NOZZLE C			
39	TOP	NOZZLE D	81	MIDDLE	NOZZLE D	123	TOP	NOZZLE C			
40	SAMPLER		82	TOP	NOZZLE D	124	SAMPLER				
41	BOTTOM	NOZZLE C	83	TOP	NOZZLE C	125	TOP	NOZZLE D			
42	TOP	NOZZLE D	84	MIDDLE	NOZZLE D	126	MIDDLE	NOZZLE D			

(a) See Figure 4.1.

(b) "Sampler" refers to Liquid Sampling System.

**TABLE A.4. Sampling Order for the Resuspension Test Using the
Concentrated Melter Feed Slurry**

2 Hour Wait-Time				6 Hour Wait-Time				24 Hour Wait-Time			
Order	Location(a)			Order	Location			Order	Location		
155	BOTTOM	NOZZLE	C	197	BOTTOM	NOZZLE	C	239	BOTTOM	NOZZLE	C
156	TOP	NOZZLE	C	198	MIDDLE	NOZZLE	C	240	TOP	NOZZLE	C
157	TOP	NOZZLE	C	199	MIDDLE	NOZZLE	D	241	TOP	NOZZLE	C
158	MIDDLE	NOZZLE	D	200	TOP	NOZZLE	C	242	MIDDLE	NOZZLE	D
159	MIDDLE	NOZZLE	C	201	TOP	NOZZLE	C	243	MIDDLE	NOZZLE	C
160	TOP	NOZZLE	D	202	BOTTOM	NOZZLE	C	244	TOP	NOZZLE	D
161	TOP	NOZZLE	D	203	TOP	NOZZLE	C	245	TOP	NOZZLE	D
162	TOP	NOZZLE	D	204	TOP	NOZZLE	D	246	TOP	NOZZLE	D
163	BOTTOM	NOZZLE	D	205	BOTTOM	NOZZLE	D	247	BOTTOM	NOZZLE	D
164	TOP	NOZZLE	D	206	MIDDLE	NOZZLE	D	248	TOP	NOZZLE	D
165	MIDDLE	NOZZLE	D	207	BOTTOM	NOZZLE	D	249	MIDDLE	NOZZLE	D
166	TOP	NOZZLE	C	208	MIDDLE	NOZZLE	C	250	TOP	NOZZLE	C
167	TOP	NOZZLE	D	209	MIDDLE	NOZZLE	D	251	TOP	NOZZLE	D
168	BOTTOM	NOZZLE	D	210	TOP	NOZZLE	C	252	BOTTOM	NOZZLE	D
169	BOTTOM	NOZZLE	C	211	BOTTOM	NOZZLE	C	253	BOTTOM	NOZZLE	C
170	BOTTOM	NOZZLE	C	212	BOTTOM	NOZZLE	C	254	BOTTOM	NOZZLE	C
171	MIDDLE	NOZZLE	C	213	MIDDLE	NOZZLE	D	255	MIDDLE	NOZZLE	C
