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# **POTENTIAL IMPACT OF BLENDING RESIDUAL SOLIDS FROM TANKS 18/19 MOUNDS WITH TANK 7 OPERATIONS**

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March 2007

Process Science & Engineering Section  
Savannah River National Laboratory  
Aiken, SC 29808

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Prepared for the U.S. Department of Energy Under Contract Number  
DEAC09-96SR18500



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## EXECUTIVE SUMMARY

High level waste tanks 18F and 19F have residual mounds of waste which may require removal before the tanks can be closed. Conventional slurry pump technology, previously used for waste removal and tank cleaning, has been incapable of removing these mounds from tanks 18F and 19F. A mechanical cleaning method has been identified that is potentially capable of removing and transferring the mound material to tank 7F for incorporation in a sludge batch for eventual disposal in high level waste glass by the Defense Waste Processing Facility. The Savannah River National Laboratory has been requested to evaluate whether the material transferred from tanks 18F/19F by the mechanical cleaning technology can later be suspended in Tank 7F by conventional slurry pumps after mixing with high level waste sludge.

The proposed mechanical cleaning process for removing the waste mounds from tanks 18 and 19 may utilize a high pressure water jet-eductor that creates a vacuum to mobilize solids. The high pressure jet is also used to transport the suspended solids. The jet-eductor system will be mounted on a mechanical crawler for movement around the bottom of tanks 18 and 19.

Based on physical chemical property testing of the jet-eductor system processed IE-95 zeolite and size-reduced IE-95 zeolite, the following conclusions were made:

- The jet-eductor system processed zeolite has a mean and median particle size (volume basis) of 115.4 and 43.3 microns in water. Preferential settling of these large particles is likely.
- The jet-eductor system processed zeolite rapidly generates settled solid yield stresses in excess of 11,000 Pascals in caustic supernates and will not be easily retrieved from Tank 7 with the existing slurry pump technology.
- Settled size-reduced IE-95 zeolite (less than 38 microns) in caustic supernate does not generate yield stresses in excess of 600 Pascals in less than 30 days.
- Preferential settling of size-reduced zeolite is a function of the amount of sludge and the level of dilution for the mixture.
- Blending the size-reduced zeolite into larger quantities of sludge can reduce the amount of preferential settling.
- Periodic dilution or resuspension due to sludge washing or other mixing requirements will increase the chances of preferential settling of the zeolite solids.
- Mixtures of Purex sludge and size-reduced zeolite did not produce yield stresses greater than 200 Pascals for settling times less than thirty days. Most of the sludge-zeolite blends did not exceed 50 Pascals. These mixtures should be removable by current pump technology if sufficient velocities can be obtained.
- The settling rate of the sludge-zeolite mixtures is a function of the ionic strength (or supernate density) and the zeolite- sludge mixing ratio.
- Simulant tests indicate that leaching of Si may be an issue for the processed Tank 19 mound material.
- Floating zeolite fines observed in water for the jet-eductor system and size-reduced zeolite were not observed when the size-reduced zeolite was blended with caustic solutions, indicating that the caustic solutions cause the fines to agglomerate.

Research on the physical properties and rheological properties of various sized zeolite particles in caustic supernate and Purex sludge simulants has generated the following recommendations:

- The Tank 18/19 mechanical cleaning program should pursue the addition of a technology that size reduces the Tank 18/19 mound material to less than 38 microns with a mean particle size distribution between 5 and 20 microns. This size reduction should be applied before or during transfer of the Tank 18/19 mound material to Tank 7.
- Vendor or site testing of the size reduction technology should include characterization for particle size distribution, settled solids yield stress and preferential settling.
- Pilot testing of the size reducing technology combined with the mechanical transport technology is recommended prior to deployment.
- Actual waste tests on Tank 19 mound material is recommended to verify that size reduction is beneficial to slurry properties as shown with the current simulant tests. Based on the current tests, the amount of Tank 19 mound material required to perform confirmatory settled solids yield stress, particle size and preferential settling test is at least 500 mL.
- Actual waste tests with the Tank 19 mound material should also examine the possibility of Si leaching from the size-reduced mound material for use in predicting the impact on disposition of the residual supernate.
- The impact of the elevated tank temperatures on the physical properties or the leaching of the settled zeolite solids should be determined. Past experience with measurement of yield stresses for actively mixed sludges at temperatures up to 50 °C has not shown much dependence of the yield stress on temperature. Settled solids yield stress has not been tested for temperature dependence (especially in the presence of a possible leaching process) in past simulant studies.

Based on the test programs described in this report, the potential for successfully removing Tank 18/19 mound material from Tank 7 with the current slurry pump technology requires the reduction of the particle size of the Tank 18/19 mound material.

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## LIST OF ACRONYMS

ACTL	Aiken County Technology Laboratory
AD	Analytical Development
cP	Centipoise
CSSA	Calculated Specific Surface Area
DWPF	Defense Waste Processing Facility
Hr	Hour
IC	Ion Chromatography
ICPES	Inductively Coupled Plasma Emission Spectrophotometer
IS	Insoluble Solids
L	Liters
M	Moles/Liter
mL	Milliliter
mm	Millimeter
NIST	National Institute of Standards and Technology
Pa	Pascals
PSD	Particle Size Distribution
SRNL	Savannah River National Laboratory
SSYS	Settled Solids Yield Stress
SRZ	Size-Reduced Zeolite

## 1.0 INTRODUCTION AND BACKGROUND

High level waste tanks 18F and 19F have residual mounds of waste which may require removal before the tanks can be closed. Conventional slurry pump technology, previously used for waste removal and tank cleaning, has been incapable of removing these mounds from tanks 18F and 19F. A mechanical cleaning method has been identified that may be capable of removing and transferring the mound material to tank 7F for incorporation in a sludge batch for eventual disposal in high level waste glass by the Defense Waste Processing Facility. The Savannah River National Laboratory has been requested to evaluate whether the material transferred from tanks 18F/19F by the mechanical cleaning technology can later be suspended in Tank 7F by conventional slurry pumps after mixing with high level waste sludge.

The primary constituent of the mounds in tanks 18/19 was the zeolite used in the cesium removal columns for treating the Tank Farm evaporator overheads prior to release to the seepage basins. Linde AW-500 zeolite (currently named IE-95 zeolite) was the primary zeolite used for cesium removal prior to Tank 19 exit from active service.<sup>1</sup> The active zeolite species for cesium removal was chabazite,  $(\text{Na}_{1.0}\text{Ca}_{1.5})[\text{Al}_4\text{Si}_8\text{O}_{24}]12\text{H}_2\text{O}$ , a calcium aluminosilicate capable of ion exchange with high selectivity for cesium.<sup>2</sup> Current estimates for the amount of material remaining in tanks 18/19 are 4300 gallons and 15000 gallons respectively.<sup>3,4</sup>

The proposed mechanical cleaning process for removing the waste mounds from tanks 18 and 19 may utilize a high pressure water jet-eductor that creates a vacuum to mobilize solids. The high pressure jet is also used to transport the suspended solids. The jet-eductor system will be mounted on a mechanical crawler for movement around the bottom of tanks 18 and 19.

Evaluation techniques used rheology measurements for fluid and settled solids properties, particle size measurement, and settling tests.

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## 2.0 APPROACH

### 2.1 Physical Property Methods Utilized

The physical properties measured during this project include weight percent solids (total solids and total dissolved solids in the supernate), slurry density, slurry rheology, settling tests and particle size distribution. The methods used to measure each of these properties are described below.

#### 2.1.1 Weight Percent Solids and Density Measurements

The weight percent solids were determined using a Mettler Toledo HR73P Halogen Moisture Analyzer. The HR73P is programmed to heat the sample to 105 °C and monitor the mass of the sample until the change in mass is less than or equal to 1 mg over a period of 130 seconds. The homogenous sample (slurry or liquid) is placed on a glass fiber pad and the pad placed in the HR73P. The HR73P weighs the initial mass of the sample, referred to as the total mass ( $m_{tt}$ ). The sample is then heated by the infrared radiation from a Halogen lamp to 105 °C (controlled by a thermocouple) to drive off all the water (assuming mass loss is only from water) and the resulting remaining mass is the total solids ( $m_{ts}$ ) in the sample. The weight percent (wt %) total solids (TS) of the sludge was determined using equation [1].

$$wt\%_{ts} = \frac{m_{ts}}{m_{tt}} \times 100\% \quad [1.]$$

A sample of the slurry is centrifuged (at 4332 gravities) to obtain the supernate. The resulting supernate is then processed through a 0.45  $\mu\text{m}$  filter. A sample of the filtered supernate is then placed on a glass fiber pad, placed in the HR73P, and weighed. The mass of sample used is the total mass of the supernate ( $m_{st}$ ). The sample is then heated by the Halogen lamp to 105 °C to drive off all the water and the resulting remaining mass is the total dissolved solids ( $m_{ds}$ ) in the supernate. The weight percent of total dissolved solids (DS) in the supernate is determined using equation [2]. This analysis assumes that all the solids in the resulting supernate are dissolved.

$$wt\%_{ds} = \frac{m_{ds}}{m_{st}} \times 100\% \quad [2.]$$

The weight percent of insoluble solids (IS) and soluble solids (SS) of the slurry are then calculated by the following conservation of mass relationships, equations [3] and [4] respectively.

$$wt\%_{is} = \frac{wt\%_{ts} - wt\%_{ds}}{100\% - wt\%_{ds}} \times 100\% \quad [3.]$$

$$wt\%_{ss} = wt\%_{ts} - wt\%_{is} \quad [4.]$$

Densities for non-settling or slowly settling fluids were determined using an Anton Paar DMA 4500 density meter. The density meter determines the density of a sample by measuring the resonant frequency of a sample-filled U tube at a specified temperature. The method used for determining the density of fine particulate material can be found in the section on the properties of the zeolite particles (section 3.1.1).

### 2.1.2 Rheology Measurements

Slurry rheology measurements were performed using a Haake RS600 rheometer at 25 °C. The rheometer uses a Searle type measuring system, where both speed and torque are measured at the rotating shaft. The rheometer was operated in the controlled rate mode for all of the data reported in this report. The primary measuring geometry used was the vane method for settled solid yield stress measurements while flow curve measurements were made using the cone and plate (60 mm Ti/2 degree) geometry. The RS600 was functionally verified using the C60/0.5 degree cone/plate geometry with a NIST traceable Newtonian oil standard on a daily basis.

Vanes have been used to measure the yield stress of non-Newtonian fluids as shown in Figure 2-1.<sup>5-18</sup> The vane is inserted into the fluid and rotated at a very slow speed. The surface area used to determine the shear stress is the surface area produced by the vane, which is cylindrical. It has been shown that this is a good assumption for determining the yield stress of the fluid as the vane slowly rotates through it.<sup>6,7,10,11</sup> The derived equation (5) assumes the stress is constant on all surfaces. The shearing due to the immersed section of the vane shaft, stress contribution of the immersed section of the shaft, and the wall effects are negligible when meeting the criteria as shown in Figure 2-1. The length of immersed shaft will need to be considered if its length starts to impact the measured stress. The exclusion of the shear stress contribution of the immersed shaft length over-estimates the shear stress.

$$\tau = \frac{\Gamma}{\frac{\pi \cdot D^3}{2} \left( \frac{H}{D} + \frac{1}{3} \right)} = A \cdot \Gamma \quad (5.)$$

Where  $\Gamma$  = measured torque (N-m or % torque)

$D$  = diameter of vane (m)

$H$  = height of vane (m)

$A$  = geometric constant (m<sup>3</sup> or Pa/% $\Gamma$ )

$\tau$  = shear stress (Pa)

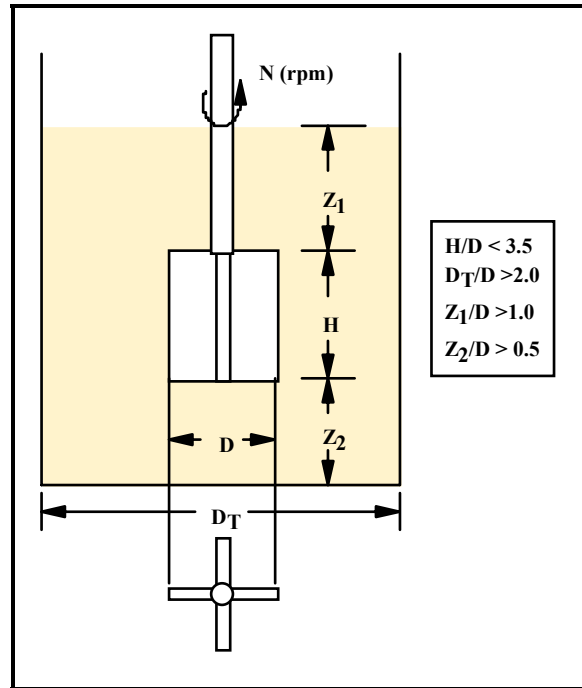


Figure 2-1 Vane Geometric Requirements

A typical stress versus time (or displacement) curve is shown in Figure 2-2. The initial response for a non-Newtonian fluid having a yield stress is typically linear and this slope is called the Hookean elastic modulus ( $G$ ). The point of departure from this linear region is called the static yield stress when the fluid starts to transition from a fully elastic to viscoelastic behavior.<sup>5</sup> At the maximum stress, the behavior of the material transitions between viscoelastic and fully viscous and is called the yield stress (also known as the dynamic yield stress).

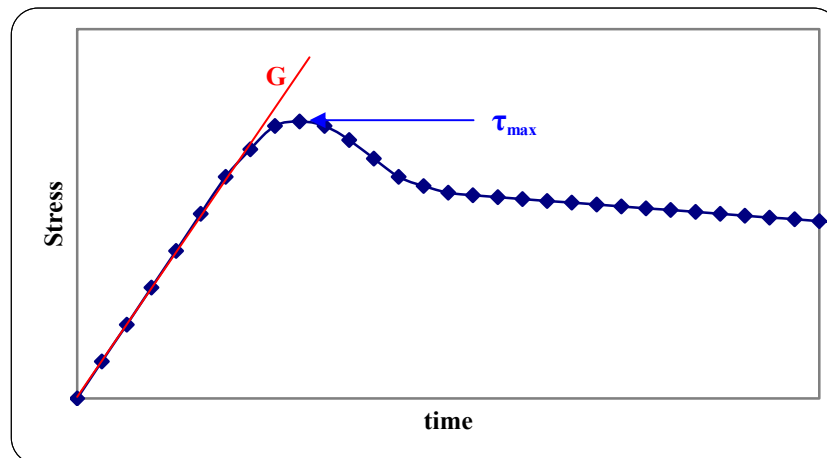


Figure 2-2 Typical Vane Torque versus Time/Displacement Curve

The vane used for this task was the Haake FL22 vane. The vane dimensions used in this task were  $D = 22$  mm and  $H = 16$  mm. The A factor for this vane was calculated and used in the RS600 to calculate the

shear stress from the measured torque, given the measured torque is in N-m. The A factor used is shown in equation 6.

$$A = \frac{A}{N \cdot m} = \frac{2}{\pi \cdot (0.022m)^3 \cdot \left( \left( \frac{16}{22} \right) + \frac{1}{3} \right)} \cdot \frac{N \cdot m}{N \cdot m} \cdot \frac{Pa}{\frac{N}{m^2}} = 56370 \frac{Pa}{N \cdot m} \quad (6.)$$

The M factor for this vane was set at 1.0 sec<sup>-1</sup>/(rad·s<sup>-1</sup>). Going through the same exercise as that shown in equation 3.4, for a rotational speed of 0.3 RPM, the controlled shear rate was 0.03 sec<sup>-1</sup>. The rotational speed was also visually verified at approximately 0.3 revolutions per minute (RPM).

Flow curves were obtained on the RS600 using the cone and plate (60 mm Ti/2 degree) geometry by linearly varying the shear rate from 0 to 600 seconds<sup>-1</sup> over a given time period. The program details for the flow curves are listed in Table 2-1. The measured shear stresses for the up and down flow curves were fitted to the Bingham Plastic rheology model (equation 7) over the shear rate range of 50 to 600 seconds<sup>-1</sup>.

$$\tau = \tau_0 + \eta_0 \dot{\gamma} \quad [7.]$$

$\tau$  = shear stress, Pa

$\tau_0$  = Bingham Yield Stress, Pa

$\dot{\gamma}$  = Shear rate, 1/seconds

$\eta_0$  = Bingham consistency, mPa.sec (or cP)

The upper limit for the fitted shear rate region was adjusted to a lower value of shear rate when necessary to avoid nonlaminar flow conditions.

**Table 2-1 Cone and Plate Geometry Rheology Program**

Program Section	Linear Shear rate, seconds <sup>-1</sup>	Time, minutes
Up Curve	0 to 600	5
Hold Period	600	1
Down Curve	600 to 0	5

### 2.1.3 Particle Size Measurements

Particle size distribution (PSD) was obtained by submitting samples to Analytical Development for analysis. Samples were analyzed with a Microtrac S3000 Tri-laser Particle Size Analyzer. The Microtrac S3000 particle size analyzer measures the particle diameters by measuring the scattered light from a laser beam projected through a stream of the fluid carrying the diluted sample particles. The amount and direction of the light scattered by the particles is measured by an optical detector array and then analyzed to determine the size distribution of the particles. The Microtrac S3000 measuring range is 0.021 to 1408 μm. It should be noted that this instrument has sonication (ultrasonic energy that breaks up agglomerations) capability; hence, PSD measurements were taken with sonication. The Microtrac S3000 stores the recorded data into volume bins, where a bin is defined by an upper and lower diameter. This volume data is then normalized to 100% after the measurement is complete. For the Microtrac S3000,



there are 64 bins covering the measuring range. The number distribution is determined from equation (8). The Microtrac S3000 software then calculates the mean number, mean surface area, mean volume, number distribution and volume distribution data for the standards and samples. The mean volume, mean surface area and mean number are determined using equation (9).

$$\left[ \begin{array}{l} n_i = \frac{6 \cdot V_i}{\pi \cdot d_i^3} \\ N_i = \frac{n_i}{\sum n_i} \times 100\% \end{array} \right] \quad (8.)$$

$$\left[ \begin{array}{l} mv(\text{mean volume}) = \frac{\sum V_i d_i}{\sum V_i} \\ ma(\text{mean surface area}) = \frac{\sum V_i}{\sum (V_i/d_i)} \\ mn(\text{mean number}) = \frac{\sum (V_i/d_i^2)}{\sum (V_i/d_i^3)} \end{array} \right] \quad (9.)$$

where:  $V_i$  = volume % in the  $i^{\text{th}}$  bin  
 $d_i$  = average bin diameter  
 $n_i$  = number of particles in the  $i^{\text{th}}$  bin, given volume %  
 $N_i$  = number % in the  $i^{\text{th}}$  bin (data normalized)

It should be noted that the mean for a volume distribution is weighted toward the larger particles while the mean for the number distribution is weighted toward the smallest particles. The calculated specific surface area in  $\text{meters}^2/\text{cm}^3$  is based on an assumption of smooth, solid spherical particles and does not reflect porosity or topology of the particles. The Microtrac S3000 was functionally checked at the beginning and at the end of the PSD measurements using NIST traceable particle size standards from Duke Scientific Corporation.

