

# Pacific Northwest National Laboratory

Operated by Battelle for the  
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## Bench-Scale Enhanced Sludge Washing and Gravity Settling of Hanford Tank S-107 Sludge

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September 1998

Prepared for the U.S. Department of Energy  
under Contract DE-AC06-76RLO 1830

PNNL-12010

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*operated by*  
BATTELLE MEMORIAL INSTITUTE  
*for the*  
UNITED STATES DEPARTMENT OF ENERGY  
*under Contract DE-AC06-76RLO 1830*

Printed in the United States of America

Available to DOE and DOE contractors from the  
Office of Scientific and Technical Information, P.O. Box 62, Oak Ridge, TN 37831;  
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Richland, Washington

## Summary

This report summarizes the work performed with sludge from Hanford Site single-shell Tank 241-S-107 during FY 98. The tests described in this report support the development of the baseline Hanford tank sludge pretreatment flowsheet that includes the enhanced sludge washing (ESW) and settle/decant processes.

ESW removes caustic and water-soluble components from the sludge in an effort to minimize the volume of high-level waste feed that would ultimately be vitrified. During each step of ESW, solid/liquid separations are required. Gravity settle/decant is an approach currently being considered and is the one reported on here.

This report provides scale-up data for the pretreatment of the Tank S-107 sludge. Enhanced sludge washing and settle/decant were performed on Tank S-107 sludge using the liter-scale settle/decant equipment. The experimental processing steps simulated those of the proposed full-scale process, including retrieval, caustic leaching, and inhibited water washing. The tests were performed remotely in 324 Building C-Cell using a stainless steel chemical leaching tank (10 liters) and a transparent plastic settling column (10 cm diameter by 1 m tall). Approximately 1000 grams of sludge were tested using this equipment. During the test, hindered-settling-rate (initial rate of decrease of the sediment height) and sludge-compaction (solid fraction in final sediment) data were obtained during each step of the ESW process. Solid and supernatant samples were taken to evaluate the removal efficiencies of radioactive and nonradioactive components from the sludge during the leaching and washing processes. In some cases, the removal of aluminum and chromium during the ESW process may be controlled by the dissolution kinetics rather than solubility limits. To better understand the time required to dissolve these analytes, an extended caustic leach test was performed at the conclusion of the ESW process.

These tests were supported by Pacific Northwest National Laboratory's (PNNL's) Radiocolloids Laboratory, which provided analyses of the decanted supernatants and sludges. Density, solids concentration, compressive yield stress, and particle size were measured to close mass balances and provide scale-up information. An empirical model was developed to scale-up the settling process to a full-scale million-gallon double-shell tank using the liter-scale settling curves and radiocolloids data.

ESW results were compared to those of Lumetta et al. (1996), who performed a similar experiment with Tank S-107 sludge on a laboratory scale (8.4 grams of sludge). In the work of Lumetta, et al. there was no initial retrieval step; only the two caustic leaches and three water washes were performed. Thus, the results can be compared only qualitatively. A summary of the results is shown in Table S.1. Note that less aluminum and phosphorus and more chromium were removed in the current experiment than in that of Lumetta et al. (1996). The differences in the results may be attributed to the solids and caustic concentration or the temperature and method of solid/liquid separation, which were different between the two tests. The bench-scale experiments were performed with higher solids concentrations than in the case of the laboratory-scale experiments. The bench-scale experiments were settled at elevated temperatures while the laboratory-scale experiment was centrifuged to separate the solids and liquids at room temperature.

In the extended caustic-leach experiment, the sludge was agitated with 3 M NaOH and at 80°C for 1 week. During that time, samples of either slurry or supernatant were taken periodically. At the conclusion of the week-long experiment, the sludge was allowed to settle, and a final sample was taken. Results of this experiment, as shown in Table S.2, indicate that 84% of the aluminum and 100% of the chromium could be removed with further leaching over and above that done during the ESW.

**Table S.1.** Comparison of Bench- and Laboratory-Scale Data Conditions and Component Distribution % (Brooks/Lumetta)

	<u>Retrieval Wash</u>	<u>Caustic Leach 1</u>	<u>Caustic Leach 2</u>	<u>Water Washes</u>	<u>Residue</u>
Free [OH]	0.01	1.98/2.1	2.9/2.8	0.01/0.01	--
Initial Wt % Solids	7.7	10.9/2.3	7.2/1.1	4.3/≈4.0	--
Al	3	22/56	18/17	6/0	52/27
Cr	41	16/35	13/18	6/1	23/47
P	48	18/92	7/6	7/0	19/2
<sup>137</sup> Cs	52	11/79	0/21	3/0	34/0

**Table S.2.** Distribution of Aluminum and Chromium Between ESW and Extended Caustic Leach<sup>(a)</sup>

	<u>Al (wt%)</u>	<u>Cr (wt%)</u>
Amount Removed in Enhanced Sludge Wash	48	77
Amount Removed in Extended Caustic Leach	36	23
Amount Remaining in Sludge Residue	16	0

(a) Based on analyte concentrations in the initial sludge.

For each step in the ESW process, the sludge settling was complete, and compaction began in the 1-meter column within 4 hours of the start of the test. During the settling, a distinct interface formed between the settling solids and supernatant. Hindered settling rates were linear, as predicted, and ranged from 3.2 cm/h (at 14.6 wt% solids, 80°C, caustic leach) to 16.7 cm/h (at 7.7 wt% solids, 80°C, retrieval step). Settling rates improved with decreased solids concentration (over the range of 4.3 to 14.9 wt%). Higher insoluble solids concentrations were achieved for the caustic-leach steps than for water-wash steps. The settle/decant process obtained high decontamination factors for both transuranics (TRU) and <sup>90</sup>Sr (as measured by the ratio of TRU and <sup>90</sup>Sr in the solids to that in the solution).

A semi-empirical model of sludge settling was developed based on the results of the bench-scale sludge-settling tests and centrifugation of smaller samples. This model is capable of predicting the hindered settling as a function of concentration (for Tank S-107 sludge with caustic and water wash solutions) and the sludge compaction for greater depths of sludge. This allows extrapolation of the experimental data to a full-scale double-shell tank (DST) or similar settling system. The results of this extrapolation are shown in Table S.3 below. Note that the caustic leaches required longer time, but are compacted to higher solids concentrations than the water washes. Higher solids concentrations required more time to settle, for both the hindered settling regime and the compaction regime. In all cases, the solids concentration in the compacted sludge is greater than 20 wt% within 10 days, which is considerably better than the 30 days assumed in the TWRS O&UP (Kirkbride 1997).

**Table S.3. Weight Percent Solids in the Compacted Sludge Attainable in a 10-meter-Tall DST at Various Settling Times**

<b><u>Condition</u></b>	<b><u>Initial Solids</u></b>	<b><u>3 days</u></b>	<b><u>10 days</u></b>	<b><u>30 days</u></b>	<b><u>Infinite</u></b>
Retrieval	5 wt%	29.4	32.3	33	33.2
Step	10 wt%	26.7	32.0	34.0	34.2
Caustic	5 wt%	9.86	31.1	33.1	33.3
Leach	10 wt%	14.0	29.7	33.3	35.8
Water Wash	5 wt%	20.0	25.3	26.3	26.5
	10 wt%	16.6	25.4	26.6	27.4

## Acknowledgments

This work was performed for Waste Feed Delivery of Lockheed Martin Hanford Company under the direction of Roger W. Powell. The authors gratefully acknowledge their financial support and managerial support. Further, we acknowledge the technical support, including test plan and report reviews provided by the project manager Randy A. Kirkbride and cognizant engineer David E. Place.

This project was a cooperative effort between PNNL and BWHC. During testing in the hot cells of the 324 Building, PNNL provided the technical direction and BWHC provided the hot-cell operators. We would like to acknowledge the project management of Ernest J. Bitten and the hot-cell operations support of David O. Jenkins, E. Dewayne Smith, Mitchell P. Marrott and Ron S. Holeman.

We acknowledge PNNL Analytical Chemistry Laboratory, especially the technical direction of Rick T. Steele and Michael W. Urie. We thank Bruce A. Reynolds for his peer review of the document and Wayne Cosby as technical editor for this document. Finally, we thank Nicole Waldo for her invaluable assistance in data recording and analysis.

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## 1.0 Introduction

The U.S. Department of Energy's (DOE's) Hanford Site has 177 underground storage tanks that contain wastes from past nuclear fuel reprocessing and waste-management operations. These tanks require remediation. The contents of these tanks will be disposed of either as high-level waste (HLW) in a deep geologic repository or as low activity waste (LAW) onsite in near-surface disposal sites. Because the cost to dispose of the high-level waste fraction is expected to be high, the waste may be pretreated before being immobilized to minimize the quantity of HLW generated.

Hanford's tanks contain a mixture of supernate, water-soluble saltcake, and water-insoluble sludge. The saltcake and supernate will be processed to remove cesium, and possibly technetium, and then immobilized as LAW. The tank sludges, on the other hand, contain the bulk of the radionuclides and will be disposed of as HLW. To minimize their impact on the final waste volume, these sludges will be retrieved from the tank and pretreated using ESW.

The ESW process first leaches the sludge with hot caustic (2 to 3 M NaOH). This step solubilizes sludge components such as aluminum, phosphorus, and chromium. The sludge is then washed with inhibited water (0.01 M NaOH/0.01 M NaNO<sub>2</sub>) to remove the added sodium as well as other water-soluble ions. The Tank Waste Remediation System Operation and Utilization Plan (TWRS O&UP) (Kirkbride 1997) uses mass-weighted average wash/leach factors of 0.91, 0.86, and 0.95 for aluminum, phosphorus, and chromium, respectively of the single-shell tank (SST) wastes. These three components are removed to decrease the volume of HLW generated and improve the quality of the final waste form produced. The transuranic elements (primary alpha emitters) and <sup>90</sup>Sr are not solubilized during ESW and should remain with the leached solids and be incorporated into the HLW.

During each step in the ESW process, solid/liquid separation techniques will be required. A candidate being considered for these separations is gravity settling. To be considered a viable separation technique, gravity settling must provide a high degree of supernate clarification and sludge compaction in an acceptable period of time. The TWRS O&UP assumes that sludges from SSTs settle in a double shell tank 1 month (rates between 1 to 2 cm/h), and that the final compacted material contains 20 wt% insoluble solids.

Recently, the U.S. Department of Energy elected to privatize several aspects of the TWRS efforts. This privatization has been divided into two phases. Phase 1 will be a proof-of-concept/commercial demonstration phase and will involve the pretreatment and LAW vitrification of approximately 6 to 13 percent of the total waste volume. Phase 1 will also allow for immobilization of a fraction of the HLW sludges. Phase 2 will be the full-scale production phase. Facilities will be sized so all of the remaining waste from the 177 tanks can be processed and immobilized by 2028.

This report describes the pretreatment of sludge from Hanford tank 241-S-107 at Pacific Northwest National Laboratory.<sup>(a)</sup> This tank will be part of the Privatization Phase 2 tanks to be remediated. It is one of the REDOX tank wastes that contain high aluminum concentrations (Lumetta et al. 1996). These REDOX tanks make up a large fraction of the total sludge inventory and have a large fraction of AlOOH (boehmite), which dissolves less readily and is in colloidal sized particles in contrast to the more common Al(OH)<sub>3</sub> (gibbsite) found in tank wastes (Lumetta et al. 1996). These

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characteristics may make the aluminum more difficult to remove and settling less efficient. For this reason, S-107 was selected as the tank of study.

This report describes the settling and compaction properties of  $\approx 1000$  grams of S-107 sludge during the various steps of simulated retrieval and ESW. The analysis of the settling and compaction data is coupled with results from the analysis of slurry samples by PNNL's Radioactive Colloids laboratory. Both of these analyses feed into a semi-empirical model used for scaling up the settling data from liter- to full-scale. The TRU and  $^{90}\text{Sr}$  concentrations in the supernate were also measured to determine the partitioning of these species between the solid and liquid phases.

The efficiency of the ESW was evaluated. The quantities of both non-radioactive elements and radioactive isotopes removed during the various stages of the ESW are presented. As an extension to the ESW, a 1-week extended caustic leach was also performed following the enhanced sludge wash to better understand the kinetics of removal of Al and Cr.

## 2.0 Experimental Methods

The settle/decant, ESW and extended caustic leaching for the S-107 material were performed from April to July 1998 using the liter-scale settle/decant equipment. A brief description of the equipment and the testing are described in the sections below.

### 2.1 Equipment Description

The settle/decant equipment consists of two processing tanks and three chemical holding tanks connected by stainless steel tubing with valves and pumps to facilitate transfer of test materials. This equipment was in the 324 Building C-Cell with supporting equipment in the C-Cell operating gallery. The test equipment process flow diagram is shown in Figure 2.1.

#### 2.1.1 Tanks

The first processing tank is the sludge receipt tank, C-202, which was used for retrieval and sludge washing and leaching functions. This 8-liter tank was equipped with an agitator, a heater, thermocouples for temperature monitoring, and inlet and outlet lines. A port on the top of the tank is used to transfer the sludge into the equipment. A funnel was used to assist the transfer of the tank waste sludge sample into the sludge receipt tank. The temperature of the tank contents can be controlled to between 25 and 110°C during chemical processing of the sludge. To reduce the effects of evaporation, vapors from this tank and the sludge settler are passed through a condenser and demister. The collected liquid is then allowed to drain back into the tanks.

The second processing tank is the sludge settler, C-201, which is approximately 10 cm in diameter and 1 m tall. The sludge settler is constructed of polysulfone, a transparent polymer that is resistant to boiling caustic and radiation. A ruler, visible from the cell window, was attached to the column so that 0 inches was near the top of the column and 36 inches (91 cm) was near the bottom. The ruler was used to observe the slurry/liquid interface level and to determine the total volume in the sludge settler. The slurry/liquid interface level is measured visually by back-lighting the column and observing the light/dark interface. The tank's temperature is controlled between 25 and 85°C by circulating water from a hot water bath through a cylindrical annulus surrounding the sludge settler. Penetrations through the top flange allow the insertion of the sample tube and the transfer of materials. The sample tube is mounted on a linear motion apparatus that enables the end of the tube to be placed at accurate depths within the sludge settler. Supernatant was then removed at these locations. This same sample tube is also used to pump supernatant out of the top of the column and into the bottom of the column enabling the sludge to be refluidized following a settling test. Once the sludge is refluidized, it can then be transferred back into the sludge receipt tank for continued chemical processing.

The dimensions of the settling column were based on an understanding of the effects of geometry on the sludge-settling characteristics. Small settling systems can provide an accurate measure of free and hindered settling that can be applied directly to larger systems if the sides of the settling column do not influence the settling rate. The column diameter must be large enough to prevent these wall effects. The column height also must be tall enough to allow accurate measurement of the sludge settling rates. Since large quantities of sludge are not readily available, the height and diameter must be balanced. Standard sludge settling methods for scale-up have used a 10-cm-diameter, 1-m-tall column (Greenberg 1992). To ensure that wall effects were indeed negligible for the sized column, a physical simulant (kaolin clay) and

a chemical simulant (C-106 simulant) were studied at Washington State University with 10- and 30-cm-diameter columns, both 1-m tall. Results showed no statistical difference between these two sizes of columns (Brooks et al. 1997).

The cold chemical tank, C-101, was used to store caustic (10 M NaOH) or inhibited water (0.01 M NaOH and 0.01 M NaNO<sub>2</sub>), which was used for the retrieval, solids resuspension, wash, and leach steps. This tank is in the operating gallery and is used with a metering pump for accurate measurement of the caustic and water added to the in-cell tanks.

The batch collection tank, C-301, and the supernate holding tank, C-302, were both used to store liquid separated from the solids by gravity settling

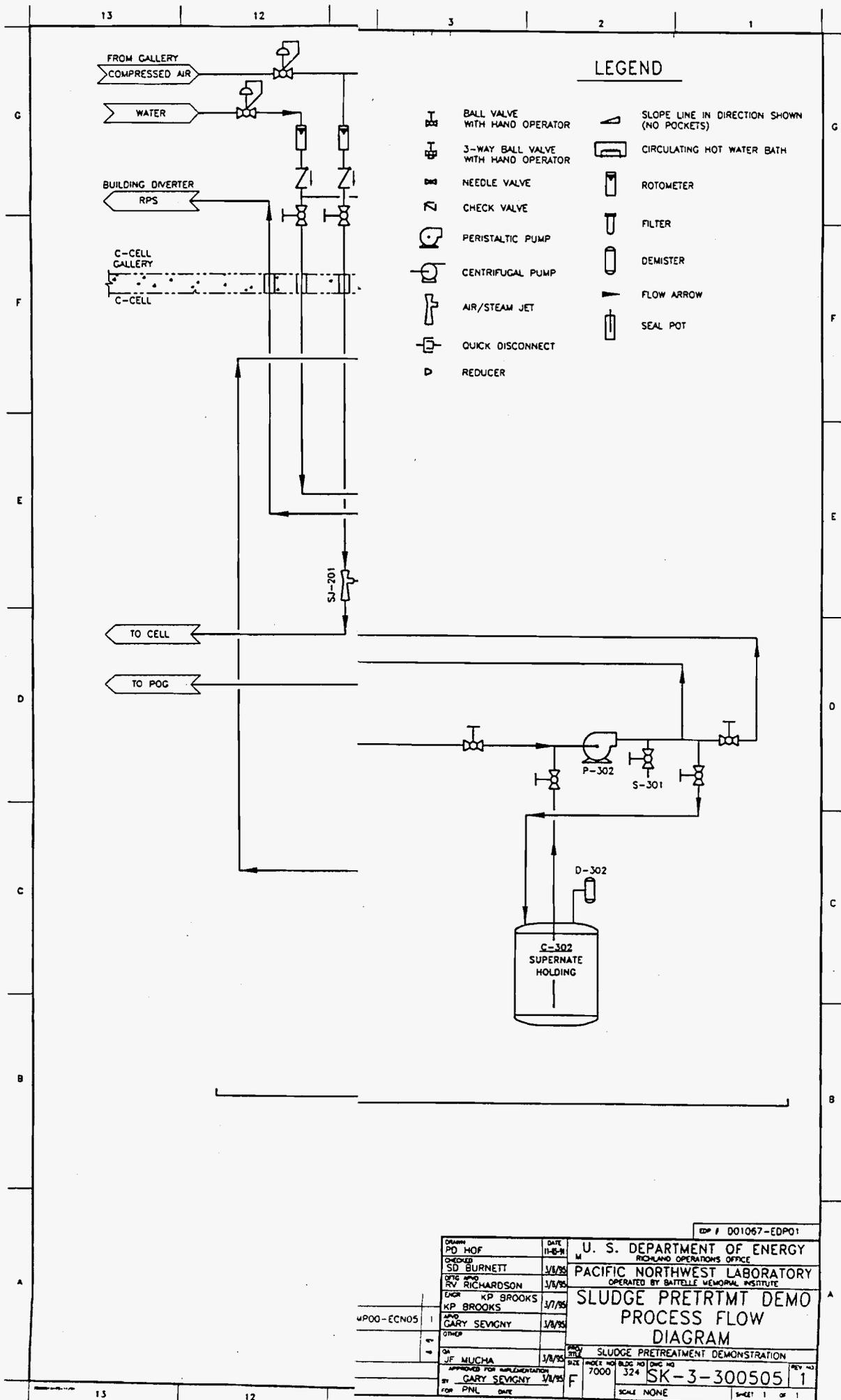
### **2.1.2 Pumps**

The pumps used to move slurry and liquid between processing stations were peristaltic pumps of various sizes. These pumps had a head that rotated against a flexible tube, thereby generating the pumping action. The pumps could be operated at any speed setting (within its range), in either forward or reverse direction, and in one of several modes. They can be set to pump at a given speed or flow rate, or to pump a set volume and shut off. The sludge was generally pumped at greater than 3.8 liter/min to prevent its settling during transfers. The supernatant, in contrast, was decanted from the settled sludge at 200 mL/min to avoid disturbing the settled sludge.

A small centrifugal pump is installed in the sludge receipt tank recirculation line. This pump attempts to simulate the shear experienced by the sludge particles during the retrieval step. These shears may break up agglomerates and reduce the particles' size, resulting in slower overall settling. This small centrifugal pump has a 3.34-cm-diameter impeller, which operates at 8000 rpm, creating a tip speed of 14 m/s, which is similar to tip speeds in the full-size mixer pumps. Although the shear profile inside a mixer pump is still very different than that in a small centrifugal pump, by matching the tip speed between the two pumps, the maximum shear should be similar.

### **2.1.3 Valves and Tubing**

The tanks and pumps described above are connected to each other through a network of stainless steel tubing mounted on a steel framework attached to a table. The table sits in a secondary containment pan on the floor in the 324 Building C-Cell. Outlets from each tank come from a dip tube at the bottom of the tank. The inlets are in the top of the tank. Valves in the tubing allow each tank and pump to be connected/disconnected from each other so the contents of the tanks can be transferred to other tanks, sampled, additions made, etc. All of the tubing fittings are stainless steel Swagelok fittings. All in-cell equipment has been designed or modified for operation with master-slave manipulators.



**LEGEND**

- BALL VALVE WITH HAND OPERATOR
- 3-WAY BALL VALVE WITH HAND OPERATOR
- NEEDLE VALVE
- CHECK VALVE
- PERISTALTIC PUMP
- CENTRIFUGAL PUMP
- AIR/STEAM JET
- QUICK DISCONNECT
- REDUCER
- SLOPE LINE IN DIRECTION SHOWN (NO POCKETS)
- CIRCULATING HOT WATER BATH
- ROTOMETER
- FILTER
- DEMISTER
- FLOW ARROW
- SEAL POT

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## 2.1.4 Instrumentation and Data Acquisition System

Liquid level and density were measured on the in-cell tanks using dip legs connected to differential pressure transducers. Temperature was measured using calibrated thermocouples. This instrumentation was connected to a data acquisition system in the C-Cell operating gallery. Results were monitored and saved throughout the test. Unfortunately, the dip legs in the sludge receipt tank became plugged during testing, and the density and liquid level could not be measured.

## 2.1.5 Photographic Data Recording

A video camera was mounted on a master slave manipulator in C-Cell during all of the settling tests. The camera was turned on before the start and turned off just after the end of the settling test. All of the settling data were thus recorded on videotape using time-lapse photography at one-sixtieth regular speed. This system also provided a means of checking what was seen through the cell window by looking at a monitor. This was helpful for distinguishing color and parallax error.

## 2.2 Gravity Settling and Sludge Washing Test

This section of the report summarizes the actual step-by-step activities conducted during the ESW and gravity settling test with the S-107 sample. The homogenization, simulated retrieval, two caustic leaches, three inhibited water washes, and an extended caustic leach were performed with material from core samples 105, 110, and 111 of S-107 sludge taken September 1995. The workplace copy of the operating procedures, including noted observations, and the laboratory record book (BNW 55983) contain the detailed description of the actual test activities.

### 2.2.1 Waste Sample Preparation

In February 1997, 11 containers of actual S-107 sludge waste were transferred from the 222-S Laboratory to the PNNL 325 Laboratory. A detailed description of sludge sampling history from Tank 241-S-107 is provided in a Tank Characterization Report (Simpson 1996). The S-107 samples were contained in wide-mouth glass containers and were stored in 325, a high-level radiochemistry facility (325A HLRF), for approximately 13 months before the enhanced-sludge-settling experiment. In April 1998, the S-107 sludge samples were removed from these containers and combined to prepare a homogeneous S-107 sludge sample for the settle decant experiment.

All 11 sludge samples were either dried or nearly dried out. A spatula was used to scrape and remove the S-107 sludge samples from the containers. The samples were transferred to a mixing vessel, and water was added to aid sludge mixing. A total of 205 grams of de-ionized water were added to the sludge. To homogenize the S-107 waste, an OMNI mixer and chamber assembly were used.<sup>(a)</sup> The mixing vessel was inserted in an iced water bath during the mixing process to minimize heating and water evaporation from S-107 sludge during sample homogenization. Since a large volume of S-107 sample was involved, and the homogenization was completed in several steps of homogenizing and blending of

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(a) The PNNL technical procedure number PNL-ALO-135 was used to homogenize the S-107 waste.

each of the composites. The composites were prepared from a primary mixing step to ensure a final uniformly homogenized S-107 sample for the ESW experiment.

Replicate samples from three locations inside the holding vessel were taken to determine the weight percent (wt%) of solids used as the starting solid mass fraction of S-107 waste in the enhanced sludge settling experiment after completing the homogenization process. Two additional samples were taken to determine the bulk density of the starting sludge. The wt% of solids was determined from the difference between the mass of each sample before and after drying in the oven at 105°C. The bulk density of samples was determined by placing the S-107 sample in a 15-mL graduated centrifuge cone and measuring the mass and volume of sludge sample after centrifuging the samples. Since the samples were very viscous, the samples were centrifuged at approximately 1000x g for 30 minutes to remove any entrapped air bubbles, which would affect the volume of sample. Following centrifugation, the total volume of sample in the centrifuge cone (centrifuged layer and supernatant layer) and the total mass of the sample in the centrifuge cone were used to determine the bulk density of samples. The wt % of solids for each sample replicate and their averaged value are presented in Table 2.1.

**Table 2.1.** Measured Solids Weight Percent and Bulk Density of S-107 Sludge Feed

Sample #	Solids Weight %	Sample #	Sludge Bulk Density (g/mL)
1	67.5	1	1.82
2	66.6		
3	68.3		
Average	67.5	2	1.81
Standard Deviation	0.84	Average	1.815
95% Confidence	1.8		

The reproducibility of the measured solids weight percent quantities in Table 2.1 suggests that the S-107 samples recovered from the containers were uniformly homogenized. The measured bulk densities were nearly identical which indicate that the sludge samples were completely packed in the graduated centrifuge cones upon centrifugation and any entrapped air was removed from the sludge samples.

## 2.2.2 Retrieval Step

An overview of the processing steps and target conditions is shown in Table 2.2 while the actual processing steps and operating conditions are shown in Figures 2.2 and 2.3. For the retrieval step, second caustic (CL2), and third water wash (WW3), two settling tests were performed targeting 5 and 10 wt% solids concentration. The target final caustic concentration for the first caustic leach (CL1) was 2 M NaOH, while the target was 3 M NaOH for the second caustic leach.

A total of 1005.4 g of S-107 sludge was transferred from the 325 Building to the 324 Building in a two-liter plastic container. Because the sludge was very viscous, water was added to the sample before it was transferred into the settle/decant equipment. After pouring, the sample container was rinsed, and the remaining inhibited water preventing corrosion (0.01M NaOH and 0.01M NaNO<sub>2</sub>) was transferred to create the targeted slurry concentration.