172	BOTTOM	NOZZLE	C	214	BOTTOM	NOZZLE	D	256	BOTTOM	NOZZLE	C
173	MIDDLE	NOZZLE	C	215	TOP	NOZZLE	D	257	MIDDLE	NOZZLE	C
174	BOTTOM	NOZZLE	C	216	TOP	NOZZLE	C	258	BOTTOM	NOZZLE	C
175	BOTTOM	NOZZLE	C	217	TOP	NOZZLE	D	259	BOTTOM	NOZZLE	C
176	MIDDLE	NOZZLE	C	218	MIDDLE	NOZZLE	D	260	MIDDLE	NOZZLE	C
177	BOTTOM	NOZZLE	D	219	MIDDLE	NOZZLE	C	261	BOTTOM	NOZZLE	D
178	TOP	NOZZLE	C	220	TOP	NOZZLE	C	262	TOP	NOZZLE	C
179	MIDDLE	NOZZLE	D	221	TOP	NOZZLE	D	263	MIDDLE	NOZZLE	D
180	BOTTOM	NOZZLE	D	222	BOTTOM	NOZZLE	D	264	BOTTOM	NOZZLE	D
181	MIDDLE	NOZZLE	C	223	MIDDLE	NOZZLE	D	265	MIDDLE	NOZZLE	C
182	MIDDLE	NOZZLE	C	224	TOP	NOZZLE	D	266	MIDDLE	NOZZLE	C
183	MIDDLE	NOZZLE	D	225	BOTTOM	NOZZLE	D	267	MIDDLE	NOZZLE	D
184	MIDDLE	NOZZLE	C	226	TOP	NOZZLE	C	268	MIDDLE	NOZZLE	C
185	TOP	NOZZLE	C	227	MIDDLE	NOZZLE	C	269	TOP	NOZZLE	C
186	BOTTOM	NOZZLE	D	228	TOP	NOZZLE	D	270	BOTTOM	NOZZLE	D
187	TOP	NOZZLE	C	229	MIDDLE	NOZZLE	D	271	TOP	NOZZLE	C
188	BOTTOM	NOZZLE	C	230	BOTTOM	NOZZLE	D	272	BOTTOM	NOZZLE	C
189	BOTTOM	NOZZLE	D	231	TOP	NOZZLE	D	273	BOTTOM	NOZZLE	D
190	MIDDLE	NOZZLE	D	232	MIDDLE	NOZZLE	C	274	MIDDLE	NOZZLE	D
191	TOP	NOZZLE	D	233	BOTTOM	NOZZLE	C	275	TOP	NOZZLE	D
192	MIDDLE	NOZZLE	D	234	BOTTOM	NOZZLE	D	276	MIDDLE	NOZZLE	D
193	BOTTOM	NOZZLE	D	235	BOTTOM	NOZZLE	C	277	BOTTOM	NOZZLE	D
194	MIDDLE	NOZZLE	D	236	MIDDLE	NOZZLE	C	278	MIDDLE	NOZZLE	D
195	TOP	NOZZLE	D	237	MIDDLE	NOZZLE	C	279	TOP	NOZZLE	D
196	TOP	NOZZLE	C	238	BOTTOM	NOZZLE	C	280	TOP	NOZZLE	C