Additional particle size information was obtained for dry solids by using standard sieves and a sonic sifter to separate solids into portions that could be weighed after sizing. Optical microscopy was also used to examine the morphology of the size-reduced zeolite particles.

### 2.1.4 Settling Tests

Settling tests were conducted in polymethylpentene (PMP) graduated cylinders which provided high transparency and good stability without leaching Silicon into caustic solutions. All of the tests in the Tank 7 supernate simulant used 500 mL PMP cylinders (48 mm inner diameter). Settling was monitored by measuring the position of the solid-liquid interface as a function of time as shown in Figure 2-3. Note that the position of the solid-liquid interface does not provide any information about the settling rate of the heavier particles and are in fact the slowest particles given the settling is unhindered. These heavier particles may have settled much faster than the liquid-solid interface.



**Figure 2-3 Size-Reduced Zeolite in 7 M Na Supernate Settling Test**

Preferential settling was determined by characterizing the composition of the settled bed using chemical analysis. To prevent disturbance of the settled bed, the free liquid was decanted from the graduated cylinder by carefully pumping the liquid to a second container. The settled bed of solids in the cylinder was then frozen at  $-82\text{ }^{\circ}\text{C}$  in an ultra-low temperature chest freezer (Thermo Forma -86C ULT Freezer). The frozen graduated cylinder was then sectioned with a band saw and the sectioned pieces identified based on the position within the settled bed of solids (Top, middle, bottom). Each frozen section was then rinsed with deionized water to remove the plastic residue from the sectioning process and examined for obvious physical evidence of zeolite. The frozen sections were then thawed and sampled for analysis. The selection of the PMP plastic graduated cylinders helped prevent contamination of the samples with either metals or glass (silicon) had the settling tests been performed in glass or metal containers.

## **2.2 Chemical Analysis Methods**

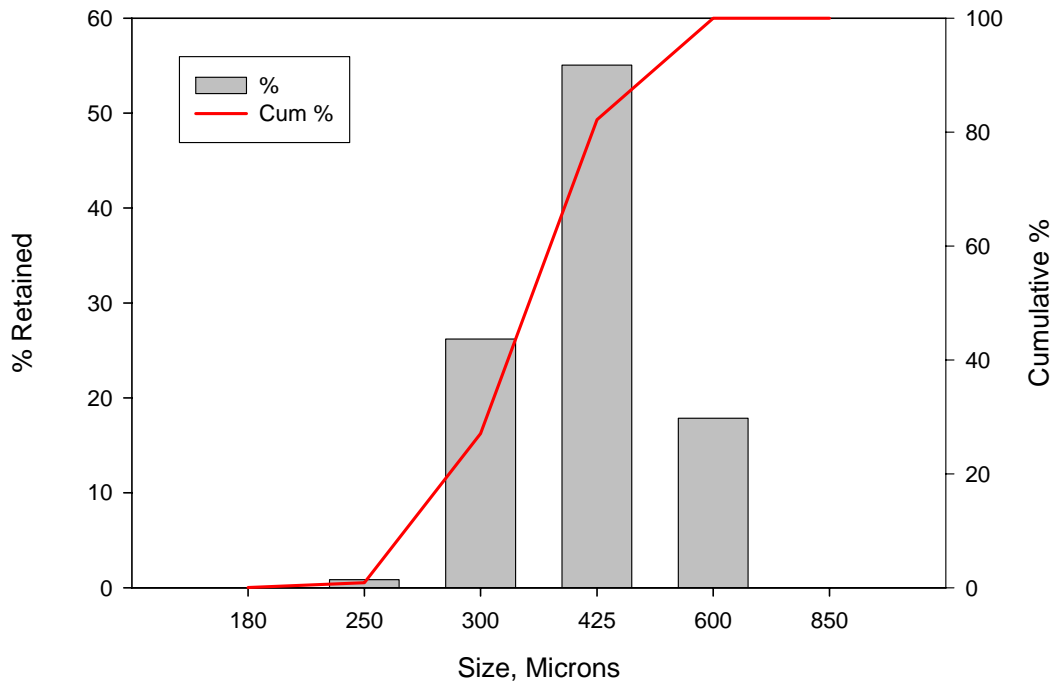
Chemical analysis methods used in studying the interactions between zeolite, Purex sludge and caustic supernates included measurements of elemental species by inductively coupled plasma emission spectrophotometry (ICPES) and ion chromatography (IC). Liquid samples were filtered through a 0.45 micron filter, diluted and analyzed. Solids were prepared for ICPES analysis by a lithium borate/lithium nitrate fusion to insure complete dissolution of the aluminosilicates

### 3.0 RESULTS

#### 3.1 Materials Tested

##### 3.1.1 Zeolite

The zeolite used in testing of the jet-eductor system mechanical cleaning system and in testing the physical properties of zeolite-sludge simulant mixtures is UOP Ionsiv IE-95 20x50 ion exchanger produced by UOP LLC. The zeolite is sold as a smaller than 20 mesh, larger than 50 mesh dry solid. Sizing the dry zeolite by sieving gave an average particle size of 420 microns and a particle size distribution (PSD) as shown in Figure 3-1.



**Figure 3-1 IE-95 Ionsiv Particle Size from Dry Sieving**

A sample of the as-received zeolite added to water and analyzed by the Microtrac gave an average particle size based on volume of 390.5 microns and a median value of 442.3 microns (see Appendix A, ).

The density of the as-received zeolite was calculated using the following procedure, but obtained from two different samples. One sample was the high sheared zeolite prepared by shearing the zeolite in water with a Silverson High Shear Mixer. The other sample was the as-received zeolite added to deionized water. The procedure is to place the solids in water in a graduated cylinder and let the particles saturate overnight. The graduated cylinder is tapped to compact the solids, the standing liquid removed, the mass and volume recorded, and the sample placed in an oven over a weekend at 105°C to evaporate the water and finally weighed. The method for calculating the density of the solids is from equation [10].

$$\frac{1}{\rho_{slurry}} = \frac{wt\%_{TS}}{100\% \cdot \rho_s} + \frac{100\% - wt\%_{TS}}{100\% \cdot \rho_{H2O}} \quad [10.]$$

where  $\rho_{\text{slurry}}$  = density of the slurry (g/ml)  
 $\rho_s$  = density of zeolite (g/ml)  
 $\rho_{\text{H}_2\text{O}}$  = density of water (g/ml)  
 $\text{wt}\%_{\text{TS}}$  = weight percent total solids - zeolite (%)

The density of the zeolite is 2.38 and 2.32 g/ml using the high sheared and as-received materials respectively. The 2.38 g/ml may be more representative, since there was much less potential for air to be trapped in the high sheared solids compared to the larger, higher porosity, as-received zeolite solids. This density is in relatively good agreement with a previous measurement for IE-95 using air pycnometry that yielded a density of 2.28 g/cm<sup>3</sup>.<sup>2</sup>

The IE-95 zeolite was analyzed by ICPES to obtain the concentrations of the major significant species necessary for potential simulant blend calculations. The results of the ICPES analysis expressed as weight % species in the solids are shown in Table 3-1.

**Table 3-1 ICPES Characterization Results for IE-95 Zeolite**

Element	Sample 1, wt%	Sample 2, wt%	Average, wt%
Al	8.54	8.76	8.65
Fe	1.99	2.00	2.00
K	1.16	1.22	1.19
Mg	0.559	0.579	0.569
Na	3.59	3.58	3.59
Si	26.1	26.8	26.5

The ratio of the amount of aluminum to silicon was calculated after converting each of the results to moles based on 100 grams of zeolite solids. The ratio is one atom of aluminum per 2.94 atoms of silicon. For chabazite, the active zeolite in IE-95, the ratio is one atom of aluminum per two atoms of silicon. The measured ratio suggests that the clay binder is high in silicon and low in aluminum in good agreement with earlier reports on the composition of the IE-95 zeolite.<sup>2</sup>

### 3.1.2 Purex Sludge Simulant

The sludge in the high level waste tanks in F area is primarily Purex sludge which is characterized by high levels of iron and uranium and a relatively small amount of aluminum. Simulated Purex sludge used in this study was obtained from an existing supply of sludge simulant. The sludge was analyzed for metals by ICPES to confirm that it would be appropriate for representing the sludge waste from the F are tanks. The characterization results are shown in Table 3-2 and they show appropriate levels of Fe, Al, Mn, and Ni in the sludge. Since the simulant is a nonradioactive simulant, uranium is not included in the mixture.

**Table 3-2 ICPES Characterization Results for Purex Sludge Simulant**

<b>Element</b>	<b>Wt% Solids</b>
<b>Al</b>	4.91
<b>Ba</b>	0.25
<b>Ca</b>	3.08
<b>Cd</b>	<0.01
<b>Ce</b>	<0.01
<b>Cr</b>	0.26
<b>Cu</b>	0.16
<b>Fe</b>	26.55
<b>K</b>	0.39
<b>La</b>	<0.1
<b>Mg</b>	0.24
<b>Mn</b>	5.6
<b>Na</b>	3.18
<b>Ni</b>	2.77
<b>P</b>	0.02
<b>S</b>	0.18
<b>Si</b>	1.62
<b>Ti</b>	0.01
<b>Zn</b>	0.30
<b>Zr</b>	2.91

The soluble anions were also confirmed to be sufficiently low to allow adjustment of the sludge supernate to represent various Tank 7 operating conditions. The supernate compositions are discussed in the next section.

The rheology of the Purex sludge simulant was measured to confirm that the simulant had the appropriate non-Newtonian rheological properties. The sludge was combined with a Tank 7 supernate simulant at 3.06 M Na to generate a nominal Tank 7 sludge simulant. The sludge simulant was 27.87 wt % total solids and 13.43 wt% insoluble solids at a density of 1.266 g/mL. The flow curve showed the expected non-Newtonian properties and was fit to the Bingham Plastic rheology model to obtain a yield stress of 5.4 Pascals based on a fit from 50 to 600 seconds<sup>-1</sup>. The Tank 7 Sludge simulant flow curve is shown in Figure 3-2. A portion of the sludge simulant was allowed to settle and was measured by the vane method for the settled solids yield stress (SSYS) over a two week period at two different depths. As shown in Figure 3-3, the SSYS varied from 4 to 10 Pascals for the shallow measurement and from 5 to 11 Pascals for the deeper measurement. The relatively small difference between the shallow and deeper measurements indicates that the settled bed of solids was relatively homogeneous. The small increase in SSYS over time was also consistent with prior SRNL experience with sludge simulants. There is no radioactive data for comparing SSYS.

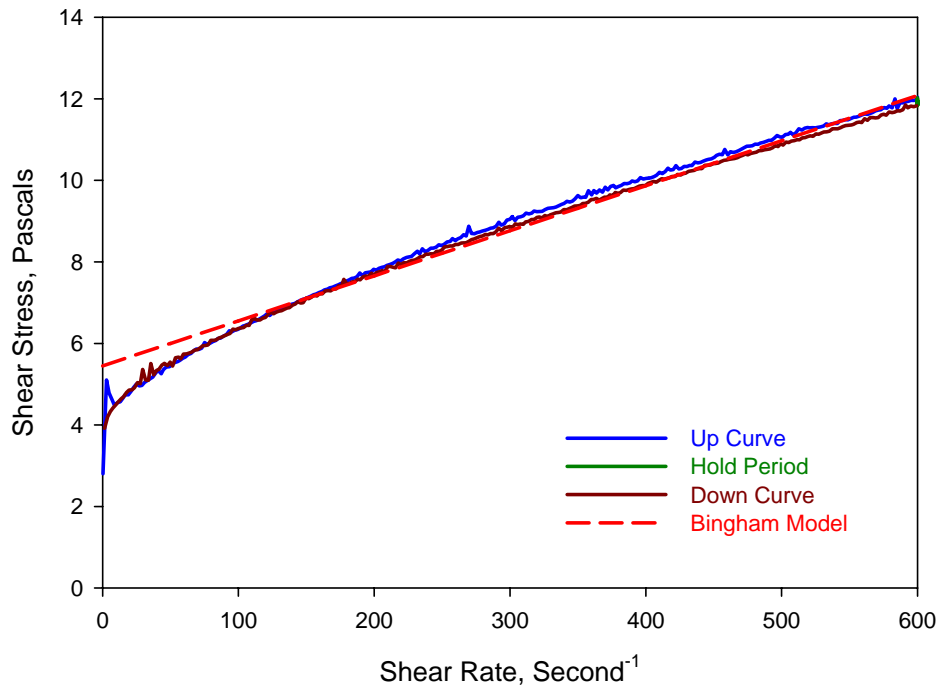


Figure 3-2 Flow Curve for the Purex Sludge Simulant in Tank 7 Supernate at 3.06 M Na

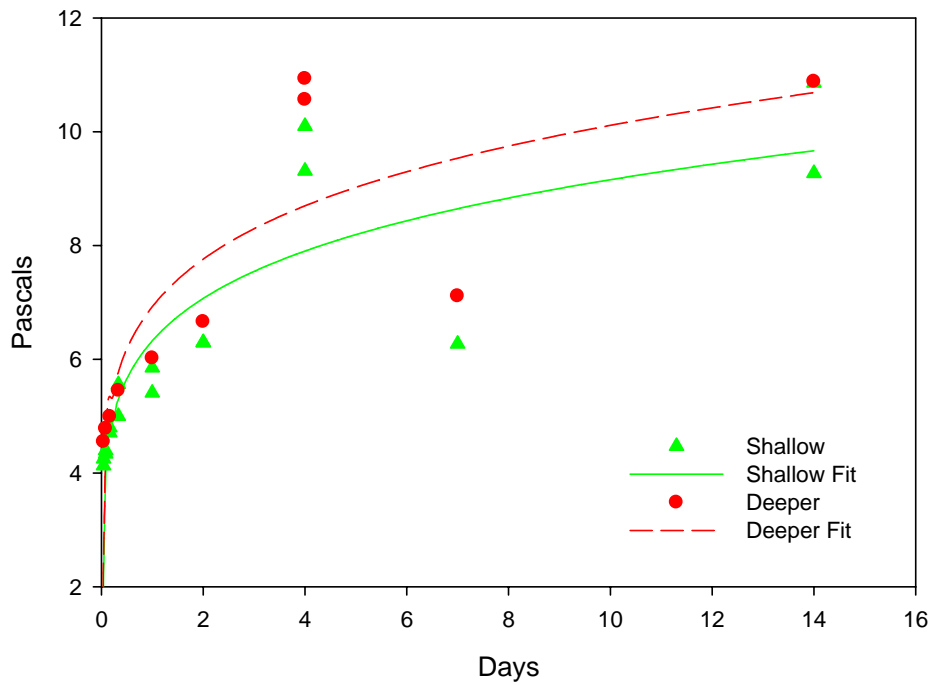


Figure 3-3 Settled Solids Yield Stress Results for Purex Sludge Simulant in Tank 7 Supernate at 3.06 M Na

### 3.1.3 Tank 7 Supernate Simulant

Three compositions of Tank 7 supernate were prepared for this study based upon information received from the SRS Planning Integration and Technology organization (Case 15c 10-07-06). Initial testing was based on a specific composition for the Tank 7 supernate as shown in column one of Table 3-3. Operation of Tank 7 for preparing sludge for feed to the DWPF involves mixing sludge and supernate from various tanks and then washing the sludge to an acceptable anion composition for vitrification. Therefore, the range of supernate exposure for the mound material is based on the possible range between un washed and washed supernate compositions. The maximum supernate composition was chosen as 7 molar in Na and the minimum was chosen as one molar in Na. Table 3-3 details the supernate composition for these two bounding cases.

**Table 3-3 Tank 7 Supernate Simulant Compositions**

Species	3.06 Molar Na Supernate Moles/Liter	One Molar Na Supernate, Moles/Liter	Seven Molar Na Supernate, Moles/Liter
<b>Sodium</b>	3.063	1.00	7.00
<b>Nitrite</b>	0.599	0.43	1.68
<b>Nitrate</b>	0.356	0.21	1.89
<b>Hydroxide</b>	1.074	0.18	1.70
<b>Chloride</b>	0.005	0.00	0.0032
<b>Sulfate</b>	0.020	0.028	0.25
<b>Fluoride</b>	0.008	0.0010	0.0064
<b>Carbonate</b>	0.446	0.053	0.528
<b>Aluminate</b>	0.069	0.019	0.17
<b>Oxalate</b>	0.009	0.0010	0.0074
<b>Phosphate</b>	0.008	0.0010	0.0064
<b>Potassium</b>	0.020	0.0069	0.059
<b>Density, g/mL</b>	1.14	1.04	1.31

## 3.2 Jet-Eductor System Processed Zeolite Tests

### 3.2.1 Preparation and Particle Size

The initial work on this study focused on whether a suitable process simulant could be produced that would match the jet-eductor system processed zeolite and the actual Tank 19 mound material. The raw IE-95 zeolite particle size is compared to the actual Tank 19 mound sample particle size in Table 3-4.<sup>2</sup> The mean particle sizes for the zeolite solids appear to be similar. Chemical composition of the Tank 19 Mound material suggests the presence of some sludge and an additional source of aluminum in the solids despite the physical appearance of the solids (granular, not sludge-like).<sup>2,19</sup> Attempts to incorporate the sludge simulant and some aluminum as alumina did not produce a granular material. The IE-95 zeolite plus water did produce granular solids which resembled the actual waste material, see Figure 3-4. Note that the actual mound material picture was taken through the Shielded Cells window with window tint removed which may have altered the picture color for the solids. Therefore, the Tank 19 mound material was chosen to be represented by only the zeolite.