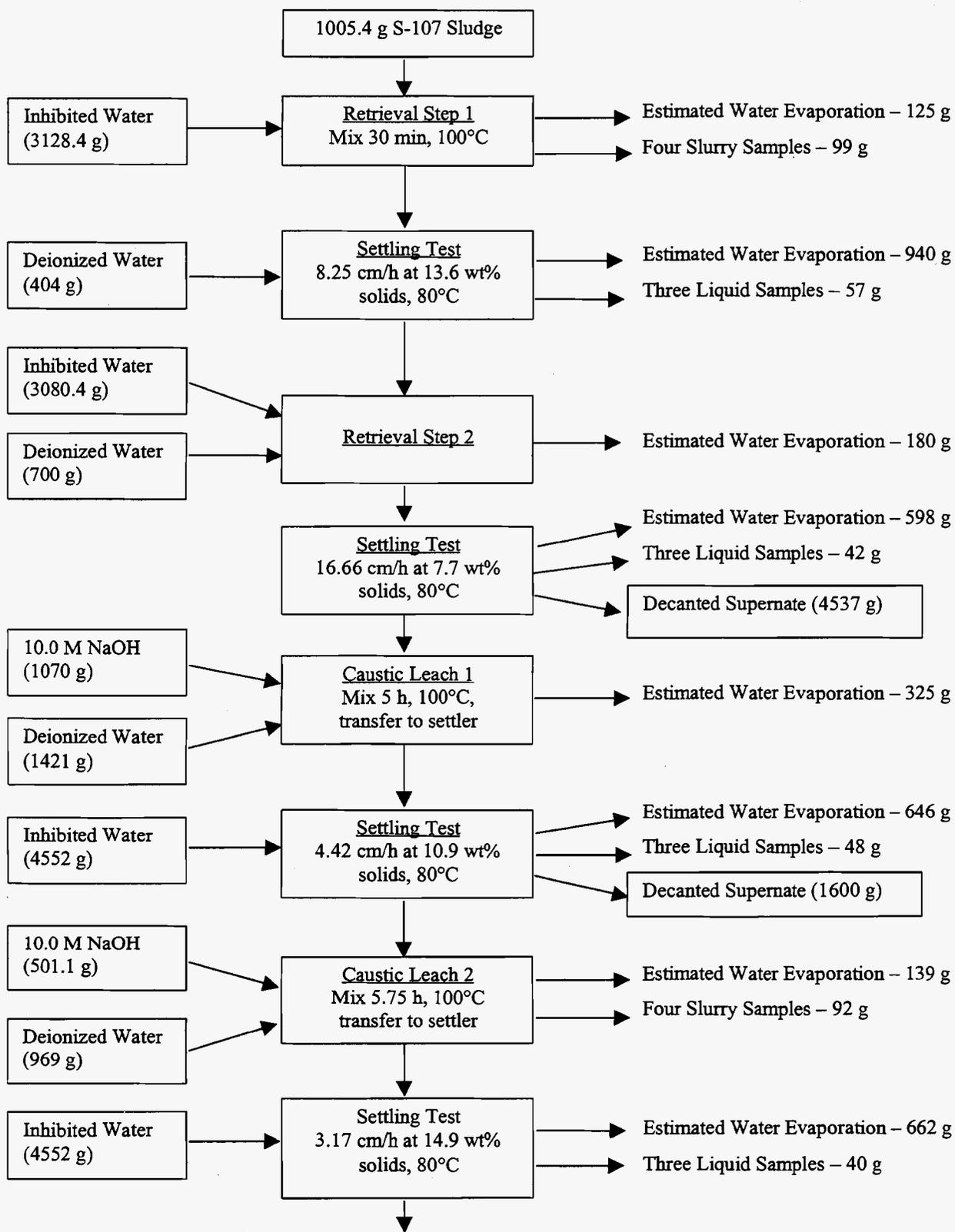
A summary of the enhanced sludge wash experiment, including each chemical addition and sample removal, is shown in Figure 2.2. Chemical additions used to meet the specified target solids

concentrations use either inhibited water or NaOH solutions. Deionized water was added to replace water lost during evaporation and the amount to be added was estimated throughout the run from available volume measurements when the slurry was pumped into the settling column. These water additions to replace water lost during evaporation were made after heating in the sludge receipt tank and during the settling tests. The settling rates shown in Figures 2.2 and 2.3 were measured as the maximum settling rate attained. The solids concentration, on the other hand, was not measured during the run and could only be estimated. The solids concentrations from samples taken after the retrieval, second caustic leach, and third water wash were measured after the testing was complete. These insoluble solids concentrations were obtained by drying the samples of both the mixed slurry and the filtered supernatant at 105°C to constant weight. Solids concentrations for each of the settling tests were calculated from these drying measurements, assuming no change in the mass insoluble solids between the first and second caustic leach nor between the first, second, and third water-wash steps.

**Table 2.2.** Summary of Target Processing and Settling Conditions for the S-107 Sample

Conditions	Retrieval	Caustic Leach	Water Wash	Extended Caustic Leach
Total Performed	1	2	3	1
Processing Solids Concentration	10%	5%	5%	5%
Final Caustic Concentration	Corrosion Inhibited Water	CL1 <sup>(a)</sup> --2M NaOH CL2 --3M NaOH	Corrosion Inhibited Water	3M NaOH
Processing Temperature	100°C	100°C	50°C	80°C
Processing Time	1 hour	5 hours	30 minutes	250 hours
Number of Settling Tests	2	3	4	1
Settling Solids Concentrations	10% and 5%	CL1 – 5% CL2 – 5% & 10%	WW1 & WW2 –5% WW3 – 5% & 10%	5%
Settling Temperature	80°C	80°C	50°C	80°C
(a) CL1 = First Caustic Leach; CL2 = Second Caustic Leach; WW1 = First Water Wash; WW2 = Second Water Wash; WW3 = Third Water Wash				

To ensure complete wetting and mixing of the sludge before the retrieval washes, the agitator was operated overnight. The slurry was then recirculated through a high-speed centrifugal pump for 30 minutes to simulate the shear from the mixer pumps during retrieval. Following this mixing, the slurry was heated at 100°C for 30 minutes. Four slurry samples were then taken.



**Figure 2.2.** Graphical Overview of the ESW Process

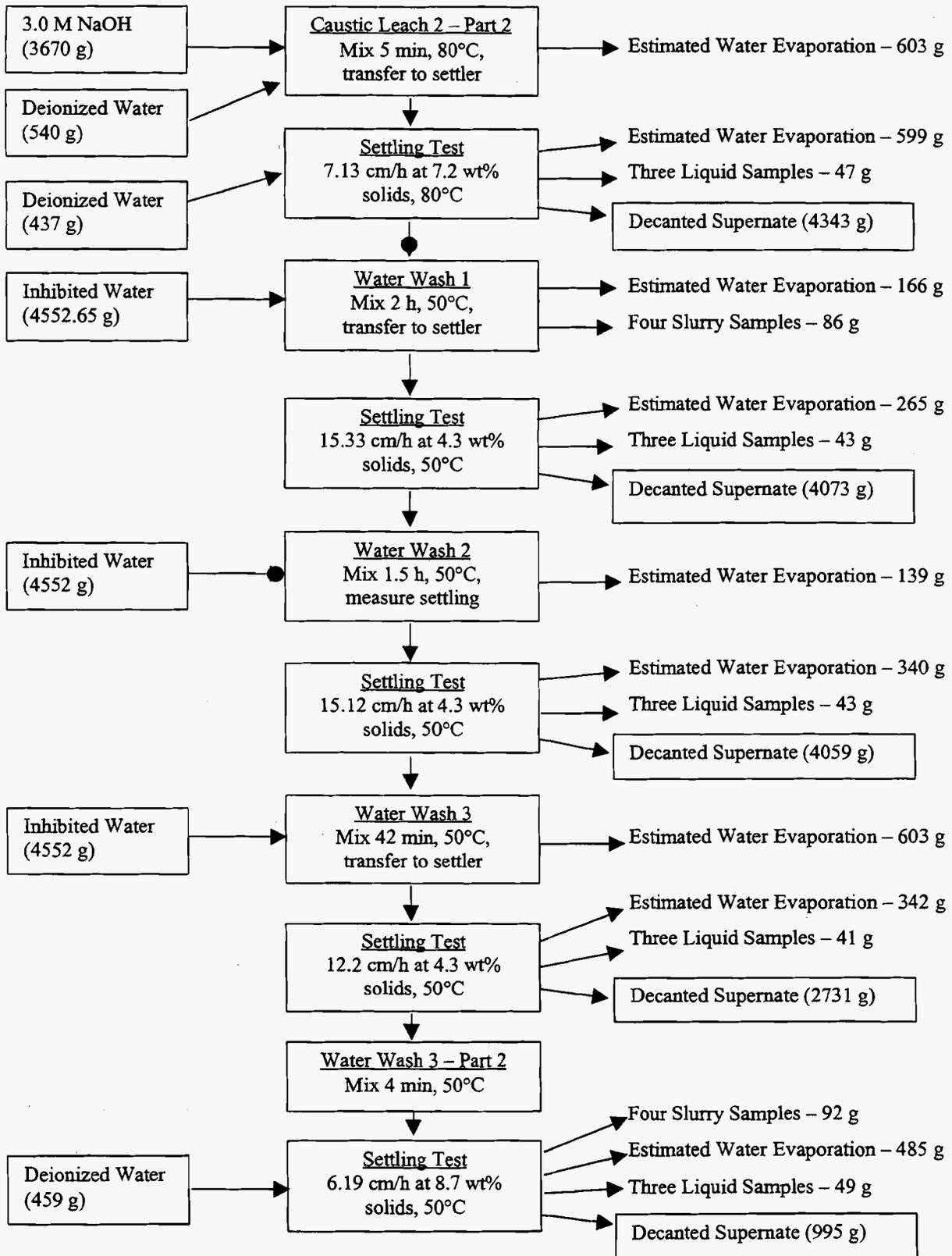


Figure 2.2. Graphical Overview of the ESW Process (Continued)

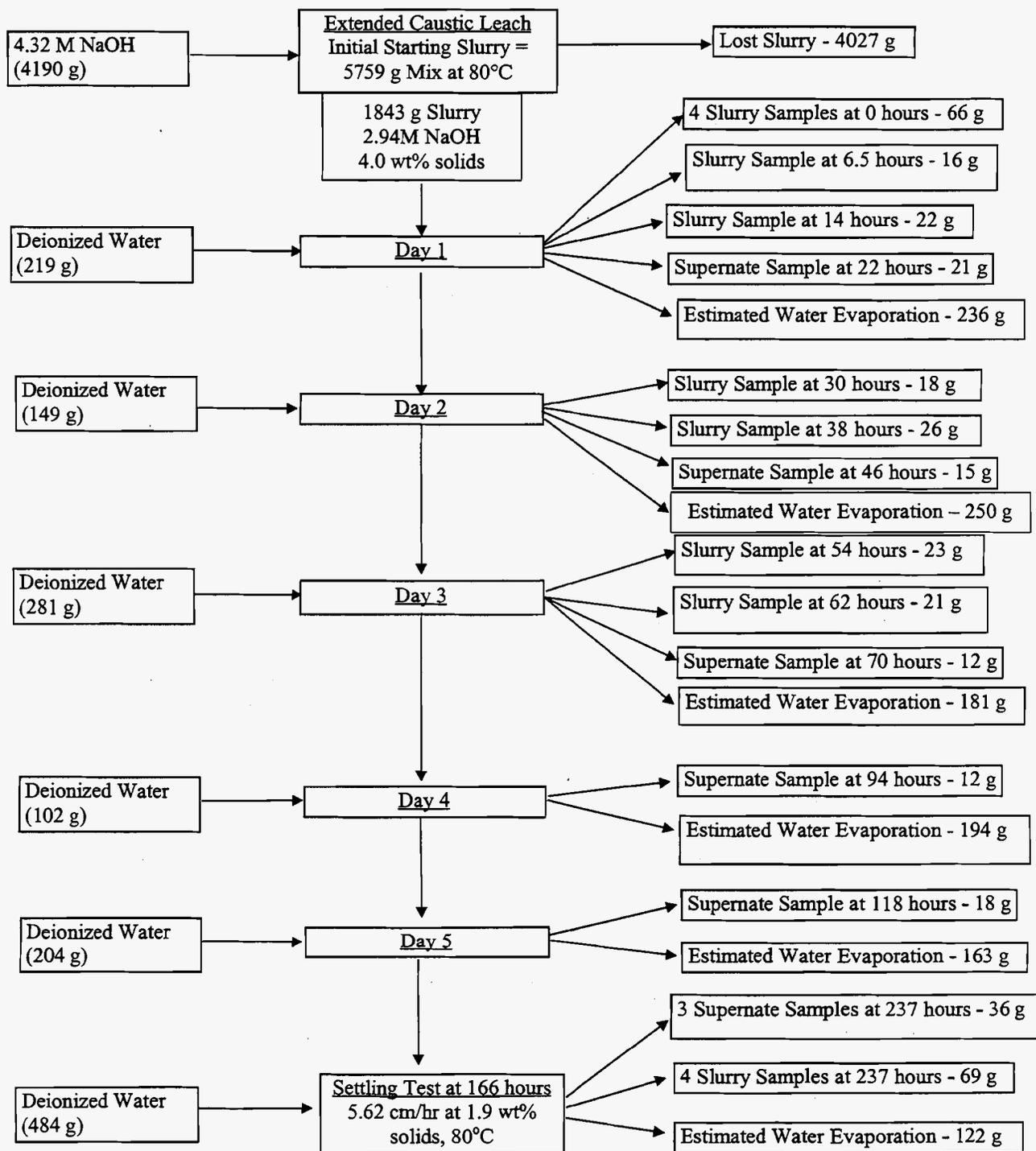


Figure 2.3. Graphical overview of the Extended Caustic Leach Process

During the retrieval step, two settling tests were performed. The first was at 13.6 wt% solids. Additional inhibited water was then added, the mixture was refluidized, and a second settling test was performed at 7.7 wt% solids. The settling rate was measured for both settling tests at 80°C. Following each settling test, three samples of supernate were taken: from 2.54 cm (1 in.) below the supernate surface, midway into the clear supernate layer, and 2.54 cm (1 in.) above the settled solids layer.

Following the sampling on the second settling test, the supernate was removed to within 1 cm of the settled solids layer and transferred into the batch collection tank. Deionized water was added to the settling column, and the sludge was fluidized by forcing liquid into the bottom of the column. The slurry was transferred back into the sludge receipt tank, and the first caustic leach procedure was begun.

### **2.2.3 First Caustic Leach**

Ten-molar sodium hydroxide was added to the sludge receipt tank targeting a 2M NaOH solution at 5 wt% insoluble solids (after leaching was complete). Since aluminum dissolution depletes hydroxide, the hydroxide concentration needed was determined based on the aluminum concentration in the Tank Characterization Report (1996) and the leaching studies of Lumetta et al. (1996). The actual free-hydroxide concentration after this leaching step was measured to be 1.98 M. The solids concentration was estimated to be 10.9 wt% insoluble solids.

The sludge was heated to 100°C and processed for 5 hours after which it was cooled to 80°C and transferred to the settling vessel. Deionized water was added to the slurry to replace the water that had evaporated. The settling was performed at 80°C, and the rate of drop of the solid/liquid interface was measured. Following the settling test, three supernate samples were taken similar to that described in Section 2.2.2. The supernate was decanted, and deionized water was added to help fluidize the mixture and transfer it back to the sludge-receipt tank.

### **2.2.4 Second Caustic Leach**

The second caustic leach was similar to the first caustic leach. A mixture was prepared targeting a 3 M NaOH final caustic concentration and a 5 wt% insoluble solids mixture. After heating at 100°C for 5.75 hours, samples of the slurry were taken. An analysis of these samples found that the final free-hydroxide concentration was 2.86 M. As with the retrieval step, two settling tests were performed, each at 80°C. The first settling test was at 14.9 wt% and then additional 3 M NaOH was added to make a 7.2 wt% insoluble solids concentration mixture. Settling rates were measured for each of these settling tests, and three samples of supernate were taken for each test. After the settling tests, the supernate was decanted and removed.

### **2.2.5 First and Second Water Wash**

Both the first and second water washes were performed as follows. Inhibited water (0.01 M NaOH/0.01 M NaNO<sub>2</sub>) was added to the compacted sludge in the column. The slurry was fluidized and transferred into the sludge receipt tank. The mixture was heated and agitated at 50°C and then transferred into the settling vessel, which was also controlled to 50°C. The insoluble solids concentrations for both these samples were estimated to be 4.3 wt%, based on the measured solids concentration from the second caustic leach and third water wash. The settling rate was measured based

on the interface height. Once settling was complete, three samples of supernate were taken, and the supernate was decanted.

### **2.2.6 Third Water Wash**

The third water wash was identical to the first and second water wash with two exceptions. First, four sludge samples were taken after this washing step. These provided a basis for the solids concentration for all three water washes. Second, rather than performing a single settling test at near 5 wt% insoluble solids, two settling tests were performed. The first was estimated to be 4.3 wt% and the second was measured at 8.7 wt%. To obtain the higher solids loading on the second settling test, supernatant was decanted to the required level and the slurry was refluidized and allowed to settle. After the settling tests, supernate samples were taken, and the supernate was decanted for continued processing.

### **2.2.7 Extended Caustic Leach**

The purpose of the extended caustic leach is to measure the kinetics of aluminum and chromium dissolution in an agitated 3 M NaOH solution at 80°C for an extended period of time. Samples were taken over the course of the experiment as shown in Figure 2.3. At the start and end of the experiment, slurry samples were taken to measure the start and end point of the metals concentration in the sludge. The experiment was performed primarily in the sludge receipt tank, allowing constant heating and agitation. Deionized water was added to replace liquid lost to evaporation. To determine the quantity of water to replace, each day the sludge was pumped into the settling column and the total slurry volume was measured. After allowing the mixture to settle for 3-5 hours, a supernate sample was taken for analysis.

It should be noted here that at the start of the experiment, tubing in one of the peristaltic pumps broke, and more than 4 kg of slurry mixture was lost. In spite of the loss, the experiment was continued with a smaller quantity of material. Since the mixture was homogenized before the spill, the smaller quantity of slurry would still be representative of the original mixture, and the experiment was continued. Therefore, the extended caustic leach described above used smaller quantities of sludge than in the ESW.

Supernate samples were taken from the settling column on the first, second, third, fourth, fifth, and tenth day. During the first 3 days of the extended caustic leach, samples were also taken every 8 hours. These samples were pulled directly from the sludge receipt tank to allow the sludge to continue to agitate during sampling. After 7 days of agitation in the sludge receipt tank, the slurry was transferred into the settling column to measure the settling rate. On the tenth day, the settling test was terminated, and the final supernate and slurry samples were taken to complete the experiment.

## **2.3 Chemical and Radiochemical Analyses**

As discussed in Section 2.2, slurry samples were taken after heating the slurry following the retrieval step, the second caustic leach step, and the third water wash step. Slurry samples were also taken before and after the extended caustic leach. For each of these steps, one 20-mL sample was taken for chemical and radiochemical analysis. Sample analysis was performed on a dried solids basis. The analyses performed on these samples are shown in Table 2.3 and include ICP-AES, TOC, AEA, GEA, and <sup>90</sup>Sr analysis

Supernatant samples were also taken during each stage of the ESW process. These samples were taken from the sludge settling column after the sludge was allowed to settle. While three samples were generally taken, due to funding limitations only one sample from each step was taken for chemical and radiochemical analysis. The samples analyzed were taken within 2.54 cm (1 in.) of the middle of the supernatant. Analyses performed on these samples are also provided in Table 2.3 and include inductively coupled plasma/atomic emission spectroscopy (ICP-AES), ion chromatography (IC), alpha energy analysis (AEA), gamma energy analysis (GEA), free hydroxide titration, and  $^{90}\text{Sr}$  analysis.

As described in Section 2.2, samples of both slurries and supernatants were taken as a function of time during the extended caustic leach test. An initial and final slurry sample was taken and analyzed to determine the composition of the sludge. The slurry samples taken during the extended caustic leach were centrifuged, and the liquid was decanted for analysis. The solids in these samples were discarded. Both the supernatant samples and the decanted slurry samples taken during the extended caustic leach were analyzed for ICP-AES. The initial and final supernate samples were also titrated for free hydroxide.

The major metallic elements were determined by ICP-AES. This method provides sufficient information to quantify the effects for each step of the ESW process on such elements as aluminum, phosphorus, chromium, iron, silicon, and sodium. The slurry samples were fused using KOH while the supernatant samples were acid digested using nitric acid.

Major soluble anions in the supernatants were determined by IC, including chloride, fluoride, nitrate, nitrite, sulfate, phosphate, and oxalate. Free-hydroxide concentration was measured on the supernatant samples using titration. This provided a means of comparing the quantities of caustic added during the leaching process and removed during the washing steps. Total inorganic carbon and total organic carbon (TIC/TOC) were also provided for the initial and final sludge samples.

Radionuclide analysis included AEA for measuring concentrations of alpha-emitting TRU elements and chemical separations followed by beta emissions counting for  $^{90}\text{Sr}$ . GEA was performed to measure the gamma-emitting isotopes, including  $^{137}\text{Cs}$ ,  $^{60}\text{Co}$ ,  $^{241}\text{Am}$ ,  $^{154}\text{Eu}$ , and  $^{155}\text{Eu}$ . Established PNNL Analytical Chemistry Laboratory procedures were used for all analyses performed, with the exception of oxalate IC analysis.

**Table 2.3. Analyses Performed on Sludges and Supernatants for the Enhanced Sludge Wash**

<b>Enhanced Sludge Wash</b>		
<b>Samples Taken After ...</b>	<b>Sample Type</b>	<b>Analyses Performed</b>
Retrieval Settling	Supernatant	ICP-AES
First Caustic Leach Settling		IC
Second Caustic Leach Settling		AEA
First Water Wash Settling		GEA
Second Water Wash Settling		<sup>90</sup> Sr
Third Water Wash Settling		OH <sup>-</sup> Titration
Retrieval Wash Step	Slurry	ICP-AES
ESW Process		TOC/TIC
		AEA
		GEA
		<sup>90</sup> Sr
<b>Extended Caustic Leach</b>		
<b>Samples Taken After ...</b>	<b>Sample Type</b>	<b>Analyses Performed</b>
6.5 hours	Slurry	Decant supernatant, discard solids and perform ICP-AES and OH <sup>-</sup> Titration on liquid
14 hours	Slurry	Decant supernatant, discard solids and perform ICP-AES on liquid
30 hours		
38 hours		
54 hours		
62 hours		
22 hours	Supernatant	ICP-AES
46 hours		
70 hours		
94 hours		
118 hours		
237 hours	Supernatant	ICP-AES OH <sup>-</sup> Titration
Initial Caustic Addition	Slurry	ICP-AES
Completion of Extended Caustic Leach	Slurry	ICP-AES

## 2.4 Radioactive Colloids Analysis

The physical characteristics of the S-107 waste were measured on slurry samples taken from the retrieval step, the second caustic leach step, the third water-wash step, the initial extended caustic leach, and the final sludge step of the extended caustic leach. The location of slurry samples from various steps of the enhanced sludge settling testing flowsheet was indicated in Figures 2.2 and 2.3. Physical characterization of the S-107 samples included the wt% of insoluble and soluble solids, slurry-bulk density and supernatant-density measurements, laboratory-scale settling rates, compressive strength, and sludge compaction.

To conduct these measurements, 20-mL scintillation vials containing slurry samples were transferred to 325 Shielded Analytical Laboratory. A list of these samples and their sludge settling process step is provided in Table 2.4.

**Table 2.4.** S-107 Slurry Sample Identity and their Sludge Settling Process Step

Settle Decant Process Step	Total Mass and Number of Scintillation Vials
Retrieval Step	63.55 g; 3 Scintillation Vials
Second Caustic Leach Step	156.87 g; 7 Scintillation Vials
Third Water Wash Step	49.58 g; 3 Scintillation Vials
Initial Extended Caustic Leach Step	32.29 g; 2 Scintillation Vials
Final Sludge Step	69.04 g; 3 Scintillation Vials

**Total Weight % of Solids in Slurry:** To determine the total wt % of solids (soluble and insoluble solids) in the slurry, all the scintillation vials associated with each settle decant step (see Table 2.3) were sub-sampled. Each scintillation vial was thoroughly homogenized, and approximately 5 grams of sub-samples were transferred to each of the three replicate drying containers. For the initial extended caustic leach material, only two replicate samples were prepared since not enough sample was available to perform all the analyses. The drying containers were dried at 105°C, and the total wt% of solids in slurry was determined from the difference between the mass of each sample before and after drying. The dried samples were saved for dried powder density.

**Solid Density:** The Micromeritics AccuPyc 1330 pycnometer was used to measure the volume of dried samples by measuring the amount of displaced gas. The pressures observed upon filling the sample chamber with ultra-high-purity helium and then discharging it into a second empty chamber allow computation of the dried-sample volume. Since a limited amount of dried sample was available, a sample chamber of 1 cc was used to maximize the experimental accuracy. The dried-solid density was measured in duplicate on dried samples from each settle-decant process step.

The dried samples from the total wt% solids measurements were used for the solid density experiment. After completing the total-wt%-solids measurements, dried sample from the replicates of each settle-decant process step were removed from the scintillation vials, and a composite sample for each settle-decant process step was prepared. A weighed portion of each composite dried sample was placed in the pycnometer, and the volume was measured. The dried-solid densities were then calculated by dividing the mass of dried solids by the measured volume. The volume for each sample was measured in duplicate.

The pycnometer was calibrated before measuring the samples, and a calibration check was made at the end. The calibration check indicated that the calibration moved <0.0008 mL during the sample measurements. A calibration check was also completed on the balance at the beginning and end of the experiment.

**Gravity Settling and Centrifugation Studies:** The remaining slurry samples for each process step were combined to prepare five composite slurry samples. Using these composites, two or three replicate slurry samples were prepared by transferring 10 mL of thoroughly homogenized slurry into 15 mL graduated centrifuged cones. All the centrifuged cones were filled to 10 mL of slurry to compare the results between process steps. The centrifuged cones were capped, and the slurries were allowed to settle for about 24 hours under gravity. The settling experiment was monitored by recording the time and

the volume of settled solids. After completing the settling experiment, all the centrifuged cones were loaded into the centrifuge at the same time and centrifuged several times by incremented steps in the rotational velocity followed by sludge-height measurements. For each rotational velocity increment, the samples were centrifuged for 30-minute intervals. The centrifugation experiment was completed by performing a total of five rotational speeds of approximately 500, 650, 900, 1100, and 1500 rpm.

**Particle Size Distribution:** A Leeds and Northrup Microtrac X100 Particle Size Analyzer was used for these analyses. This device can measure particle and agglomerate diameters between 0.12 to 704 microns. The analyzer works by analyzing light scattered by the particles in a dilute suspension. The amount and direction of the light scattered by the particles is measured by an array of optical detectors and then analyzed to determine the size distribution of the particles.<sup>(b)</sup> To measure PSD, a sample is added manually to a sample reservoir. It mixes with the re-circulating fluid so that a stream of well dispersed particles passes through the sample cell for analysis.

The recirculating fluid was prepared for each process step by reproducing the solution of similar electrolyte molarities in sodium hydroxide as the actual supernatant. A 0.1 M sodium nitrate concentration was used in the recirculating fluid makeup as a qualitative estimate of the soluble species concentration. In Table 2.1, the recirculating fluid used for the PSD analyses are compared with the actual supernatant concentrations.

**Table 2.5.** Concentration of Electrolyte and Soluble Species for PSD Analysis Recirculating Fluid and S-107 Supernatant

Settle Decant Process Step	Recirculating Fluid for PSD	Measured S-107 Supernatant Concentration
Retrieval Step	0.53 M NaOH; 0.1 M NaNO <sub>3</sub>	0.53 M NaOH; 3 g/L NO <sub>3</sub> <sup>-</sup>
Second Caustic Leach Step	1.75 M NaOH; 0.1 M NaNO <sub>3</sub>	1.75 M NaOH; 0.3 g/L NO <sub>3</sub> <sup>-</sup>
Third Water Wash Step	0.046 M NaOH; 0.1 M NaNO <sub>3</sub>	0.046 M NaOH; 0.4 g/L NO <sub>3</sub> <sup>-</sup>
Initial Extended Caustic Leach Step	1.96 M NaOH; 0.1 M NaNO <sub>3</sub>	1.96 M NaOH
Final Sludge Step	1.96 M NaOH; 0.1 M NaNO <sub>3</sub>	N/A

Samples for the particle-size analysis were extracted after the slurry samples for each process step were combined, and a well homogenized slurry feed stock explicit to each settle decant process step was prepared. A small amount (approximately 5mL) of slurry was transferred into the reservoir of the particle-size analyzer to produce a suspension with sufficient particles to make an accurate determination of the PSD. The PSD analyses were performed in duplicate for each settle decant process step. Each sample was analyzed after applying a variety of circulation time, circulation flow rate, and sonication treatments. The treatments in successive order included 1) circulation at 40 mL/s and PSD analyses after 1, 5, and 10 minutes of total circulation time, 2) circulation at 60 mL/s and PSD analyses after 15 and 20

(b) The instrument combines low-angle laser light scattering (LALLS) with 90 degree scattering at three different wavelengths and orthogonal polarities. This combination will extend the size range to a lower size than is usually used for forward light scattering alone. The forward light scattering and Fraunhofer theory are used to analyze particles coarser than 2 microns. The Mie theory and 90-degree scatter are used for smaller-sized particles.

minutes of total circulation time, 3) circulation at 60 mL/s with 40 W sonication for 90 seconds, and 4) circulation at 40 mL/s with 40 W sonication for 90 seconds.

The instrument performance was checked with two NIST traceable standards from Duke Scientific Corporation.

**Supernatant Density:** After the final centrifugation increment, the supernatant layer was decanted and filtered to remove any residual solid particles from the supernatant. The density of supernatant was determined by extracting a 1.0-mL volume of supernatant using a pipette and measuring its mass.

**Weight% Soluble Solids in Supernatant:** Two or three replicates of approximately 8 mL of filtered supernatant samples for each settling step were dried at 105°C. The wt% of soluble solids in the supernatant was determined from the difference between the mass of each sample before and after drying in the oven.

**Calculated Weight % of Insoluble Solids in Slurry:** The wt % of insoluble solids in the slurry was calculated from the wt% of total solids (soluble and insoluble solids) in slurry, the wt% of soluble solids in supernatant, and the ratio of soluble solids to water in the supernatant.

## 2.5 Theoretical Analysis

The sedimentation rates of a liter-scale process are useful only if they can be extrapolated to large production-scale systems. A transient sedimentation model has been developed that incorporates the primary features necessary to accurately predict the sedimentation behavior of a tank-waste settling column. Both the sedimentation test results and the analytical data are used to determine the appropriate coefficients for each stage in the ESW procedure.

Hanford tank waste contains particles of widely varying size and composition. The smallest particles are less than a micron ( $\mu\text{m}$ ) in diameter and may exhibit colloidal behavior, while the largest particles may be hundreds of microns in diameter. Depending on the chemistry of the solution, the small colloidal particles may aggregate to form large porous flocs. The rate of sedimentation of each individual particle or floc depends on its size and relative density. When sedimentation begins, the large, dense particles and flocs quickly settle to the bottom. Therefore, the small particles and low-density flocs control the rate of sedimentation.