(a) See Figure 4.1.

TABLE A.5. Sampling Order for the Resuspension Test Using the Reference Melter Feed Slurry

2 Hour Wait-Time			6 Hour Wait-Time			24 Hour Wait-Time		
Order	Location(a)		Order	Location		Order	Location	
155	MIDDLE	NOZZLE D	197	TOP	NOZZLE C	239	MIDDLE	NOZZLE D
156	MIDDLE	NOZZLE D	198	BOTTOM	NOZZLE D	240	MIDDLE	NOZZLE D
157	BOTTOM	NOZZLE C	199	BOTTOM	NOZZLE D	241	BOTTOM	NOZZLE C
158	BOTTOM	NOZZLE C	200	BOTTOM	NOZZLE D	242	BOTTOM	NOZZLE C
159	MIDDLE	NOZZLE C	201	BOTTOM	NOZZLE C	243	MIDDLE	NOZZLE C
160	MIDDLE	NOZZLE C	202	TOP	NOZZLE C	244	MIDDLE	NOZZLE C
161	MIDDLE	NOZZLE C	203	BOTTOM	NOZZLE D	245	MIDDLE	NOZZLE C
162	BOTTOM	NOZZLE D	204	MIDDLE	NOZZLE D	246	BOTTOM	NOZZLE D
163	BOTTOM	NOZZLE D	205	TOP	NOZZLE C	247	BOTTOM	NOZZLE D
164	MIDDLE	NOZZLE D	206	MIDDLE	NOZZLE C	248	MIDDLE	NOZZLE D
165	MIDDLE	NOZZLE C	207	TOP	NOZZLE D	249	MIDDLE	NOZZLE C
166	BOTTOM	NOZZLE C	208	TOP	NOZZLE D	250	BOTTOM	NOZZLE C
167	TOP	NOZZLE D	209	MIDDLE	NOZZLE D	251	TOP	NOZZLE D
168	MIDDLE	NOZZLE D	210	TOP	NOZZLE C	252	MIDDLE	NOZZLE D
169	BOTTOM	NOZZLE C	211	BOTTOM	NOZZLE C	253	BOTTOM	NOZZLE C
170	MIDDLE	NOZZLE C	212	MIDDLE	NOZZLE C	254	MIDDLE	NOZZLE C
171	TOP	NOZZLE D	213	MIDDLE	NOZZLE C	255	TOP	NOZZLE D
172	TOP	NOZZLE C	214	TOP	NOZZLE D	256	TOP	NOZZLE C
173	TOP	NOZZLE C	215	BOTTOM	NOZZLE D	257	TOP	NOZZLE C
174	MIDDLE	NOZZLE D	216	MIDDLE	NOZZLE C	258	MIDDLE	NOZZLE D
175	MIDDLE	NOZZLE C	217	BOTTOM	NOZZLE C	259	MIDDLE	NOZZLE C
176	TOP	NOZZLE D	218	MIDDLE	NOZZLE D	260	TOP	NOZZLE D
177	BOTTOM	NOZZLE C	219	TOP	NOZZLE D	261	BOTTOM	NOZZLE C
178	TOP	NOZZLE C	220	BOTTOM	NOZZLE D	262	TOP	NOZZLE C
179	TOP	NOZZLE D	221	MIDDLE	NOZZLE D	263	TOP	NOZZLE D
180	MIDDLE	NOZZLE D	222	TOP	NOZZLE C	264	MIDDLE	NOZZLE D
181	BOTTOM	NOZZLE D	223	MIDDLE	NOZZLE C	265	BOTTOM	NOZZLE D
182	TOP	NOZZLE C	224	TOP	NOZZLE D	266	TOP	NOZZLE C
183	BOTTOM	NOZZLE C	225	TOP	NOZZLE C	267	BOTTOM	NOZZLE C
184	TOP	NOZZLE D	226	TOP	NOZZLE C	268	TOP	NOZZLE D
185	BOTTOM	NOZZLE D	227	TOP	NOZZLE D	269	BOTTOM	NOZZLE D
186	BOTTOM	NOZZLE C	228	MIDDLE	NOZZLE C	270	BOTTOM	NOZZLE C
187	TOP	NOZZLE D	229	MIDDLE	NOZZLE D	271	TOP	NOZZLE D
188	TOP	NOZZLE D	230	BOTTOM	NOZZLE C	272	TOP	NOZZLE D
189	MIDDLE	NOZZLE D	231	BOTTOM	NOZZLE C	273	MIDDLE	NOZZLE D
190	TOP	NOZZLE C	232	BOTTOM	NOZZLE D	274	TOP	NOZZLE C
191	BOTTOM	NOZZLE D	233	BOTTOM	NOZZLE C	275	BOTTOM	NOZZLE D
192	BOTTOM	NOZZLE D	234	TOP	NOZZLE D	276	BOTTOM	NOZZLE D
193	MIDDLE	NOZZLE C	235	MIDDLE	NOZZLE D	277	MIDDLE	NOZZLE C
194	BOTTOM	NOZZLE D	236	MIDDLE	NOZZLE D	278	BOTTOM	NOZZLE D
195	TOP	NOZZLE C	237	BOTTOM	NOZZLE C	279	TOP	NOZZLE C
196	TOP	NOZZLE C	238	MIDDLE	NOZZLE C	280	TOP	NOZZLE C