Figure 3-4 Actual Tank 19 Mound Sample Compared to IE-95 Zeolite + Water

Tests were also performed to determine if the application of a period of high shear mixing could produce a zeolite particle size that reproduces the particle sizes from the jet-eductor system process. The jet-eductor system processed zeolite does show some reduction in particle size due to the generation of lots of small particles as evidenced by the change in the mean particle size from 390.5 to 115.4 microns shown in Table 3-4. The actual particle size distribution (PSD) for the jet-eductor system processed zeolite shows a strong bimodal distribution with some large (less damaged) particles remaining (see Figure A- 3 in Appendix A). The high sheared zeolite is much smaller (5.3 microns, for PSD see Figure A- 4 in Appendix A) and was observed to be much more fluid upon settling. Considerable effort and time would have been required to match the PSD for the jet-eductor system by use of a high shear mixer. Therefore, sufficient jet-eductor system processed zeolite was obtained for the planned settled solids yield stress and settling rate tests.

Table 3-4 Particle Size Results for the Zeolite Simulants Compared to Actual Tank 19 Material

Material Description	Size Range		Volume Basis	
	Min	Max	Mean	Median
	µm	µm	µm	µm
As-Received Zeolite in Water	0.486	1408.0	390.5	442.3
Tank 19 From WSRC-TR-2002-00288	0.972	704.0	541.0	292.0
Processed jet-eductor system Zeolite in Water	0.972	704.0	115.4	43.3
High Sheared Zeolite Using High Shear Mixer in Water	0.409	37.0	5.3	4.2

### 3.2.2 Settled Solids Yield Stress Results

The initial tests conducted with the jet-eductor system processed zeolite were settled solids yield stress (SSYS) tests with water and caustic supernates. These tests bounded the settling of zeolite and sludge where the zeolite settles through the sludge to form mounds as observed in Tank 19. Figure 3-5 shows the SSYS results as a function of time and depth for water and for a Tank 19 supernate composition. The dependence on depth shows that the settled bed of solids is not very homogenous and that the position of measurement has a significant impact on the result. The high magnitude of the SSYS for all of the measurements indicate that standard slurry pumps would have difficulty suspending and removing the settled bed from a waste tank without obstructions. For Tank 7, the many obstructions would definitely preclude effective removal of the settled processed zeolite. Note that the SSYS increased extremely



rapidly for the jet-eductor system processed zeolite in a caustic supernate. For example, the value plotted at 4 days is actually a greater than result since the sample exceeded the maximum range for the Haake RS600 rheometer and the vane that was used.

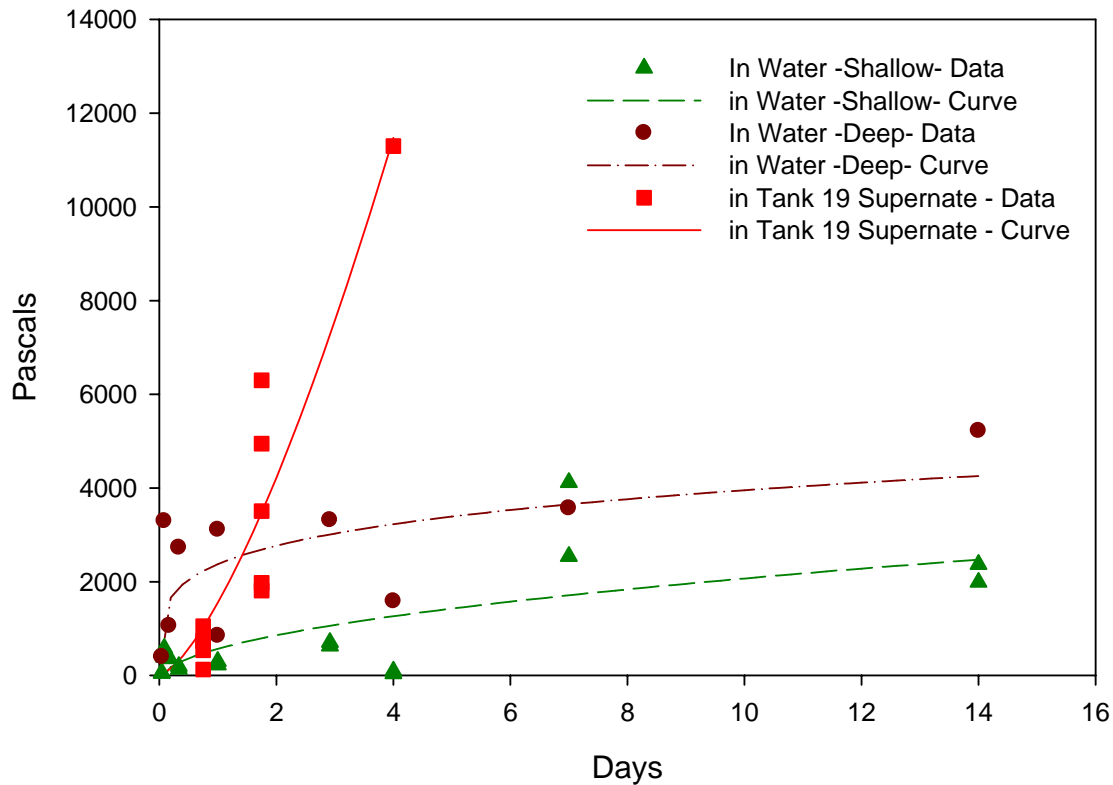


Figure 3-5 Settled Solids Yield Stress Results for Jet-Eductor System Processed Zeolite

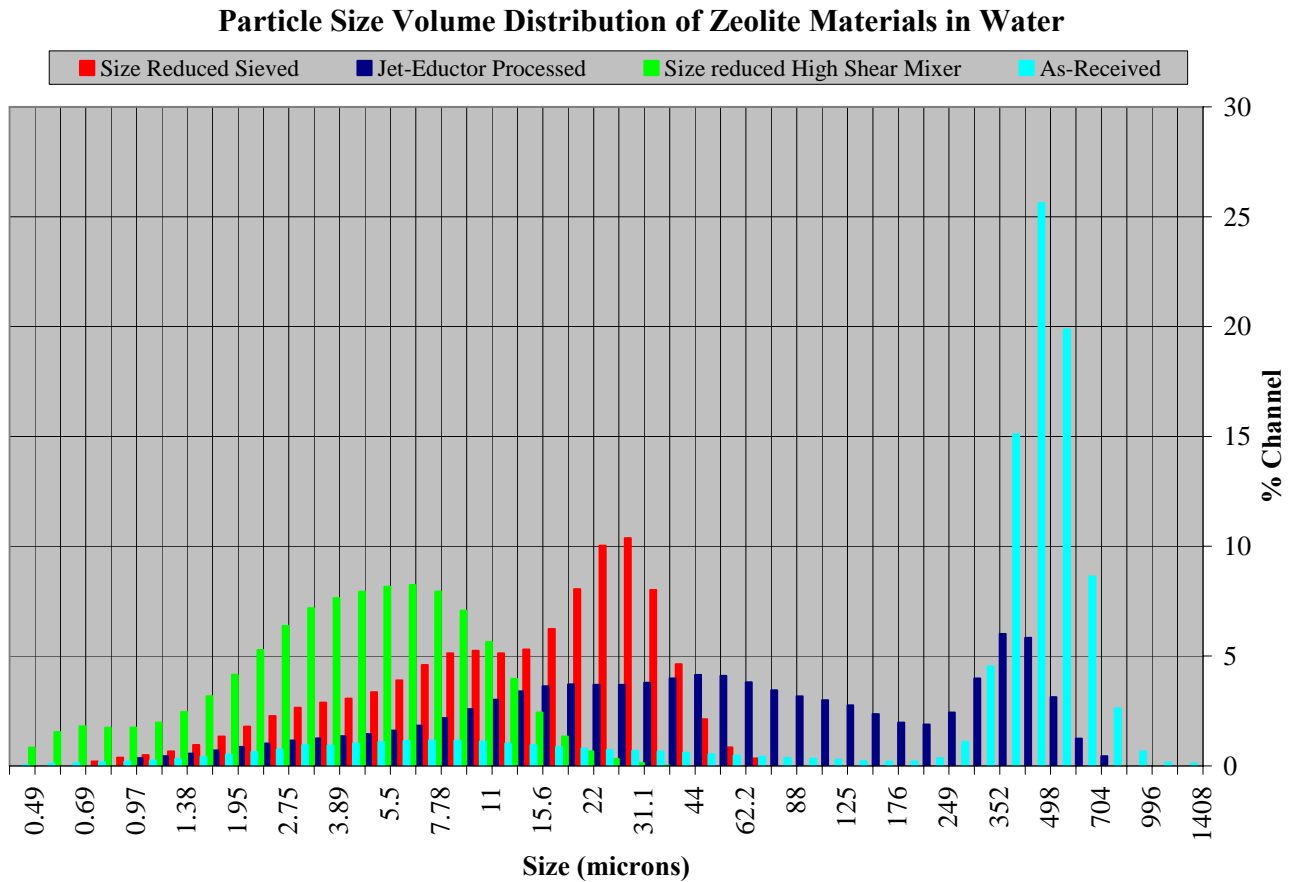
Mixtures of the jet-eductor system processed zeolite with the Purex sludge simulant also showed preferential settling of the larger zeolite particles suggesting that the settled solids results shown above probably apply to the jet-eductor system material combined with Purex sludge simulant. Since the high shear mixed zeolite (with the much smaller particle size) gave a more fluid bed, the remainder of the testing focused on whether size-reduced zeolite would be acceptable in Tank 7.

### 3.3 Size-Reduced Zeolite Testing

#### 3.3.1 Preparation and Test Mixtures

A small batch of UOP IONSIV IE-95 zeolite was charged to an alumina fortified porcelain jar with grinding media and placed on a U.S. Stoneware 755RMV jar mill and ground for approximately 30 minutes per pass. The processed zeolite was transferred to a Fritsch Analysette 3 PRO vibratory sieve shaker where it passed through a series of ASTM E-11 specification mesh screens from Fisher Scientific Company to remove larger particles. The smallest mesh size screen used was 400 mesh, which corresponds to particle size of less than 38 microns. All particles that passed through the 400 mesh screen were isolated and set aside. Particles caught on larger screens were milled and sieved again with additional raw zeolite until about 1.3 kg of sub-38 micron particles were collected. A comparison of the

PSD (volume basis) for the size-reduced zeolite to the other tested forms of zeolite is shown in Figure 3-6. All of the remaining tests utilized the size-reduced zeolite.



**Figure 3-6 Particle Size Distribution (Volume Basis) for Zeolite Materials in Water**

As mentioned previously, operation of Tank 7 involves several factors. The tank is used as sludge blending tank for F-Area sludges from tanks 1-8. This can lead to high levels of insoluble solids and concentrated supernates. Sludge washing produces less concentrated supernates and much less concentrated insoluble solids loadings prior to settling. The production of hydrogen by radiolysis of the high level waste requires periodic mixing of the tank sludge to prevent accumulation of hydrogen in the settled sludge. These factors were used to establish the range of conditions for the size-reduced zeolite tests. Conditions tested were: Insoluble Solids Content (3 wt % minimum, 12 wt % nominal), Supernate Composition (1.0 M Na minimum, 7.0 M Na maximum) and Settling time (30 days maximum). The test mixtures prepared are shown in Table 3-5. These test mixtures were used for settled solids yield stress measurements, settling rate tests, preferential settling tests and leaching of silica tests.

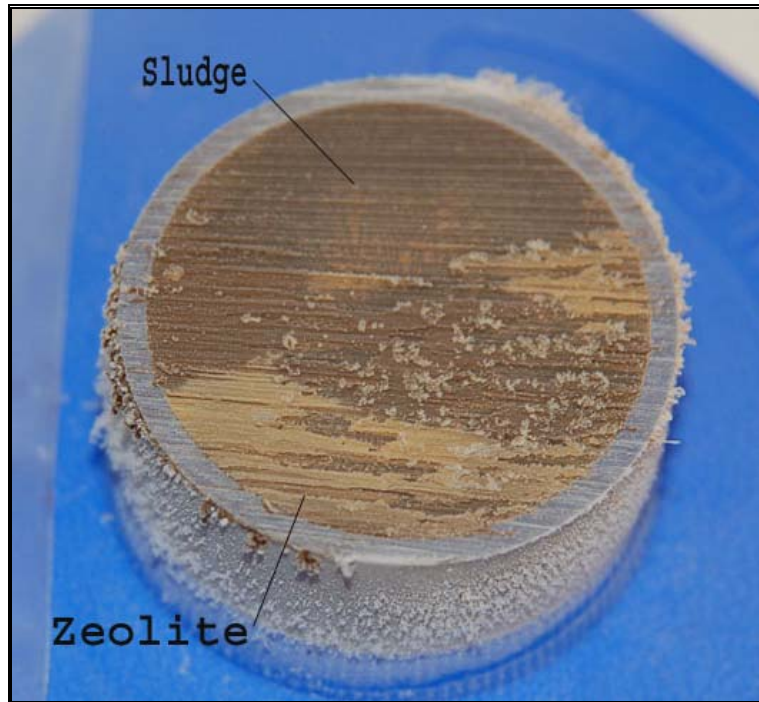
**Table 3-5 Size-Reduced Zeolite Test Mixtures**

<b>Sample</b>	<b>Test Description</b>
T19-SRZ-1	SRZ + 7 M Tank 7 Sup. at 12 wt% IS
T19-SRZ-2	SRZ + 1 M Tank 7 Sup. at 12 wt% IS
T19-SRZ-3	SRZ + DI H <sub>2</sub> O at 12 wt% IS
T19-SRZ-4	SRZ + 7 M Sludge + 7 M Tank 7 Sup. at 12 wt% IS
T19-SRZ-5	SRZ + 7 M Sludge(2X) + 7 M Tank 7 Sup. at 12 wt% IS
T19-SRZ-6	SRZ + 7 M Sludge + 7 M Tank 7 Sup. at 3 wt% IS
T19-SRZ-7	SRZ + 7 M Sludge(0.5X) + 7 M Tank 7 Sup. at 12 wt% IS
T19-SRZ-8	SRZ + 7 M Sludge(0.5X) + 7 M Tank 7 Sup. at 3 wt% IS
T19-SRZ-9	SRZ + 1 M Sludge + 1 M Tank 7 Sup. at 12 wt% IS
T19-SRZ-10	SRZ + 1 M Sludge(2X) + 1 M Tank 7 Sup. at 12 wt% IS
T19-SRZ-11	SRZ + 1 M Sludge(2X) + 1 M Tank 7 Sup. at 3 wt% IS
T19-SRZ-12	SRZ + 1 M Sludge(0.5) + 1 M Tank 7 Sup. at 3 wt% IS
T19-SRZ-13	7 M Sludge + 7M Tank 7 Sup. at 3 wt% IS
T19-SRZ-14	1 M Sludge + 1 M Tank 7 Sup. at 3 wt% IS
T19-SRZ-15	SRZ + 1 M Tank 7 Sup. at 3 wt% IS
T19-SRZ-16	SRZ + 7 M Tank 7 Sup. at 3 wt% IS

### 3.3.2 Preferential Settling Tests

Settled columns of tests T19-SRZ-4 through T19-SRZ-12 were frozen and sectioned to allow analysis for evidence of preferential settling. Physical observation was made of each section for obvious evidence of preferential settling. Each section was sampled and the samples were analyzed by ICPEs for Al, Ca, Fe, Mg, Mn, Na, Ni and Si. The primary indicator for the zeolite in each section was the Si content with the Ca content as a secondary indicator. The primary indicators for sludge were the results for Fe, Mn, or Ni. If the settled bed of zeolite-sludge was homogeneous, then the ratio of Si to Fe or Si to Ni would be constant across the three samples of the bed. The same applies to the Ca/Fe or the Ca/Ni ratios. The enrichment in zeolite was calculated by comparing the Si/Fe or Ca/Fe for the section to the same ratio for the top section. Similar results were also obtained using the Si/Ni and Ca/Ni ratios as a crosscheck since Ni was not expected to be present in the IE-95 zeolite..

Observation of the individual slices only showed one mixture that had obvious separation of zeolite from sludge. Figure 3-7 shows the bottom sample from T19-SRZ-9. The uneven distribution is probably due to the channeling that occurs when particles that are denser force less dense particles aside. Most of the sections did not show distinct evidence of preferential settling.



**Figure 3-7 50:50 Sludge:Zeolite at 12 wt Insoluble Solids in 1 Molar Na, Bottom Sample**

Chemical analysis of the sections showed that preferential settling was present in nearly all of the settling tests. Table 3-6 and Table 3-7 give the degree of zeolite enrichment in the top, middle and bottom sections from each settling test for the 7 Molar and 1 Molar supernate tests respectively. The least amount of preferential settling was observed for tests with twice as much sludge solids as zeolite solids with the solids loading starting at 12 wt % insoluble solids. However, dilution of the same mixture of insoluble solids to 3 wt % leads to a definite increase in zeolite in the bottom section. The 3 wt % results suggest that the periodic dilution to 3 wt % insoluble solids for sludge washing or for hydrogen release could increase the chances for zeolite to produce deposits under the sludge (especially in locations with flow obstructions for the Tank 7 slurry pumps). Since similar tests for preferential settling were not performed on the jet-eductor system processed zeolite combined with sludge, it is not possible to determine if the preferential settling is less with the size-reduced zeolite. However, the jet-eductor system processed zeolite is expected to further preferentially settle since it has 50% of its mass in particles greater than 43 microns.

**Table 3-6 Preferential Settling Results for Zeolite-Sludge Mixtures in 7 Molar Na Supernate**

Test Mixture	Sample location	Zeolite Enrichment		Preferential Settling (Y,N)
		Silicon	Calcium	
50% Sludge 50% Zeolite Starting at 12 wt% Insoluble Solids	Top	100	100	
	Middle	117	137	Y
	Bottom	116	139	
50% Sludge 50% Zeolite Starting at 3 wt% Insoluble Solids	Top	100	100	
	Middle	110	103	Y
	Bottom	201	279	
67% Sludge 33% Zeolite Starting at 12 wt% Insoluble Solids	Top	100	100	
	Middle	101	100	N
	Bottom	101	99	
33% Sludge 67% Zeolite Starting at 12 wt % Insoluble Solids	Top	100	100	
	Middle	114	127	Y
	Bottom	114	126	
33% Sludge 67% Zeolite Starting at 3 wt % Insoluble Solids	Top	100	100	
	Middle	143	131	Y
	Bottom	332	405	

**Table 3-7 Preferential Settling Results for Zeolite-Sludge Mixtures in 1 Molar Na Supernate**

Test Mixture	Sample location	Zeolite Enrichment		Preferential Settling (Y,N)
		Silicon	Calcium	
50% Sludge 50% Zeolite Starting at 12 wt% Insoluble Solids	Top	100	100	
	Middle	100	144	Y
	Bottom	107	140	
67% Sludge 33% Zeolite Starting at 12 wt% Insoluble Solids	Top	100	100	
	Middle	105	115	Y
	Bottom	105	112	
67% Sludge 33% Zeolite Starting at 3 wt% Insoluble Solids	Top	100	100	
	Middle	119	137	Y
	Bottom	158	363	
33% Sludge 67% Zeolite Starting at 3 wt % Insoluble Solids	Top	100	100	
	Middle	118	122	Y
	Bottom	142	252	

Caution must be exercised in translating these tests to the actual waste tank since the sludge simulant does not include uranium. The presence of uranium in the sludge may increase the settling velocity of the sludge particles relative to the zeolite particles and minimize the degree of preferential settling. However,

the presence of mounds of zeolite in Tank 19 despite the unobstructed use of slurry pumps suggest that the settling rate of larger particles of zeolite does exceed that of the actual sludge (including uranium). In our opinion, the current preferential settling results are conservative in that they indicate that the potential exists for preferential settling with the actual waste.