Samples such as those used in this work, which have a relatively high solids loading, exhibit a sharp, well-defined interface that appears almost immediately between the clear supernatant liquid and an opaque region that contains the suspended solids. For such suspensions, the sedimentation velocity is monitored by noting the position of the interface as a function of time. As the sediment settles, the sediment layer becomes thinner, and the average solids loading in the sediment layer increases until the sediment compresses to its equilibrium solids-loading profile.

A settling curve is the height of the slurry-liquid interface as a function of time. In cases with relatively high loading, it can be divided into two regions, the hindered settling region and the compaction region. The hindered settling region is more or less a straight line followed by an asymptotic slowing of the settling during compaction. The compaction continues until an equilibrium is reached. The point where the hindered settling slows to compaction is known as the gel point ( $\phi_g$ ). It occurs when the volume fraction of the solids ( $\phi$ ) is sufficiently high that the agglomerates form a network. The

suspension then can take on the form of a solid structure. Compressive stresses on the system can be transmitted via the network, and the structure can then, at least partially, support itself. In this case, the compression rate of the sediment is controlled by a combination of the hydrodynamic drag of the interstitial fluid being squeezed out of the network and particle bonds breaking and re-forming as the agglomerates are being crushed by the weight of the sediment above.

The computational sedimentation model predicts the solids density profile as a function of time, based on information obtained from both settling experiments and laboratory tests of the suspensions of interest. From this information, we can derive the height of the sediment as a function of time. To be accurate, this model must reflect the two major aspects of the sedimentation process as follows:

- *Hindered Settling* – The settling rate of a suspension for a given particle distribution and solution chemistry depends only on the local solids loading. The rate is independent of the overall dimensions of the system. For example, if a 5-wt% particle suspension settles at 5 cm/h in a liter-scale column (assuming no wall effects), it will settle at 5 cm/h in a full-scale tank until it reaches the sediment layer. An expression must be developed that relates local solids loading to the hindered settling rate.
- *Sediment Compression* – As the total solids loading per unit area increases, the final height of the sediment increases. However, as the additional weight of solids is added, the sediment is compressed, resulting in a higher average solids density in the sediment. An expression must be developed that relates the local solids density to the compressive force on the sediment.

### 2.5.1 Hindered Settling

In the hindered settling region, the solids concentration is below the gel point,  $\phi_g$ , for that suspension, and the particle agglomerates interact only through hydrodynamic forces. The velocities,  $u$ , of the agglomerates in this region were taken from Buscall and White (1987) as

$$u = \frac{u_0(1-\phi)}{r(\phi)} \quad (2.1)$$

where  $u_0$  is the Stokes settling velocity at infinite dilution, and  $r(\phi)$  is a dimensionless hydrodynamic interaction parameter. The term  $(1 - \phi)$  results from the fact that the volume displacement of downward-flowing solids must be compensated by an equal upward volume flow of solution. The term converts the relative velocity of solids to solution into a reference-frame velocity. The Stokes settling velocity,  $u_0$ , for solid spherical particles is given by the expression

$$u_0 = \frac{2a^2 \Delta\rho g}{9\eta_s} \quad (2.2)$$

where  $a$  is the particle radius,  $\Delta\rho$  is the solid-liquid density difference, and  $\eta_s$  is the solution viscosity. For particle agglomerates containing many primary particles, the radius becomes the effective agglomerate radius, and the density is given by the relative density of the agglomerate. The hydrodynamic interaction parameter,  $r(\phi)$ , can take many forms, but one possible expression is

$$r(\phi) = \left(1 - \frac{\phi}{\phi_{ref}}\right)^n \quad (2.3)$$

where  $\phi_{ref}$  is a reference volume fraction. For particle agglomerates, the value for  $\phi_{ref}$  must be greater than the gel point for that system.

Using experimental hindered settling data over a range of solids volume fractions, ( $\phi$ ), the parameters  $u_0$ ,  $\phi_{ref}$ , and  $n$  can be determined. The Stokes settling velocity expression generally is applied to monodispersed particle-size systems. However, since the interface height is controlled by the settling rate of the smallest particle size, these equations can also be applied to the polydispersed sludges studied in this work.

## 2.5.2 Sediment Compression

As discussed above, when the particle volume fraction is sufficiently high, a network of connected aggregates forms, and the suspension takes on the form of a solid structure. In particular, compressive stresses on the system can be transmitted via the network throughout the system, and the structure then possesses the ability to support itself. As the network pressure,  $P$ , is increased, either mechanically with a piston or through gravitation forces, the network structure will resist further compression until the forces become strong enough that the structure begins to deform irreversibly. This network pressure at any vertical location is the relative weight per unit area of the sediment above that location. The relative weight, in turn, is calculated by multiplying the integral of the volume fraction of solids above the location of interest by the acceleration of gravity,  $g$ , and by the difference between the solid and liquid densities.

The compressive yield stress,  $P_y(\phi)$ , is defined as the value of the network pressure at which the flocculated suspension at volume fraction,  $\phi$ , will no longer resist compression elastically and will start to yield and irreversibly consolidate. The compressive yield stress is an implicit function of many variables, including the size, shape, composition, and relative number of particles involved and the interparticle forces (which, in turn, depend on the solution chemistry). At concentrations less than the gel point, the aggregates are not connected and act as independent units. At the gel point, these aggregates become interconnected throughout the container to the extent that they are able to support a load. At concentrations greater than the gel point, the compressive yield stress is typically modeled using a power law curve of the following type:

$$P_y(\phi) = c \left[ \left( \frac{\phi}{\phi_g} \right) - 1 \right]^m \quad \phi > \phi_g \quad (2.4)$$

with  $m$  varying between 4 and 10 (Landman et al. 1988).

The parameters  $c$  and  $m$  for the power-law curve may be determined using equilibrium sediment-height data when the network pressure ( $P$ ) is equal to the compressive yield stress  $P_y(\phi)$ . The only data required are the solid and liquid densities, the overall weight or volume percent of particulate solids in the sediment, and the final sediment height. The primary disadvantage of relying only on standard sediment-height data is that the range is limited by the heights of the test columns used, which are typically much smaller than the full-scale applications that we wish to model.

The range of sediment compression data can be extended by measuring the sediment heights of samples that have been centrifuged at different speeds. For these centrifuge tests, the compressive yield stress is based on the integral of the relative artificial weight of the solids created by the centrifuge at each location in the sediment. These data, together with the equilibrium gravity sedimentation data, are used to determine the expression parameters. A computer program has been written to optimize the power-law parameters ( $c$ ,  $\phi_g$ , and  $n$  or  $m$ ) by performing a least-squares fit based on the sediment heights using a simulated annealing approach. The exponents are restricted to the ranges specified above.

### 2.5.3 Overall Sedimentation Model

The sedimentation model divides the system into two regions. In the upper region, the solids concentration is below the gel point,  $\phi_g$ , for that suspension, and the particle agglomerates interact only through hydrodynamic forces. The velocities of the solids in this region are expressed in Equation (2.1).

In the lower region, the solids concentration is above the gel point,  $\phi_g$ , and the particle agglomerates interact through both hydrodynamic forces, represented by  $r(\phi)$ , and solid network pressure,  $P$ . The velocities of the agglomerates in this region are given by the expression taken from Buscall and White (1987)

$$u = \frac{u_0(1-\phi)}{r(\phi)} \left( 1 + \frac{\partial P / \partial z}{(\Delta\rho g\phi)} \right) \quad (2.5)$$

where  $P$  is the network pressure at elevation,  $z$ , and the term  $\Delta\rho g\phi$  is the change in gravitational head per unit elevation. Note that for regions that have no network pressure, the last term is zero. For sediments that have reached equilibrium, the change in network pressure is equal to the negative of the change in gravitational head, resulting in a net velocity of zero.

In Equation (2.4), we described the compressive yield stress,  $P_y(\phi)$ , of a suspension. If the network pressure,  $P$ , is less than or equal to the compressive yield stress, the network is strong enough to support the weight of the sediment, and no change occurs. However, when the network pressure exceeds the compressive yield stress, the network consolidates irreversibly until the volume fraction,  $\phi$ , increases to the point where the yield stress equals the network pressure. This changes at a rate controlled by the dynamic compressibility,  $\kappa(\phi)$ . The network velocity is controlled by the expression from Buscall and White (1987)

$$\frac{\partial u}{\partial z} = \frac{\kappa(\phi)}{\phi} [P - P_y(\phi)] \quad P \geq P_y(\phi) \quad = 0 \quad P < P_y(\phi) \quad (2.6)$$

When Equation (2.5) is substituted into this expression, we obtain a second-order differential equation for the network pressure,

$$\frac{\partial}{\partial z} \left[ \frac{(1-\phi)}{r(\phi)} \left( 1 + \frac{\partial P / \partial z}{\Delta\rho g\phi} \right) \right] = \frac{\kappa(\phi)}{u_0} [P - P_y(\phi)] \quad (2.7)$$

where the right-hand term is zero when  $P < P_y(\phi)$ .

A computational model has been developed that combines the hindered settling model, the network pressure model, and the aggregation kinetics model (when appropriate) to predict the sedimentation behavior of suspensions. Both time and elevation are discretized using a finite-difference formulation. The following procedure is followed for each time step:

- The total solids volume fraction,  $\phi$ , is calculated at each elevation based on a measured value of the overall mixture and mass balance equations. If the total solids volume fraction exceeds the gel point ( $\phi > \phi_g$ ), the node is considered part of the sediment layer. The elevation node that represents the top of the sediment layer is located. This divides the system into two regions.
- In the upper region, the hindered settling velocities are calculated by using expressions of the form presented in Equation (2.1). The velocities are used to calculate solids transport from one elevation to another using an upwind-differencing formulation. Because this term is explicit in time, the time step,  $\Delta t$ , is restricted by the Courant limit (Anderson et al. 1984),

$$\Delta t = \frac{\Delta x}{u} \quad (2.8)$$

- In the lower region, the network pressure is calculated using Equation (2.7). The network pressure at the top of the sediment layer is assumed to be zero. The network values allow the sediment velocity at each elevation to be calculated using Equation (2.6), which is also subsequently applied to the calculation of the solids transport for that time step.

The unknown parameters that must be defined to use this model for any particular suspension are the Stokes settling velocity ( $u_0$ ), the compressive yield stress,  $P_y(\phi)$ , the hydrodynamic interaction parameter,  $r(\phi)$ , and the dynamic compressibility,  $\kappa(\phi)$ . Separate values for these parameters were determined for the retrieval, caustic leach, and water wash steps. All of the settling test and centrifuge data from each of these ESW steps were combined to determine these parameters. The transient model was then iteratively solved, and the parameters were adjusted to minimize the error between these data and the model results.

## 3.0 Results and Discussion

The results of the bench-scale enhanced sludge washing, extended caustic leach, and settling tests are discussed below in Sections 3.1, 3.2 and 3.3, respectively. The results of the Radioactive Colloids Laboratory are described in Section 3.4. The results of a model that uses the results from Sections 3.3 and 3.4 to predict the settling of S-107 sludge at full scale are presented in Section 3.5.

### 3.1 Results of Enhanced Sludge Wash

Results of the caustic leaching and washing experiments performed on S-107 sludge are presented in the following sections. Tables 3.1, 3.2, 3.3, and 3.6 present the effects of the partitioning/leach and wash efficiency measured for the enhanced-sludge-wash experiments on the nonradioactive components of the sludge. Tables 3.4, 3.5, and 3.7 provide the analogous results on the sludge radionuclides. Table 3.8 provides a comparison between the laboratory-scale ESW test (Lumetta et al. 1996) and these liter-scale results. Similar to the work by Lumetta et al., the values in Tables 3.1 to 3.7 describe the mass of each component dissolved in that particular step and account for interstitial liquid remaining from previous steps.

Tables 3.1, 3.2 and 3.4 provide the concentrations of the nonradioactive and radioactive analytes in each of the process streams. These were determined by laboratory analysis. The mass of analyte dissolved in each process step is also presented in these tables. As previously mentioned, these masses have been corrected for interstitial liquid carried over from previous steps. Tables 3.3 and 3.5 display how the analytes are distributed between the process streams. The percentages shown are the mass of analyte found in each stream divided by the sum of the masses of the analyte for all streams. Finally, Tables 3.6 and 3.7 show the mass recoveries that can be represented as follows:

$$Recovery = \frac{Analyte_{InitialSludge}}{\sum Analyte_{Leached} + Analyte_{Residue}} * 100 \quad (3.1)$$

The mass recovery is the mass of analyte present in the initial sludge compared to the mass of analyte removed in each process step plus that which remains in the sludge residue.

Al, Cr, P, Na, and Si are the five most significant non-radioactive analytes present in the sludge that are removed by the ESW process. The remaining analytes presented in the tables are provided for completeness, but will not be discussed. Three primary observations were made: 1) nearly half of the chromium and phosphorus were removed during the retrieval step using no more than inhibited water, 2) the larger fraction of Al, Cr, and P was removed during the first caustic leaching; there was, however, still a significant quantity of these analytes removed by the second caustic leach, and 3) the free hydroxide concentration was approximately 2 M in the first caustic leach and 3 M in the second caustic leach. The higher caustic concentration used in the second caustic leach may have aided in the further removal of each of these analytes. During the water washes, a small fraction of these analytes was removed.

As discussed previously, the TWRS O&UP assumes that 91, 86, and 95% of the Al, P, and Cr would be leached from the SSTs, respectively. These are compared to the values achieved in the S-107 liter-scale leaching of 48.4, 81.2, and 77.0% for Al, P, and Cr, respectively. None of these values were

achieved during the ESW of S-107. While the P and Cr are reasonably close to the target values, Al falls significantly short of this target. This comparison is for reference only and does not necessarily indicate that future processing campaigns will fail to meet their objectives.

The recoveries for Al, Cr, and P are all reasonably good. Al is less than 100% with 94% recovered while Cr and P are over 100% at 108 and 104%, respectively. Because of the large addition of NaOH during the caustic leaching process, the Na recovery is difficult to measure, and the Na recovery and removal estimates are relatively poor. No total organic and inorganic carbon measurements were made during the ESW. Only an initial and final sample were measured. Thus, the 38 and 37% recoveries in this case indicate the fraction of total organic and inorganic carbon, respectively, that were removed during the ESW.

As would be expected, the primary radionuclide leached during the ESW is  $^{137}\text{Cs}$ . More than half of it was removed during the initial retrieval step. The first caustic leach removed an additional 11%, but 34% of the Cs was not removed during the ESW process. As would be expected, the TRU isotopes (Pu, Am, and Cm) as well as the  $^{90}\text{Sr}$  were not significantly removed during the ESW process.

A comparison of the results from the laboratory-scale ESW process from Lumetta et al. (1996) to that done on the liter-scale in this study are shown in Table 3.8. In the work done by Lumetta et al., only 8.4 grams of sludge were used as compared to 1005 grams used in this study. The Lumetta work did not perform a retrieval step on the sludge before the caustic leaches. Thus, the results of the retrieval wash were not compared to the results of Lumetta et al.

## 3.2 Results of Extended Caustic Leach

As described in Section 2.2.7, following the ESW, the sludge was leached a third time with 3 M NaOH over an extended time to measure the leaching kinetics of various constituents. The amounts of the non-radioactive concentrations (in  $\mu\text{g/mL}$ ) and the mass of these constituents (in  $\mu\text{g}$ ) in the supernatant solutions are presented in Tables 3.9 and 3.10, respectively. The initial wt% of the undissolved solids at the beginning of the extended caustic wash was measured to be 4.0%. The wt% of undissolved solids at the end of each sampling time, which was needed to calculate the mass of the supernatant, was then calculated based on the initial measured wt% minus the amount of  $\text{Al}(\text{OH})_3$  that was dissolved into the solution during that sampling period. It was assumed here that only the Al dissolved in the supernatant makes a significant contribution to the total weight of the undissolved solids. The percent of the non-radioactive constituents recovered into the supernatant solutions based on the measurement of the total solids concentration at the beginning of the extended caustic leach stage is listed in Table 3.11. The data in Table 3.11 for the two major constituents, namely Al and Cr, are schematically represented in Figures 3.1 and 3.2, respectively.

From the data, it can be seen that the laboratory-scale ESW process was more efficient in the removal of Al, P, Si, and  $^{137}\text{Cs}$  as compared to this work. This is especially significant in the case of the Al, where more than 50% of the Al remained in the sludge leached in this study as compared to 27% in the work of Lumetta, et al. In contrast, the Cr-removal efficiencies were higher in this study than that of Lumetta et al. Similar results have been seen in the C-106 and C-107 studies (Brooks et al. 1996; Brooks et al. 1997). In both the laboratory-scale and the liter-scale studies, more Si was removed during the second caustic leach (at higher NaOH concentration) than during the first caustic leach.

**Table 3.1. Concentrations of Nonradioactive Components in Various Process Streams**

Analyte	Retrieval Wash		First Caustic Leach		Second Caustic Leach		First Water Wash	
	Conc., µg/g	Mass, µg	Conc., µg/g	Mass, µg	Conc., µg/g	Mass, µg	Conc., µg/g	Mass, µg
Ag	< 0.12	342	< 0.56	< 1.73E+3	< 0.54	< 1.32E+3	< 0.12	< 682
Al	7.99E+2	4.74E+6	1.34E+4	4.12E+7	8.65E+3	3.34E+7	2.64E+3	1.19E+7
B	< 0.24	684	< 1.12	< 3.46E+3	< 1.09	< 2.64E+3	< 0.24	< 1.36E+3
Ba	0.074	211	< 0.22	692	< 0.22	< 528	0.099	563
Bi	0.51	1.45E+3	< 2.23	< 6.92E+3	< 2.17	< 5.28E+3	< 0.48	< 2.73E+3
Ca	1.10	3.14E+3	6.70	2.03E+4	9.70	1.39E+4	0.30	< 6.82E+3
Cd	< 0.072	205	< 0.33	< 1.04E+3	< 0.33	< 792	< 0.072	< 409
Cr	132	7.83E+5	118	3.11E+5	70	2.55E+5	22.6	1.03E+5
Cu	< 0.12	342	0.74	2.29E+3	1.60	2.82E+3	< 0.12	< 682
Fe	0.55	1.57E+3	2.40	7.21E+3	5.00	8.69E+3	0.51	1094
Li	3.41	9.72E+3	1.30	2.60E+3	1.10	793	0.17	569
Mg	< 0.48	1369	< 2.23	< 6.92E+3	< 2.17	< 5.28E+3	< 0.48	< 2.73E+3
Mn	< 0.24	684	< 1.12	< 3.46E+3	< 1.09	< 2.64E+3	< 0.24	< 1.36E+3
Mo	5.81	1.66E+4	1.70	2.84E+3	1.09	< 2.64E+3	< 0.24	< 1.36E+3
Na	9.52E+3	5.65E+7	5.70E+4	1.73E+8	6.32E+4	3.03E+8	1.75E+4	7.66E+7
Nd	< 0.48	1369	< 2.23	< 6.92E+3	< 2.17	< 5.28E+3	< 0.48	< 2.73E+3
P	70	4.12E+5	59.7	1.56E+5	24.5	6.31E+4	8.10	3.72E+4
Pb	< 0.48	1369	< 2.23	< 6.92E+3	3.50	8.51E+3	< 0.48	< 2.73E+3
Si	31.6	1.87E+5	153	4.61E+5	357	1.96E+6	98	4.28E+5
Sr	< 0.072	205	< 0.33	< 1.04E+3	< 0.33	< 792	< 0.072	< 409
Th	7.20	2.05E+4	< 22.3	< 6.92E+4	21.7	< 5.28E+4	< 4.80	< 2.73E+4
Ti	< 0.12	342	< 0.56	< 1.73E+3	< 0.54	< 1.32E+3	< 0.12	< 682
U	< 9.6	27371	< 44.6	< 1.38E+5	< 43.4	< 1.06E+5	< 9.60	< 5.46E+4
V	0.66	1.88E+3	< 1.12	< 3.46E+3	< 1.09	< 2.64E+3	< 0.24	< 1.36E+3
Zn	0.36	1.03E+3	2.20	6.67E+3	2.30	2.41E+3	0.46	1.78E+3
Zr	< 0.24	684	< 1.12	< 3.46E+3	< 1.09	< 2.64E+3	< 0.24	< 1.36E+3
TOC		No Analysis Performed		No Analysis Performed		No Analysis Performed		No Analysis Performed
TIC		No Analysis Performed		No Analysis Performed		No Analysis Performed		No Analysis Performed

Mass values represent the mass of material dissolved in a given step. These results have been corrected for interstitial liquid carried over from the previous step. A value of zero represents more material predicted to be carried over in the interstitial liquid than was determined analytically.

Analyte	Second Water Wash		Third Water Wash		Leached Solids	
	Conc., µg/g	Mass, µg	Conc., µg/g	Mass, µg	Conc., µg/g	Mass, µg
Ag	< 0.12	< 689	< 0.13	< 698	220	6.92E+4
Al	724	9.09E+4	199	5.49E+4	3.14E+5	9.81E+7
B	0.25	< 1.38E+3	< 0.25	< 1.40E+3	87	2.74E+4
Ba	0.050	< 275	< 0.050	< 279	103	3.24E+4
Bi	0.50	< 2.75E+3	< 0.50	< 2.79E+3	180	5.66E+4
Ca	0.15	387	< 1.25	< 6.98E+3	3.78E+3	1.19E+6
Cd	0.074	< 413	< 0.075	< 419	18	5.66E+3
Cr	7.69	9.05E+3	2.63	3.45E+3	1.43E+3	4.42E+5
Cu	0.12	< 689	< 0.13	< 698	65	2.04E+4
Fe	0.22	436	0.14	467	1.64E+4	5.16E+6
Li	0.15	< 826	< 0.15	< 838	36	1.13E+4
Mg	0.50	< 2.75E+3	< 0.50	< 2.79E+3	520	1.64E+5
Mn	0.25	< 1.38E+3	< 0.25	< 1.40E+3	1.34E+3	4.21E+5
Mo	0.25	< 1.38E+3	< 0.25	< 1.40E+3	< 49	< 1.5E+4
Na	5.45E+3	4.25E+6	1.85E+3	2.34E+6	4.51E+4	9.15E+6
Nd	0.50	< 2.75E+3	< 0.50	< 2.79E+3	150	4.72E+4
P	5.14	1.65E+4	2.90	8.68E+3	540	1.62E+5
Pb	0.50	< 2.75E+3	< 0.50	< 2.79E+3	90	2.83E+4
Si	29.4	1.71E+4	18.0	5.76E+4	1.69E+4	5.28E+6
Sr	0.074	< 413	< 0.075	< 419	1.89E+3	5.94E+5
Th	4.95	< 2.75E+4	< 5.00	< 2.79E+4	< 983	< 3.1E+5
Ti	0.12	< 689	< 0.13	< 698	150	4.72E+4
U	9.90	< 5.51E+4	< 10.0	< 5.58E+4	3.17E+4	9.97E+6
V	0.25	< 1.38E+3	< 0.25	< 1.40E+3	< 49	< 1.5E+4
Zn	0.25	< 1.38E+3	< 0.25	< 1.40E+3	92	2.89E+4
Zr	0.25	< 1.38E+3	< 0.25	< 1.40E+3	400	1.26E+5
TOC		No Analysis Performed			2.30E+3	7.24E+5
TIC		No Analysis Performed			6.06E+3	1.91E+6

Mass values represent the mass of material dissolved in a given step. These results have been corrected for interstitial liquid carried over from the previous step. A value of zero represents more material predicted to be carried over in the interstitial liquid than was determined analytically.

Table 3.2. Anion Concentration in Various Process Solutions<sup>(a)</sup>

Analyte	Retrieval Wash		First Caustic Leach		Second Caustic Leach		First Water Wash	
	Conc., µg/g	Mass, g	Conc., µg/g	Mass, g	Conc., µg/g	Mass, g	Conc., µg/g	Mass, g
F <sup>-1</sup>	D.L.	D.L.	0	0	0	0	0	0
Cl <sup>-1</sup>	553	1.576	D.L.	D.L.	509	1.239	274	1.372
NO <sub>2</sub> <sup>-1</sup>	1.012E+4	28.854	3.355E+3	6.165	1.183E+3	0	642	3.220
Br <sup>-1</sup>	D.L.	D.L.	D.L.	D.L.	0	0	D.L.	D.L.
NO <sub>3</sub> <sup>-2</sup>	1.541E+4	43.933	5.125E+3	9.441	1.885E+3	0	444	1.846
PO <sub>4</sub> <sup>-3</sup>	529	1.509	894	2.552	521	0	309	1.569
SO <sub>4</sub> <sup>-2</sup>	448	1.277	D.L.	D.L.	D.L.	D.L.	D.L.	D.L.

Analyte	Second Water Wash		Third Water Wash	
	Conc., µg/g	Mass, g	Conc., µg/g	Mass, g
F <sup>-1</sup>	0	0	0	0
Cl <sup>-1</sup>	D.L.	D.L.	254	0.726
NO <sub>2</sub> <sup>-1</sup>	593	2.344	631	1.802
Br <sup>-1</sup>	0	0	0	0
NO <sub>3</sub> <sup>-2</sup>	D.L.	D.L.	D.L.	D.L.
PO <sub>4</sub> <sup>-3</sup>	0	0	0	0
SO <sub>4</sub> <sup>-2</sup>	D.L.	D.L.	D.L.	D.L.

(a) D.L. denotes a value that was too low to report (i.e., the error too great for the value to be reportable). A value of zero indicates no value obtained for that particular anion.

**Table 3.3. Distribution of Nonradioactive Inorganic Sludge Components Between the Various Washing Functions**

Analyte	Component Distribution, %											
	Retrieval Wash			First			Second			Third		
	Wash	Caustic Leach	Water Wash	Caustic Leach	Water Wash	Caustic Leach	Water Wash	Caustic Leach	Water Wash	Caustic Leach	Water Wash	Residue
Ag	< 0.49%	< 2.5%	< 1.0%	< 1.0%	< 1.0%	< 1.0%	< 1.0%	< 1.0%	< 1.0%	< 1.0%	< 1.0%	> 92.1%
Al	2.5%	21.8%	6.3%	17.6%	6.3%	17.6%	6.3%	17.6%	0.048%	0.048%	0.029%	51.8%
B	< 2.5%	< 12.6%	< 5.0%	< 9.6%	< 5.0%	< 9.6%	< 5.0%	< 9.6%	5.0%	5.0%	5.1%	> 60.1%
Ba	0.64%	< 2.1%	< 1.7%	< 1.6%	< 1.7%	< 1.6%	< 1.7%	< 1.6%	0.8%	0.8%	0.8%	> 92.3%
Bi	2.5%	< 11.9%	< 4.7%	< 9.1%	< 4.7%	< 9.1%	< 4.7%	< 9.1%	4.7%	4.7%	4.8%	> 97.7%
Ca	0.26%	< 1.7%	< 0.56%	< 1.1%	< 0.56%	< 1.1%	< 0.56%	< 1.1%	0.032%	0.032%	0.57%	> 62.2%
Cd	3.6%	< 18.3%	< 7.2%	< 14.0%	< 7.2%	< 14.0%	< 7.2%	< 14.0%	7.3%	7.3%	7.4%	> 95.8%
Cr	41.1%	< 16.3%	< 5.4%	< 13.4%	< 5.4%	< 13.4%	< 5.4%	< 13.4%	0.47%	0.47%	0.18%	> 42.1%
Cu	< 1.3%	9.0%	2.7%	11.0%	2.7%	11.0%	2.7%	11.0%	2.7%	2.7%	2.7%	> 70.6%
Fe	0.030%	0.14%	0.021%	0.17%	0.021%	0.17%	0.021%	0.17%	0.008%	0.008%	0.009%	> 80.0%
Li	38.9%	10.4%	2.3%	3.2%	2.3%	3.2%	2.3%	3.2%	3.3%	3.3%	3.3%	> 38.6%
Mg	0.8%	< 4.2%	< 1.7%	< 3.2%	< 1.7%	< 3.2%	< 1.7%	< 3.2%	1.7%	1.7%	1.7%	> 45.3%
Mn	< 0.16%	< 0.8%	< 0.32%	< 0.63%	< 0.32%	< 0.63%	< 0.32%	< 0.63%	0.33%	0.33%	0.33%	> 86.6%
Mo	85.4%	14.6%	7.0%	13.6%	7.0%	13.6%	7.0%	13.6%	7.1%	7.1%	7.2%	> 97.4%
Na	9.0%	27.7%	12.3%	48.5%	12.3%	48.5%	12.3%	48.5%	0.7%	0.7%	0.37%	0%
Nd	2.9%	< 14.7%	< 5.8%	< 11.2%	< 5.8%	< 11.2%	< 5.8%	< 11.2%	5.8%	5.8%	5.9%	> 1.5%
P	48.2%	18.2%	4.3%	7.4%	4.3%	7.4%	4.3%	7.4%	1.9%	1.9%	1.0%	> 53.7%
Pb	3.7%	< 18.8%	< 7.4%	< 23.1%	< 7.4%	< 23.1%	< 7.4%	< 23.1%	7.5%	7.5%	7.6%	> 18.9%
Si	2.2%	5.5%	5.1%	23.3%	5.1%	23.3%	5.1%	23.3%	0.2%	0.2%	0.7%	> 31.9%
Sr	0.035%	< 0.17%	< 0.069%	< 0.13%	< 0.069%	< 0.13%	< 0.069%	< 0.13%	0.070%	0.070%	0.070%	> 76.9%
Th	100%	< 337%	< 133%	< 257%	< 133%	< 257%	< 133%	< 257%	134%	134%	136%	> 99.4%
Tl	0.73%	< 3.7%	< 1.4%	< 2.8%	< 1.4%	< 2.8%	< 1.4%	< 2.8%	1.5%	1.5%	1.5%	0%
U	0.27%	< 1.4%	< 0.55%	< 1.1%	< 0.55%	< 1.1%	< 0.55%	< 1.1%	0.55%	0.55%	0.56%	> 88.4%
V	100%	< 184%	< 72.5%	< 140%	< 72.5%	< 140%	< 72.5%	< 140%	73.2%	73.2%	74.2%	> 95.6%
Zn	2.5%	16.3%	4.4%	5.9%	4.4%	5.9%	4.4%	5.9%	3.4%	3.4%	3.4%	0%
Zr	0.54%	< 2.7%	< 1.1%	< 2.1%	< 1.1%	< 2.1%	< 1.1%	< 2.1%	1.1%	1.1%	1.1%	> 64.1%
												> 70.9%
												> 91.3%

Mass values represent the mass of material dissolved in a given step. These results have been corrected for interstitial liquid carried over from the previous step. A value of zero represents more material predicted to be carried over in the interstitial liquid than was determined analytically.