(a) See Figure 4.1.

TABLE A.6. Sampling Order for the Resuspension Test Using the Dilute Melter Feed Slurry

2 Hour Wait-Time				6 Hour Wait-Time				24 Hour Wait-Time			
Order	Location(a)			Order	Location			Order	Location		
155	BOTTOM	NOZZLE	C	197	BOTTOM	NOZZLE	C	239	BOTTOM	NOZZLE	C
156	TOP	NOZZLE	C	198	BOTTOM	NOZZLE	C	240	TOP	NOZZLE	C
157	BOTTOM	NOZZLE	D	199	TOP	NOZZLE	D	241	BOTTOM	NOZZLE	D
158	TOP	NOZZLE	D	200	MIDDLE	NOZZLE	D	242	TOP	NOZZLE	D
159	TOP	NOZZLE	D	201	MIDDLE	NOZZLE	C	243	TOP	NOZZLE	D
160	BOTTOM	NOZZLE	D	202	BOTTOM	NOZZLE	D	244	BOTTOM	NOZZLE	D
161	MIDDLE	NOZZLE	C	203	TOP	NOZZLE	C	245	MIDDLE	NOZZLE	C
162	MIDDLE	NOZZLE	C	204	MIDDLE	NOZZLE	D	246	MIDDLE	NOZZLE	C
163	MIDDLE	NOZZLE	D	205	MIDDLE	NOZZLE	D	247	MIDDLE	NOZZLE	D
164	TOP	NOZZLE	D	206	MIDDLE	NOZZLE	C	248	TOP	NOZZLE	D
165	TOP	NOZZLE	C	207	MIDDLE	NOZZLE	D	249	TOP	NOZZLE	C
166	BOTTOM	NOZZLE	C	208	TOP	NOZZLE	D	250	BOTTOM	NOZZLE	C
167	BOTTOM	NOZZLE	D	209	BOTTOM	NOZZLE	C	251	BOTTOM	NOZZLE	D
168	BOTTOM	NOZZLE	D	210	TOP	NOZZLE	C	252	BOTTOM	NOZZLE	D
169	TOP	NOZZLE	C	211	BOTTOM	NOZZLE	C	253	TOP	NOZZLE	C
170	TOP	NOZZLE	C	212	MIDDLE	NOZZLE	C	254	TOP	NOZZLE	C
171	TOP	NOZZLE	D	213	BOTTOM	NOZZLE	D	255	TOP	NOZZLE	D
172	TOP	NOZZLE	C	214	MIDDLE	NOZZLE	D	256	TOP	NOZZLE	C
173	MIDDLE	NOZZLE	C	215	MIDDLE	NOZZLE	C	257	MIDDLE	NOZZLE	C
174	BOTTOM	NOZZLE	D	216	BOTTOM	NOZZLE	D	258	BOTTOM	NOZZLE	D
175	BOTTOM	NOZZLE	C	217	TOP	NOZZLE	C	259	BOTTOM	NOZZLE	C
176	MIDDLE	NOZZLE	D	218	MIDDLE	NOZZLE	C	260	MIDDLE	NOZZLE	D
177	MIDDLE	NOZZLE	D	219	BOTTOM	NOZZLE	D	261	MIDDLE	NOZZLE	D
178	BOTTOM	NOZZLE	C	220	BOTTOM	NOZZLE	C	262	BOTTOM	NOZZLE	C
179	BOTTOM	NOZZLE	C	221	TOP	NOZZLE	D	263	BOTTOM	NOZZLE	C
180	MIDDLE	NOZZLE	D	222	TOP	NOZZLE	C	264	MIDDLE	NOZZLE	D
181	BOTTOM	NOZZLE	D	223	TOP	NOZZLE	D	265	BOTTOM	NOZZLE	D
182	MIDDLE	NOZZLE	C	224	TOP	NOZZLE	C	266	MIDDLE	NOZZLE	C
183	TOP	NOZZLE	D	225	TOP	NOZZLE	D	267	TOP	NOZZLE	D
184	MIDDLE	NOZZLE	D	226	MIDDLE	NOZZLE	C	268	MIDDLE	NOZZLE	D
185	TOP	NOZZLE	C	227	TOP	NOZZLE	C	269	TOP	NOZZLE	C
186	TOP	NOZZLE	D	228	MIDDLE	NOZZLE	D	270	TOP	NOZZLE	D
187	BOTTOM	NOZZLE	D	229	BOTTOM	NOZZLE	D	271	BOTTOM	NOZZLE	D
188	BOTTOM	NOZZLE	C	230	TOP	NOZZLE	D	272	BOTTOM	NOZZLE	C
189	TOP	NOZZLE	D	231	MIDDLE	NOZZLE	C	273	TOP	NOZZLE	D
190	MIDDLE	NOZZLE	D	232	TOP	NOZZLE	D	274	MIDDLE	NOZZLE	D
191	MIDDLE	NOZZLE	C	233	BOTTOM	NOZZLE	C	275	MIDDLE	NOZZLE	C
192	MIDDLE	NOZZLE	C	234	BOTTOM	NOZZLE	D	276	MIDDLE	NOZZLE	C
193	TOP	NOZZLE	C	235	MIDDLE	NOZZLE	D	277	TOP	NOZZLE	C
194	MIDDLE	NOZZLE	D	236	TOP	NOZZLE	C	278	MIDDLE	NOZZLE	D
195	MIDDLE	NOZZLE	C	237	BOTTOM	NOZZLE	D	279	MIDDLE	NOZZLE	C
196	BOTTOM	NOZZLE	C	238	BOTTOM	NOZZLE	C	280	BOTTOM	NOZZLE	C

(a) See Figure 4.1.