### 3.3.3 Settled Solids Yield Stress Results

Settled solids yield stress measurements were made on portions of the test mixtures that were initially mixed and then allowed to stand undisturbed for up to 30 days. Results are available for the 12 wt % IS tests but not for the 3 wt % IS tests. The beds of solids for the 3 wt % IS tests were either too thin to perform the vane measurements for our current vanes (see Figure 2-1) or inaccessible to our instrument when sufficient solids were present.

If preferential settling occurs with the size-reduced zeolite, the SSYS for the size-reduced zeolite in caustic supernate becomes the major issue. Figure 3-8 shows the time dependence of the SSYS for the size-reduced zeolite in 7 Molar Na supernate for up to 36 days of settling time. The size-reduced zeolite does not build SSYS of greater than 600 Pascals in less than 30 days compared to the jet-eductor system processed zeolite which exceeded 11000 Pascals in less than 5 days. Reducing the size of the zeolite particles does improve the fluid properties of the settled zeolite should the zeolite settle separately from the sludge. Note that Figure 3-8 also shows the mixture of an equal amount of insoluble sludge solids with the zeolite further reduces the maximum SSYS obtained within 30 days to about 200 Pascals.

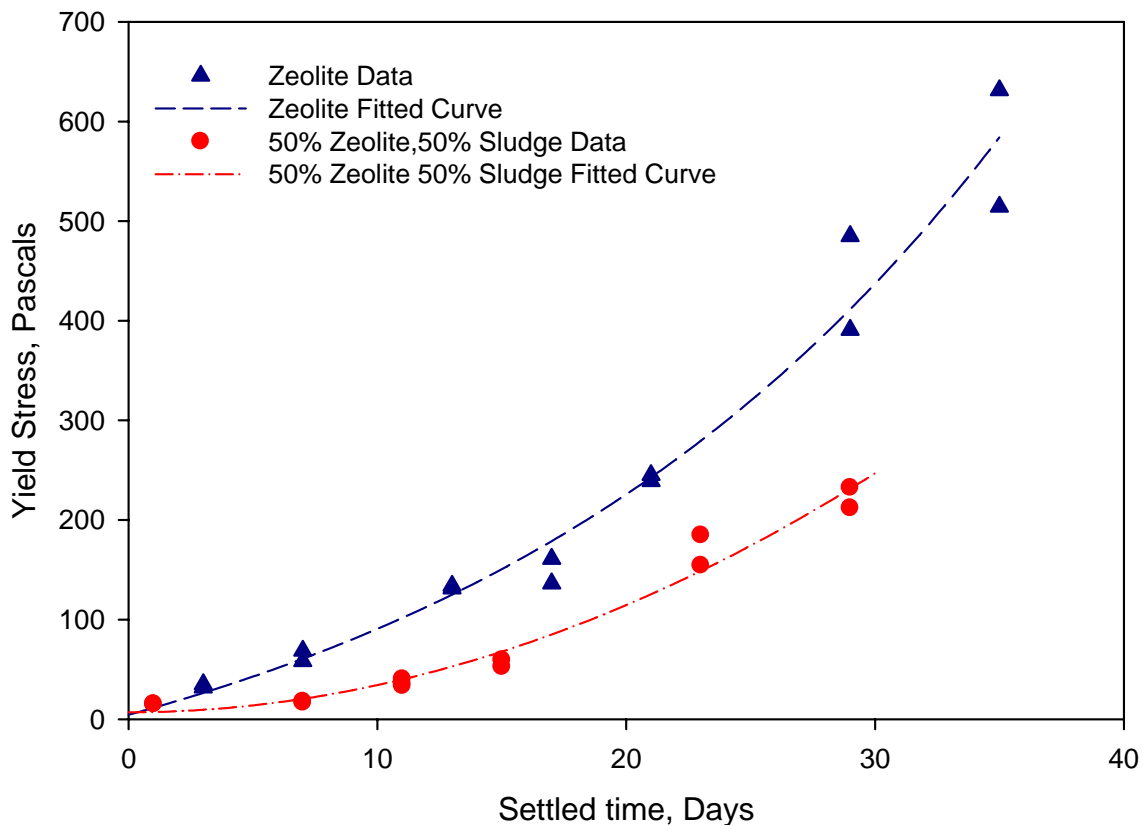
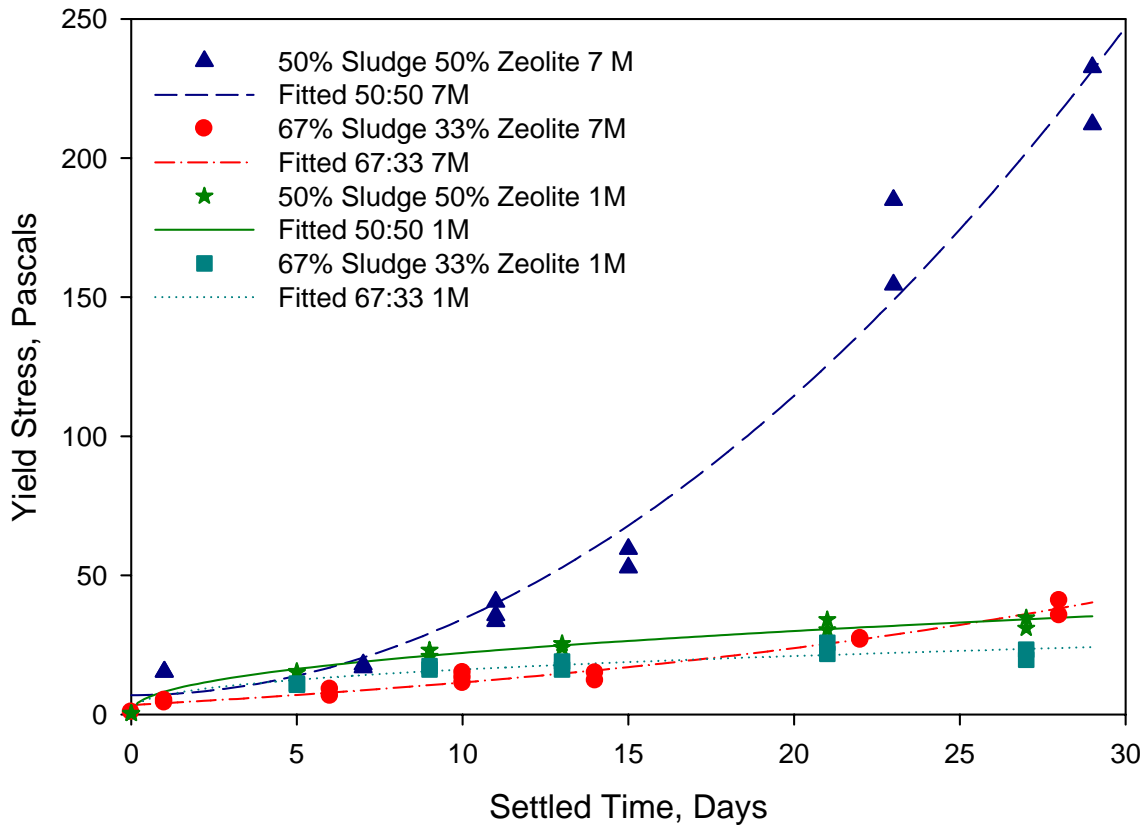


Figure 3-8 Size-Reduced Zeolite at 12 wt% Insoluble Solids in 7 Molar Na Supernate

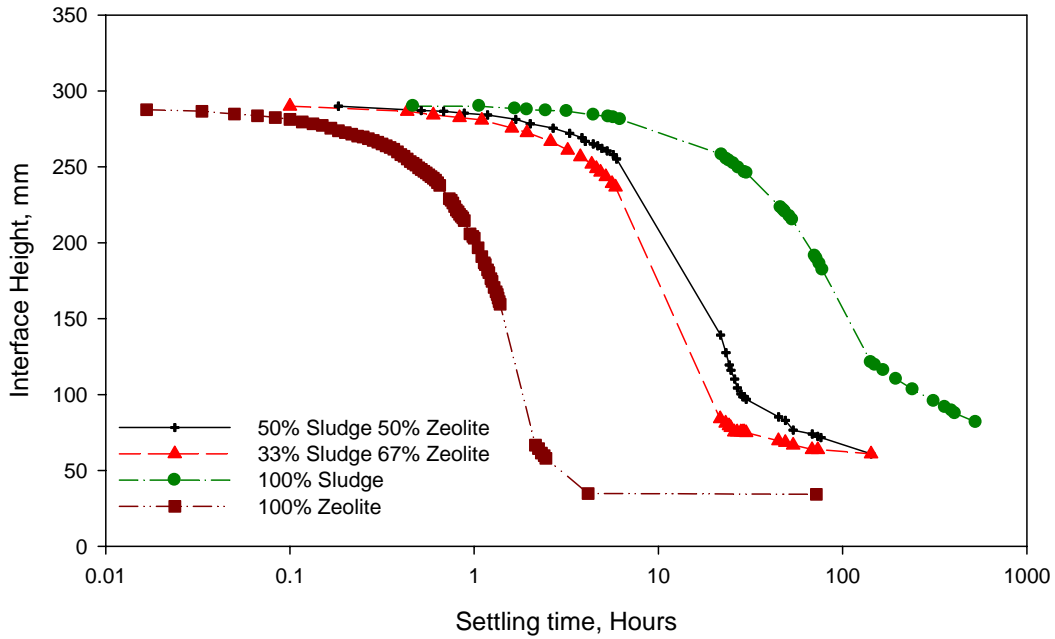
A comparison of all of the SSYS results for sludge-zeolite mixtures in both 7 and 1 Molar Na supernates is shown in Figure 3-9. In general, the more sludge the smaller the increase in SSYS as a function of time for periods of under thirty days. Based upon the SSYS results from the size-reduced zeolite only and from the mixtures with sludge, the transfer of mound material from Tank 19 to Tank 7 appears to be compatible with Tank 7 operation if periodic mixing is applied to the waste. Extrapolation of these curves to much longer time periods without mixing is risky. The majority of the blends SSYS are below 50 Pa, a settled sludge that should easily resuspend.



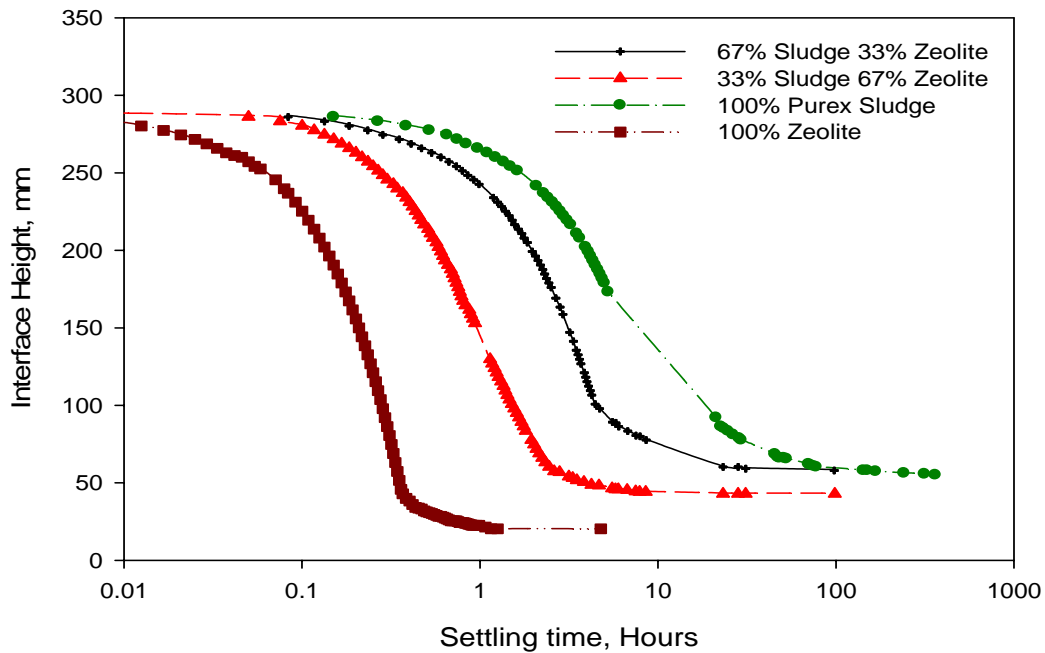
**Figure 3-9 Size-Reduced Zeolite Mixed with Purex Sludge at 12 wt % Insoluble Solids in Seven Molar Na and One Molar Na Supernates**

### 3.3.4 Settling Tests

The settling of the size-reduced zeolite and sludge test mixtures was measured as a function of time to give information that can be compared to actual waste settling data. The settling data for the 3 wt % insoluble solids test mixture in seven Molar and one Molar supernates are shown in Figure 3-10 and Figure 3-11 respectively. Note that the time axis is in a log scale to allow easy display of both fast and slow settling mixtures. In general, the mixtures with higher percentages of zeolite settled faster. Conversely, increased sludge slowed the settling of the mixture with 100% sludge settling the slowest. The settling rate was also a function of the sodium molarity (supernate density, supernate viscosity) with the slowest settling mixtures containing the highest concentration of soluble salts.



**Figure 3-10 Settling Plot for Size-Reduced Zeolite Sludge Mixtures at 3 wt% Insoluble Solids in Seven Molar Na Supernate**



**Figure 3-11 Settling Plot for Size-Reduced Zeolite Sludge Mixtures at 3 wt % Insoluble Solids in One Molar Na Supernate**

Similar shaped curves were obtained for the settling of the 12 wt % IS test mixtures. The maximum settling rates were calculated for each of the test mixtures based upon portion of the curves that

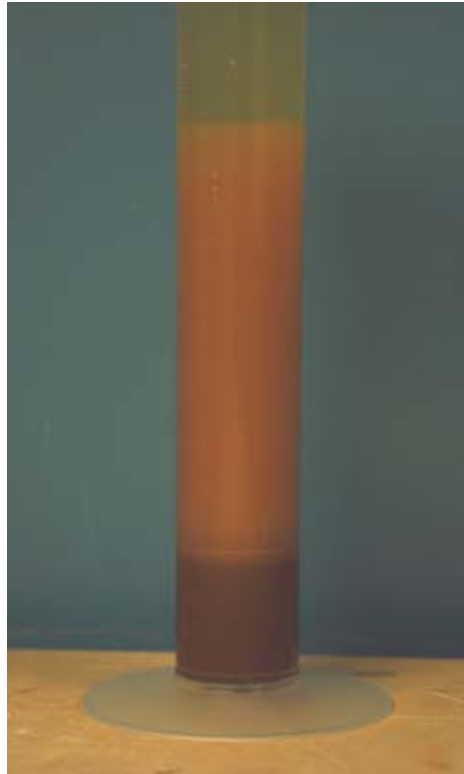


represented a nearly steady settling rate (unhindered settling region). Table 3-8 gives the maximum settling rate for each test in mm/hour or inches/hour for comparison to actual waste data when that is available.

**Table 3-8 Maximum Measured Settling Rates**

<b>Material Tested</b>	<b>Na, Moles/L</b>	<b>Initial wt% IS</b>	<b>Rate, mm/Hr</b>	<b>Rate, inches/Hr</b>
100% SRZ in 1 M (12 wt%)	1	12	78.1	3.075
50% SRZ + 50% Sludge + 1 M (12 wt% IS)	1	12	23.6	0.929
33% SRZ + 67% Sludge + 1 M (12 wt% IS)	1	12	14.8	0.583
33% SRZ + 67% Sludge + 1 M (3 wt% IS)	1	3	36.3	1.429
67% SRZ + 33% Sludge + 1 M (3 wt% IS)	1	3	133	5.236
100% Sludge + 1 M (3 wt% IS)	1	3	22.2	0.874
100% SRZ + 1 M (3 wt% IS)	1	3	680	26.772
100% SRZ + 7 M (12 wt%)	7	12	7.6	0.299
50% SRZ + 50% Sludge + 7 M (12 wt% IS)	7	12	2.3	0.091
33% SRZ + 67% Sludge + 7 M (12 wt% IS)	7	12	0.08	0.003
67% SRZ + 33% Sludge + 7 M (12 wt% IS)	7	12	3.1	0.122
50% SRZ + 50% Sludge + 7 M (3 wt% IS)	7	3	5.9	0.232
67% SRZ + 33% Sludge + 7 M T (3 wt% IS)	7	3	9.1	0.358
100% Sludge + 7 M (3 wt% IS)	7	3	1.4	0.055
100% SRZ + 7 M (3 wt% IS)	7	3	103.9	4.091

Settling data for the size-reduced zeolite in water is not included because of the presence of fines that did not agglomerate with the larger particles and tended to settle extremely slowly with no evidence of a clearly defined settling interface. Instead, the larger particles were observed to quickly settle through the mixture. Similar observations of the nonsettling or extremely slow settling fines were also made on the jet-eductor system processed zeolite. The tests with the size-reduced zeolite in the caustic supernate did not produce a nonsettling fines phase presumably due to the high ionic strength suppressing the boundary layer charge sufficiently to allow agglomeration to take place. Chemical analysis of the nonsettling zeolite fines did not show a significant difference from the bulk IE-95 material.



**Figure 3-12 Settling Test of Size-Reduced Zeolite in Water after 60 days**

### **3.3.5 Particle Size Results for the Size-Reduced Zeolite Tests**

The particle size results for the size-reduced zeolite tests are shown in Table 3-9. The mean particle size on a volume basis varied from 16.4 microns down to 3.8 microns with the smaller average size due to the presence of the Purex sludge simulant. The measured particle size distribution on a volume basis for each of the materials listed in Table 3-9 can be found in Appendix A.