**Table 3.4. Concentrations of Radioactive Components in Various Process Streams**

Analyte	Retrieval Wash		First Caustic Leach		Second Caustic Leach		First Water Wash	
	Conc., $\mu\text{Ci/g}$	Actvty, $\mu\text{Ci}$						
Total Alpha	6.09E-5	0.174	7.00E-5	0.192	5.92E-5	4.26E-2	3.87E-5	0.199
$^{239/240}\text{Pu}$	6.13E-5	0.175	4.72E-5	0.121	9.22E-5	0.156	7.52E-6	9.46E-3
$^{241}\text{Am}/^{238}\text{Pu}$	1.87E-5	5.32E-2	3.31E-5	9.47E-2	1.19E-5	< 0.029	1.69E-5	9.18E-2
$^{243/244}\text{Cm}$	2.86E-6	8.16E-3	5.98E-6	1.74E-2	< 4E-6	< 0.010	4.20E-6	2.39E-2
$^{60}\text{Co}$	< 1.E-4	< 0.29	< 5.E-5	< 0.16	< 1.E-4	< 0.24	3.95E-4	2.24
$^{137}\text{Cs}$	14	3.99E+4	4.58	8.34E+3	2.85	< 74	0.401	1.25E+3
$^{154}\text{Eu}$	< 4.E-4	< 1.1	< 2.E-4	< 0.62	< 3.E-4	< 0.73	< 4.E-5	< 0.23
$^{155}\text{Eu}$	< 8.E-3	< 23	< 4.E-3	< 12	< 3.E-3	< 7.3	< 4.E-4	< 2.27
$^{241}\text{Am}$	< 8.E-3	< 23	< 4.E-3	< 12	< 3.E-3	< 7.3	< 4.E-4	< 2.27
$^{90}\text{Sr}$	< 3.E-2	< 86	4.57E-3	14.2	8.70E-3	14.5	4.35E-3	21.6

Analyte	Second Water Wash		Third Water Wash		Leached Solids	
	Conc., $\mu\text{Ci/g}$	Actvty, $\mu\text{Ci}$	Conc., $\mu\text{Ci/g}$	Actvty, $\mu\text{Ci}$	Conc., $\mu\text{Ci/g}$	Actvty, $\mu\text{Ci}$
Total Alpha	2.24E-5	6.66E-2	8.62E-5	0.449	1.71	539
$^{239/240}\text{Pu}$	6.39E-6	2.43E-2	3.17E-5	0.167	1.58	496
$^{241}\text{Am}/^{238}\text{Pu}$	1.12E-5	3.68E-2	3.27E-5	0.166	0.553	174
$^{243/244}\text{Cm}$	2.65E-6	8.50E-3	< 3.E-6	< 0.017	3.81E-3	1.20
$^{60}\text{Co}$	< 2.E-5	< 0.11	< 2.E-5	< 0.11	5.84E-2	18.4
$^{137}\text{Cs}$	0.166	325	0.137	522	84.5	2.62E+4
$^{154}\text{Eu}$	< 4.E-5	< 0.22	< 4.E-5	< 0.22	0.252	79.3
$^{155}\text{Eu}$	< 3.E-4	< 1.7	< 3.E-4	< 1.7	0.149	46.9
$^{241}\text{Am}$	< 3.E-4	< 1.7	< 3.E-4	< 1.7	0.525	165
$^{90}\text{Sr}$	3.12E-3	10.8	2.57E-3	9.80	837	2.63E+5

Mass values represent the mass of material dissolved in a given step. These results have been corrected for interstitial liquid carried over from the previous step. A value of zero represents more material predicted to be carried over in the interstitial liquid than was determined analytically.

**Table 3.5. Distribution of Radioactive Sludge Components**

Analyte	Component Distribution, %												
	Retrieval Wash		First Caustic Leach		Second Caustic Leach		First Water Wash		Second Water Wash		Third Water Wash		Residue
	<	0.032%	<	0.035%	<	0.008%	<	0.037%	<	0.012%	<	0.083%	
<b>Total Alpha</b>	<	0.032%	<	0.035%	0.008%	<	0.037%	<	0.012%	<	0.083%	>	99.8%
<sup>239/240</sup> Pu	<	0.035%	<	0.024%	0.031%	<	0.0019%	<	0.005%	<	0.034%	>	99.9%
<sup>241</sup> Am/ <sup>238</sup> Pu	<	0.030%	<	0.054%	0.017%	<	0.053%	<	0.021%	<	0.095%	>	99.7% < 99.7%
<sup>243/244</sup> Cm	<	0.65%	<	1.4%	0.77%	<	1.9%	<	0.68%	<	1.3%	>	93.3% < 95.4%
<sup>60</sup> Co	<	1.4%	<	0.75%	1.2%	<	11%	<	0.54%	<	0.54%	>	84.7% < 89.1%
<sup>137</sup> Cs	<	52%	<	11%	0.097%	<	1.6%	<	0.42%	<	0.68%	>	34.1% < 34.2%
<sup>154</sup> Eu	<	1.4%	<	0.78%	0.92%	<	0.29%	<	0.28%	<	0.28%	>	96.0%
<sup>155</sup> Eu	<	49%	<	26%	16%	<	4.9%	<	3.6%	<	3.6%	>	70.9%
<sup>241</sup> Am	<	14%	<	7.5%	4.4%	<	1.4%	<	1.0%	<	1.0%	>	70.9%
<sup>90</sup> Sr	<	0.033%	<	0.005%	0.006%	<	0.008%	<	0.004%	<	0.004%	>	99.9% < 100%

Mass values represent the mass of material dissolved in a given step. These results have been corrected for interstitial liquid carried over from the previous step. A value of zero represents more material predicted to be carried over in the interstitial liquid than was determined analytically.

**Table 3.6. Mass Recoveries for Nonradioactive Sludge Components**

Analyte	Total Mass, $\mu\text{g}$		Recovery, %
	Direct Analysis	Summation Method	
Ag	5.10E+4	6.92E+4	136%
Al	2.00E+8	1.89E+8	95%
B	6.76E+4	2.74E+4	40%
Ba	3.05E+4	3.32E+4	109%
Bi	6.30E+4	5.81E+4	92%
Ca	1.20E+6	1.23E+6	102%
Cd	0	5.66E+3	
Cr	1.77E+6	1.91E+6	108%
Cu	2.90E+4	2.56E+4	88%
Fe	4.92E+6	5.18E+6	105%
Li	2.30E+4	2.50E+4	109%
Mg	1.59E+5	1.64E+5	103%
Mn	3.82E+5	4.21E+5	110%
Mo	3.82E+4	1.94E+4	51%
Na	6.52E+7	6.25E+8	957%
Nd	0	4.72E+4	
P	8.21E+5	8.56E+5	104%
Pb	0	3.68E+4	
Si	5.82E+6	8.38E+6	144%
Sr	6.82E+5	5.94E+5	87%
Th	0	2.05E+4	
Ti	5.63E+4	4.72E+4	84%
U	1.13E+7	9.97E+6	88%
V	0	1.88E+3	
Zn	5.56E+4	4.08E+4	73%
Zr	8.85E+4	1.26E+5	142%
TOC	1.88E+6	7.24E+5	38%
TIC	5.06E+6	1.91E+6	38%

It should be noted that there were several differences between the two experiments. The Lumetta work was done at a lower solids loading for all steps, allowing more solution to contact the sludge. Thus, although the final Al concentrations in the first caustic leach solutions are nearly identical for both this work and that of Lumetta et al, a significantly larger fraction of the total was removed in the laboratory-scale work. However, based on the Al/Na equilibrium data developed by Barney et al. (1976), these solutions should not be limited by solubility. Another difference is that Lumetta et al. used a centrifuge operated at ambient temperatures, while this work used gravity settling operated for a week or more at elevated temperatures for the solid/liquid separation. Finally, the sludge samples were also different in composition. The S-107 sludge studied by Lumetta et al. contained 1.75 times the phosphorus and twice the chromium as the liter-scale experiment.

**Table 3.7. Recoveries for Radioactive Sludge Components**

Analyte	Total Activity, $\mu\text{Ci}$		Recovery, %
	Direct Analysis	Summation Method	
<b>Total Alpha</b>	521	540	104%
<sup>239/240</sup> Pu	522	497	95%
<sup>241</sup> Am/ <sup>238</sup> Pu	182	174	96%
<sup>243/244</sup> Cm	0.570	1.26	220%
<sup>60</sup> Co	174	20.6	12%
<sup>137</sup> Cs	7.25E+4	7.65E+4	106%
<sup>154</sup> Eu	62.4	79.3	127%
<sup>155</sup> Eu	22.8	46.9	206%
<sup>241</sup> Am	171	165	96%
<sup>90</sup> Sr	2.74E+5	2.63E+5	96%

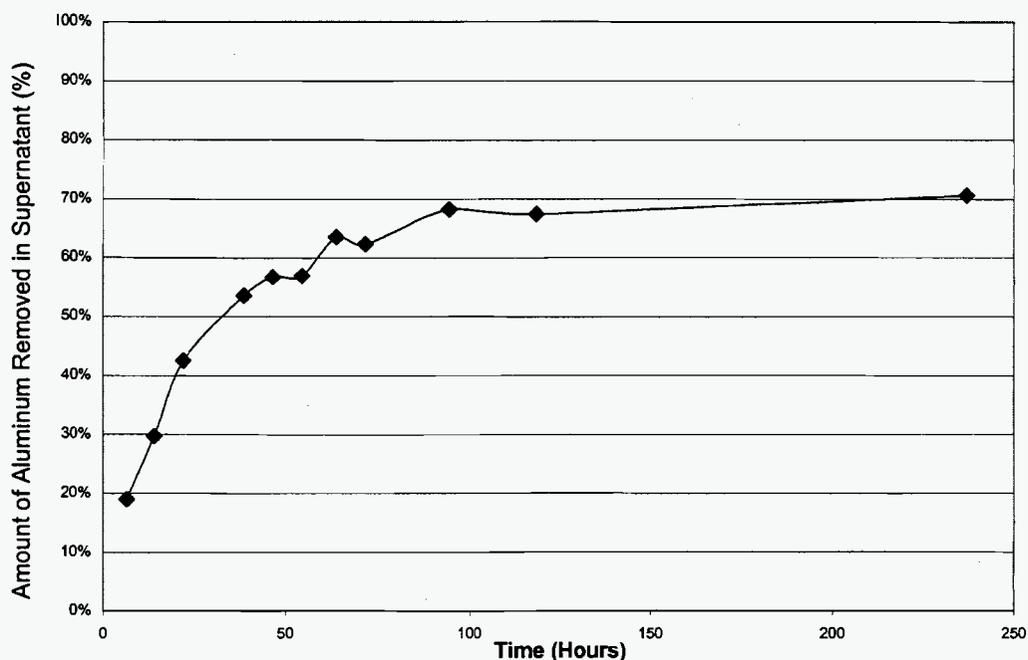
**Table 3.8. Distribution of Various Tank S-107 Components Compared to the Data from Lumetta et al. (1996)**

(% from this study/% from Lumetta)

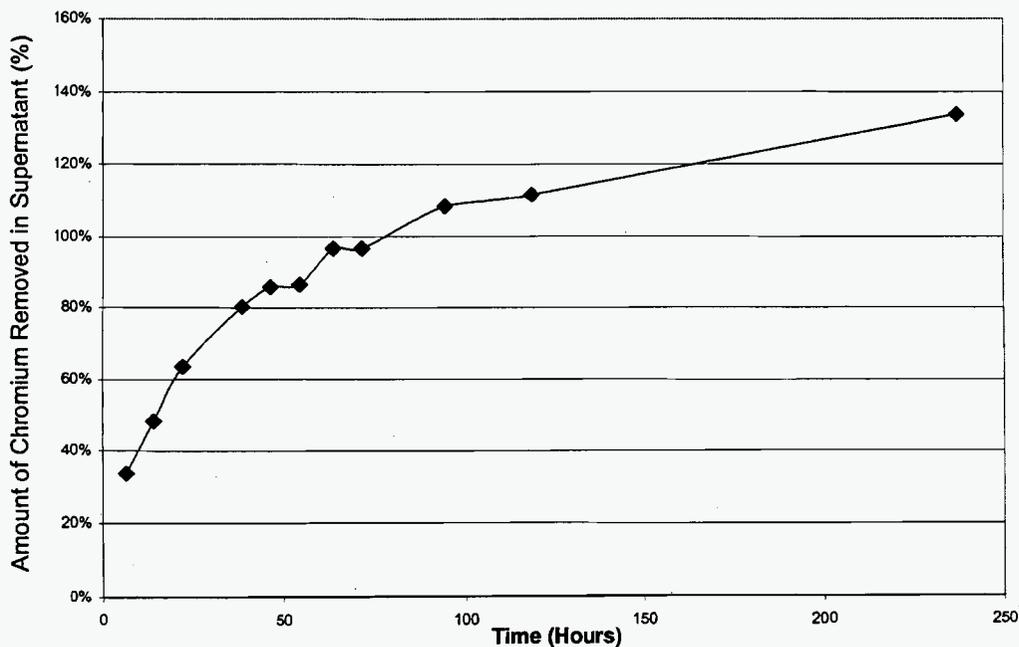
	<u>Retrieval Wash</u>	<u>Caustic Leach 1</u>	<u>Caustic Leach 2</u>	<u>Water Washes</u>	<u>Residue</u>
Al	2	22/56	18/17	6/0	52/27
Cr	41	16/35	13/18	6/1	23/47
Fe	0.04	0.1/0	0.2/3	0/0	100/96
P	48	18/92	7/6	7/0	19/2
Si	2	6/5	24/67	6/17	63/12
<sup>137</sup> Cs	52	11/79	0.1/21	3/0	34/0
<sup>90</sup> Sr	0.03	0.00/0	0.01/0	0.02/0	100/100
<b>Total Alpha</b>	0.03	0.04/0	0.01/0	0.2/0	100/100

The data in the Tables and Figures illustrate some very interesting results. First, the amount of major targeted constituents, namely Al and Cr, leached into the supernatant increase with increasing contact time (cf. Table 3.9 and Figures 3.1 and 3.2). After about 100 h of leaching, all of the Cr was extracted into the supernatant (the >100% data in Figure 3.2 are attributed to analytical errors and errors in the computation of the wt% undissolved solids). However, after a contact period of 240 h, the amount of Al leached was about 70%. Also, the Al leaching profiles in Table 3.9 and Figure 3.1 indicate that prolonging the contact time does not lead to higher amounts of Al in the supernatant. Interestingly, the final Al concentration in the supernate of the extended caustic leach is very similar to the concentration in

the first caustic leach during the enhanced sludge wash. There may in fact be a lower Al solubility than predicted by Barney et al. (1976). An interesting feature of the extended caustic leaching step was that no significant leaching of the minor constituents, such as B, Ca, and Si, was observed. For example, the average amounts of B, Ca, and Si leached into the supernatant (cf. Table 3.11) during the extended caustic leaching step were in all cases less than 10%.



**Figure 3.1.** Removal of Al from the Supernatant During the Extended Caustic Leach



**Figure 3.2.** Removal of Chromium from the Supernatant During the Extended Caustic Leach

**Table 3.9. Concentration of Nonradioactive Components in the Extended Caustic Wash Supernatant Solutions as a Function of Time**

Analyte	Sample Time in Hours											
	Slurry 6.50	Slurry 14.00	Supernatant 22.00	Slurry 38.50	Supernatant 46.50	Slurry 54.50	Slurry 64.00	Supernatant 72.00	Supernatant 94.50	Supernatant 118.50	Supernatant 237.00	
Al	2.48E+03	3.86E+03	5.58E+03	7.39E+03	8.04E+03	8.03E+03	9.04E+03	8.95E+03	1.04E+04	1.02E+04	1.48E+04	
B	6.62E+01	3.94E+01	3.32E+01	2.89E+01	3.77E+01	2.81E+01	3.47E+01	4.29E+01	3.51E+01	2.67E+01	3.16E+01	
Ca	2.24E+01	0.00E+00	0.00E+00	2.46E+01	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	1.10E+01	1.57E+01	
Cr	1.41E+01	2.03E+01	2.69E+01	3.57E+01	3.92E+01	3.93E+01	4.44E+01	4.48E+01	5.31E+01	5.47E+01	9.03E+01	
Cu	0.00E+00	6.50E-01	7.95E-01	0.00E+00	8.85E-01	6.72E-01	0.00E+00	7.84E-01	9.41E-01	8.06E-01	1.08E+00	
Fe	4.14E+00	4.59E+00	6.50E+00	3.58E+00	7.43E+00	2.91E+00	2.69E+00	6.31E+00	7.12E+00	5.71E+00	4.26E+00	
Mo	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	2.58E+00	
Na	6.82E+04	7.21E+04	7.69E+04	7.71E+04	7.69E+04	7.57E+04	7.63E+04	7.10E+04	7.53E+04	6.78E+04	8.85E+04	
P	9.30E+00	9.18E+00	8.06E+00	7.06E+00	9.18E+00	8.29E+00	7.17E+00	8.18E+00	9.52E+00	8.96E+00	1.68E+01	
Pb	0.00E+00	0.00E+00	2.58E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	
Si	3.62E+02	1.70E+02	2.56E+02	1.23E+02	2.48E+02	1.23E+02	1.23E+02	2.50E+02	2.08E+02	1.55E+02	1.81E+02	
Sr	0.00E+00	0.00E+00	0.00E+00	0.00E+00	4.70E-01	0.00E+00	0.00E+00	5.04E-01	7.62E-01	7.39E-01	8.96E-01	
Zn	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	1.23E+00	0.00E+00	0.00E+00	1.68E+00	

µg/mL

Concentration of Species (µg/mL) = ICP Data (µg/g) \* Supernatant Density (g/mL)

**Table 3.10. Mass of Nonradioactive Components in the Extended Caustic Wash Supernatant Solutions as a Function of Time**

Analyte	Sample Time in Hours										
	Slurry 6.50	Slurry 14.00	Supernatant 22.00	Slurry 38.50	Supernatant 46.50	Slurry 54.50	Slurry 64.00	Supernatant 72.00	Supernatant 94.50	Supernatant 118.50	Supernatant 237.00
Al	4.23E+06	6.62E+06	9.47E+06	1.19E+07	1.26E+07	1.27E+07	1.42E+07	1.39E+07	1.52E+07	1.50E+07	1.57E+07
B	1.13E+05	6.75E+04	5.63E+04	4.66E+04	5.93E+04	4.44E+04	5.44E+04	6.66E+04	5.13E+04	3.91E+04	3.36E+04
Ca	3.83E+04	0.00E+00	0.00E+00	3.98E+04	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	1.61E+04	1.67E+04
Cr	2.41E+04	3.47E+04	4.57E+04	5.77E+04	6.16E+04	6.22E+04	6.95E+04	6.95E+04	7.78E+04	8.02E+04	9.61E+04
Cu	0.00E+00	1.11E+03	1.35E+03	0.00E+00	1.39E+03	1.06E+03	0.00E+00	1.22E+03	1.38E+03	1.18E+03	1.14E+03
Fe	7.09E+03	7.86E+03	1.10E+04	5.79E+03	1.17E+04	4.60E+03	4.21E+03	9.78E+03	1.04E+04	8.38E+03	4.53E+03
Mo	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	2.74E+03
Na	1.17E+08	1.24E+08	1.31E+08	1.24E+08	1.21E+08	1.20E+08	1.19E+08	1.10E+08	1.10E+08	9.94E+07	9.42E+07
P	1.59E+04	1.57E+04	1.37E+04	1.14E+04	1.44E+04	1.31E+04	1.12E+04	1.27E+04	1.39E+04	1.31E+04	1.79E+04
Pb	0.00E+00	0.00E+00	4.38E+03	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Si	6.19E+05	2.92E+05	4.36E+05	1.99E+05	3.89E+05	1.95E+05	1.93E+05	3.87E+05	3.05E+05	2.27E+05	1.93E+05
Sr	0.00E+00	0.00E+00	0.00E+00	0.00E+00	7.39E+02	0.00E+00	0.00E+00	7.82E+02	1.12E+03	1.08E+03	9.54E+02
Zn	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	1.91E+03	0.00E+00	0.00E+00	1.79E+03

Mass of Species in Solution ( $\mu\text{g}$ ) = ICP Data ( $\mu\text{g/g}$ ) \* Mass of Supernatant (g)

Mass of Supernatant (g) = Mass of Slurry (g) \* (100 - Wt% Solids)/100

**Table 3.11. Percent Removal of Non-radioactive Components During the Extended Caustic Leach Step**

Analyte	Sample Time in Hours											
	Slurry 6.50	Slurry 14.00	Supernatant 22.00	Slurry 38.50	Supernatant 46.50	Slurry 54.50	Supernatant 64.00	Slurry 72.00	Supernatant 94.50	Supernatant 118.50	Supernatant 237.00	
Al	19.0%	29.7%	42.5%	53.5%	56.7%	57.0%	63.5%	62.3%	68.2%	67.4%	70.6%	
B	20.3%	12.1%	10.1%	8.4%	10.6%	8.0%	9.7%	11.9%	9.2%	7.0%	6.0%	
Ca	7.4%	0.0%	0.0%	7.7%	0.0%	0.0%	0.0%	0.0%	0.0%	3.1%	3.2%	
Cr	33.6%	48.3%	63.5%	80.2%	85.7%	86.4%	96.6%	96.7%	108.1%	111.5%	133.7%	
Cu	0.0%	4.2%	5.1%	0.0%	5.3%	4.0%	0.0%	4.6%	5.2%	4.5%	4.3%	
Fe	1.4%	1.6%	2.2%	1.1%	2.3%	0.9%	0.8%	1.9%	2.1%	1.7%	0.9%	
Mo	--	--	--	--	--	--	--	--	--	--	--	
Na	126.4%	133.8%	141.6%	134.8%	131.0%	129.7%	129.4%	119.3%	119.4%	107.6%	102.0%	
P	13.2%	13.0%	11.3%	9.4%	12.0%	10.8%	9.3%	10.5%	11.5%	10.9%	14.8%	
Pb	0.0%	0.0%	8.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	
Si	13.5%	6.4%	9.5%	4.3%	8.5%	4.3%	4.2%	8.5%	6.7%	5.0%	4.2%	
Sr	0.0%	0.0%	0.0%	0.0%	0.8%	0.0%	0.0%	0.9%	1.3%	1.2%	1.1%	
Zn	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	8.1%	0.0%	0.0%	7.6%	

### 3.3 Results of the Liter-Scale Settling Experiments

As discussed in Section 2.0, settling tests were performed for each step of the ESW process. In the retrieval, second caustic leach, and third water wash, two settling tests were performed for each of these steps at both high and low solids concentrations. A settling test was also performed after the extended caustic leach. The settling conditions, velocities, and solids concentrations for these tests are shown in Table 3.12.

The settling rates seen from these data are higher than the assumptions used in the TWRS O&UP (Kirkbride 1997). The assumed settling rates are 1-2 cm/h as compared to the experimentally measured hindered settling values of 3 to 16 cm/h achieved for the S-107 sludge. The final compaction values, on the other hand, are in some cases below the TWRS O&UP assumptions. The final compaction required was 20 wt% as compared to the experimentally measured values of between 15.7 and 32.7 wt% insoluble solids for the S-107 sludge. As discussed previously, the settling region can be scaled directly to full scale since the size of the settling vessel does not impact its rate. The level of compaction, however, is based on the height of the sludge layer. A taller column of sludge would exert more force and further compact the sludge below it than a shorter column. Thus, these solids concentration results with  $\approx 12$  to 16 cm of compacted sludge are not directly scaleable to a full-scale system, but provide a lower bound on the full-scale sludge compaction. The sludge in the full-scale system should compact to a greater degree than seen here. The model results described in Section 3.5 provide a means of scaling up the compaction results.

The solids and the supernate separated with a single, very distinct interface in all of the settling experiments as seen in Figure 3.3. In all cases, the interface formed within the first 6 minutes of settling. For most settling experiments, the solution clarified within 10 minutes from the formation of the interface. Clarity is based on there being no visible particles in solution. Only for the retrieval steps did the solution remain cloudy during the initial stages of settling. For the retrieval step at low initial solids concentration, the solution remained cloudy during the first 30 minutes after the formation of the interface. This cloudiness was the result of fine particles remaining suspended in the supernate even after the bulk of the sludge material had settled. As these fine particles settled, the solution cleared. In the cases where the supernate was cloudy, the settling interface used to measure the settling rate was that of the bulk solids rather than the remaining fine particles.

In the second and third water wash, the slurry foamed when pumped into the column. After the foam had dissipated, some solids remained on the top of the supernate. These solids remained for several hours before they disappeared. The foaming and floating particles were not seen on any other settling tests. This formation of stable foams during the later water washes may result from lower solution ionic strength of these solutions.

Table 3.12. Tank S-107 Settling Test Results

<u>Process Step</u>	<u>Temperature</u> (°C)	<u>Initial Solid</u> <u>Concentration</u> (wt%)	<u>Hindered</u> <u>Settling</u> <u>Rate (cm/h)</u>	<u>Final Solid</u> <u>Concentration</u> (wt%)
Retrieval 1	80	13.6	8.2	32.7
Retrieval 2	80	7.7	16.7	31.6
1 <sup>st</sup> Caustic Leach	80	10.9	4.4	24.8
2 <sup>nd</sup> Caustic Leach 1	80	14.6	3.2	27.1
2 <sup>nd</sup> Caustic Leach 2	80	7.2	7.1	26.2
1 <sup>st</sup> Water Wash	50	4.3	15.3	15.7
2 <sup>nd</sup> Water Wash	50	4.3	15.1	16.6
3 <sup>rd</sup> Water Wash 1	50	4.3	12.2	16.6
3 <sup>rd</sup> Water Wash 2	50	8.7	6.2	17.5
Extended Caustic Leach	80	1.8	5.6	3.9



Figure 3.3. S-107 Sludge Settling in the Liter-Scale Settling Equipment. Note the distinct interface between supernate and slurry.

Several trends can also be seen from the settling results (See Table 3.12). The hindered settling rate appears to be strongly concentration dependent. Increases in solids concentration resulted in a proportionate decrease in the hindered settling rate. This is seen consistently with the retrieval, caustic leach, and water wash steps, which were each performed at both high and low solids loading. Furthermore, the sludge settles fastest during the retrieval step, next fastest during the water washes, and slowest during the caustic leaches. This is consistent with expectations since 3 M NaOH has a viscosity twice that of water, and the Stokes settling velocity is inversely proportional to viscosity. The slower settling rate for the water washes than the retrieval might also be due to the lower temperature and resultant lower viscosity. In any case, the reduction in settling rate at lower temperatures has also been shown in past studies (Brooks et al. 1996).

The settling curves for the above data are shown in Figure 3.4. The settling curves are similar to those typically seen for hindered settling as described in Section 2.5. The hindered settling region is a more or less a straight line followed by a slowing of the settling during compaction. The maximum settling rate is taken from a linear regression of the hindered settling region. For the tests shown here, hindered settling is complete within 1 to 4 h. Compaction required usually 80 to 100 h.