TABLE A.7. Sample Analysis Order for the Homogeneity and Sampling System Test Using the Concentrated Melter Feed Slurry

Analysis Order	Sample Order (a)	Analysis Order	Sample Order	Analysis Order	Sample Order	Analysis Order	Sample Order
1	23	42	113	83	144	124	86
2	98	43	153	84	76	125	96
3	15	44	125	85	88	126	17
4	64	45	34	86	107	127	38
5	123	46	141	87	66	128	CONTROL
6	60	47	92	88	90	129	72
7	36	48	CONTROL	89	135	130	93
8	47	49	19	90	28	131	120
9	14	50	73	91	5	132	26
10	150	51	67	92	134	133	143
11	31	52	12	93	87	134	121
12	99	53	110	94	103	135	27
13	102	54	116	95	151	136	74
14	45	55	68	96	CONTROL	137	20
15	83	56	24	97	13	138	62
16	CONTROL (b)	57	65	98	147	139	97
17	57	58	79	99	61	140	52
18	6	59	84	100	11	141	3
19	139	60	56	101	18	142	95
20	128	61	101	102	49	143	63
21	41	62	127	103	126	144	CONTROL
22	10	63	8	104	32	145	91
23	114	64	CONTROL	105	112	146	140
24	94	65	43	106	108	147	100
25	51	66	44	107	146	148	137
26	16	67	21	108	75	149	4
27	9	68	59	109	77	150	7
28	71	69	58	110	29	151	111
29	106	70	78	111	104	152	118
30	130	71	81	112	CONTROL	153	22
31	35	72	42	113	124	154	2
32	CONTROL	73	122	114	131	155	133
33	48	74	119	115	136	156	142
34	1	75	80	116	39	157	40
35	55	76	30	117	82	158	115
36	33	77	149	118	132	159	148
37	37	78	89	119	69	160	CONTROL
38	53	79	46	120	129	161	105
39	85	80	CONTROL	121	70	162	145
40	154	81	117	122	50	163	109
41	152	82	25	123	54	164	138

(a) See Table A.1 for description of sample locations.

(b) A 250 ml sample was taken during the Homogeneity and Sampling System Test using the concentrated melter feed slurry. Aliquots from this sample were analyzed every 16th sample to determine if long-term analytical effects existed.

TABLE A.8. Sample Analysis Order for the Homogeneity and Sampling System Test Using the Reference Melter Feed Slurry

Analysis Order	Sample Order ^(a)	Analysis Order	Sample Order	Analysis Order	Sample Order	Analysis Order	Sample Order
1	34	42	26	83	14	124	96
2	66	43	3	84	95	125	57
3	94	44	114	85	62	126	153
4	136	45	129	86	91	127	86
5	110	46	104	87	83	128	CONTROL
6	100	47	134	88	123	129	19
7	143	48	CONTROL	89	132	130	98
8	99	49	30	90	20	131	6
9	92	50	42	91	1	132	85
10	81	51	13	92	107	133	44
11	84	52	126	93	17	134	55
12	60	53	67	94	88	135	106
13	145	54	117	95	18	136	135
14	61	55	23	96	CONTROL	137	118
15	124	56	121	97	74	138	77
16	CONTROL ^(b)	57	54	98	119	139	24
17	89	58	36	99	115	140	151
18	10	59	102	100	90	141	11
19	78	60	33	101	65	142	12
20	149	61	103	102	93	143	8
21	120	62	131	103	87	144	CONTROL
22	46	63	29	104	105	145	68
23	122	64	CONTROL	105	63	146	51
24	47	65	109	106	112	147	113
25	25	66	111	107	49	148	127
26	39	67	40	108	144	149	154
27	21	68	64	109	31	150	97
28	130	69	76	110	128	151	108
29	150	70	137	111	52	152	15
30	138	71	142	112	CONTROL	153	70
31	75	72	69	113	41	154	27
32	CONTROL	73	7	114	101	155	80
33	73	74	82	115	71	156	50
34	2	75	133	116	37	157	38
35	59	76	140	117	9	158	5
36	148	77	152	118	22	159	141
37	35	78	16	119	43	160	CONTROL
38	4	79	48	120	56	161	53
39	125	80	CONTROL	121	147	162	79
40	139	81	45	122	32	163	146
41	28	82	116	123	58	164	72

(a) See Table A.2 for description of sample locations.

(b) A 250 ml sample was taken during the Homogeneity and Sampling System Test using the concentrated melter feed slurry. Aliquots from this sample were analyzed every 16th sample to determine if long-term analytical effects existed.