**Table 3-9 Particle Size Results for Size-Reduced Zeolite Tests**

Material Description	Size Range		Volume Basis	
	Min	Max	Mean	Median
	µm	µm	µm	µm
As-Received Zeolite in Water	0.486	1408.0	390.5	442.3
Tank 19 From WSRC-TR-2002-00288	0.972	704.0	541.0	292.0
Processed jet-eductor system Zeolite in Water	0.972	704.0	115.4	43.3
High Sheared Zeolite Using High Shear Mixer in Water	0.409	37.0	5.3	4.2
T19-SRZ-3, Size Reduced Zeolite + DI Water	0.688	62.2	15.0	13.0
T19-SRZ-1, Size Reduced Zeolite + 7M Tank 7 Supernate	1.156	176.0	16.4	11.8
T19-SRZ-2, Size Reduced Zeolite + 1M Tank 7 Supernate	1.156	31.1	10.9	10.5
T19-SRZ-4, Size Reduced Zeolite + 7M Sludge/Supernate – 12 wt% IS	0.486	37.0	7.9	5.4
T19-SRZ-5, Size Reduced Zeolite + (2X)7M Sludge/Supernate – 12 wt% IS	0.578	22.0	6.5	4.7
T19-SRZ-6, Size Reduced Zeolite + 7M Sludge/Supernate – 3 wt% IS	0.688	18.5	5.6	4.5
T19-SRZ-7, Size Reduced Zeolite + (0.5X)7M Sludge/Supernate – 12 wt% IS	0.972	18.5	6.6	5.3
T19-SRZ-8, Size Reduced Zeolite + (0.5X)7M Sludge/Supernate – 3 wt% IS	0.818	26.2	8.4	6.6
T19-SRZ-9, Size Reduced Zeolite + 1M Sludge/Supernate – 12 wt% IS	0.578	22.0	7.1	4.5
T19-SRZ-10, Size Reduced Zeolite + (2X)1M Sludge/Supernate – 12 wt% IS	0.578	22.0	5.2	3.5
T19-SRZ-11, Size Reduced Zeolite + (2X)1M Sludge/Supernate – 3 wt% IS	0.688	15.6	4.1	3.1
T19-SRZ-12, Size Reduced Zeolite + (0.5X)1M Sludge/Supernate – 3 wt% IS	0.972	22.0	6.9	5.1
T19-SRZ-13, 7M Sludge/Supernate - 3 wt% IS	0.486	18.5	6.9	3.1
T19-SRZ-14, 1M Sludge/Supernate - 3 wt% IS	0.486	15.6	3.8	2.6

### 3.3.6 Leaching of Silicon from Zeolite During Testing

The supernate from the size-reduced zeolite tests and from the original Tank 7 supernate simulants were submitted for analysis for soluble Si and Ca to determine if leaching of the IE-95 zeolite was occurring during the tests. All of the test mixtures sampled had contact times with the supernate of at least 30 days. The results of ISCPES analysis are shown in Table 3-10. Significant amounts of Si were leached from the size-reduced IE-95 resin with the largest amounts leached in the highest concentration supernate simulant. Some leaching of Si from the Purex sludge simulant is also present but at lower levels. The Ca results suggest that the leaching is from the clay binder phase of the IE-95 resin and not the chabazite phase since Ca dissolution appears to be low compared to Si.

**Table 3-10 Elemental Leach Results for Size-Reduced Zeolite Tests**

Test Description	Supernate Na, Moles/Liter	Si, mg/Liter	Ca, mg/Liter
7 M Na Supernate	7	3.8	3.0
1 M Na Supernate	1	0.5	1.3
SRZ + 7 M Tank 7 Sup. at 12 wt% IS	7	2795	4.9
SRZ + 1 M Tank 7 Sup. at 12 wt% IS	1	133.5	23.6
SRZ + 7 M Sludge + 7 M Tank 7 Sup. at 3 wt% IS	7	62.4	7.8
SRZ + 7 M Sludge(0.5X) + 7 M Tank 7 Sup. at 3 wt% IS	7	113.1	5.8
SRZ + 1 M Sludge + 1 M Tank 7 Sup. at 12 wt% IS	1	56.9	2.6
SRZ + 1 M Sludge(2X) + 1 M Tank 7 Sup. at 12 wt% IS	1	38.5	2.0
SRZ + 1 M Sludge(2X) + 1 M Tank 7 Sup. at 3 wt% IS	1	48.3	2.1
SRZ + 1 M Sludge(0.5) + 1 M Tank 7 Sup. at 3 wt% IS	1	101.3	2.9
7 M Sludge + 7M Tank 7 Sup. at 3 wt% IS	7	91.0	2.8
1 M Sludge + 1 M Tank 7 Sup. at 3 wt% IS	1	5.7	2.0

The leaching of Si from the zeolite material in the simulant tests may not necessarily occur with the actual Tank 19 mound material since the actual waste has already seen years of exposure to the caustic F area supernates. The potential may exist for leaching of Si when the actual waste material is fractured to produce smaller particles as this may expose fresh surfaces for the caustic supernates to attack. Leach tests with actual Tank 19 mound material that has been size-reduced is suggested to determine if disposition of the zeolite-contacted supernate will be an issue for the tank farm evaporators.

## 4.0 CONCLUSIONS

Based on physical chemical property testing of jet-eductor system processed IE-95 zeolite and size-reduced IE-95 zeolite the following conclusions were made:

- The jet-eductor system processed zeolite has a mean and median particle size (volume basis) of 115.4 and 43.3 microns in water. Preferential settling of these large particles is likely.
- The jet-eductor system processed zeolite rapidly generates settled solid yield stresses in excess of 11000 Pascals in caustic supernates and will not be easily retrieved from Tank 7 with the existing slurry pump technology.
- Settled size-reduced IE-95 zeolite (less than 38 microns) in caustic supernate does not generate yield stresses in excess of 600 Pascals in less than 30 days.
- Preferential settling of size-reduced zeolite is a function of the amount of sludge and the level of dilution for the mixture.
- Blending the size-reduced zeolite into larger quantities of sludge can reduce the amount of preferential settling.
- Periodic dilution or resuspension due to sludge washing or other mixing requirements will increase the chances of preferential settling of the zeolite solids.
- Mixtures of Purex sludge and size-reduced zeolite did not produce yield stresses greater than 200 Pascals for settling times less than thirty days. Most of the sludge-zeolite blends did not exceed 50 Pascals. These mixtures should be removable by current pump technology if sufficient velocities can be obtained.
- The settling rate of the sludge-zeolite mixtures is a function of the ionic strength (or supernate density) and the zeolite- sludge mixing ratio.
- Simulant tests indicate that leaching of Si may be an issue for the processed Tank 19 mound material.
- Floating zeolite fines observed in water for the jet-eductor system and size-reduced zeolite were not observed when the size-reduced zeolite was blended with caustic solutions, indicating that the caustic solutions cause the fines to agglomerate.

Based on the test programs described in this report, the potential for successfully removing Tank 18/19 mound material from Tank 7 with the current slurry pump technology requires the reduction of the particle size of the Tank 18/19 mound material.

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## 5.0 RECOMMENDATIONS/PATH FORWARD

Research on the physical properties and rheological properties of various sized zeolite particles in caustic supernate and Purex sludge simulants has generated the following recommendations for additional work that is recommended for support of the mechanical tank cleaning program:

- The Tank 18/19 mechanical cleaning program should pursue the addition of a technology that size reduces the Tank 18/19 mound material to less than 38 microns with a mean particle size distribution between 5 and 20 microns. This size reduction should be applied before or during transfer of the Tank 18/19 mound material to Tank 7.
- Vendor or site testing of the size reduction technology should include characterization for particle size distribution, settled solids yield stress and preferential settling.
- Pilot testing of the size reducing technology combined with the mechanical transport technology is recommended prior to deployment.
- Actual waste tests on Tank 19 mound material is recommended to verify that size reduction is beneficial to slurry properties as shown with the current simulant tests. Based on the current tests, the amount of Tank 19 mound material required to perform confirmatory settled solids yield stress, particle size and preferential settling test is at least 500 mL.
- Actual waste tests with the Tank 19 mound material should also examine the possibility of Si leaching from the size-reduced mound material for use in predicting the impact on disposition of the residual supernate.
- The impact of the elevated tank temperatures on the physical properties or the leaching of the settled zeolite solids should be determined. Past experience with measurement of yield stresses for actively mixed sludges at temperatures up to 50 °C has not shown much dependence of the yield stress on temperature. Settled solids yield stress has not been tested for temperature dependence (especially in the presence of a possible leaching process) in past simulant studies.

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## **7.0 ACKNOWLEDGEMENTS**

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**APPENDIX A.**

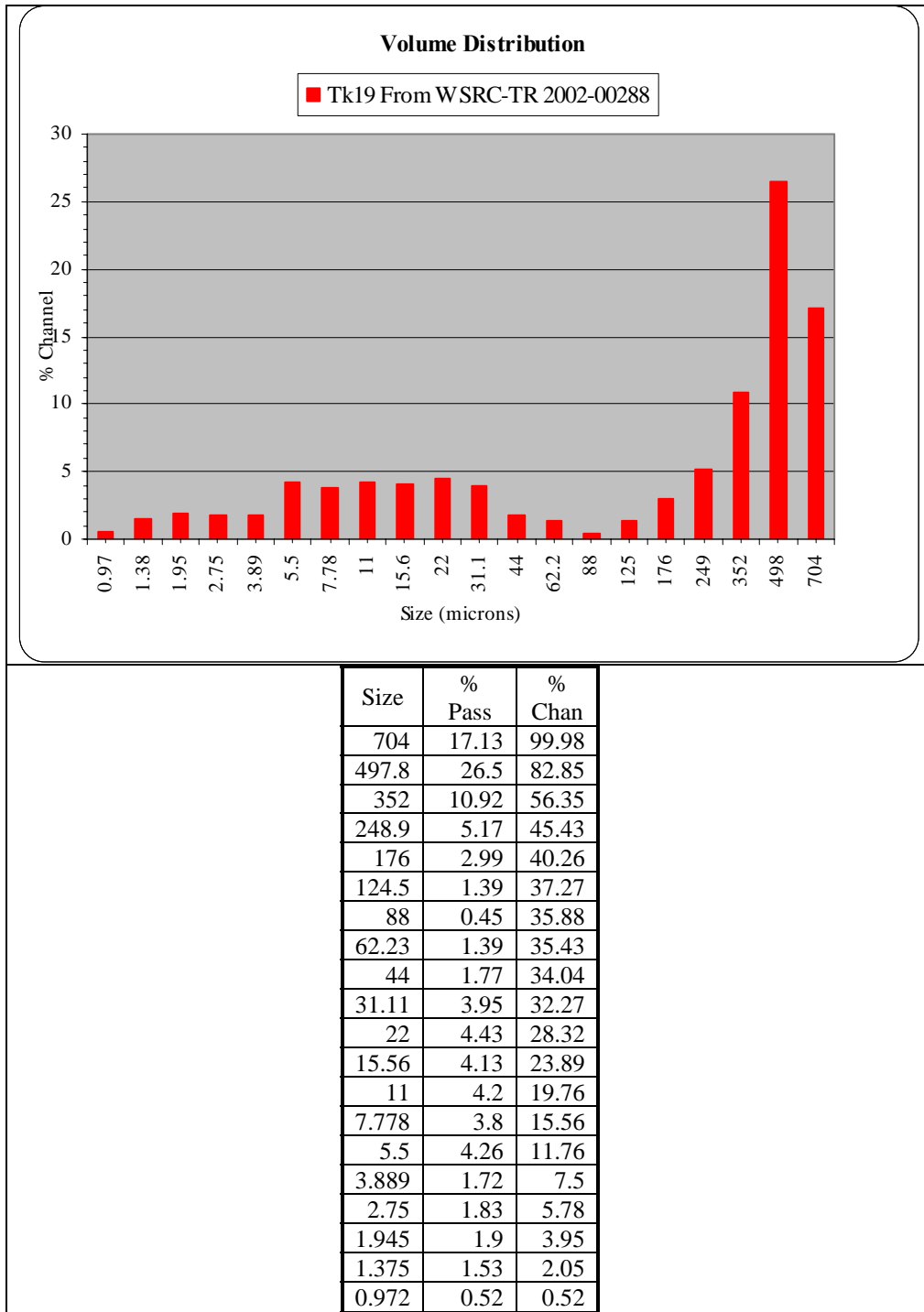
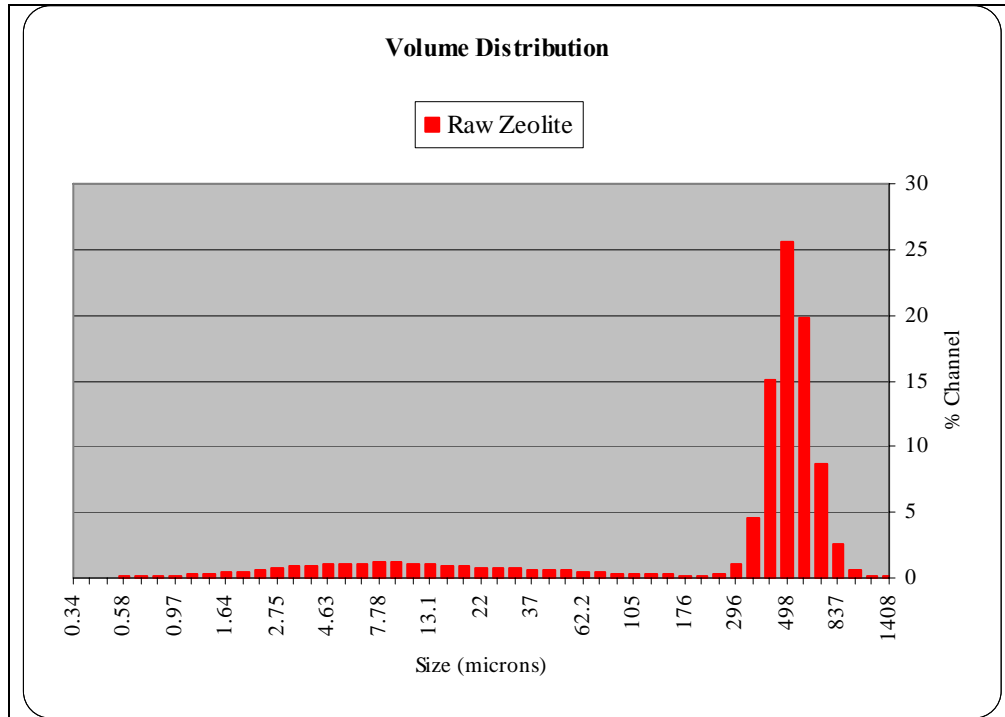
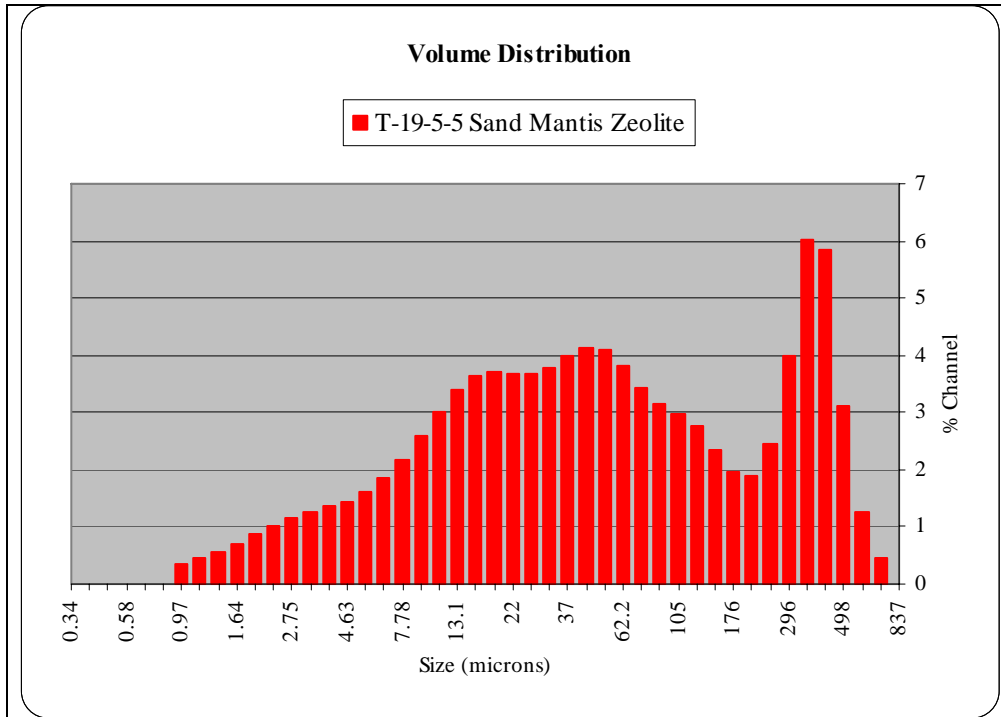


Figure A-1 Tank 19 From WSRC-TR-2002-00288



Size	% Pass	% Chan	Size	% Pass	% Chan	Size	% Pass	% Chan
1408	100.00	0.11	31.11	16.96	0.69	0.688	0.28	0.13
1184	99.89	0.15	26.16	16.27	0.73	0.578	0.15	0.10
995.6	99.74	0.67	22.00	15.54	0.79	0.486	0.05	0.05
837.2	99.07	2.62	18.50	14.75	0.86	0.409	0.00	0.00
704.0	96.45	8.63	15.56	13.89	0.94	0.344	0.00	0.00
592.0	87.82	19.87	13.08	12.95	1.02	0.289	0.00	0.00
497.8	67.95	25.62	11.00	11.93	1.10	0.243	0.00	0.00
418.6	42.33	15.10	9.250	10.83	1.15	0.204	0.00	0.00
352.0	27.23	4.54	7.778	9.68	1.16	0.172	0.00	0.00
296.0	22.69	1.09	6.541	8.52	1.14	0.145	0.00	0.00
248.9	21.60	0.35	5.500	7.38	1.09	0.122	0.00	0.00
208.3	21.25	0.20	4.625	6.29	1.02	0.102	0.00	0.00
176.0	21.05	0.19	3.889	5.27	0.93	0.086	0.00	0.00
148.0	20.86	0.23	3.270	4.34	0.94	0.072	0.00	0.00
124.5	20.63	0.29	2.750	3.50	0.74	0.061	0.00	0.00
104.7	20.34	0.33	2.312	2.76	0.63	0.051	0.00	0.00
88.00	20.01	0.37	1.945	2.13	0.52	0.043	0.00	0.00
74.00	19.64	0.42	1.635	1.61	0.41	0.036	0.00	0.00
62.23	19.22	0.47	1.375	1.20	0.32	0.030	0.00	0.00
52.33	18.75	0.54	1.156	0.88	0.25	0.026	0.00	0.00
44.00	18.21	0.60	0.972	0.63	0.19			
37.00	17.61	0.65	0.818	0.44	0.16			