The settling data were normalized according to formulas recommended by Graham MacLean<sup>(a)</sup>:

$$t^* = t v_{\max} / z_0 \quad \text{and} \quad z^* = z / z_0 \quad 3.2$$

where  $t^*$  and  $z^*$  are the normalized time and height,  $t$  is the dimensional time,  $v_{\max}$  is the maximum settling velocity, and  $z$  and  $z_0$  are the dimensional interface height and initial height, respectively. By non-dimensionalizing the data, the shapes of the settling curves can be compared with similar experiments performed in containers with other geometries. The data are shown in Figure 3.5.

In some cases, the solids concentration is of interest instead of the interface height. Figure 3.6 provides the same data in terms of solids concentration versus time. As can be seen from this figure, final compaction is relatively independent of the initial solids concentration. These results are reasonable considering that the final compaction is based on sludge height, which was nearly constant throughout all tests. The rate of compaction seems to decrease as a function of the ESW step. Additionally, the final solids concentration in the sediment decreases as a function of the ESW step (i.e., retrieval > caustic leach > water wash). This could be the result of changes in the particle-particle interactions during the course of the settling process. For example, at the low ionic strength of the water washes, repulsive electrostatic forces between particles become more significant, making the compaction of the sludge more difficult.

The settling test following the extended caustic leach was done at considerably lower solids concentration. An estimated 80% of the original insoluble solids in the sludge had dissolved during the ESW and extended leaching processes. This final settling test compacted very slowly and only slightly.

The final solids concentration in the sediment was less than 5 wt% after 50 hours. Based on these results and those to be discussed in Section 3.4, the characteristics of the sludge following the extended caustic leach are very different than the other samples studied. Because of the small primary particles and the easily broken, large agglomerates, this material may be difficult to separate efficiently with both sedimentation and filtration.

As mentioned previously, for a solid/liquid separation technique to be considered viable, the TRU and <sup>90</sup>Sr concentrations in the final LLW product must be minimized. The decontamination factor (DF) is a measure of the ability of settle/decant to keep these radionuclides in the solids and prevent them from

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(a) Westinghouse Hanford Company Internal Memo, From Graham MacLean To Dave Place, June 18, 1996.

entering the supernate, either as dissolved material or as colloidal particles. High DFs indicate both that a solid/liquid separation technique is viable and that the ESW does not dissolve these radionuclides. The DFs are calculated as a ratio of radionuclides in the solids to that in the liquid. They are shown in Table 3.13 for each step as well as for a composite. The DFs for TRU are between 1065 and 5904 while the DFs for  $^{90}\text{Sr}$  are between  $>3786$  to 29360. The TWRS Privatization Contract (1996) requires that the LAW immobilized product be less than 100 nCi/g TRU and less than  $20 \text{ Ci/m}^3$   $^{90}\text{Sr}$ . Assuming each supernate would be individually vitrified in a 20 wt%  $\text{Na}_2\text{O}$  glass matrix, the TRU would be less than 6.7 nCi/g and the  $^{90}\text{Sr}$  less than  $0.52 \text{ Ci/m}^3$  in all cases, indicating compliance to the Privatization Contract for S-107 using settle decant. By blending the individual streams, the radionuclides are further diluted.

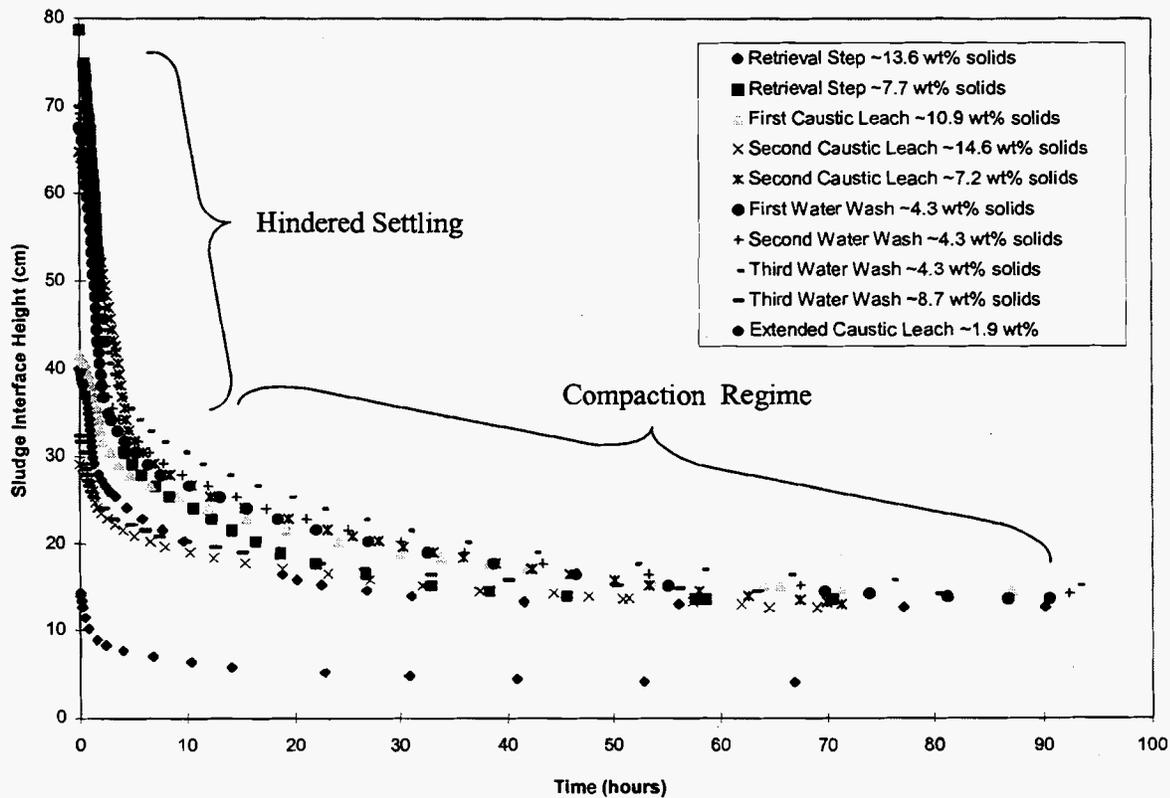


Figure 3.4. Dimensional Settling Curves for Each Step of the S-107 Enhanced Sludge Wash

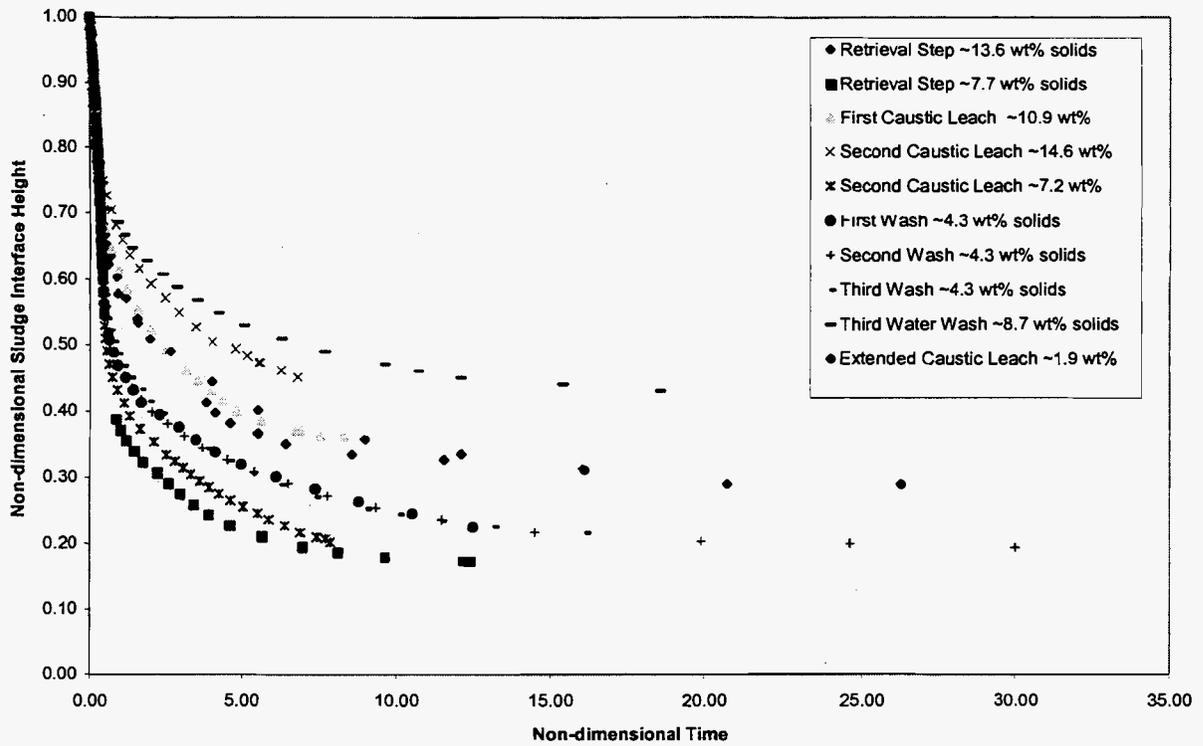


Figure 3.5. Non-Dimensional Settling Curves for Each Step of the S-107 Enhanced Sludge Wash

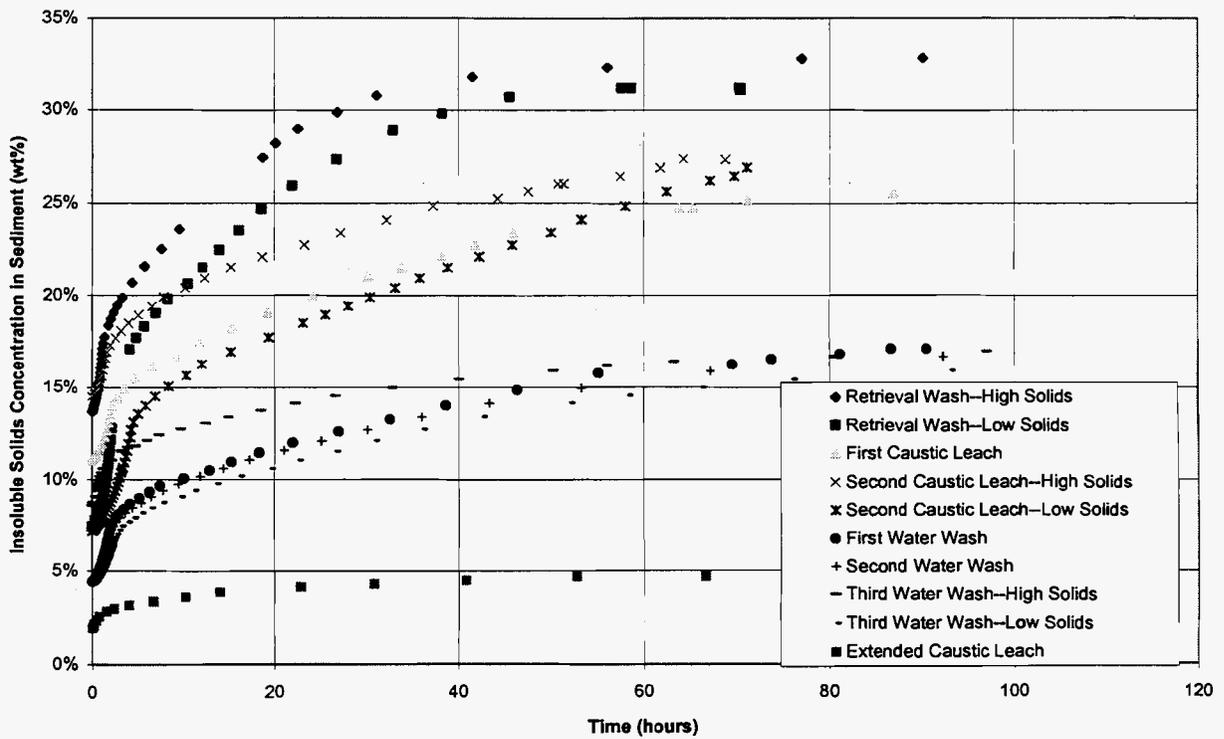


Figure 3.6. Insoluble Solids Concentration for Each Step of the S-107 Enhanced Sludge Wash

**Table 3.13. TRU and <sup>90</sup>Sr Separation Efficiencies**

	<b>Initial Sludge</b>	<b>Retrieval Step</b>	<b>Caustic Leach 1</b>	<b>Caustic Leach 2</b>	<b>Water Wash 1</b>	<b>Water Wash 2</b>	<b>Water Wash 3</b>	<b>Overall Blend</b>
<b>Sample Weight (g)</b>	512.11	1747	1479	1085.71	4151.53	4199.74	4132	16794.98
<b>Measured TRU in Sample (mCi/g)</b>	0.741	6.09E-05	7.00E-05	5.92E-05	3.87E-05	2.24E-05	8.62E-05	5.62E-05
<b>Measured Sr in Sample (mCi/g)</b>	387.5	3.00E-02	4.57E-03	8.70E-03	4.35E-03	3.12E-03	2.57E-03	8.88E-03
<b>Total TRU (mCi)</b>	3.79E+02	0.11	0.10	0.06	0.16	0.09	0.36	0.94
<b>Total Sr-90 (uCi)</b>	1.98E+05	52.41	6.76	9.45	18.06	13.08	10.62	149.21
<b>Decontamination Factor for TRU</b>	--	<b>3567</b>	<b>3665</b>	<b>5904</b>	<b>2362</b>	<b>4043</b>	<b>1065</b>	<b>402</b>
<b>Decontamination Factor for Sr-90</b>	--	<b>3786</b>	<b>29360</b>	<b>21009</b>	<b>10988</b>	<b>15169</b>	<b>18687</b>	<b>1330</b>
<b>Sodium in Supernate (ug/g)</b>	--	2.12E+04	5.70E+04	7.02E+04	1.75E+04	5.36E+03	1.86E+03	1.79E+04
<b>g LLW glass/g supernate</b>	--	0.15	0.39	0.48	0.12	0.04	0.01	0.12
<b>Sr-90 (Ci/m<sup>3</sup> glass)</b>	--	<b>0.52</b>	<b>0.03</b>	<b>0.05</b>	<b>0.09</b>	<b>0.21</b>	<b>0.50</b>	<b>0.18</b>
<b>TRU (nCi/g glass)</b>	--	<b>0.42</b>	<b>0.18</b>	<b>0.12</b>	<b>0.32</b>	<b>0.61</b>	<b>6.77</b>	<b>0.46</b>

The liter-scale settling/decant curves can not only be used for scale up to a full-scale settle/decant system, but can also be used to design a continuous-feed clarifier/thickener. A clarifier/thickener in a shielded facility could replace a double shell tank. Because the process can be operated continuously, the size of the process is reduced. A clarifier/thickener was sized for the retrieval, second caustic leach, and third water wash using the approach of Christian (1994) and the above settling data. In this approach, the settling rate is assumed to be an exponential function of solids concentration. It then uses the settling rate, mass, and material balances to determine the solids flux. Solids flux can then be related back to the diameter of the thickener. The depth of the tank is not a factor, but typical depths are between 2.5 and 5 meters.

For the approach taken here, it is assumed that the slurries will be fed to the system at 15 gpm at 5 wt% solids. The solids would be removed from the system at an underflow concentration of >20 wt%. The effluent is assumed to have no solids. Under these conditions, the clarifier/thickener would be 4.65, 6.6, and 7.0 meters in diameter for the retrieval, caustic leach, and water-wash steps, respectively.

### 3.4 Radiological Colloids Analysis

The results of the measured wt% of total solids (soluble and insoluble) in the slurry and soluble solids in the supernatant are described below. Measurements of the density of the slurry, supernatant, and dried solids and the particle size distribution (PSD) of the homogenized slurry samples are also provided in this section. A centrifugation study measuring the compaction of sludge at high g-forces is also discussed. These results provide insight into how the ESW experiment modifies the colloidal characteristics of S-107 sludge. Furthermore, these analyses assist in the mass-balance closure and in benchmarking the theoretical model.

The measured wt% of total solids (soluble and insoluble) in the slurry, the soluble solids in the supernatant, and the calculated insoluble solids in the slurry are provided in this section. For each process step, 2 to 3 replicates, the averaged value, and the estimated measurement error are presented in Table 3.15. The reproducibility of the measured and calculated solids wt% quantities for all the process steps in all cases suggest that the S-107 slurry samples were uniformly homogenized.

The measured densities of the bulk slurry and the supernatant are listed in Table 3.16. These quantities were nearly identical for the replicate samples. In the case of bulk densities, the results indicate that the slurry samples were thoroughly homogenized, and each extracted sample was a representative of the sludge.

The results of the dried-solid densities for each process step are presented in Table 3.17. The data indicate that the density of solids (dried-solid mixture) increases from 2.69 g/cc in the retrieval step to a maximum of 3.11 g/cc in the third water-wash step and then back down to 2.39 g/cc for the final sludge step. The increasing solids density values in the initial steps suggest that a significant portion of the low-density materials were dissolved as the S-107 slurry was washed or treated with the caustic solution. For example, a total of 47 wt % of aluminum inventory in the S-107 sludge (see Table 3.3) was removed from the solid phase during ESW. It is presumed that the removal of the Al components in various mineral phases such as gibbsite, with typical specific gravity's of 2.4 (Brady 1991), gradually increases the solid-phase density to a maximum of 3.11 g/cc in the third water-wash step. Based on similar hypothesis, the solid-phase density decreased after adding a large amount of caustic solution (specific gravity of 2.13 for the NaOH) during the extended caustic leach step. Ultimately, the solid-phase density of the final S-107 sludge increased to 2.39 g/cc as an additional of 38 wt % of Al species (see Tables S.2 and 3.10) were removed at the end of extended caustic leaching step.

**Table 3.14.** Measured Solids wt% of S-107 Slurries

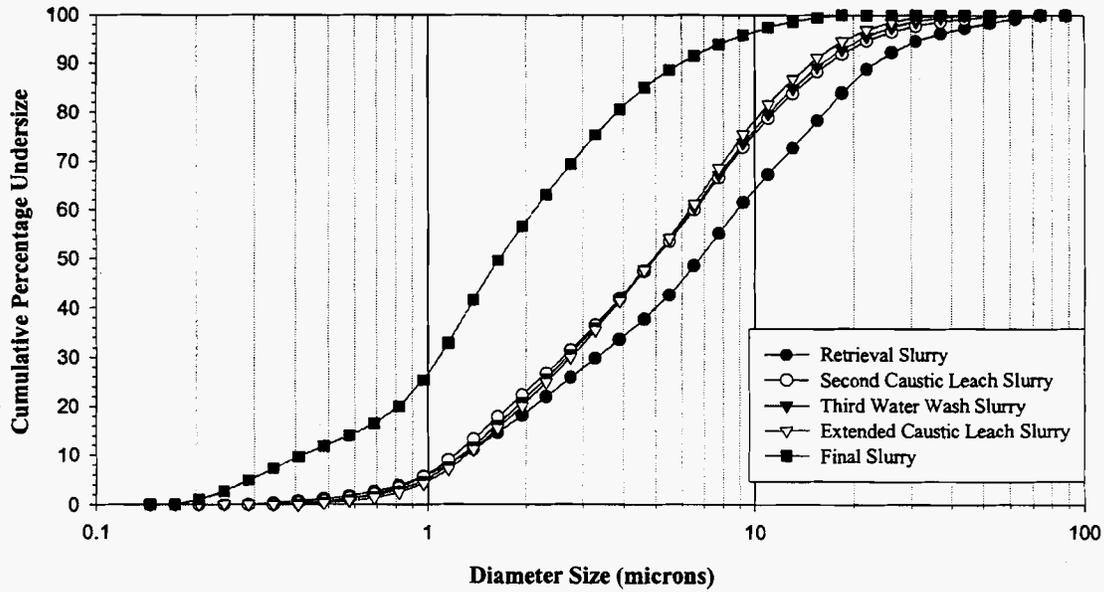
Enhanced Sludge Settling Step	Measured Total Solids in Slurry, Soluble & insoluble (Wt %)	Measured Soluble Solids In Supernatant (Wt %)	Calculated % Ratio of Soluble Solids to Water in Supernatant	Calculated Insoluble Solids (Wt %)
Retrieval Step	16.05	5.17	5.45	11.39
	15.87	5.26	5.55	11.20
	16.87	5.15	5.43	12.36
Average	16.26	5.19	5.48	11.65
Standard Deviation	0.53	0.06	0.06	0.62
95% Confidence	1.15	0.12	0.13	1.33
Second Caustic Leach Step	15.29	8.53	9.33	7.39
	15.71	8.18	8.91	8.20
	15.68	8.25	8.99	8.09
Average	15.56	8.32	9.08	7.90
Standard Deviation	0.23	0.19	0.22	0.44
95% Confidence	0.50	0.4	0.48	0.95
Third Water Wash Step	9.56	1.27	1.29	8.4
	10.10	1.09	1.10	9.11
	10.06	1.06	1.07	9.10
Average	9.91	1.14	1.15	8.87
Standard Deviation	0.30	0.12	0.12	0.41
95% Confidence	0.64	0.25	0.26	0.88
Initial Extended Caustic Leach Step	13.75	12.09	11.72	3.65
	14.15	11.92	11.40	4.37
Average	13.95	12.01	11.56	4.01
Standard Deviation	0.28	0.12	0.23	0.51
Final Sludge Step	17.86	16.48	19.23	2.06
	17.78	17.56	20.82	0.65
	17.57	17.26	20.70	0.51
Average	17.73	17.10	20.25	1.07
Standard Deviation	0.15	0.56	0.88	0.85
95% Confidence	0.32	1.20	1.90	1.84

Figure 3.7 presents the PSD plots of S-107 slurries on a volume-weighted basis for each process stream. Each point represents the percentage of total slurry with particle size less than or equal to the given diameter. The PSDs of samples are compared after circulating each "as received" slurry in the instrument re-circulation line for 10 minutes at 40 mL/s. The plots indicate that in all five process streams, the particles or agglomerates are less than 60 microns in diameter.

**Table 3.15.** Measured Slurry Bulk Density and the Supernatant Density of S-107 Samples

Enhanced Sludge Settling Step	Slurry Bulk Density (g/mL)	Supernatant Density (g/mL)
Retrieval Step	1.13	1.0278, 1.0469, 1.0406
	1.12	1.0344, 1.0302, 1.0267
	1.14	1.0454, 1.0477, 1.0371
Average	1.13	1.0374
Standard Deviation	0.01	0.0082
95% Confidence	0.02	0.0024
Second Caustic Leach Step	1.15	1.0606, 1.0424, 1.0342
	1.14	1.0842, 1.0887, 1.0843
	1.15	1.0806, 1.0849, 1.0853
Average	1.15	1.0717
Standard Deviation	0.01	0.0207
95% Confidence	0.01	0.0060
Third Water Wash Step	1.10	1.0154, 1.0192, 1.0211
	1.08	1.0180, 1.0190, 1.0188
	1.09	
Average	1.09	1.0186
Standard Deviation	0.01	0.0019
95% Confidence	0.02	0.0010
Initial Extended Caustic Leach Step	1.18	1.1202, 1.1232, 1.1202
	1.20	1.1184, 1.1207, 1.1171
Average	1.19	1.1199
Standard Deviation	0.02	0.0021
95% Confidence	-	0.0011
Final Sludge Step	1.21	1.1534, 1.1560, 1.1690
	1.22	1.1706, 1.1725, 1.1820
	1.21	1.1765, 1.1780, 1.1781
Average	1.21	1.1707
Standard Deviation	0.00	0.0099
95% Confidence	0.01	0.0029

In addition, the percentages of larger particles and the mean volume-weighted distribution of particles or agglomerates decreased as the S-107 was subjected to high-caustic concentrations. For example, on a volume-weighted distribution, approximately 65% of the particles in the retrieval step slurry were less than 10 microns, whereas approximately 80% of particles after the second caustic leach were less than 10 microns in diameter. After the extended caustic leach, more than 96% of the particles were less than 10 microns in diameter (see Figure 3.7). With smaller particles within the slurry, the magnitude of surface forces acting on the particles dominate the body or bulk forces and interparticle interactions become significant. A summary of the particle sizes, on a volume-weighted basis, is presented in Table 3.18.



**Figure 3.7.** PSD of S-107 Slurries on a Volume-Weighted Basis for Each Process Stream in Cumulative Under-Size-Percentage Distribution

**Table 3.16.** Measured Dried Solids of S-107 Samples.

Enhanced Sludge Settling Step	Solid Density (g/cc)
Retrieval Step	2.685
	2.686
Average	2.686
Standard Deviation	0.001
Second Caustic Leach Step	2.857
	3.000
	2.857
Average	2.905
Standard Deviation	0.082
Third Water Wash Step	3.105
	3.112
Average	3.109
Standard Deviation	0.004
Initial Extended Caustic Leach Step	2.183
	2.183
Average	2.183
Standard Deviation	0.000
Final Sludge Step	2.391
	2.391
Average	2.391
Standard Deviation	0.000

The lower settling rate of the second caustic leach and final extended caustic leach may be attributed to these interparticle interactions, which in turn impact the type and density of the agglomerates formed. Not only does the high-caustic solution have a higher viscosity, which in turn slows the settling, but it is postulated that the agglomerates under these conditions are less compact and dense than during the water-wash steps. A high molarity electrolyte solution decreases the thickness of the diffused double layer surrounding particles, which in turn minimizes the strength of repulsive forces between the particles. When the repulsive forces are weakened, the attractive forces (van der Waals and London Dispersion forces) between the particles dominate, and the particles tend to aggregate rapidly and non-selectively. Under these conditions, weakly bound, open agglomerate structures of low density are formed (Hiemenz and Rajagopalan 1997). These agglomerates, in turn, settle more slowly than the more dense, compact agglomerate structures of the water-wash and retrieval steps.

Volume- and number-weighted histograms of the slurries are presented in Figures 3.8 and 3.9. In contrast to all other samples, >99% of the particles in the “final slurry” were smaller than 4 microns on a number-weighted distribution<sup>(a)</sup>. The slower settling rate of the extended caustic leach may be directly related to the small size of the particles in this step (see Figure 3.6).

**Table 3.17.** Summary of Cumulative Under-Size-Percentage Distribution and Mean Volume-Weighted Distribution for each Settle Decant Process Step.

Settle Decant Process Step	10 Percentile (microns)	50 Percentile (microns)	95 Percentile (microns)	Mean Volume (microns)
Retrieval Step	1.284	6.766	31.94	10.35
Second Caustic Leach Step	1.133	4.858	21.70	7.197
Third Water Wash Step	1.272	4.913	21.01	7.103
Initial Extended Caustic Leach Step	1.311	4.926	18.88	6.710
Final Sludge Step	0.418	1.649	8.442	2.615

The number of sub-micron particles did not change significantly between the second caustic leach and the third water wash. There were, however, more sub-micron particles in the retrieval step and less in the sample taken before the extended caustic-leach step. In these cases, the differences can be attributed to either dissolution or agglomeration of these small particles. Higher caustic concentrations will promote agglomeration of the sub-micron particles or, if they are caustic soluble, it will dissolve them. Most likely these sub-micron particles from the retrieval step were dissolved since they did not reappear during the third water wash. The reason for the reduction in these sub-micron particles from the sample taken at the beginning of the extended caustic-leach step is more difficult to determine.

(a) In a poly-dispersed slurry system containing a wide range of solid phases and sizes such as the S-107 samples, there is a large difference between the number-weighted basis and the volume-weighted basis. The number-weighted PSD is computed by counting each particle and by weighting all the particle diameters equally. The volume-weighted PSD, however, is weighted by the volume of each particle measured, which is proportional to the cube of the particle diameter. In this case, larger particles are treated as more important in the distribution than the smaller particles.