TABLE A.9. Sample Analysis Order for the Homogeneity and Sampling System Test Using the Dilute Melter Feed Slurry

Analysis Order	Sample Order ^(a)	Analysis Order	Sample Order	Analysis Order	Sample Order	Analysis Order	Sample Order
1	36	42	61	83	8	124	43
2	71	43	79	84	130	125	90
3	63	44	120	85	82	126	78
4	23	45	107	86	1	127	52
5	144	46	114	87	16	128	CONTROL
6	41	47	140	88	75	129	89
7	77	48	CONTROL	89	125	130	150
8	17	49	91	90	22	131	145
9	118	50	101	91	45	132	129
10	132	51	142	92	127	133	117
11	20	52	112	93	54	134	74
12	70	53	122	94	98	135	76
13	139	54	12	95	60	136	73
14	111	55	95	96	CONTROL	137	92
15	81	56	10	97	47	138	50
16	CONTROL ^(b)	57	151	98	102	139	124
17	103	58	128	99	13	140	93
18	106	59	9	100	18	141	2
19	141	60	97	101	5	142	15
20	115	61	30	102	46	143	86
21	85	62	80	103	67	144	CONTROL
22	3	63	119	104	42	145	49
23	133	64	CONTROL	105	39	146	126
24	65	65	153	106	48	147	11
25	57	66	108	107	40	148	27
26	24	67	110	108	25	149	44
27	37	68	38	109	94	150	6
28	88	69	137	110	149	151	53
29	148	70	87	111	55	152	104
30	68	71	99	112	CONTROL	153	28
31	121	72	154	113	96	154	69
32	CONTROL	73	135	114	14	155	84
33	59	74	113	115	7	156	58
34	72	75	32	116	105	157	33
35	143	76	116	117	19	158	134
36	56	77	83	118	152	159	138
37	131	78	62	119	64	160	CONTROL
38	109	79	100	120	29	161	66
39	31	80	CONTROL	121	136	162	26
40	146	81	34	122	51	163	123
41	147	82	4	123	21	164	35

(a) See Table A.2 for description of sample locations.

(b) A 250 ml sample was taken during the Homogeneity and Sampling System Test using the concentrated melter feed slurry. Aliquots from this sample were analyzed every 16th sample to determine if long-term analytical effects existed.

TABLE A.10. Sample Analysis Order for the Resuspension Tests Using the Concentrated Melter Feed Slurry

Analysis Order	Sample Order ^(a)	Analysis Order	Sample Order	Analysis Order	Sample Order	Analysis Order	Sample Order
1	163	35	221	69	206	103	248
2	217	36	233	70	205	104	263
3	157	37	195	71	237	105	275
4	186	38	160	72	190	106	268
5	177	39	162	73	214	107	261
6	191	40	180	74	189	108	273
7	168	41	172	75	209	109	264
8	211	42	213	76	228	110	254
9	200	43	203	77	170	111	262
10	175	44	234	78	193	112	CONTROL
11	226	45	166	79	236	113	258
12	225	46	196	80	CONTROL	114	246
13	185	47	230	81	201	115	249
14	204	48	CONTROL	82	210	116	271
15	184	49	198	83	158	117	247
16	CONTROL ^(b)	50	238	84	197	118	272
17	183	51	165	85	155	119	259
18	227	52	212	86	229	120	239
19	231	53	199	87	232	121	251
20	164	54	216	88	187	122	260
21	194	55	202	89	169	123	266
22	156	56	176	90	270	124	257
23	192	57	207	91	250	125	274
24	223	58	167	92	245	126	277
25	218	59	179	93	278	127	244
26	235	60	159	94	280	128	CONTROL
27	208	61	224	95	267	129	279
28	215	62	174	96	CONTROL	130	265
29	173	63	219	97	256	131	253
30	181	64	CONTROL	98	240	132	243
31	161	65	171	99	276	133	242
32	CONTROL	66	188	100	252	134	269
33	178	67	220	101	241		
34	222	68	182	102	255		

(a) See Table A.1 for description of sample locations.

(b) A 250 ml sample was taken during the Homogeneity and Sampling System Test using the concentrated melter feed slurry. Aliquots from this sample were analyzed every 16th sample to determine if long-term analytical effects existed.

TABLE A.11. Sample Analysis Order for the Resuspension Test Using the Reference Melter Feed Slurry