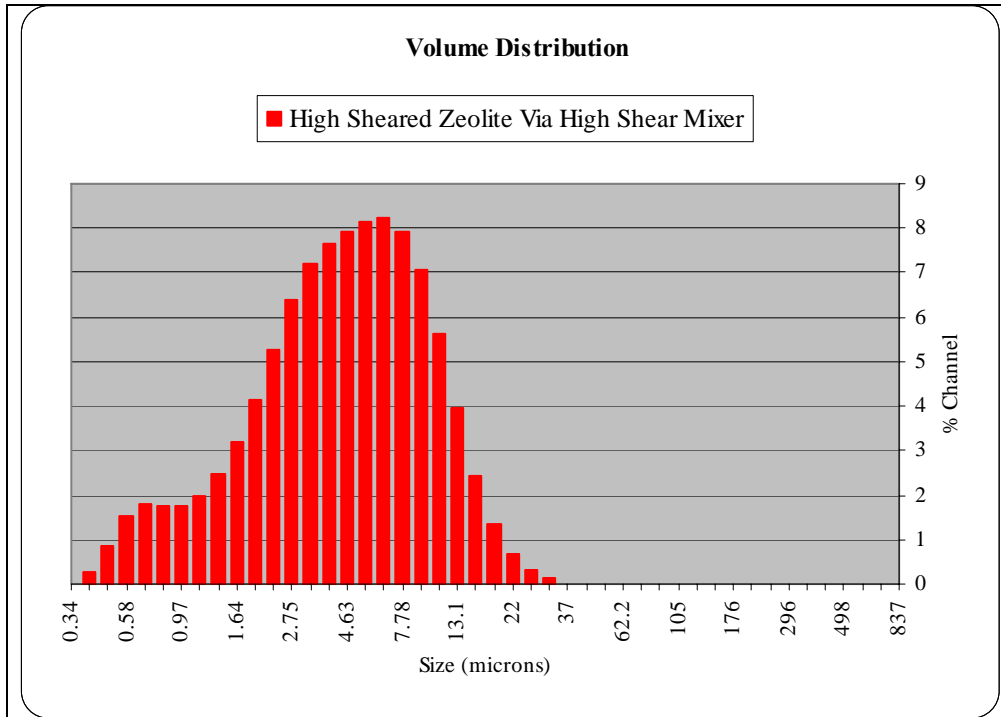
Figure A- 2 As-Received Zeolite in Water



Size	% Pass	% Chan	Size	% Pass	% Chan	Size	% Pass	% Chan
1408	100.00	0.00	31.11	42.31	3.79	0.688	0.00	0.00
1184	100.00	0.00	26.16	38.52	3.69	0.578	0.00	0.00
995.6	100.00	0.00	22.00	34.83	3.69	0.486	0.00	0.00
837.2	100.00	0.00	18.50	31.14	3.71	0.409	0.00	0.00
704.0	100.00	0.44	15.56	27.43	3.63	0.344	0.00	0.00
592.0	99.56	1.25	13.08	23.80	3.39	0.289	0.00	0.00
497.8	98.31	3.13	11.00	20.41	3.02	0.243	0.00	0.00
418.6	95.18	5.84	9.250	17.39	2.59	0.204	0.00	0.00
352.0	89.34	6.01	7.778	14.80	2.18	0.172	0.00	0.00
296.0	83.33	3.98	6.541	12.62	1.84	0.145	0.00	0.00
248.9	79.35	2.44	5.500	10.78	1.61	0.122	0.00	0.00
208.3	76.91	1.89	4.625	9.17	1.45	0.102	0.00	0.00
176.0	75.02	1.97	3.889	7.72	1.35	0.086	0.00	0.00
148.0	73.05	2.36	3.270	6.37	1.26	0.072	0.00	0.00
124.5	70.69	2.76	2.750	5.11	1.16	0.061	0.00	0.00
104.7	67.93	2.99	2.312	3.95	1.02	0.051	0.00	0.00
88.00	64.94	3.16	1.945	2.93	0.87	0.043	0.00	0.00
74.00	61.78	3.44	1.635	2.06	0.71	0.036	0.00	0.00
62.23	58.34	3.81	1.375	1.35	0.56	0.030	0.00	0.00
52.33	54.53	4.10	1.156	0.79	0.44	0.026	0.00	0.00
44.00	50.43	4.14	0.972	0.35	0.35			
37.00	46.29	3.98	0.818	0.00	0.00			

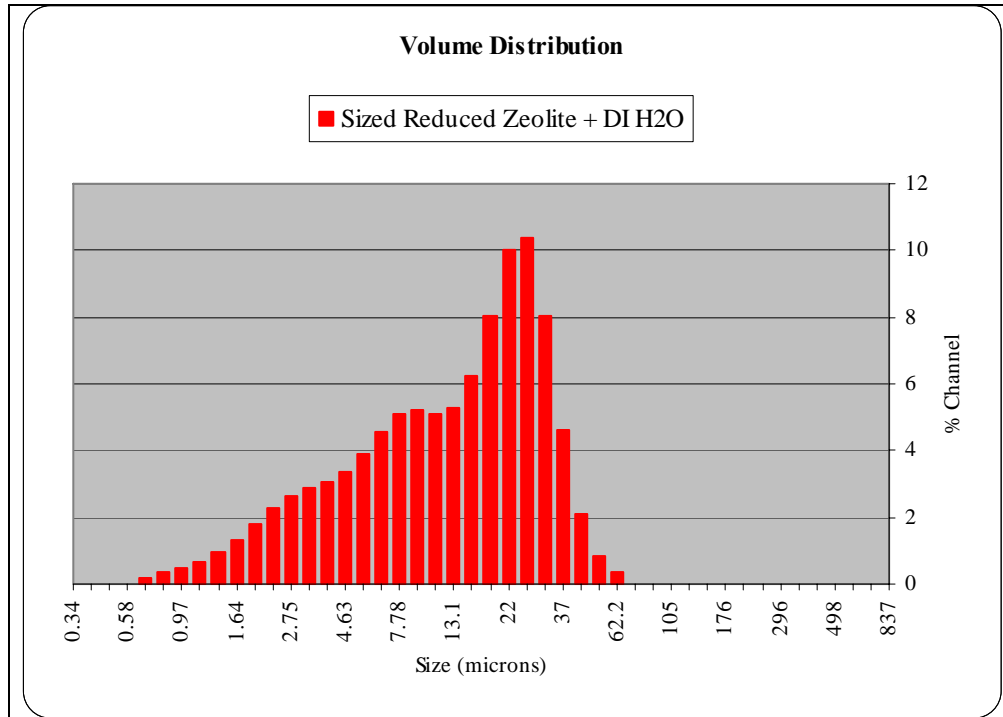
Figure A- 3 Jet-Eductor System Zeolite in Water





Size	% Pass	% Chan	Size	% Pass	% Chan	Size	% Pass	% Chan
1408	100.00	0.00	31.11	99.98	0.13	0.688	4.44	1.80
1184	100.00	0.00	26.16	99.85	0.31	0.578	2.64	1.54
995.6	100.00	0.00	22.00	99.54	0.67	0.486	1.10	0.84
837.2	100.00	0.00	18.50	98.87	1.34	0.409	0.26	0.26
704.0	100.00	0.00	15.56	97.53	2.44	0.344	0.00	0.00
592.0	100.00	0.00	13.08	95.09	3.96	0.289	0.00	0.00
497.8	100.00	0.00	11.00	91.13	5.64	0.243	0.00	0.00
418.6	100.00	0.00	9.250	85.49	7.07	0.204	0.00	0.00
352.0	100.00	0.00	7.778	78.42	7.94	0.172	0.00	0.00
296.0	100.00	0.00	6.541	70.48	8.24	0.145	0.00	0.00
248.9	100.00	0.00	5.500	62.24	8.15	0.122	0.00	0.00
208.3	100.00	0.00	4.625	54.09	7.93	0.102	0.00	0.00
176.0	100.00	0.00	3.889	46.16	7.64	0.086	0.00	0.00
148.0	100.00	0.00	3.270	38.52	7.18	0.072	0.00	0.00
124.5	100.00	0.00	2.750	31.34	6.37	0.061	0.00	0.00
104.7	100.00	0.00	2.312	24.97	5.28	0.051	0.00	0.00
88.00	100.00	0.00	1.945	19.69	4.15	0.043	0.00	0.00
74.00	100.00	0.00	1.635	15.54	3.18	0.036	0.00	0.00
62.23	100.00	0.00	1.375	12.36	2.46	0.030	0.00	0.00
52.33	100.00	0.00	1.156	9.90	1.97	0.026	0.00	0.00
44.00	100.00	0.00	0.972	7.93	1.75			
37.00	100.00	0.02	0.818	6.18	1.74			

Figure A- 4 High Sheared Zeolite Using High Shear Mixer in Water



Size	% Pass	% Chan	Size	% Pass	% Chan	Size	% Pass	% Chan
1408	100.00	0.00	31.11	92.06	8.02	0.688	0.20	0.20
1184	100.00	0.00	26.16	84.04	10.38	0.578	0.00	0.00
995.6	100.00	0.00	22.00	73.66	10.03	0.486	0.00	0.00
837.2	100.00	0.00	18.50	63.63	8.05	0.409	0.00	0.00
704.0	100.00	0.00	15.56	55.58	6.23	0.344	0.00	0.00
592.0	100.00	0.00	13.08	49.35	5.30	0.289	0.00	0.00
497.8	100.00	0.00	11.00	44.05	5.13	0.243	0.00	0.00
418.6	100.00	0.00	9.250	38.92	5.25	0.204	0.00	0.00
352.0	100.00	0.00	7.778	33.67	5.13	0.172	0.00	0.00
296.0	100.00	0.00	6.541	28.54	4.59	0.145	0.00	0.00
248.9	100.00	0.00	5.500	23.95	3.89	0.122	0.00	0.00
208.3	100.00	0.00	4.625	20.06	3.36	0.102	0.00	0.00
176.0	100.00	0.00	3.889	16.70	3.07	0.086	0.00	0.00
148.0	100.00	0.00	3.270	13.63	2.89	0.072	0.00	0.00
124.5	100.00	0.00	2.750	10.74	2.65	0.061	0.00	0.00
104.7	100.00	0.00	2.312	8.09	2.27	0.051	0.00	0.00
88.00	100.00	0.00	1.945	5.82	1.79	0.043	0.00	0.00
74.00	100.00	0.00	1.635	4.03	1.34	0.036	0.00	0.00
62.23	100.00	0.34	1.375	2.69	0.95	0.030	0.00	0.00
52.33	99.66	0.85	1.156	1.74	0.67	0.026	0.00	0.00
44.00	98.81	2.12	0.972	1.07	0.49			
37.00	96.69	4.63	0.818	0.58	0.38			

Figure A- 5 T19-SRZ-3, Size Reduced Zeolite + DI Water

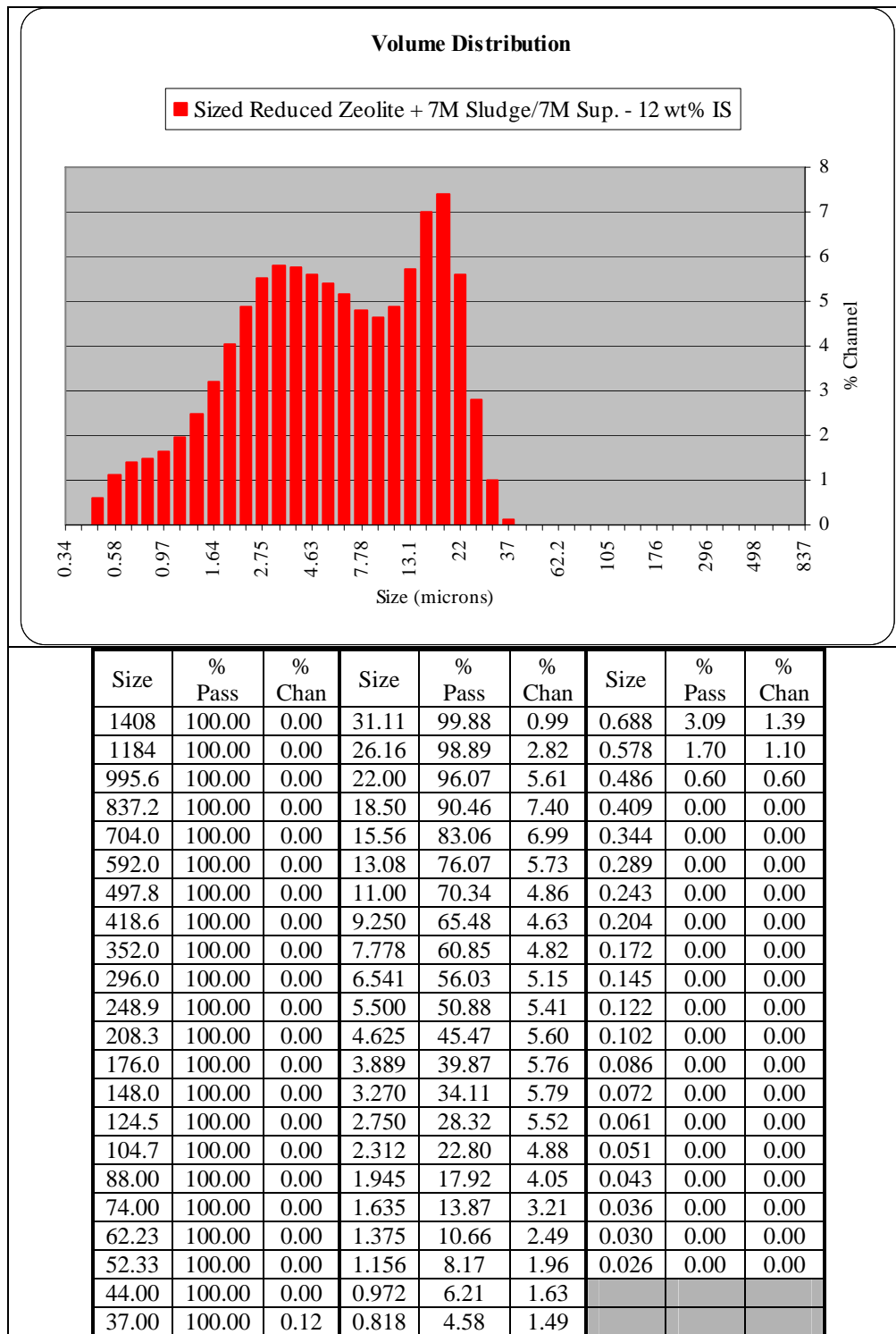


Figure A- 6 T19-SRZ-4, Size Reduced Zeolite + 7M Sludge/Supernate – 12 wt% IS

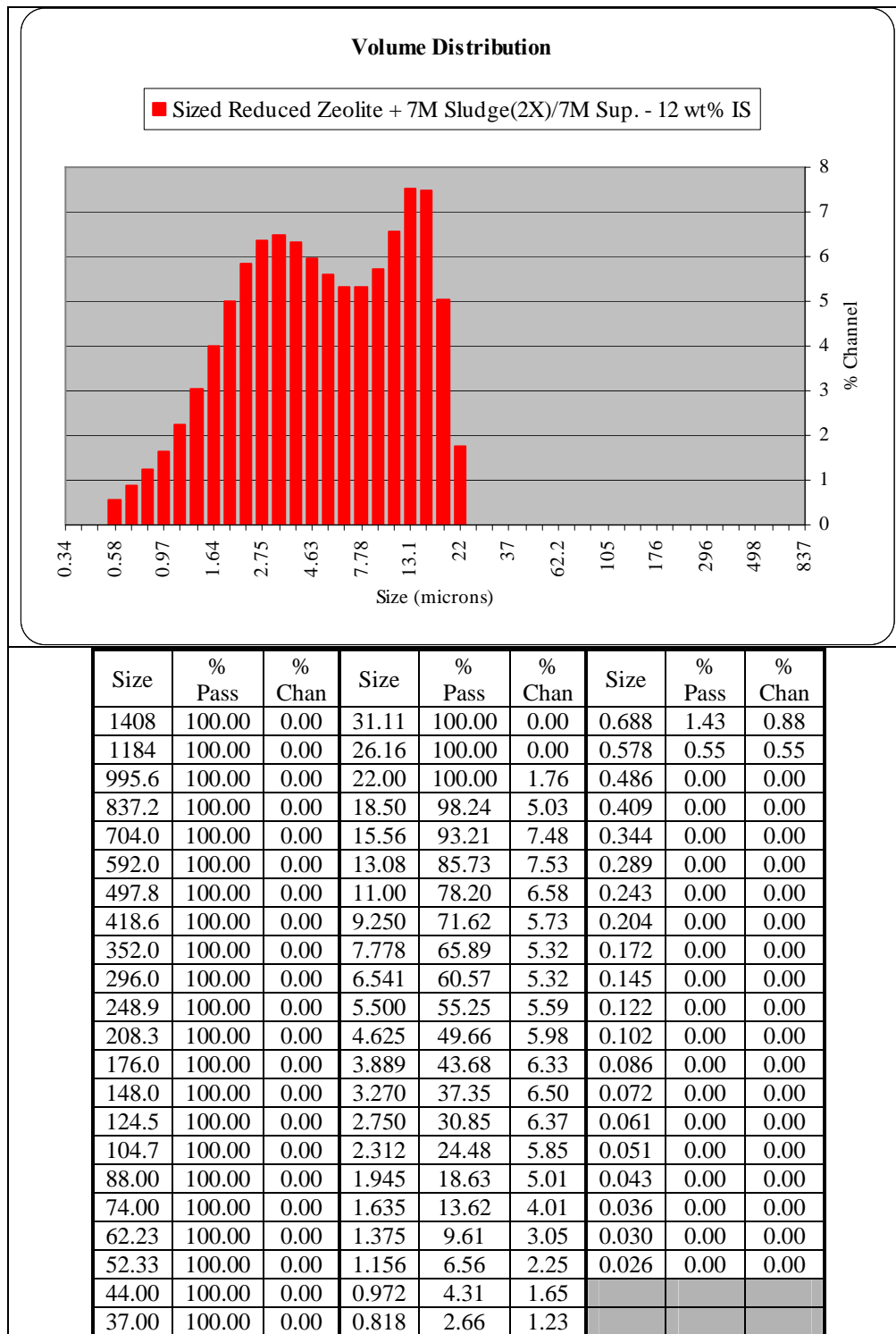


Figure A- 7 T19-SRZ-5, Size Reduced Zeolite + (2X)7M Sludge/Supernate – 12 wt% IS