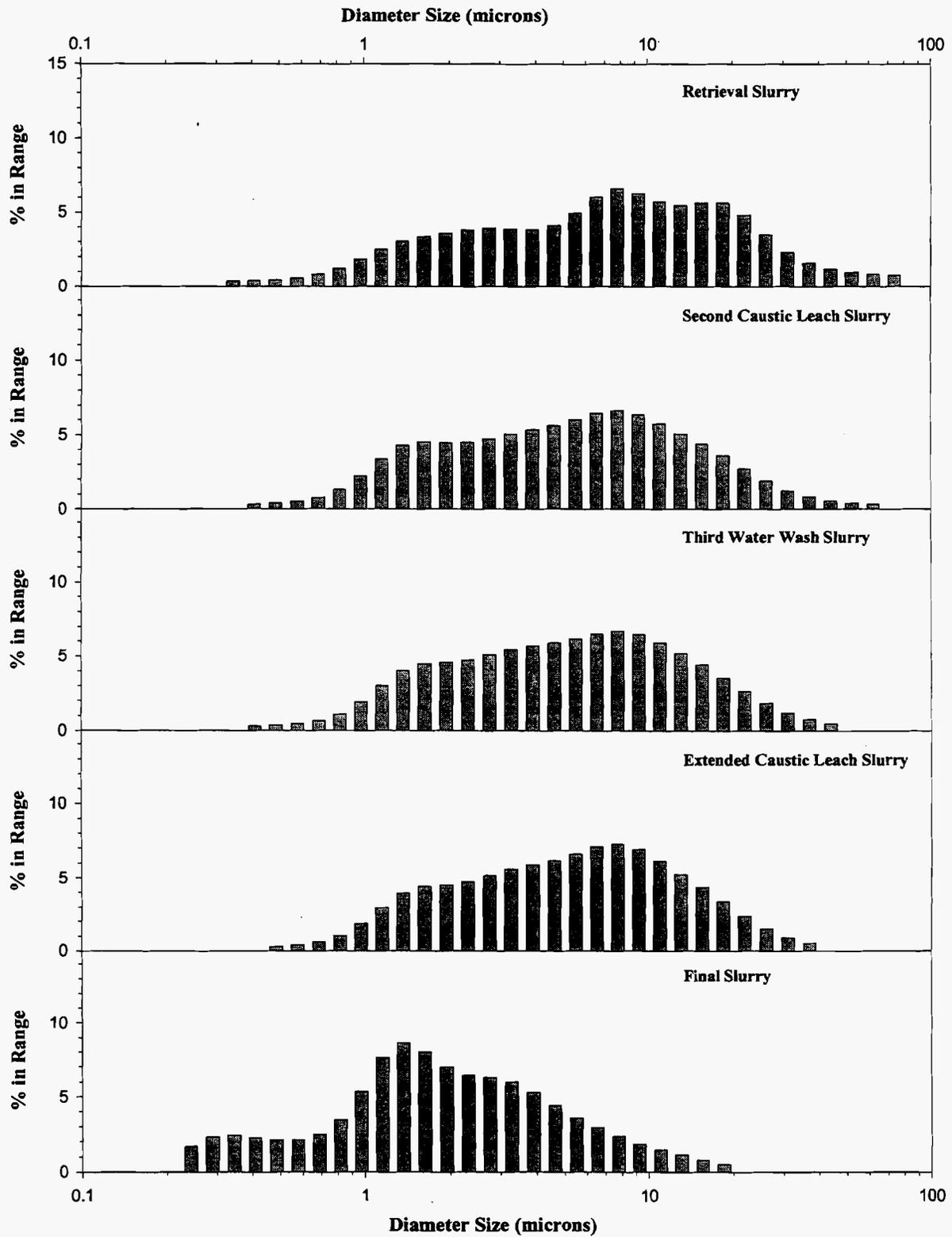


Figure 3.8 Volume-Weighted Histogram of "As Received" Slurries for Each Process Stream

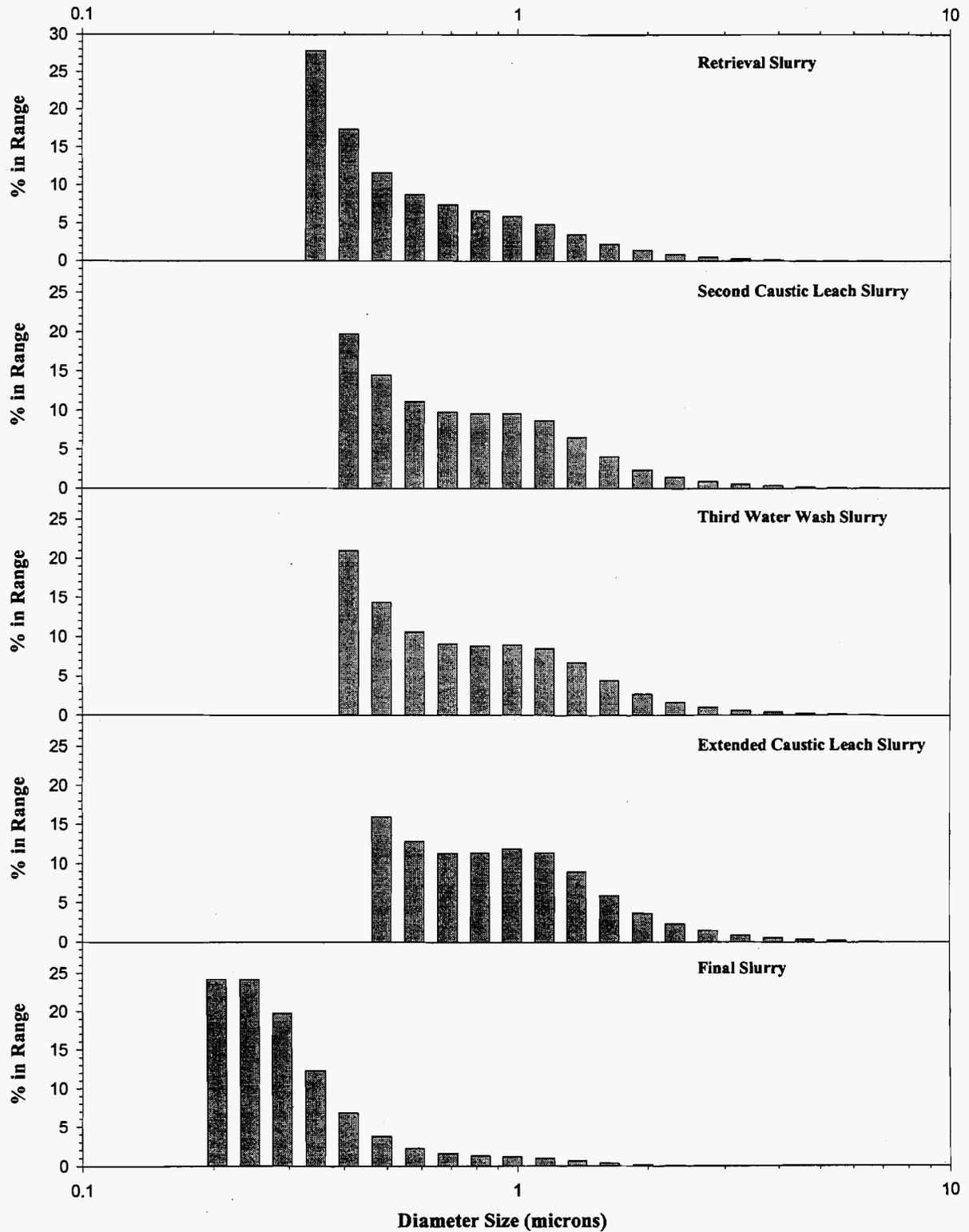
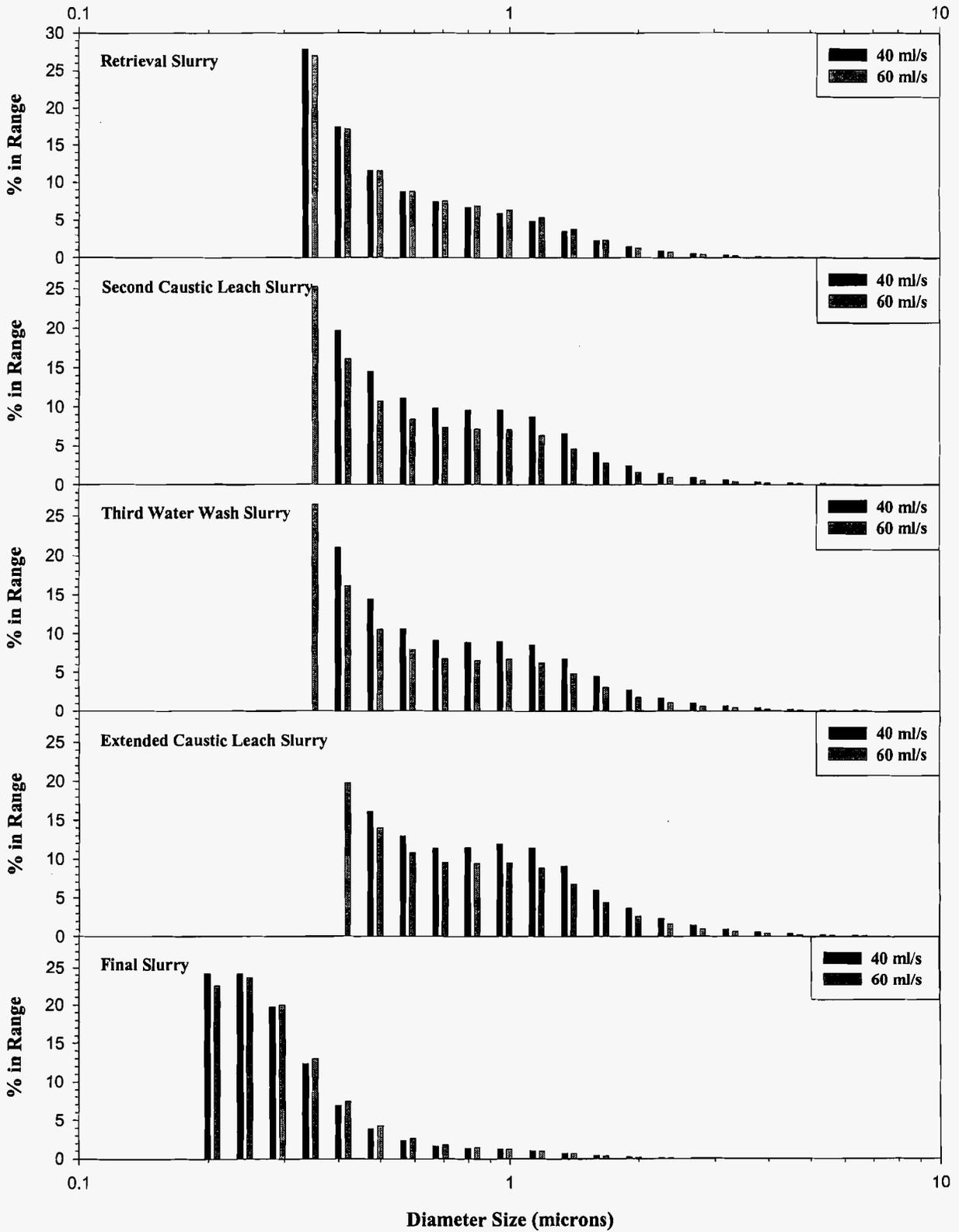


Figure 3.9. Number-Weighted Histogram of “As Received” Slurries for Each Process Stream

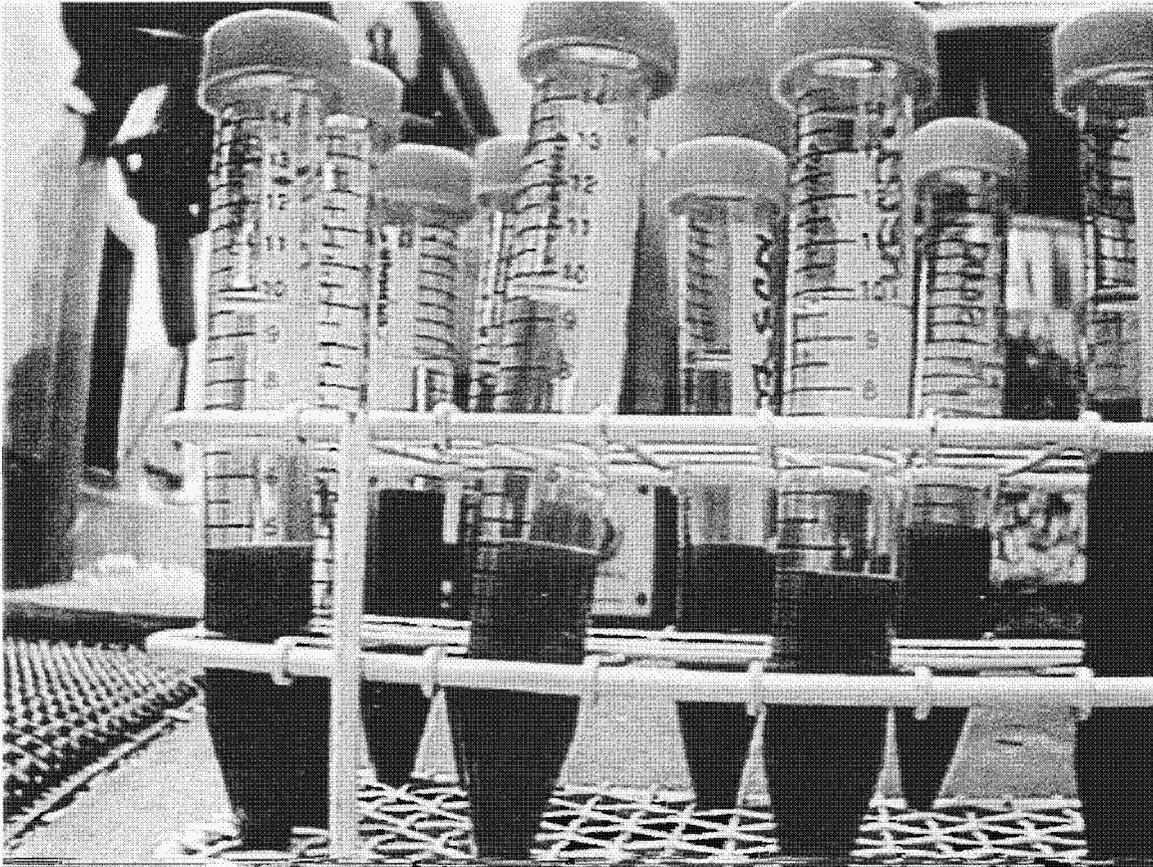
Figure 3.10 compares the PSD histograms of S-107 slurries on a number-weighted basis for each process stream after circulating the “as received” slurries in the instrument re-circulation line for 10 minutes at 40 mL/s, and after 20 minutes of total circulation time at 60 mL/s. These plots indicate that as the circulation flow rate increased from 40 mL/s to 60 mL/s, a significant fraction of agglomerates in the second caustic leach, extended caustic leach and the final slurry broke down. In contrast, the PSD distribution of samples from the retrieval step and the third water wash step was not influenced by changing the flow rate. These results may suggest that the strength of agglomerates in the retrieval step and the third water wash step (both at low ionic strength) are greater than the other steps (at high ionic strength). The agglomerate breakage in the second caustic leach, extended caustic leach, and the final sludge further supports the weakly bound, less compact agglomerate hypothesis discussed previously.

In Figure 3.11, a video snapshot of the replicate samples for the retrieval step after settling for 24 h is shown. The sediment volume reading is 4.5 mL for all three replicates shown in the foreground. Similar consistency can be seen in the second caustic leach and final sludge samples. The reproducibility of the observed sediment volume or height quantities in these video snapshots suggests that the sample in each graduated centrifuge cone gives a good representation of the homogenized slurry in the retrieval step.

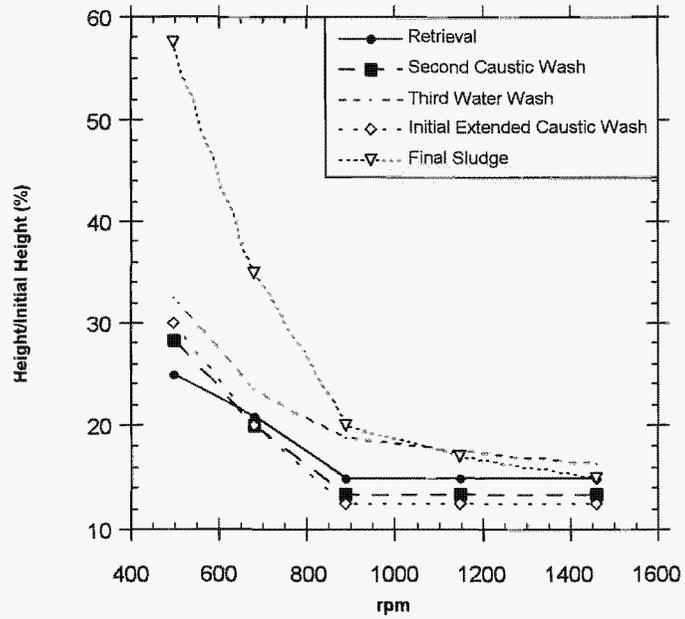
The ratio of the height of the centrifuged layer to the initial slurry height as a function of centrifugation rotational velocity is shown in Figure 3.8. These results were used to estimate the compressive-yield-stress values. These results indicate that the initial extended-caustic-wash and the second-caustic-wash samples reached maximum compression at rotational velocity of about 900 rpm while the final sludge sample at the same electrolyte concentration did not. The low compaction of final sludge sample is consistent with the liter-scale tests and, as previously discussed, may be explained by the dissolution of some solid phases as the S-107 slurry was subjected to the extended caustic leach. As the particles dissolved, the agglomerates became less poly-dispersed, resulting in a reduced ability of the particles to compact.



**Figure 3.10.** Number-Weighted Histogram of “As Received” Slurries at 40 mL/s and at 60 mL/s for Each Process Stream



**Figure 3.11.** Replicate Samples for the Retrieval Step After Settling for 24 Hours. The sediment volume is 4.5 mL for all three replicates (in foreground).



**Figure 3.12.** S-107 Sedimentation Rate in a Centrifuge

### 3.5 Theoretical Analysis

The results obtained from both the settling tests and the laboratory analysis were used to develop the expressions used in the following theoretical settling model. The results presented in this section are restricted to systems with the same particle size, the same component distribution, and the same solution chemistry as provided in the Tank S-107 sample.

#### 3.5.1 Hindered Settling

Expressions predicting the hindered settling rate as a function of solids loading were developed for the retrieval, caustic-leach, and the water-wash steps, based on the forms presented in Section 2.5. The coefficients were determined by performing a least-squares fit on the measured settling rates. The hindered settling rates, in cm/h, for all retrieval are given by the expression

$$u = \frac{27.3(1 - \phi)}{\left(1 - \frac{\phi}{0.176}\right)^{-3.97}} \quad \phi < \phi_g \quad (3.3)$$

where the suspension temperature is 85°C. Values for the solid-volume fractions,  $\phi$ , were determined using the measured supernate density and an assumed solid-particle density of 2.5 gm/cm<sup>3</sup>.<sup>(b)</sup> The hindered settling rate in cm/h for all caustic leach tests is given by the expression

$$u = \frac{10.87(1 - \phi)}{\left(1 - \frac{\phi}{0.189}\right)^{-4.0}} \quad \phi < \phi_g \quad (3.4)$$

where the suspension temperature is 80°C. The hindered settling rate in cm/h for all water wash tests is given by the expression

$$u = \frac{22.6(1 - \phi)}{\left(1 - \frac{\phi}{0.131}\right)^{-4.0}} \quad \phi < \phi_g \quad (3.5)$$

It is not surprising that the hindered settling expressions for the retrieval, caustic leach step, and water wash would be different from each other. The higher ionic strength of the caustic solution reduces the size of the electrical double layer, resulting in a change in the size and density of the particle aggregates as evidenced by the results in Section 3.4. The particle size and density are different for the retrieval step than for the water washes due primarily to the Al removal.

#### 3.5.2 Compressive Yield Stress

Expressions predicting the compression yield stress as a function of solids loading were developed for the retrieval steps, caustic leaches, and wash steps, based on the forms in Equation (2.4). The coefficients were determined by performing a least-squares fit on both the measured equilibrium

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(b) The solid density used for these calculations was estimated to be 2.5 g/cm<sup>3</sup>.

sediment heights resulting from the settling tests and those from centrifuge data. The compressive yield stress, in  $g/cm^2$  for all retrieval steps, is given by the expression

$$P_y(\phi) = 0.1826 \left[ \frac{\phi}{0.0759} - 1 \right]^{4.0} \quad \phi > \phi_g \quad (3.6)$$

The compressive yield stress in  $g/cm^2$  for all caustic leach tests is given by the expression

$$P_y(\phi) = 0.2454 \left[ \frac{\phi}{0.059} - 1 \right]^{4.0} \quad \phi > \phi_g \quad (3.7)$$

The compressive yield stress in  $g/cm^2$  for all water wash tests is given by the expression

$$P_y(\phi) = 0.0871 \left[ \frac{\phi}{0.0432} - 1 \right]^{4.0} \quad \phi > \phi_g \quad (3.8)$$

The dynamic compressibility factors for the retrieval steps, caustic leach tests, and water wash tests are shown in Table 3.18 below.

**Table 3.18.** Dynamic Compressibility Factors Fit to the Experimental Data

ESW Step	Dynamic Compressability Factor ( $\kappa$ ) $\times 10^{-5}$
Retrieval	0.97
Caustic Leach	0.24
Water Wash	0.33

These parameters were developed by calculating the sediment height for each sedimentation test or centrifuge measurement and optimizing these unknown values to minimize the error between the experiment and the model. The calculations for the sedimentation tests were based on the estimated and measured solids loading provided by the mass-balance calculations. In almost all cases, the predicted sediment heights were within 10% of the measured values. Such results give confidence that these expressions will provide accurate sediment-height predictions for both high and low solids-loading situations.

### 3.5.3 Transient Sedimentation Model

The hindered settling-rate expressions and the compressive yield-stress expressions are combined with the transient sedimentation model described previously to provide a computational model for predicting the sedimentation behavior of Tank S-107 pretreatment settle-decant systems.

The model was validated by comparing model results with the recorded settling data for the actual S-107 sludge. The model was used to calculate the entire density and network-stress profile at each

moment in time. The location of the top of the sediment was interpolated from the density profile. Examples of these comparisons are shown in Figures 3.13, 3.14, and 3.15 for the first retrieval step, first caustic-leach, and first water-wash cases, respectively. For each step in the ESW, the data from all of the settling and centrifugation curves are combined to develop the unknown parameters rather than producing individual expressions for each settling curve. That being the case, the model fits some settling curves better than others. In the case of the first retrieval step and the first water wash, the settling rates are reasonably well predicted. In contrast, the first caustic leach is less well predicted. Curves from other settling tests are shown in Appendix E. In each case, the initial settling velocity and the final sediment height appear to be relatively well predicted. The region of greatest discrepancy is the transition area between the settling of particles before contact with the sediment layer and the slow compression of the sediment layer.

### 3.5.4 Extrapolation to Full-Scale Tank Settling

The usefulness of this model is demonstrated by predicting the settling behavior of pretreatment settle-decant operations performed in a full-scale Hanford HLW tank. The height of the suspension is assumed to be 10 m, which is roughly equivalent to the height of existing waste in many of the double-shell tanks. Predictions were made for the retrieval, the caustic leach, and the wash steps (See Figures 3.16, 3.17 and 3.18). It should be emphasized that these results apply only to S-107 and similar sludges over the conditions investigated here.

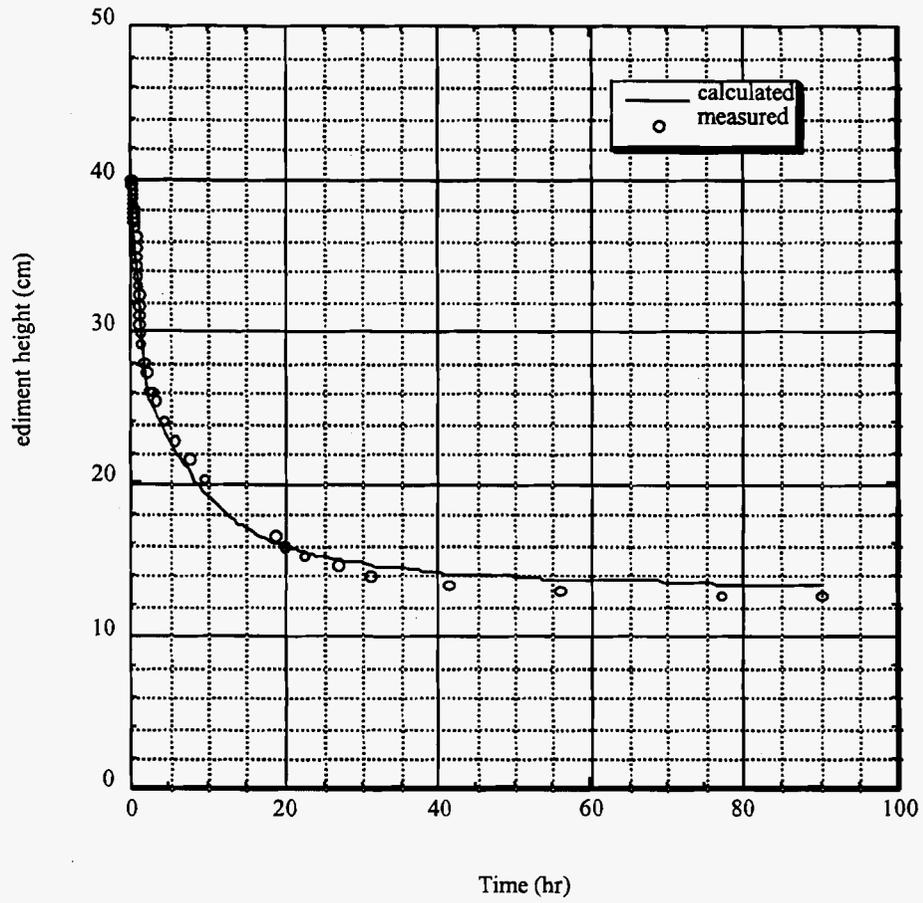
The retrieval-step simulation was performed assuming both 5 and 10 wt% solids loading and a temperature of 80°C. As was seen with the liter-scale settling tests, the settling and compaction occurred very quickly. Within 2 and 2.5 days, respectively, all the free-falling solids contacted the sediment layer. At this time, the solids concentration was well over 20 wt% in the sediment. After 3 days of settling, the sediment layer contained an average of 29.4 and 26.7 wt% solids for the initial solids concentrations of 5 and 10 wt%, respectively.

The caustic-leach simulation was also performed assuming both 5 and 10 wt% solids loading and a temperature of 80°C. The initial hindered settling rate of the caustic leach was slower than in the case of the retrieval step. It took approximately 5 and 6.5 days for all the free-falling solids to contact the sediment layer for initial loadings of 5 and 10 wt% solids, respectively. After 10 days of settling, the sediment layer contained an average of 31.1 and 29.7 wt% solids for the initial solids concentrations of 5 and 10 wt%, respectively.

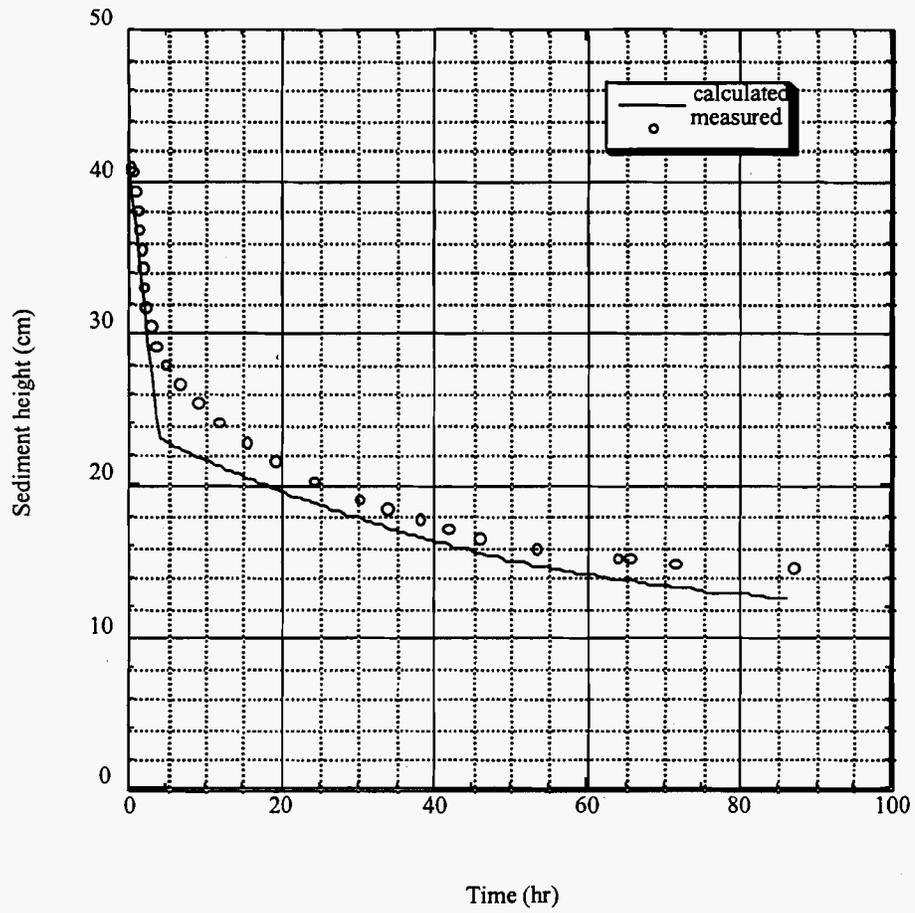
The water-wash simulation was performed assuming both 5 and 10 wt% solids loading and a temperature of 90°C. The water-wash steps had higher initial settling rates than the caustic leach, but did not compact to the same extent as the caustic leach. It took approximately 2.5 and 4.5 days for all the free-falling solids to contact the sediment layer for initial loadings of 5 and 10 wt% solids, respectively. After 10 days of settling, the sediment layer contained an average of 25.3 and 25.4 wt% solids for the initial solids concentrations of 5 and 10 wt%, respectively.