<u>Analysis Order</u>	<u>Sample Order^(a)</u>	<u>Analysis Order</u>	<u>Sample Order</u>	<u>Analysis Order</u>	<u>Sample Order</u>	<u>Analysis Order</u>	<u>Sample Order</u>
1	191	35	193	69	195	103	266
2	178	36	214	70	189	104	269
3	197	37	173	71	167	105	273
4	230	38	159	72	235	106	272
5	179	39	203	73	204	107	275
6	169	40	233	74	228	108	249
7	224	41	161	75	198	109	254
8	162	42	200	76	229	110	267
9	211	43	223	77	170	111	258
10	199	44	216	78	165	112	CONTROL
11	212	45	174	79	188	113	253
12	213	46	181	80	CONTROL	114	264
13	185	47	194	81	222	115	261
14	155	48	CONTROL	82	210	116	242
15	221	49	218	83	157	117	260
16	CONTROL ^(b)	50	202	84	208	118	278
17	226	51	207	85	190	119	274
18	176	52	238	86	164	120	245
19	206	53	186	87	175	121	256
20	187	54	156	88	171	122	277
21	163	55	182	89	158	123	262
22	236	56	196	90	279	124	240
23	231	57	220	91	252	125	241
24	160	58	168	92	268	126	265
25	225	59	172	93	255	127	271
26	184	60	192	94	263	128	CONTROL
27	237	61	183	95	247	129	248
28	180	62	217	96	CONTROL	130	259
29	227	63	215	97	276	131	244
30	166	64	CONTROL	98	243	132	280
31	234	65	177	99	246	133	239
32	CONTROL	66	232	100	251	134	250
33	201	67	205	101	257		
34	219	68	209	102	270		

(a) See Table A.2 for description of sample locations.

(b) A 250 ml sample was taken during the Homogeneity and Sampling System Test using the concentrated melter feed slurry. Aliquots from this sample were analyzed every 16th sample to determine if long-term analytical effects existed.

TABLE A.12. Sample Analysis Order for the Resuspension Test Using the Dilute Melter Feed Slurry

Analysis Order	Sample Order ^(a)	Analysis Order	Sample Order	Analysis Order	Sample Order	Analysis Order	Sample Order
1	160	35	173	69	184	103	246
2	185	36	205	70	177	104	259
3	231	37	219	71	175	105	260
4	186	38	200	72	230	106	265
5	206	39	210	73	228	107	277
6	176	40	226	74	216	108	252
7	237	41	221	75	161	109	270
8	178	42	157	76	199	110	278
9	218	43	225	77	203	111	254
10	164	44	194	78	180	112	CONTROL
11	159	45	172	79	189	113	264
12	191	46	215	80	CONTROL	114	242
13	238	47	217	81	202	115	272
14	156	48	CONTROL	82	213	116	266
15	188	49	179	83	162	117	273
16	CONTROL ^(b)	50	190	84	208	118	257
17	212	51	214	85	155	119	243
18	220	52	163	86	169	120	274
19	196	53	233	87	207	121	241
20	223	54	192	88	209	122	240
21	234	55	222	89	235	123	244
22	182	56	187	90	271	124	280
23	165	57	193	91	250	125	258
24	224	58	174	92	269	126	253
25	181	59	229	93	261	127	245
26	227	60	166	94	256	128	CONTROL
27	201	61	168	95	263	129	239
28	195	62	170	96	CONTROL	130	275
29	211	63	204	97	255	131	249
30	197	64	CONTROL	98	248	132	267
31	236	65	158	99	279	133	268
32	CONTROL	66	198	100	262	134	251
33	171	67	232	101	276		
34	167	68	183	102	247		

(a) See Table A.3 for description of sample locations.

(b) A 250 ml sample was taken during the Homogeneity and Sampling System Test using the concentrated melter feed slurry. Aliquots from this sample were analyzed every 16th sample to determine if long-term analytical effects existed.

APPENDIX B

STATISTICAL ANALYSES RESULTS

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Plots of the total solids or elemental weight percents after removing the effects of the factors studied in the experiment (residual plots) should resemble a random band of values when plotted by analytical order. However, as seen in the residual plots shown in Figures B.1 through B.24, there are definite trends across analytical order. These long-term effects account for a sizable portion of the total analytical variation.

Plots of the analytical determinations for the control samples are shown in Figures B.25 through B.31. These plots also reveal the long-term analytical variation. The magnitude of the analytical uncertainty will directly affect the number of analyses that will be required during hot operations. If the analytical uncertainties can be reduced, the required number of feed analyses for characterization and waste form qualification purposes can be reduced. Moreover, if the analytical uncertainty is reduced, the cold chemical addition recipe will be more accurate, thereby improving the ability to produce a glass composition nearer the optimal target composition. The benefits gained by reducing the analytical uncertainty seem to warrant some effort towards such a reduction.

FIGURE B.1. Plot of Residuals Versus Analytical Order for Aluminum (Al) - Homogeneity and Sampling System Test with Concentrated Melter Feed Slurry.

FIGURE B.2. Plot of Residuals Versus Analytical Order for Boron (B) - Homogeneity and Sampling System Test with Concentrated Melter Feed Slurry.