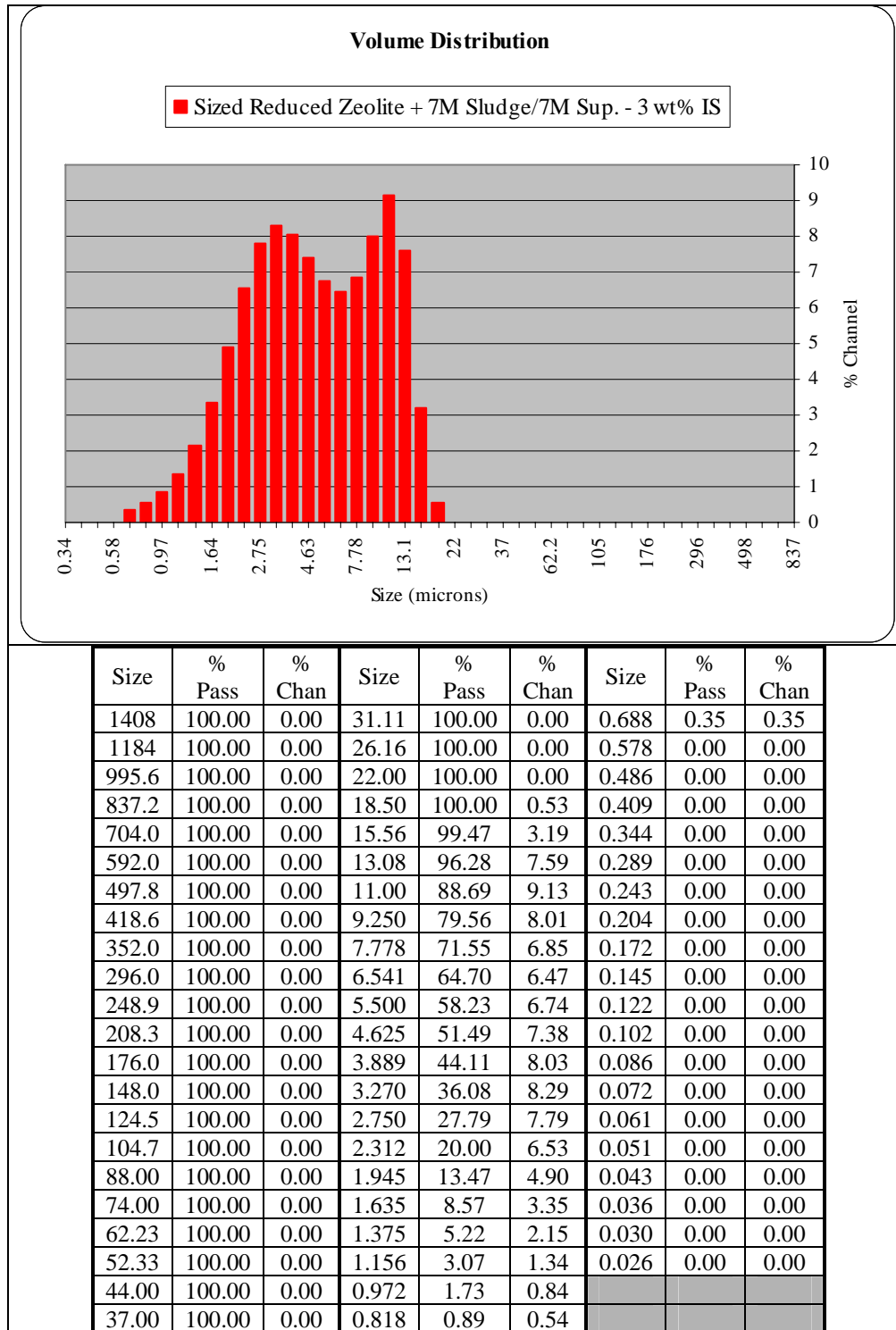


Figure A- 8 T19-SRZ-6, Size Reduced Zeolite + 7M Sludge/Supernate – 3 wt% IS

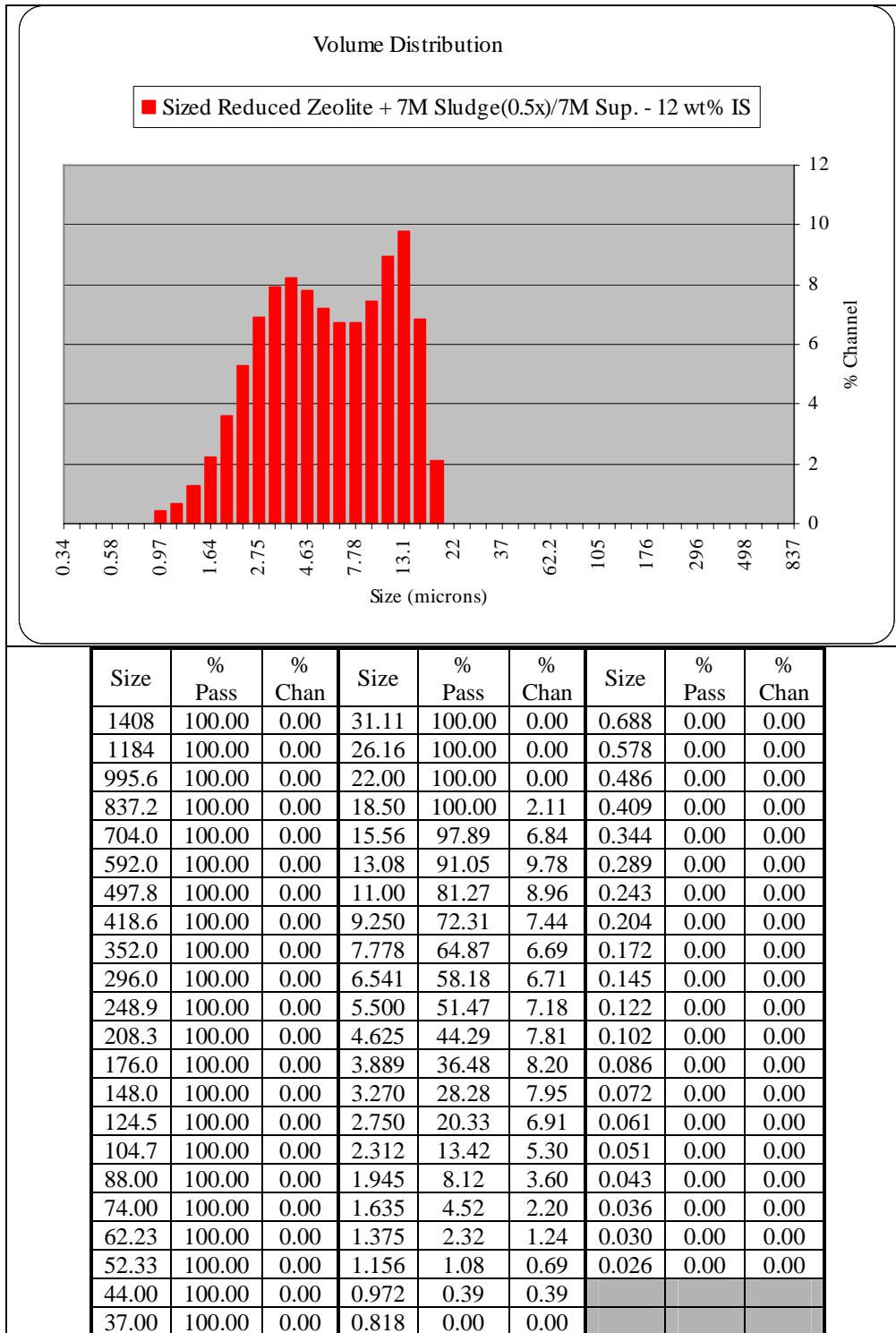


Figure A- 9 T19-SRZ-7, Size Reduced Zeolite + (0.5X)7M Sludge/Supernatant – 12 wt% IS

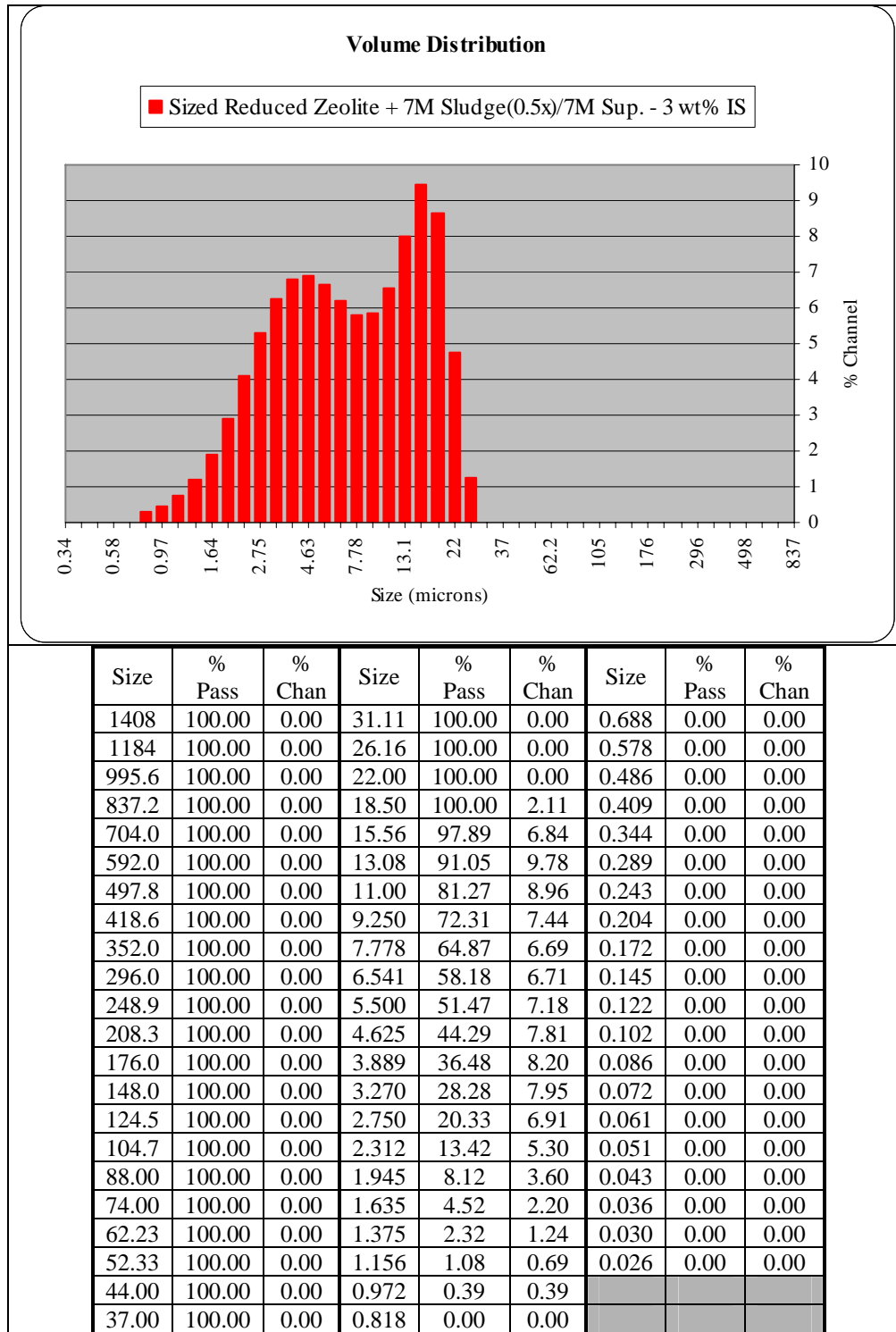


Figure A- 10 T19-SRZ-8, Size Reduced Zeolite + (0.5X)7M Sludge/Supernate – 3 wt% IS

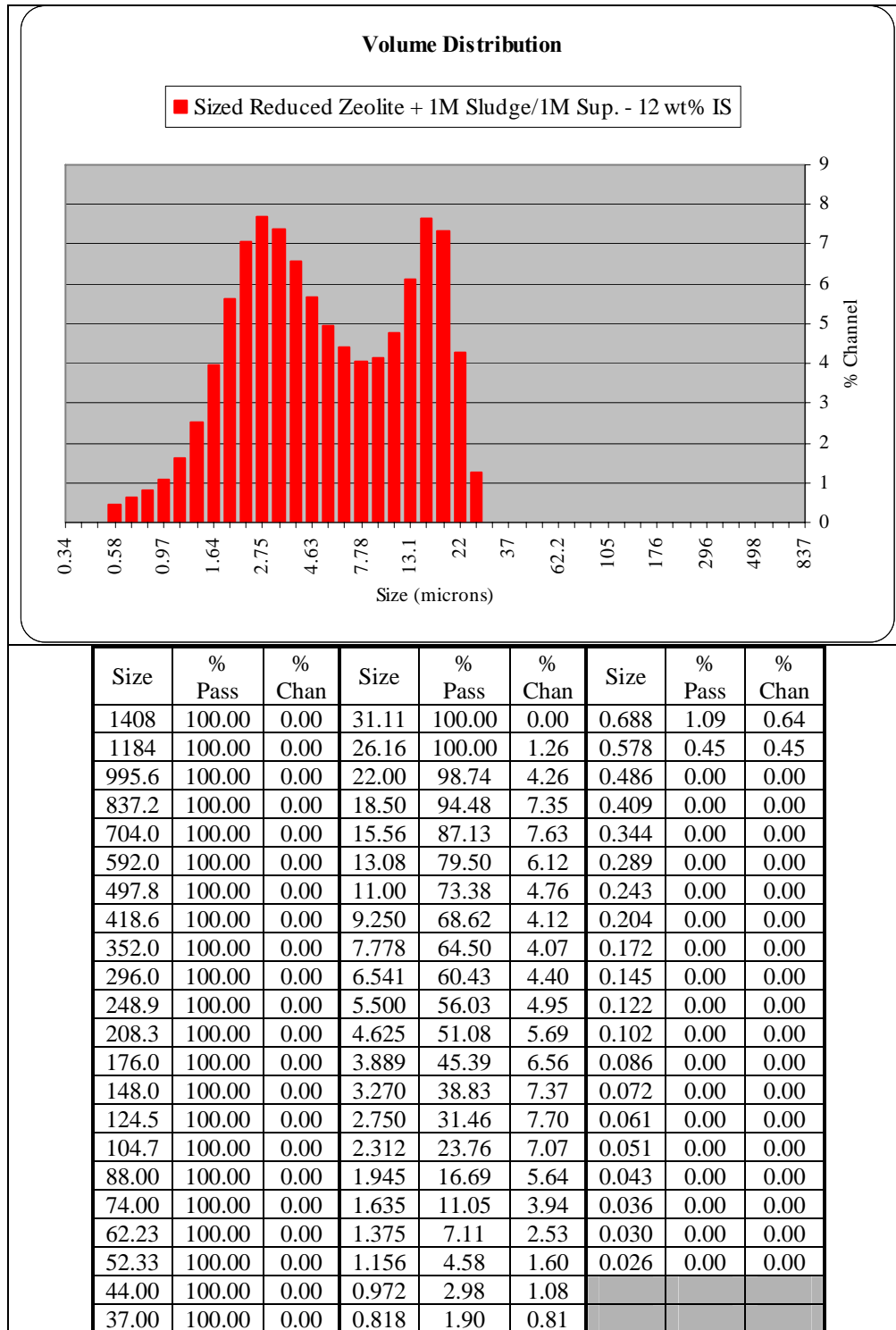


Figure A- 11 T19-SRZ-9, Size Reduced Zeolite + 1M Sludge/Supernate – 12 wt% IS



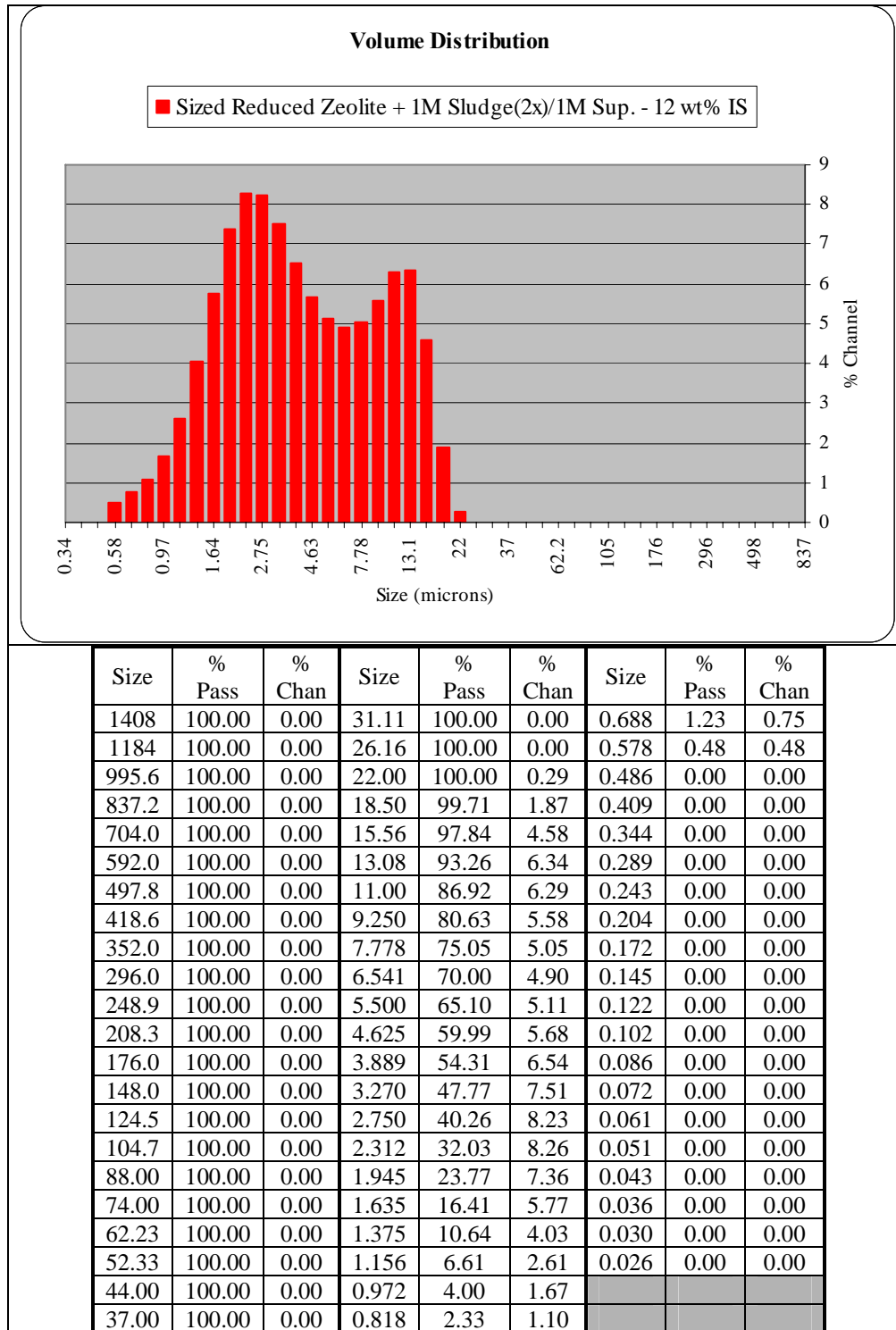


Figure A- 12 T19-SRZ-10, Size Reduced Zeolite + (2X)1M Sludge/Supernate – 12 wt% IS