For all three ESW steps, the final sediment concentration was considerably higher for the million-gallon tank than for the liter-scale process. This was to be expected because the greater sludge depth would allow increased compaction of the sludge.

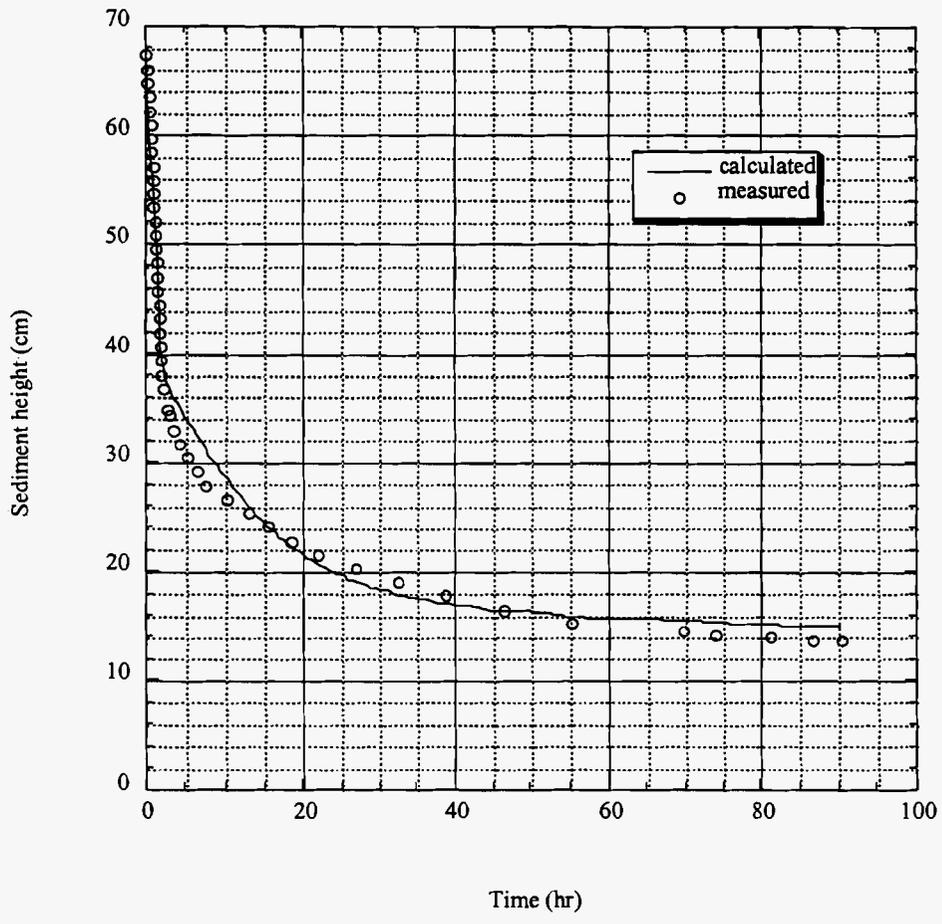
These simulations demonstrate that sedimentation data collected using laboratory-scale equipment can be extrapolated, using physically based computational sedimentation models, to predict the dynamic behavior of production-scale sedimentation systems.



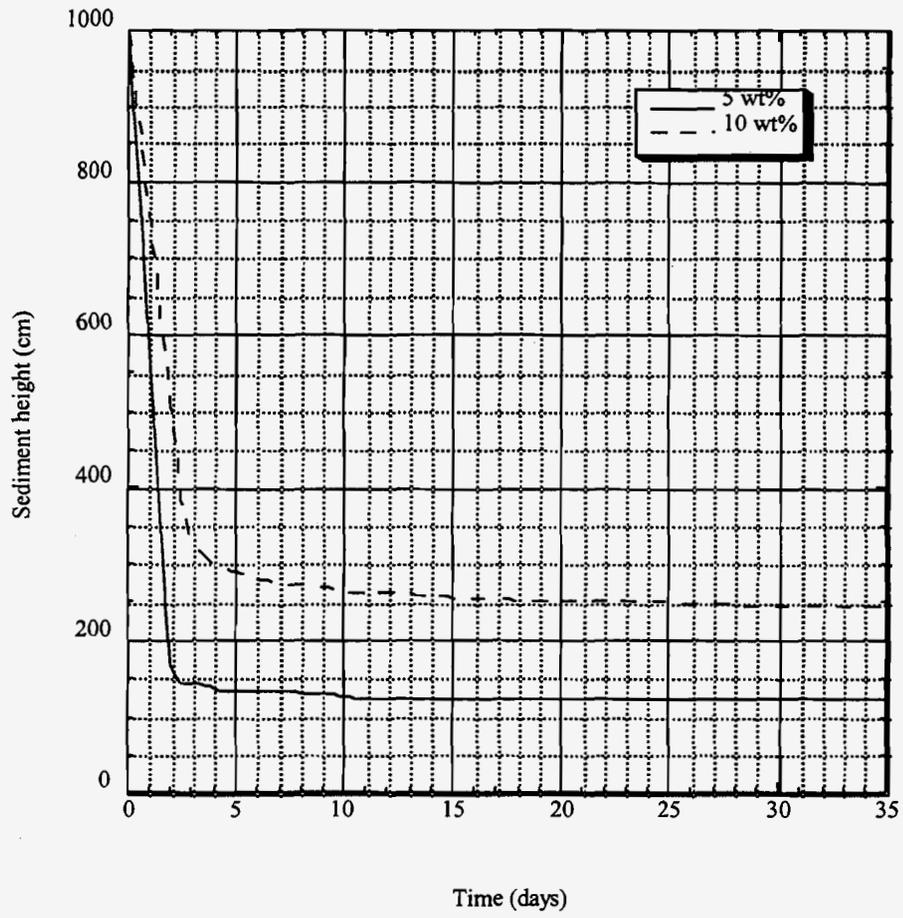
**Figure 3.13.** Comparison Between Predicted and Measured Sludge Interface Heights for the First Retrieval Step



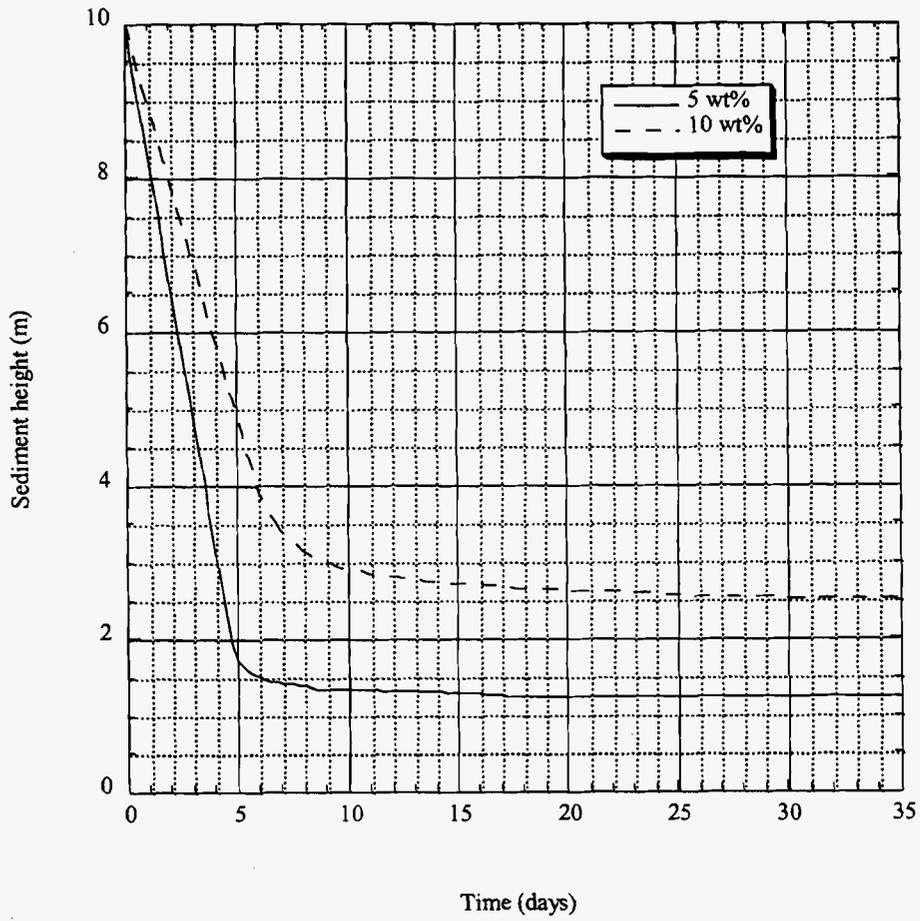
**Figure 3.14.** Comparison Between Predicted and Measured Sludge Interface Heights for the First Caustic Leach



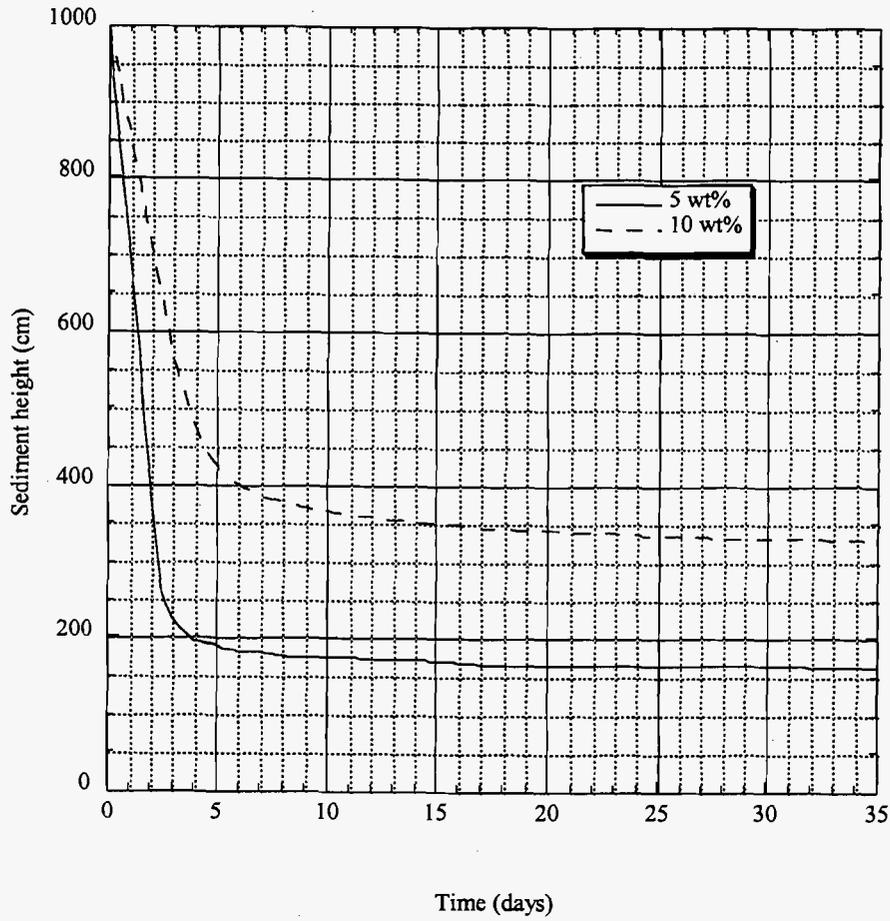
**Figure 3.15.** Comparison Between Predicted and Measured Sludge Interface Heights for the First Water Wash



**Figure 3.16.** Prediction of the Sludge Interface Height vs Time for S-107 Retrieval in a Full-Scale Tank



**Figure 3.17.** Prediction of the Sludge Interface Height vs Time for S-107 Caustic Leach in a Full-Scale Tank



**Figure 3.18.** Prediction of the Sludge Interface Height vs Time for S-107 Water Wash in a Full-Scale Tank

## 4.0 Conclusions

Of the major nonradioactive components, those that were significantly removed with enhanced sludge washing (ESW) and extended caustic leaching (ECL) were Al, Cr, and P. In all cases, the percentage removed for S-107 during the ESW is lower than that estimated in the TWRS O&UP (Kirkbride 1997) baseline. In the case of Cr and Al, the extended caustic leach significantly increased the total component removal. A comparison of the fraction of each of these components removed as compared to the TWRS baseline is shown in Table 4.1. The TWRS O&UP averages for all SSTs are shown for comparison only and do not necessarily imply that future processing will fail to meet its objectives.

**Table 4.1.** Major Non-Radioactive Component Removal Compared to TWRS O&UP Baseline

Component	S-107 Removal During ESW	S-107 Removal during ESW & ECL	TWRS O&UP Baseline for all SST
Al	47%	85%	91%
Cr	78%	100%	95%
P	82%	84%	86%

Roughly half of the Cr and P were removed during the retrieval step of the ESW. A small fraction of these components were also removed in subsequent steps. The aluminum was primarily removed during the caustic leach steps. Al is the primary constituent in the sludge and consists of 28% of the total mass on a dry basis. (If the aluminum is bohemite or gibbsite, these aluminum compounds would consist of 80% of the dried sludge).

During the extended caustic leach, the remaining Cr was removed from the sludge. The Al removal increased from 47% following the ESW to 85% following the extended caustic leach. The majority of the Al was removed during the first 100 hours of the extended leaching process. Very little was removed after that time. The majority of the Cr was removed within the first 80 hours.

The results of these tests differ somewhat from those of the laboratory-scale tests with S-107 performed by Lumetta et al. (1996) where more P and Al were removed while less Cr was leached. These differences could be attributable to differences in scale, starting material, and experimental conditions. The material studied here, for example, had significantly more aluminum and phosphorus and significantly less chromium than the material studied by Lumetta et al. Additionally, in the liter-scale apparatus, the sludge-receipt-tank level is measured by air bubblers. The higher Cr removal obtained here could be attributed to the oxidation of the Cr(III) to Cr(VI) by this contact with oxygen at elevated temperatures during both the leaching and washing steps.

Of the radioactive components, a significant amount of  $^{137}\text{Cs}$  (70%) was removed during the enhanced sludge wash. Only a very small fraction of the remaining radionuclides were removed, including  $^{90}\text{Sr}$  (0.03%) and TRU elements (0.2%). These results are consistent with other ESW tests. All of the supernatants (both individually and as a blend) removed from these washing steps, once vitrified as low level waste glasses (at 20 wt%  $\text{Na}_2\text{O}$ ), would be significantly less than the required 100 nCi/g in TRU elements and 20 Ci/m<sup>3</sup> in  $^{90}\text{Sr}$ .

Gravity settle/decant appears to be a viable approach to solid/liquid separations for Tank S-107 sludge considering its settling rate. The solids in the compacted sludge, however, was lower than the TWRS O&UP assumption of 20 wt% during the caustic leach and water wash steps. The solids generally

settled as a single, distinct interface at initial rates ranging from 3.2 to 16.7 cm/hr. This is significantly higher than the TWRS O&UP assumption for settling rate of 1-2 cm/hr.

The initial settling rate was generally slower for the caustic leach steps than for the retrieval and water wash steps. The final solids concentration in the sediment generally decreased for each step of the ESW process, reaching a maximum of 32.7 during the retrieval to a minimum of 15.1 wt% for the final water wash. Higher sludge layers encountered during full-scale operations will increase these solids concentrations even more and may bring them well beyond the TWRS assumption of 20 wt%.

A computational sedimentation model has been developed that incorporates the important features affecting the rate of sediment settling. Information obtained from both column settling tests and Colloids Laboratory tests was used to develop expressions for hindered settling rates and compressive yield stress. These expressions were incorporated into the transient sedimentation model, which was then validated by comparing predictions with both settling data for the bench-scale experiments and compression data for the centrifuged sludge experiments. The usefulness of this model was then demonstrated by performing simulations of solid-liquid separation through sedimentation in a full-scale waste tank for both leach and wash steps at different solids loadings. The model has verified that the solids concentration of S-107 in the compacted sediment layer in a full-scale 10-m-tall tank would be greater than the TWRS assumption of 20 wt% solids.

Although the empirical model provides estimates of settling on a large scale with varying solids concentrations and enhanced sludge washing steps, it could be improved. Further study of the sludge chemistry would enable the results of the model to be extrapolated to other particle size distributions, compositions, and solution chemistries.

## 5.0 References

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## **Appendix A**

### **Material Balance for the Liter-Scale Test**

# Appendix A: Material Balance for the Liter-Scale Test

Table A.1

Date/Time	Description	Settling Column Level (in)	Volume (mL)	Mass (g)	Density (g/mL)	Caustic Molarity (M)	Total Solids (g)	Weight Percent Undissolved Solids	Water Soluble Solids (g)	Leach Soluble Solids (g)	Insoluble Solids (g)
	Initial Test Sample	NA	554	1005.37	1.82	0	678.38				
	Amount lost	NA	2	3.41	1.82	0	2.30	67.5%			
	Effective Test Sample	NA	552	1001.96	1.82	0	676.08	67.5%			
							543.94	54.3%			
28-Apr-98	<b>Retrieval Wash</b>										
	Inhibited Water Added to C-202	NA	3128	3128.40	1.00	NA	NA	NA	NA	NA	NA
	Evaporation	NA	125	124.94	1.00	NA	NA	NA	NA	NA	NA
29-Apr-98	Slurry Sample -001	NA	24	26.97	1.13	0	3.66	13.6%	NA	0	0
	Slurry Sample -002	NA	16	17.95	1.13	0	2.44	13.6%	NA	0	0
	Slurry Sample -003	NA	21	24.19	1.13	0	3.29	13.6%	NA	0	0
	Slurry Sample -004	NA	17	19.98	1.13	0	2.66	13.6%	NA	0	0
	Lost Slurry	NA	9	10.00	1.13	0	1.36	13.6%	NA	0	0
	Initial Settling Conditions	20.125	3468	3919	1.13	0	532.2	13.6%	NA	0	0
	Evaporation	NA	940	939.71	1.00	NA	NA	NA	NA	NA	NA
1-May-98	Addition of DI water	NA	404	404	1.00	NA	NA	NA	NA	NA	NA
	Conditions after settling	22.75	2932	3383	1.15	0	532.2	15.7%	NA	0	0
	Final Settled Solids	31	1249	1624	1.30	0	532.20	32.76%			
4-May-98	Top Supernate Sample -005	NA	15	15.63	1.04	0	0	0	NA	0	0
	Middle Supernate Sample -006	NA	12	12.64	1.04	0	0	0	NA	0	0
	Bottom Supernate Sample -008	NA	13	13.27	1.04	0	0	0	NA	0	0
	Lost Supernate Sample	NA	15	15.33	1.04	0	0	0	NA	0	0
5-May-98	Deionized Water added	NA	700	700	1.00	NA	NA	NA	NA	NA	NA
	Inhibited Water added to C-202	NA	3080	3080	1.00	NA	NA	NA	NA	NA	NA
	Evaporation	NA	180	180	1.00	NA	NA	NA	NA	NA	NA
5-May-98	<b>Second Retrieval Wash</b>										
	Initial conditions	5.375	6478	6927	1.069	0	532.2	7.7%	NA	0	0
	Evaporation	NA	597	597	1.00	NA	NA	NA	NA	NA	NA
8-May-98	Top Supernate Sample -009	NA	14	13.93	1.02	0	0	0	NA	0	0
	Middle Supernate Sample -010	NA	14	14.14	1.02	0	0	0	NA	0	0
	Bottom Supernate Sample -011	NA	14	13.89	1.02	0	0	0	NA	0	0
	Final Column Conditions	8.50	5841	6288.23	1.08	0	532.2	8.5%	NA	0	0
	Supernatant removed	NA	4362	4449.49	1.02	0	0	0	NA	0	0
	Remaining slurry in C-201	29.875	1478	1838.74	1.24	0	532.2	28.9%	NA	0	0
	Final Settled Solids	30.625	1325	1682.62	1.27	0	532.20	31.63%			
	Deionized Water added	NA	1421	1421.40	1.00	NA	NA	NA	NA	NA	NA
	Caustic Added	NA	794	1068.52	1.35	10	NA	NA	NA	NA	NA
	Deionized Water added	NA	202	301.60	1.00	NA	NA	NA	NA	NA	NA
8-May-98	Evaporation	NA	325	325	1.00	NA	NA	NA	NA	NA	NA
	<b>Caustic Leach 1</b>										
	Initial Settling Column Conditions	19.625	5370	4206.09	1.18	2.2	458.4	10.9%	NA	0	0
	Top Supernate Sample -012	NA	15	16.44	1.12	2.2	0	0	NA	0	0
	Middle Supernate Sample -013	NA	14	15.35	1.12	2.2	0	0	NA	0	0
	Bottom Supernate Sample -014	NA	14	15.27	1.12	2.2	0	0	NA	0	0
	Evaporation	NA	646	646	1.00	NA	NA	NA	NA	NA	NA
	Conditions after Leaching	23	2831	3512.08	1.22	2.7	458.4	13.1%	NA	0	0
	Final Settled Solids	30.25	1402	1849.56	1.32	2.7	458.45	24.79%			
	Supernate removed	NA	1422	1605.20	1.12	2.7	0	0	NA	0	0

Date/Time	Description	Settling Column Level (in)	Volume (mL)	Mass (g)	Density (g/mL)	Caustic Molarity (M)	Total Solids (g)	Weight Percent Undissolved Solids	Water Soluble Solids (g)	Leach Soluble Solids (g)	Insoluble Solids (g)
	Final conditions	30	453	1906.89	1.31	2.7	458.4	24.0%	NA	0	0
	Caustic Addition	NA	372	591.14	1.35	10	NA	NA	NA	NA	NA
	Deionized Water added	NA	766	765.8	1.00	NA	NA	NA	NA	NA	NA
	Slurry Sample -015	NA	22	26.82	1.23	2.9	3.99	14.9%	NA	NA	3.99
	Slurry Sample -017	NA	17	20.85	1.25	2.9	3.10	14.9%	NA	NA	3.10
	Slurry Sample -018	NA	18	21.91	1.25	2.9	3.26	14.9%	NA	NA	3.26
	Slurry Sample -019	NA	18	22.34	1.23	2.9	3.33	14.9%	NA	NA	3.33
	Evaporation	NA	144	144	1.00	NA	NA	NA	NA	NA	NA
	Deionized Water added	NA	203	203.4	1.00	NA	NA	NA	NA	NA	NA
12-May-98	<b>Caustic Leach 2</b>										
	Initial Settling Column Conditions	24.5	2575	3141.55	1.22	2.9	459	14.6%	NA	NA	458.89
	Deionized Water Added	NA	340	340	1.00	NA	NA	NA	NA	NA	NA
	Evaporation	NA	662	662	1.00	NA	NA	NA	NA	NA	NA
15-May-98	Top Supernate Sample -020	NA	14	17.17	1.12	3.30	0	0	NA	NA	0
	Middle Supernate Sample -021	NA	10	11.70	1.12	3.30	0	0	NA	NA	0
	Bottom Supernate Sample -022	NA	11	12.83	1.12	3.30	0	0	NA	NA	0
	Final Conditions	26.25	2218	2780.16	1.25	3.30	458.9	16.5%	NA	NA	459
	Final Settled Solids	31	1249	1694.44	1.36	3.30	458.89	27.08%			
18-May-98	Caustic Addition	NA	3277	5670.43	1.12	3.00	NA	NA	NA	NA	NA
	Deionized Water Added	NA	540	540.00	1.00	NA	NA	NA	NA	NA	NA
	Evaporation	NA	603	603	1.00	NA	NA	NA	NA	NA	NA
	<b>Caustic Leach 2-Part 2</b>										
	Initial Column Conditions	10.5	5432	6387.70	1.18	3.16	458.9	7.2%	NA	NA	459
	Deionized Water Added	NA	437	437.32	1.00	NA	NA	NA	NA	NA	NA
	Evaporation	NA	599	599.15	1.00	NA	NA	NA	NA	NA	NA
21-May-98	Top Supernate Sample -023	NA	17	19.34	1.12	3.16	0	0	NA	NA	0
	Middle Supernate Sample -024	NA	12	13.76	1.12	3.16	0	0	NA	NA	0
	Bottom Supernate Sample -025	NA	13	14.22	1.12	3.16	0	0	NA	NA	0
	Conditions after Leaching	11.5	5228	6178.55	1.18	3.16	458.9	7.4%	NA	NA	459
	Supernate removed	NA	3378	4342.86	1.12	3.16	0	0	NA	NA	0
	Final Conditions	30.5	1351	1835.69	1.36	3.16	458.9	25.0%	NA	NA	459
	Final Settled Solids	30.9	1274	1749.98	1.37	3.16	458.89	26.22%			
	Inhibited Water Addition	NA	4553	4552.65	1.00	NA	NA	NA	NA	NA	NA
	Evaporation	NA	166	166.23	1.00	NA	NA	NA	NA	NA	NA
	Slurry Sample -026	NA	13	14.77	1.15	0.74	1.0339	7.0%	NA	NA	1.03
	Slurry Sample -027	NA	19	22.05	1.15	0.74	1.5435	7.0%	NA	NA	1.54
	Slurry Sample -028	NA	22	25.40	1.15	0.74	1.778	7.0%	NA	NA	1.78
	Slurry Sample -029	NA	21	24.26	1.15	0.74	1.6982	7.0%	NA	NA	1.70
22-May-98	<b>Water Wash 1</b>										
	Initial Settling Column Conditions	9.375	5582	6136	1.08	0.74	263.8	4.3%	NA	NA	264
	Evaporation	NA	265	265	1.00	NA	NA	NA	NA	NA	NA
26-May-98	Top Supernate Sample -030	NA	14	14.12	1.03	0.78	0	0	NA	NA	0
	Middle Supernate Sample -031	NA	14	14.03	1.03	0.78	0	0	NA	NA	0
	Bottom Supernate Sample -032	NA	14	14.24	1.03	0.78	0	0	NA	NA	0
	Conditions after Settling	10.875	5356	5828.27	1.09	0.78	263.8	4.5%	NA	NA	264
	Final Settled Solids	30.625	1325	1676.74	1.27	0.78	263.82	15.73%			

Date/Time	Description	Settling Column Level (in)	Volume (mL)	Mass (g)	Density (g/mL)	Caustic Molarity (M)	Total Solids (g)	Weight Percent Undissolved Solids	Water Soluble Solids (g)	Leach Soluble Solids (g)	Insoluble Solids (g)
	Supernate Decanted and Transferred to C-301	NA	3854	4073	1.03	0.78	0	0	NA	NA	0
	Sludge in C-201	30.25	1402	1756	1.25	0.78	263.8	15.0%	NA	NA	264
	Inhibited Water Added	NA	4552	4552.00	1.00	NA	NA	NA	NA	NA	NA
	Evaporation	NA	139	138.73	1.00	NA	NA	NA	NA	NA	NA
26-May-98	<b>Water Wash 2</b>										
	Initial Settling Column Conditions	8.625	5815	6168.84	1.06	0.15	263.8	4.3%	NA	NA	264
	Evaporation	NA	340	340.47	1.00	NA	NA	NA	NA	NA	NA
1-Jun-98	Top Supernate Sample -033	NA	14	14.12	1.02	0.20	0	0	NA	NA	0
	Middle Supernate Sample -034	NA	14	14.52	1.02	0.20	0	0	NA	NA	0
	Bottom Supernate Sample -035	NA	14	14.39	1.02	0.20	0	0	NA	NA	0
	Conditions after Settling	10.5	5431	5785	1.06	0.20	263.8	4.6%	NA	NA	264
	Final Settled Solids	30.675	1315	1585.64	1.21	0.20	263.82	16.64%			
	Supernate Decanted and Transferred to C-301	NA	3980	4059	1.02	0.20	0	0	NA	NA	0
	Sludge in C-201	30	1453	1726	1.19	0.20	263.8	15.3%	NA	NA	264
	Inhibited Water Added	NA	4552	4552	1.00	NA	NA	NA	NA	NA	NA
	Evaporation	NA	88	88	1.00	NA	NA	NA	NA	NA	NA
1-Jun-98	<b>Water Wash 3</b>										
	Initial Settling Column Conditions	8.125	5917	6190	1.05	0.05	263.8	4.3%	NA	NA	
9-Jun-98	Top Supernate Sample -036	NA	14	14.77	1.02	0.05	0	0	NA	NA	0
	Middle Supernate Sample -037	NA	13	13.81	1.02	0.05	0	0	NA	NA	0
	Bottom Supernate Sample -038	NA	13	13.45	1.02	0.05	0	0	NA	NA	0
	Evaporation	NA	343	342.62	1.00	NA	NA	NA	NA	NA	NA
	Conditions after Settling	10	5534	5806.99	1.05	0.05	263.8	4.5%	NA	NA	
	Final Settled Solids	30.25	1402	1591.68	1.14	0.05	263.82	16.57%			
	Supernate Decanted and Transferred to C-301	NA	2704	2758.16	1.02	0.05	0	0	NA	NA	0
9-Jun-98	<b>Water Wash 3 - Part 2</b>										
	Initial settling Conditions	23.25	2830	3048.82	1.08	0.05	263.8	8.7%	0	0	
	Evaporation	NA	485	484.69	1.00	NA	NA	NA	NA	NA	NA
	Final Settling Conditions	25.625	2346	2564.13	1.09	0.06	263.8	10.3%	0	0	
15-Jun-98	Top Supernate Sample -039	NA	16	16.78	1.02	0.06	0	0	NA	NA	0
	Middle Supernate Sample -040	NA	16	16.00	1.02	0.06	0	0	NA	NA	0
	Bottom Supernate Sample -041	NA	16	16.21	1.02	0.06	0	0	NA	NA	0
	Addition of DI water	NA	459	459.10	1.00	NA	NA	NA	NA	NA	NA
	Slurry Sample -042	NA	17	18.54	1.09	0.05	1.64	8.9%	NA	NA	1.64
	Slurry Sample -043	NA	24	26.33	1.09	0.05	2.34	8.9%	NA	NA	2.34
	Slurry Sample -044	NA	13	14.29	1.09	0.05	1.27	8.9%	NA	NA	1.27
	Slurry Sample -045	NA	7	7.53	1.09	0.05	0.67	8.9%	NA	NA	0.67
	Lost Slurry	NA	23	25.00	1.09	0.05	2.22	8.9%	NA	NA	2.22
	Evaporation	NA	250	250.38	1.00	NA	NA	NA	NA	NA	NA
17-Jun-98	Final Conditions	25.25	2422	2632.13	1.09	0.05	255.7	9.7%	0	0	256
	Final Settled Solids	30.5	1331	1464.27	1.08	0.05	255.68	17.46%			
	Supernate Decanted and Transferred to C-301	NA	995	1014.80	1.02	0	0	0	NA	NA	0
	Sludge in C-202	30.125	1407	1617.33	1.13	0.05	255.7	15.8%	NA	NA	256
	Addition of Caustic	NA	3573	4190.00	1.17	4.32	NA	NA	NA	NA	NA
	Lost Slurry	NA	3496	4027	1.15	3.10	177.3	4.4%	NA	NA	177