FIGURE B.3. Plot of Residuals Versus Analytical Order for Calcium (Ca) - Homogeneity and Sampling System Test with Concentrated Melter Feed Slurry.

FIGURE B.4. Plot of Residuals Versus Analytical Order for Iron (Fe) - Homogeneity and Sampling System Test with Concentrated Melter Feed Slurry.

FIGURE B.5. Plot of Residuals Versus Analytical Order for Lithium (Li) - Homogeneity and Sampling System Test with Concentrated Melter Feed Slurry.

FIGURE B.6. Plot of Residuals Versus Analytical Order for Sodium (Na) - Homogeneity and Sampling System Test with Concentrated Melter Feed Slurry.

FIGURE B.7. Plot of Residuals Versus Analytical Order for Silica (Si) - Homogeneity and Sampling System Test with Concentrated Melter Feed Slurry.

FIGURE B.8. Plot of Residuals Versus Analytical Order for Aluminum (Al) - Homogeneity and Sampling System Test with Reference Melter Feed Slurry.

FIGURE B.9. Plot of Residuals Versus Analytical Order for Boron (B) - Homogeneity and Sampling System Test with Reference Melter Feed Slurry.

FIGURE B.10. Plot of Residuals Versus Analytical Order for Calcium (Ca) - Homogeneity and Sampling System Test with Reference Melter Feed Slurry.

FIGURE B.11. Plot of Residuals Versus Analytical Order for Iron (Fe) - Homogeneity and Sampling System Test with Reference Melter Feed Slurry.

FIGURE B.12. Plot of Residuals Versus Analytical Order for Lithium (Li) - Homogeneity and Sampling System Test with Reference Melter Feed Slurry.

FIGURE B.13. Plot of Residuals Versus Analytical Order for Sodium (Na) - Homogeneity and Sampling System Test with Reference Melter Feed Slurry.

FIGURE B.14. Plot of Residuals Versus Analytical Order for Silica (Si) - Homogeneity and Sampling System Test with Reference Melter Feed Slurry.

FIGURE B.15. Plot of Residuals Versus Analytical Order for Aluminum (Al) - Homogeneity and Sampling System Test with Dilute Melter Feed Slurry.

FIGURE B.16. Plot of Residuals Versus Analytical Order for Boron (B) - Homogeneity and Sampling System Test with Dilute Melter Feed Slurry.

FIGURE B.17. Plot of Residuals Versus Analytical Order for Calcium (Ca) - Homogeneity and Sampling System Test with Dilute Melter Feed Slurry.

FIGURE B.18. Plot of Residuals Versus Analytical Order for Iron (Fe) - Homogeneity and Sampling System Test with Dilute Melter Feed Slurry.

FIGURE B.19. Plot of Residuals Versus Analytical Order for Lithium (Li)-Homogeneity and Sampling System Test with Dilute Melter Feed Slurry.

FIGURE B.20. Plot of Residuals Versus Analytical Order for Sodium (Na) - Homogeneity and Sampling System Test with Dilute Melter Feed Slurry.

FIGURE B.21. Plot of Residuals Versus Analytical Order for Silica (Si) - Homogeneity and Sampling System Test with Dilute Melter Feed Slurry.

FIGURE B.22. Plot of Residuals Versus Analytical Order for Total Solids - Resuspension Test with Concentrated Melter Feed Slurry.

FIGURE B.23. Plot of Residuals Versus Analytical Order for Total Solids - Resuspension Test with Reference Melter Feed Slurry.

FIGURE B.24. Plot of Residuals Versus Analytical Order for Total Solids - Resuspension Test with Dilute Melter Feed Slurry.

FIGURE B.25. Plot of Normalized Weight Percent Aluminum (Al) Versus Analytical Order for the Control Samples

FIGURE B.26. Plot of Normalized Weight Percent Boron (B) Versus Analytical Order for the Control Samples

FIGURE B.27. Plot of Normalized Weight Percent Calcium (Ca) Versus Analytical Order for the Control Samples

FIGURE B.28. Plot of Normalized Weight Percent Iron (Fe) Versus Analytical Order for the Control Samples

FIGURE B.29. Plot of Normalized Weight Percent Lithium (Li) Versus Analytical Order for the Control Samples

FIGURE B.30. Plot of Normalized Weight Percent Sodium (Na) Versus Analytical Order for the Control Samples

FIGURE B.31. Plot of Normalized Weight Percent Silica (Si) Versus Analytical Order for the Control Samples

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