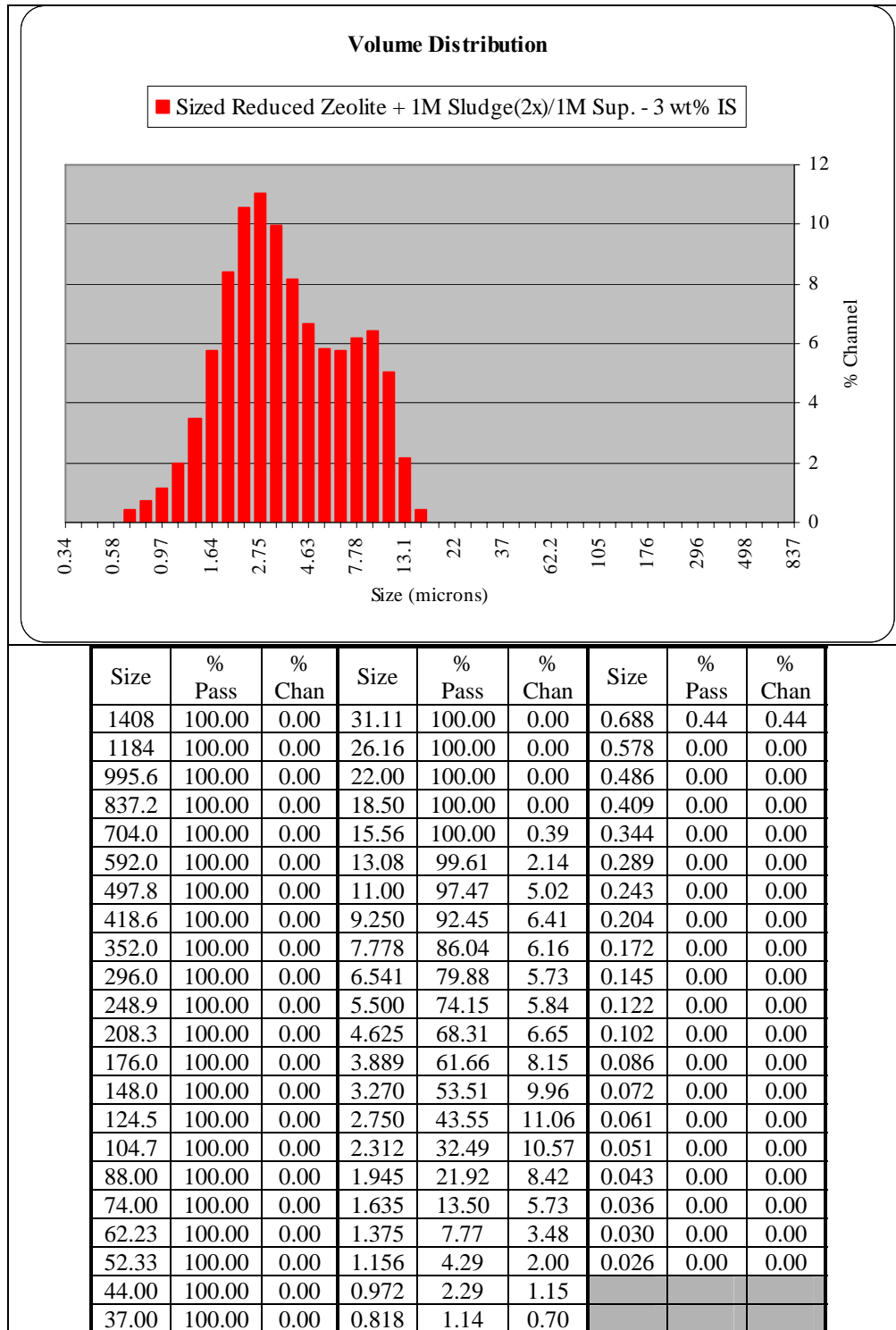


Figure A- 13 T19-SRZ-11, Size Reduced Zeolite + (2X)1M Sludge/Supernate – 3 wt% IS

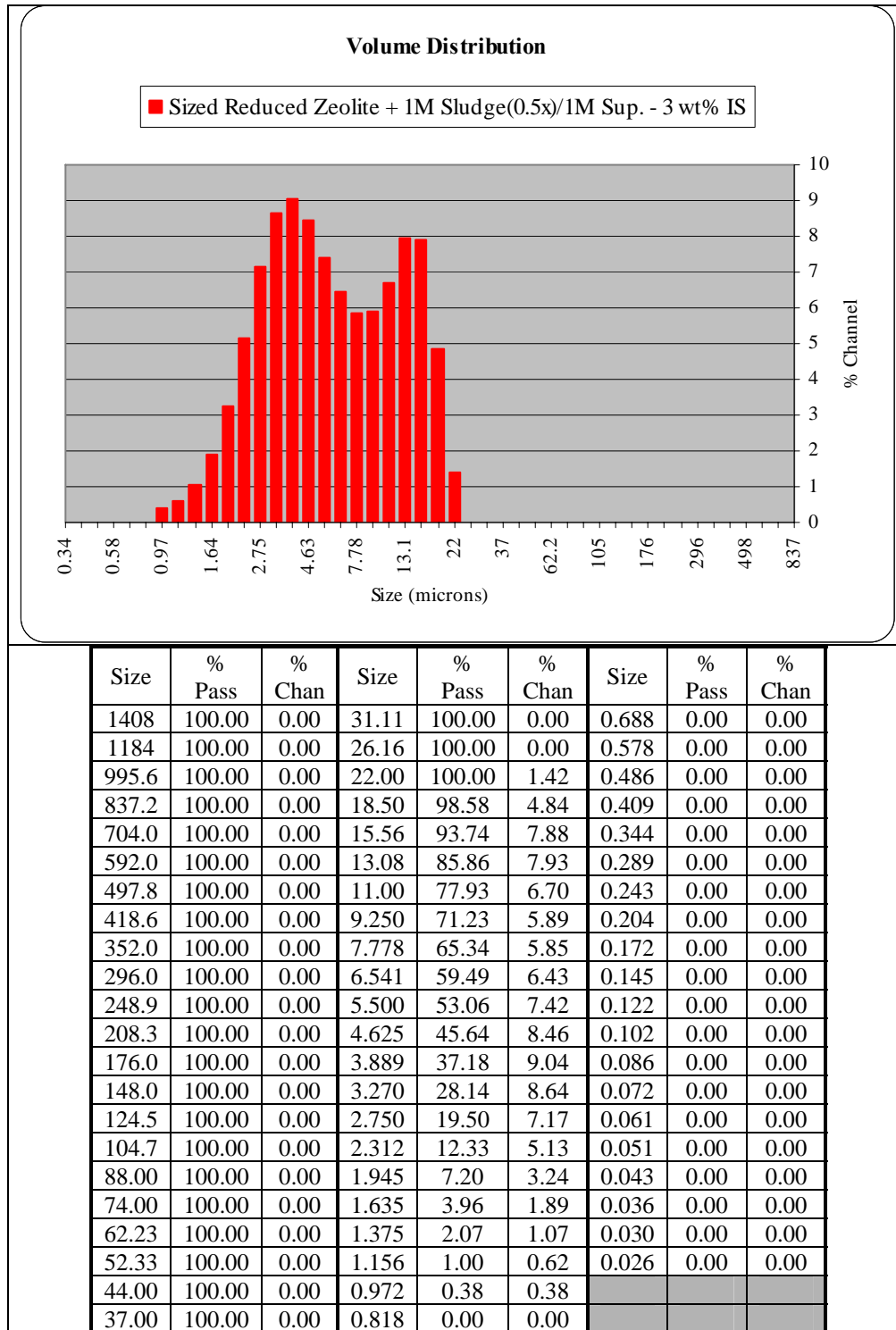
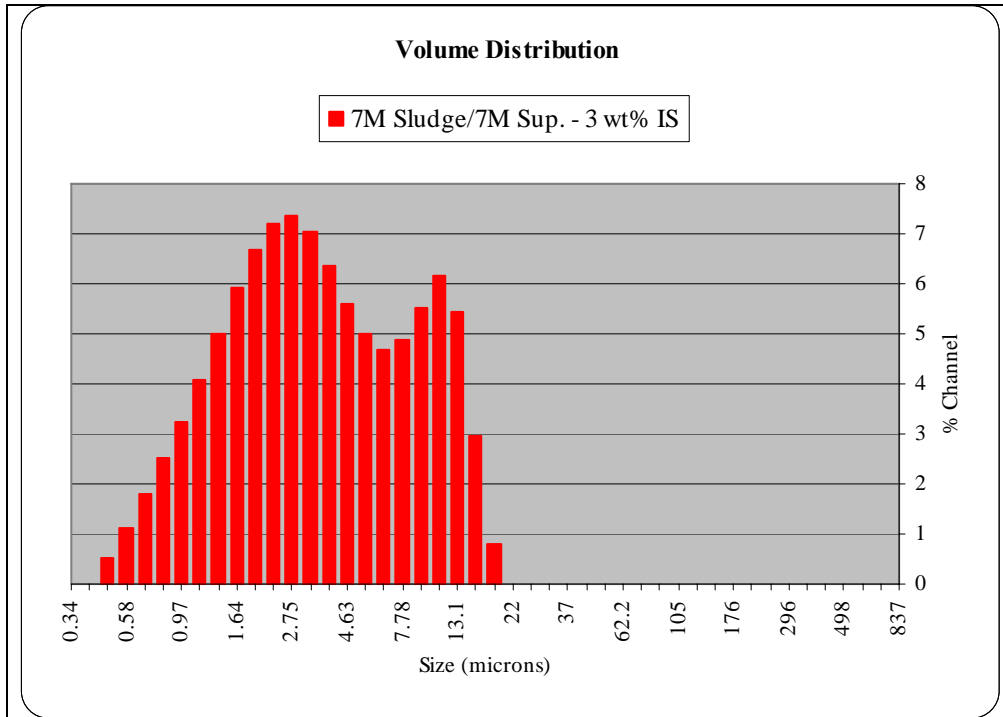
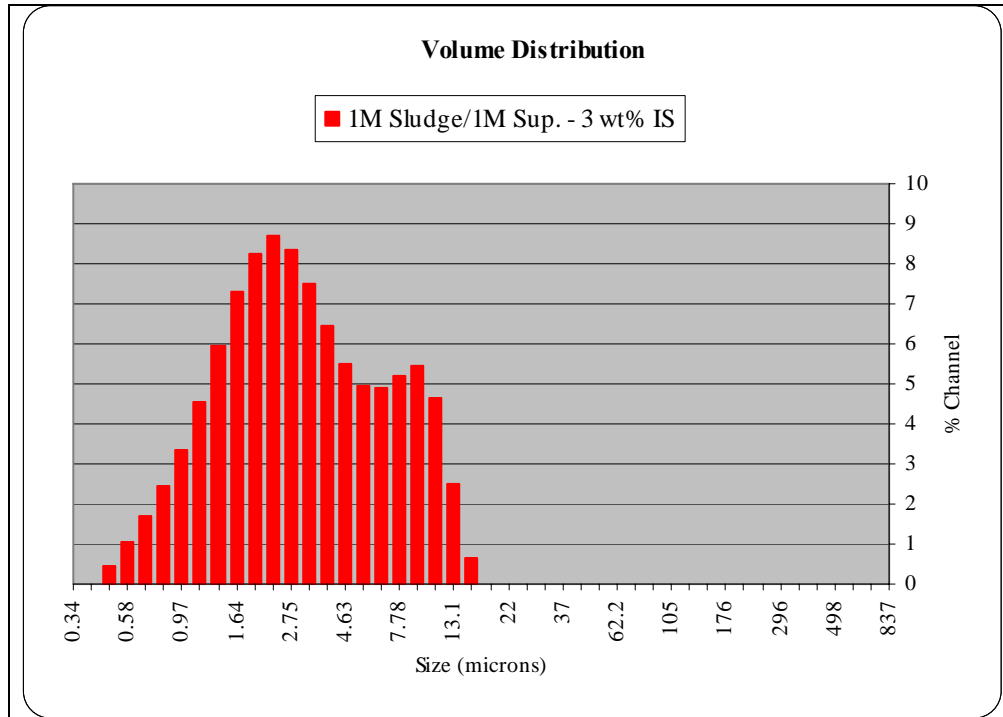


Figure A- 14 T19-SRZ-12, Size Reduced Zeolite + (0.5X)1M Sludge/Supernate – 3 wt% IS



Size	% Pass	% Chan	Size	% Pass	% Chan	Size	% Pass	% Chan
1408	100.00	0.00	31.11	100.00	0.00	0.688	3.44	1.80
1184	100.00	0.00	26.16	100.00	0.00	0.578	1.64	1.10
995.6	100.00	0.00	22.00	100.00	0.00	0.486	0.54	0.54
837.2	100.00	0.00	18.50	100.00	0.79	0.409	0.00	0.00
704.0	100.00	0.00	15.56	99.21	2.95	0.344	0.00	0.00
592.0	100.00	0.00	13.08	96.26	5.46	0.289	0.00	0.00
497.8	100.00	0.00	11.00	90.80	6.17	0.243	0.00	0.00
418.6	100.00	0.00	9.250	84.63	5.54	0.204	0.00	0.00
352.0	100.00	0.00	7.778	79.09	4.89	0.172	0.00	0.00
296.0	100.00	0.00	6.541	74.20	4.70	0.145	0.00	0.00
248.9	100.00	0.00	5.500	69.50	4.98	0.122	0.00	0.00
208.3	100.00	0.00	4.625	64.52	5.60	0.102	0.00	0.00
176.0	100.00	0.00	3.889	58.92	6.38	0.086	0.00	0.00
148.0	100.00	0.00	3.270	52.54	7.05	0.072	0.00	0.00
124.5	100.00	0.00	2.750	45.49	7.35	0.061	0.00	0.00
104.7	100.00	0.00	2.312	38.14	7.22	0.051	0.00	0.00
88.00	100.00	0.00	1.945	30.92	6.70	0.043	0.00	0.00
74.00	100.00	0.00	1.635	24.22	5.92	0.036	0.00	0.00
62.23	100.00	0.00	1.375	18.30	5.01	0.030	0.00	0.00
52.33	100.00	0.00	1.156	13.29	4.09	0.026	0.00	0.00
44.00	100.00	0.00	0.972	9.20	3.25			
37.00	100.00	0.00	0.818	5.95	2.51			

Figure A- 15 T19-SRZ-13, 7M Sludge/Supernate - 3 wt% IS



Size	% Pass	% Chan	Size	% Pass	% Chan	Size	% Pass	% Chan
1408	100.00	0.00	31.11	100.00	0.00	0.688	3.22	1.72
1184	100.00	0.00	26.16	100.00	0.00	0.578	1.50	1.03
995.6	100.00	0.00	22.00	100.00	0.00	0.486	0.47	0.47
837.2	100.00	0.00	18.50	100.00	0.00	0.409	0.00	0.00
704.0	100.00	0.00	15.56	100.00	0.63	0.344	0.00	0.00
592.0	100.00	0.00	13.08	99.37	2.48	0.289	0.00	0.00
497.8	100.00	0.00	11.00	96.89	4.67	0.243	0.00	0.00
418.6	100.00	0.00	9.250	92.22	5.47	0.204	0.00	0.00
352.0	100.00	0.00	7.778	86.75	5.22	0.172	0.00	0.00
296.0	100.00	0.00	6.541	81.53	4.91	0.145	0.00	0.00
248.9	100.00	0.00	5.500	76.62	4.97	0.122	0.00	0.00
208.3	100.00	0.00	4.625	71.65	5.50	0.102	0.00	0.00
176.0	100.00	0.00	3.889	66.15	6.43	0.086	0.00	0.00
148.0	100.00	0.00	3.270	59.72	7.52	0.072	0.00	0.00
124.5	100.00	0.00	2.750	52.20	8.37	0.061	0.00	0.00
104.7	100.00	0.00	2.312	43.83	8.69	0.051	0.00	0.00
88.00	100.00	0.00	1.945	35.14	8.26	0.043	0.00	0.00
74.00	100.00	0.00	1.635	26.88	7.30	0.036	0.00	0.00
62.23	100.00	0.00	1.375	19.58	5.96	0.030	0.00	0.00
52.33	100.00	0.00	1.156	13.62	4.57	0.026	0.00	0.00
44.00	100.00	0.00	0.972	9.05	3.37			
37.00	100.00	0.00	0.818	5.68	2.46			

Figure A- 16 T19-SRZ-14, 1M Sludge/Supernate - 3 wt% IS

**Distribution:**

R. E. Edwards, SRNL  
J. C. Griffin, 773-A  
S. L. Marra, 773-A  
D. A. Crowley, 999-W  
C. C. Herman, 773-42A  
T. B. Calloway, 999-W  
D. B. Burns, 786-5A  
N. E. Bibler, 773-A  
C.M. Jantzen, 773-A  
J. R. Harbour, 773-42A  
C. A. Langton, 773-43A  
N. F. Chapman, 766-H  
C. D. Banaszewski, 766-H  
B. R. Hess, 704-71F  
J. M. Gillam, 766-H  
A. P. Fellingner, 723-A  
D. Kremetz, 723-A  
J. R. Gordon, 730-A