Table. A.2

Date/Time	Description	Settling Column Level (in)	Volume (mL)	Mass (g)	Density (g/mL)	Caustic Molarity (M)	Undissolved Solids (g)	Weight Percent Undissolved Solids	
26-Jun-98	<b>Extended Caustic Leach</b>								
	Initial Column Conditions	29.750	1504	1842.87	1.23	3.10	75.56	4.10%	
	Addition of DI water	NA	219	218.75	1.00	NA	NA	NA	
	Slurry Sample -046	NA	13	15.20	1.19	2.71	NA	4.01%	
	Slurry Sample -047	NA	13	15.00	1.19	2.71	NA	4.01%	
	Slurry Sample -048	NA	15	17.20	1.19	2.71	NA	4.01%	
	Slurry Sample -049	NA	15	17.25	1.19	2.71	NA	4.01%	
	Slurry Sample -050	NA	13	15.62	1.19	2.71	62.76	3.14%	
27-Jun-98	Slurry Sample -051	NA	19	22.40	1.19	2.71	55.88	2.82%	Sample Analyzed
	Supernatant Sample -052	NA	19	21.19	1.12	2.71	47.62	2.43%	Sample Analyzed
	Evaporation	NA	240	240.15	1.00	NA	NA	NA	
	Final Column Conditions	30.375	1376	1696.47	1.23	3.11	47.62	2.97%	
	Addition of DI water	NA	149	148.72	1.00	NA	NA	NA	
	Slurry Sample -053	NA	15	18.23	1.19	2.84	47.62	2.97%	
28-Jun-98	Slurry Sample -054	NA	22	26.47	1.19	2.84	40.51	2.22%	Sample Analyzed
	Supernatant Sample -055	NA	13	14.55	1.12	2.84	38.47	2.14%	Sample Analyzed
	Evaporation	NA	251	251.23	1.00	NA	NA	NA	
	Final Column Conditions	31.125	1273	1534.72	1.25	3.33	38.47	2.46%	
	Addition of DI water	NA	281	280.77	1.00	NA	NA	NA	
	Slurry Sample -056	NA	20	23.33	1.19	2.78	38.31	2.11%	Sample Analyzed
29-Jun-98	Slurry Sample -057	NA	18	20.84	1.19	2.78	34.08	1.90%	Sample Analyzed
	Supernatant Sample -058	NA	10	11.51	1.12	2.78	34.88	1.97%	Sample Analyzed
	Evaporation	NA	182	182.36	1.00	NA	NA	NA	
	Final Column Conditions	30.875	1274	1577.45	1.24	3.12	34.88	2.33%	
	Addition of DI water	NA	102	102.00	1.00	NA	NA	NA	
30-Jun-98	Supernatant Sample -059	NA	10	11.82	1.19	2.92	31.06	1.85%	Sample Analyzed
	Evaporation	NA	194	194.11	1.00	NA	NA	NA	
	Final Column Conditions	31.375	1172	1473.52	1.26	3.33	31.06	2.09%	
	Addition of DI water	NA	204	204.06	1.00	NA	NA	NA	
	Supernatant Sample -060	NA	15	18.18	1.19	2.90	31.57	1.88%	Sample Analyzed
	Evaporation	NA	163	163.27	1.00	NA	NA	NA	
	Final Column Conditions	31.250	1158	1496.13	1.25	3.24	31.57	2.09%	
	Addition of DI water	NA	178	178.00	1.00	NA	NA	NA	
	Evaporation	NA	305	305.43	1.00	NA	NA	NA	
3-Jul-98	Addition of DI water	NA	306	306.00	1.00	NA	NA	NA	
	Initial Settling Conditions	30.375	1376	1674.70	1.22	2.87	31.57	1.88%	
	Evaporation	NA	434	433.67	1.00	NA	NA	NA	
	Final Settling Conditions	32.500	943	1241.03	1.32	3.96	31.57	3.91%	Sample Analyzed
	Top Supernatant Sample -061	NA	10	11.25	1.17	3.96	NA	NA	
	Middle Supernatant Sample -062	NA	9	10.22	1.17	3.96	NA	NA	Sample Analyzed
	Bottom Supernatant Sample -063	NA	12	13.58	1.17	3.96	NA	NA	
	Evaporation	NA	123	122.68	1.00	NA	NA	NA	
	Final Column Conditions	33.250	790	1082.80	1.37	4.46	48.52	4.48%	
	Slurry Sample -064	NA	14	16.97	1.21	4.46	0.76	4.48%	
	Slurry Sample -065	NA	14	17.38	1.21	4.46	0.77	4.48%	
	Slurry Sample -066	NA	14	17.52	1.21	4.46	0.79	4.48%	
	Slurry Sample -067	NA	14	17.39	1.21	4.46	0.78	4.48%	
	Remaining Slurry	NA	732	1013.69	1.38	4.46	45.43	4.48%	
	* All boxes highlighted in gray represent known values. All other values have been calculated from these numbers.								

## **Appendix B**

### **Detailed Operations Description**

## Appendix B: Detailed Operations Description

### B.1 Retrieval Slurry Preparation

#### B.1.1 Preparation of Tank Waste Sample

Sludge sampling of single-shell tank 241-S-107 was performed in September 1995. Three eight-segment core samples were taken and analyzed. These samples were stored in the 222-S laboratory. In February of 1998, 11 of these samples were transported to the 325 building. These samples were combined together to form the slurry for the settling experiments. The original core samples and their recovered weights during homogenization are as follows:

<u>Sample Number</u>	<u>Sample Weight</u>
C105 S6	68.964 g
C105 S7	136.382 g
C105 S7	150.973 g
C105 S8	33.548 g
C110 S7	119.560 g
C110 S8	128.578 g
C111 S6	81.393 g
C111 S6	70.492 g
C111 S6	49.054 g
C111 S7	101.159 g
C111 S7	97.317 g

The total weight of these samples was 1,037.42 grams. Many of the sludge samples were relatively dry and could not be homogenized without water addition. We added 205 grams of deionized water to the sludge, and the mixture was homogenized with an Omni mixer. Three samples were taken to determine the initial weight percent solids. Two additional samples were taken to determine the initial sludge density. The final mass of the sample used for the settling experiment was 1005.367 g. This was transferred to the 324 building and poured into the sludge-receipt tank in the hot cell on April 28, 1998. The sludge addition and retrieval steps were performed under procedure 3I-SOP-REC-F-27, Rev. 4. This procedure was completed on May 8, 1998.

### B.2 First Retrieval Wash and Settling Test

#### B.2.1 Add Inhibited Water, Heat, and Agitate

The amount of inhibited water (0.01 M NaOH and 0.01 M NaNO<sub>2</sub>) to be added for the first retrieval wash was determined to be 3128 grams to create a slurry with a solids concentration of 13.6 wt%. This was transferred to the Cold Chemical Tank (C-101). Some of this water was added to the sample container and mixed with a screwdriver before pouring the sludge into the sludge-receipt tank (C-202). The sludge was sufficiently thick even after addition of water to require a screw driver to poke the sludge through the funnel. After placing the sludge in the sludge-receipt tank, the sample container

was rinsed four times with approximately 400 mL of this water to ensure that none remained. All of this liquid was poured into the sludge-receipt tank. Less than one gram of sludge material remained in the container after rinsing. The remainder of the liquid in the cold chemical tank was transferred directly to the sludge-receipt tank. The agitator was turned on in the sludge-receipt tank. While in the sludge-receipt tank, the slurry was recirculated through the high-speed, centrifugal pump P-204 for 30 minutes.

The off-gas system was set for heating, and the heater in the sludge-receipt tank was turned on. While the slurry was heating, the agitator continued to operate. The slurry in the sludge-receipt tank was heated to 100°C and held at that temperature for 30 minutes while being mixed by the agitator. Meanwhile, the temperature of the water in the circulating hot-water bath was heated to 80°C.

### **B.2.2 Slurry Sampling**

When the slurry had mixed for 30 minutes at 100°C, the heater was turned off. Approximately 180 g of water evaporated during this time. One slurry sample (SPD-S107-01) was taken out of sample port S-202 during recirculation. Approximately 10 g of slurry were lost during the first sampling due to overfilling of the sample bottle. Then half of the slurry in the sludge-receipt tank was transferred to the settling column. Two additional samples (SPD-S107-02 and SPD-S107-03) were removed, and half of the remaining slurry was transferred to the settler. One final sample (SPD-S107-04) was taken, and the rest of the slurry was transferred to the column. The sample weights were 26.97, 17.95, 24.19, and 19.58 grams, respectively. The solids/liquid interface heights in each of the sample bottles was compared after 24 hours, and it was found that all of the samples had the same heights relative to total volume.

### **B.2.3 First Retrieval Wash Settling Test**

The slurry was transferred from the sludge-receipt tank to the sludge settler on April 29, 1998. The solids in the sludge settler were then resuspended by 1) placing the sample tube approximately 2 inches below the liquid level and 2) circulating the slurry through the sample tube, pump P-202, and back through a port in the bottom of the sludge settler. The slurry was circulated for 5 minutes, and then the settling test began. The hot water bath was at 80°C. At the start of the settling test, the slurry volume was 3468 mL, corresponding to a height of 20 1/8 inches in the settler.

During regular work hours, the solids/liquid interface was visually observed and recorded on data sheets. The entire settling process was also recorded on videotape (SPD-S107-01 and SPD-S107-02) so that the entire settling rate and solids/liquid interface could be documented. Electronic data were monitored and recorded every 10 minutes on a data disk (S107-1data.xls) automatically by the data acquisition system. The settling test was terminated after 5 days (120 hours). Approximately 940 g of water evaporated during the settling test. At 43 hours into the test, 404 mL of deionized water were added to the column to account for evaporation. The final total volume was 2932 mL, corresponding to a column height of 22 3/4 inches. The final volume of solids was 1249 mL, corresponding to a height of 31 inches in the sludge settler. The test was completed on May 4, 1998.

### **B.2.4 Axial Sampling**

Four samples (SPD-S107-05 through SPD-S107-08) of the supernate were taken at axial elevations of 24, 27, and 30 inches from the top of the sludge settler. The top sample was taken twice because the first sample missed the bottle and approximately 15 grams of supernate was lost. Sample bottle SPD-S107-07 was dropped while trying to obtain the bottom supernate sample and was replaced

by SPD-S107-08. The samples were obtained by 1) inserting the sample tube into the supernate to the desired sample location (upper samples first), 2) running the peristaltic pump (P-301) clockwise to draw the sample into the tube, 3) raising the sample tube out of the sludge settler, 4) placing the sample bottle under the sample tube, and 5) running the pump (P-301) counter clockwise to discharge the sample into the bottle. The samples were weighed, and the top, middle, and bottom supernate sample weights were 15.33, 12.64, and 13.27 grams, respectively. The supernate was not removed after the completion of this settling test. Approximately 180 g of water evaporated between the time the first settling test was finished and the second retrieval wash settling test began.

## **B.3 Second Retrieval Wash and Settling Test**

### **B.3.1 Inhibited Water Addition**

We added 3080 grams of inhibited water (0.01 M NaOH and 0.01 M NaNO<sub>2</sub>) for the second retrieval wash to lower the solids concentration to 7.7 wt%. The water was added to the cold chemical tank and then transferred to the settling column. We also added 700 mL of deionized water to the settling column to account for evaporation losses.

On May 5, 1998, the solids in the sludge settler were then resuspended by 1) placing the sample tube approximately 2 inches below the liquid level and 2) circulating the slurry through the sample tube, pump P-202, and back through a port in the bottom of the sludge settler. After five minutes, the sample tube was raised until it cleared the top of the supernate. The pump continued to run for another minute to clear the line.

### **B.3.2 Second Retrieval Wash Settling Test**

Pump P-202 was shut off and the settling test began. The hot-water bath was maintained at a temperature of 80°C. At the start of the settling test, the slurry volume was 6478 mL, corresponding to a height of 5 3/8 inches in the settler. Approximately 597 g of water evaporated during the settling test.

During regular work hours, the solids/liquid interface was visually observed and recorded on data sheets. Also, the entire settling process was also recorded on videotape (SPD-S107-02), so that the entire settling rate and solids/liquid interface could be documented. Electronic data were monitored and recorded every 10 minutes on a data disk (S107-1data.xls) automatically by the data acquisition system. The settling test was terminated after 3 days (72 hours), on May 8, 1998. The final total volume was 5841 mL, corresponding to a column height of 8 1/2 inches. The final volume of solids was 1325 mL, corresponding to a height of 30 5/8 inches in the sludge settler.

### **B.3.3 Axial Sampling**

Three samples (SPD-S107-09 through SPD-S107-11) of the supernate were taken at axial elevations of 10, 20, and 29 inches from the top of the sludge settler (measured on the sludge settler ruler). The samples were obtained by 1) inserting the sample tube into the supernate to the desired sample location (upper samples first), 2) running the peristaltic pump (P-301) clockwise to draw the sample into the tube, 3) raising the sample tube out of the sludge settler, 4) placing the sample bottle under the sample tube, and 5) running the pump (P-301) counter clockwise to discharge the sample into the bottle. The samples were weighed and the top, middle, and bottom supernate sample weights were 13.93, 14.18, and 13.89 grams, respectively.

### **B.3.4 Supernate Decant and Transfer**

Supernate was decanted by lowering the sample tube to within 2 inches of the solids layer and slowly pumping the supernate into the batch collection tank, C-301. As the liquid level came near the end of the sample tube, the sample tube was incrementally lowered until it was within  $\frac{3}{4}$  inches of the solids level. Supernate removed in the process was 4362 mL, determined from observations of the liquid height in the sludge settler before and after decanting. The supernate was then transferred from the batch collection tank to the supernate holding tank, C-302.

### **B.3.5 Deionized Water Addition**

The amount of deionized water to be added for the first caustic leach and settling test was calculated to be 1421 grams. This amount of water was weighed and added to the cold chemical tank. The deionized water was pumped into the sludge settler. The solids in the sludge settler were then resuspended by 1) placing the sample tube approximately 2 inches below the liquid level and 2) circulating the slurry through the sample tube, pump P-202, and back through a port in the bottom of the sludge settler. After four minutes, the valve at the bottom of the sludge settler was opened, and the slurry was transferred to the sludge-receipt tank. The pump continued to run for a minute to clear the line. The agitator in the sludge-receipt tank was turned on.

## **B.4 First Caustic Leach and Settling Test**

The First Caustic Leach and Settling Test was performed using procedure 3I-SOP-REC-F-38, Rev. 4. It was started on May 8, 1998, and completed on May 12, 1998.

### **B.4.1 Add Caustic Solution, Heat, and Agitate**

The amount of 10 M NaOH needed to achieve the desired final concentration of 2 M NaOH was calculated to be 1070 grams to create a slurry with a solids concentration of 10.9 wt%. This amount of caustic leach was added to the cold chemical tank and was pumped through the settler column to rinse out the column. It was then transferred into the sludge-receipt tank.

On May 8, 1998, the off-gas system was set for heating, and the heater in the sludge-receipt tank was turned on. While the slurry was heating, the agitator continued to operate. The slurry in the sludge-receipt tank was heated to 100°C and held at that temperature for 7.5 hours while mixing with the agitator. Meanwhile, the temperature of the water in the circulating hot water bath was heated to 80°C.

### **B.4.2 First Caustic Leach Settling Test**

When the slurry had mixed at 100°C for 7.5 hours, and the temperature in the circulating hot water bath was at 80°C, the slurry was transferred from the sludge-receipt tank to the sludge settler. It was estimated that approximately 325 g of water evaporated while the slurry was in the sludge-receipt tank. We added 202 grams of deionized water to the cold chemical tank and transferred it to the sludge-receipt tank. This was then added to the slurry in the settler column. The solids were resuspended, and the settling test began. At the start of the settling test, the slurry volume was 3570 mL, corresponding to a height of 19  $\frac{5}{8}$  inches in the sludge settler.

During regular work hours, the solids/liquid interface was visually observed and recorded on data sheets. Also, the entire settling process was also recorded on videotape (SPD-S107-003) so that the entire settling rate and solids/liquid interface could be documented. Electronic data were monitored and recorded every 10 minutes on a data disk (S107-1data.xls) automatically by the data-acquisition system. The settling test was terminated on May 12, 1998, after 3 days and 15 hours (87 hours). The final total volume was 2881 mL, corresponding to a column height of 23 inches. The final volume of solids was 1402 mL, corresponding to a height of 30 ¼ inches in the sludge settler. Approximately 646 grams of water evaporated during the settling test.

### **B.4.3 Axial Sampling**

Three samples (SPD-S107-12 through SPD-S107-14) of the supernate were taken at axial elevations of 24, 27, and 29 inches from the top of the sludge settler (measured on the sludge settler ruler). The samples were obtained by 1) inserting the sample tube into the supernate to the desired sample location (upper samples first), 2) running the peristaltic pump (P-301) clockwise to draw the sample into the tube, 3) raising the sample tube out of the sludge settler, 4) placing the sample bottle under the sample tube, and 5) running the pump (P-301) counter clockwise to discharge the sample into the bottle. The samples were weighed and the top, middle, and bottom supernate sample weights were 16.44, 15.85, and 15.27 grams, respectively.

### **B.4.4 Supernate Decant and Transfer**

Supernate was decanted by lowering the sample tube to within 2 inches of the solids layer and slowly pumping the supernate into the batch collection tank, C-301. As the liquid level came near the end of the sample tube, the sample tube was incrementally lowered until it was within ¼ inch of the solids level. Supernate removed in the process was 1429 mL, determined from observations of the liquid height in the sludge settler before and after decanting. The supernate was not transferred from the batch collection tank to the supernate holding tank, C-302, as was originally planned, to allow the bubbler line of the holding tank to unplug.

## **B.5 Second Caustic Leach and Settling Test**

The Second Caustic Leach and Settling Test was performed using Procedure 3I-SOP-REC-F-39, Rev. 4. This procedure was started on May 12, 1998, and completed on May 21, 1998.

### **B.5.1 Deionized Water/Caustic Leach Addition and Solids Resuspension**

The amount of 10 M NaOH needed to achieve the desired final concentration of 3 M NaOH was calculated to be 501 grams, to create a slurry with a solids concentration of 14.6 wt%. This amount of caustic leach was added along with 766 grams of deionized water to the cold-chemical tank. All of the caustic except 500 mL was transferred to the sludge settler.

The solids in the sludge settler were then resuspended by 1) placing the sample tube approximately 2 inches below the liquid level and 2) circulating the slurry through the sample tube, pump P-202, and back through a port in the bottom of the sludge settler. After 2 minutes, the valve was opened to move the slurry into the sludge-receipt tank. The pump continued to run for another minute to clear the line. The rest of the caustic left in the cold chemical tank was then transferred into the settling

column and pumped through to rinse the column. The valve was then opened to transfer the caustic to the sludge-receipt tank. The pump continued to run for another minute to clear the line. The agitator was turned on in the sludge-receipt tank.

On May 12, 1998, the off-gas system was set for heating, and the heater in the sludge-receipt tank was turned on. While the slurry was heating, the agitator continued to operate. The slurry in the sludge-receipt tank was heated to 100°C and held at that temperature for 5.75 hours while mixing with the agitator. Meanwhile, the temperature of the water in the circulating hot water bath was heated to 80°C. Approximately 139 grams of water evaporated during the sludge preheating.

### **B.5.2 Slurry Sampling**

When the slurry had mixed for 5.75 hours at 100°C, the heater was turned off. At this point, four sludge samples (SPD-S107-15 and SPD-S107-17 through SPD-S107-19) were taken from the sludge-receipt tank through sample port S-202. Sample bottle SPD-S107-16 was dropped before a sample had been collected, so no material was lost. The sample weights were 26.82, 20.83, 21.91, and 22.34 grams, respectively. The solids/liquid interface heights in each of the sample bottles were compared after 24 hours, and it was found that all of the samples had the same heights relative to total volume.

### **B.5.3 Second Caustic Leach Settling Test**

The slurry in the sludge-receipt tank was transferred to the settling column on May 12, 1998. We added 203 g of deionized water to the settler to account for evaporation. The solids in the sludge settler were then resuspended by 1) placing the sample tube approximately 2 inches below the liquid level and 2) circulating the slurry through the sample tube, pump P-202, and back through a port in the bottom of the sludge settler. The pump was run for 4 minutes, and then the sample tube was raised above the top of the slurry. The pump continued to run for another minute to clear the line and was then shut off. Immediately after this, the settling-test began. At the start of the settling test, the slurry volume was 2575 mL, corresponding to a height of 24 1/2 inches in the sludge settler.

During regular work hours, the solids/liquid interface was visually observed and recorded on data sheets. Also, the entire settling process was also recorded on videotape (SPD-S107-04) so that the entire settling rate and solids/liquid interface could be documented. Electronic data were monitored and recorded every 10 minutes on a data disk (S107-1data.xls) automatically by the data-acquisition system. The settling test was terminated on May 15, 1998, after 69 hours. Approximately 662 grams of water evaporated during the settling test. We added 340 grams of deionized water 39 hours into the test to account for evaporation losses. The final total volume was 2218 mL, corresponding to a column height of 26 ¼ inches. The final volume of solids was 1249 mL, corresponding to a height of 31 inches in the sludge settler.

### **B.5.4 Axial Sampling**

Three samples (SPD-S107-20 through SPD-S107-22) of the supernate were taken at axial elevations of 27, 29, and 30 inches from the top of the sludge settler (measured on the sludge settler ruler). The samples were obtained by 1) inserting the sample tube into the supernate to the desired sample location (upper samples first), 2) running the peristaltic pump (P-301) clockwise to draw the sample into the tube, 3) raising the sample tube out of the sludge settler, 4) placing the sample bottle under the sample tube, and 5) running the pump (P-301) counter clockwise to discharge the sample into

the bottle. The samples were weighed, and the top, middle, and bottom supernate sample weights were 15.17, 11.70, and 12.83 grams, respectively.

### **B.5.5 Caustic Addition and Solids Resuspension**

At this point, an additional 3670 grams of 3 M caustic (NaOH) were added to the settling column so that the settling test could be performed at 7.2 wt% solids. The solids were resuspended, and a new settling test was begun. At the start of the settling test, the slurry volume was 5432 mL, corresponding to a height of 10 ½ inches in the sludge settler.

During regular work hours, the solids/liquid interface was visually observed and recorded on data sheets. Also, the entire settling process was also recorded on videotape (SPD-S107-005) so that the entire settling rate and solids/liquid interface could be documented. Electronic data were monitored and recorded every 10 minutes on a data disk (S107-1data.xls) automatically by the data-acquisition system. The first settling test was terminated after 68 hours because the solids/liquid interface had fallen below the screen of the camera 4 hours after the test began. The solids were resuspended, and the settling test was started again. The settling test was terminated on May 21, 1998, after approximately 3 days (71.5 hours). We added 977 grams of deionized water to the column during the settling tests to account for evaporation losses. Approximately 1202 grams of water evaporated during the two tests. The final total volume was 5279 mL, corresponding to a column height of 11 ¼ inches. The final volume of solids was 1274 mL, corresponding to a height of 30 7/8 inches in the sludge settler.

### **B.5.6 Axial Sampling**

Three samples (SPD-S107-23 through SPD-S107-25) of the supernate were taken at axial elevations of 13, 21, and 30 inches from the top of the sludge settler (measured on the sludge settler ruler). The samples were obtained by 1) inserting the sample tube into the supernate to the desired sample location (upper samples first), 2) running the peristaltic pump (P-301) clockwise to draw the sample into the tube, 3) raising the sample tube out of the sludge settler, 4) placing the sample bottle under the sample tube, and 5) running the pump (P-301) counter clockwise to discharge the sample into the bottle. The samples were weighed and the top, middle, and bottom supernate sample weights were 19.34, 13.76, and 14.22 grams, respectively.

### **B.5.7 Supernate Decant and Transfer**

Supernate was decanted by lowering the sample tube to within 2 inches of the solids layer and slowly pumping the supernate into the batch-collection tank, C-301. As the liquid level came near the end of the sample tube, the sample tube was incrementally lowered until it was within 3/8 inches of the solids level. Supernate removed in the process was 3878 mL, determined from observations of the liquid height in the sludge settler before and after decanting. The supernate was not transferred from the batch collection tank to the supernate holding tank, C-302.

## **B.6 First Water Wash and Settling Test**

The First Water Wash and Settling Test was performed using Procedure 3I-SOP-REC-F-40, Rev. 4. This procedure was started on May 21, 1998, and completed on May 26, 1998.

### **B.6.1 Inhibited Water Addition and Solids Resuspension**

The amount of inhibited water (0.01 M NaOH and 0.01 M NaNO<sub>2</sub>) to be added to obtain 4.3 wt% solids was determined to be 4553 grams. The inhibited water was weighed and added to the cold chemical tank. All but approximately 1000 mL of the water was then transferred to the sludge settler.

The solids were resuspended and mixed for one minute. Valve P-1-01-V01 was then opened to transfer the slurry to the sludge-receipt tank. The remaining water in the cold-chemical tank was transferred to the sludge settler to rinse out the column and was then transferred to the sludge-receipt tank. The agitator was turned on.

### **B.6.2 Agitate and Heat**

On May 21, 1998, the off-gas system was set for heating, and the heater in the sludge-receipt tank was turned on. While the slurry was heating, the agitator continued to operate. The slurry in the sludge-receipt tank was heated to 50°C and held at that temperature for 30 minutes while mixing with the agitator. Meanwhile, the temperature of the water in the circulating hot water bath was heated to 50°C.

### **B.6.3 Slurry Sampling**

Four slurry samples (SPD-S107-26 through SPD-S107-29) were collected through sample port S-202. The sample weights were 14.77, 22.05, 25.40, and 24.26 grams, respectively. The solids/liquid interface heights in each of the sample bottles was compared after 24 hours, and it was found that all of the samples had the same heights relative to total volume.

### **B.6.4 First Water-Wash Settling Test**

The slurry in the sludge-receipt tank was transferred to the settler on May 21, 1998. The solids were allowed to settle overnight. On the morning of May 22, 1998, the solids were resuspended and mixed for 10 minutes. The gravity settling test began immediately thereafter. At the start of the settling test, the slurry volume was 5662 mL, corresponding to a height of 9 3/8 inches in the sludge settler. The solids were resuspended again 4 hours later due to problems with the recording equipment.

During regular work hours, the solids/liquid interface was visually observed and recorded on data sheets. Also, the entire settling process was also recorded on videotape (SPD-S107-06), so that the entire settling rate and solids/liquid interface could be documented. Electronic data were monitored and recorded every 10 minutes on a data disk (S107-2data.xls) automatically by the data-acquisition system. The settling test was terminated on May 26, 1998, after 92 hours. The final total volume was 5381 mL, corresponding to a column height of 10 3/4 inches. The final volume of solids was 1325 mL, corresponding to a height of 30 5/8 inches in the sludge settler. Approximately 431 grams of water evaporated throughout the process.

### **B.6.5 Axial Sampling**

Three samples (SPD-S107-30 through SPD-S107-32) of the supernate were taken at axial elevations of 12, 21, and 30 inches from the top of the sludge settler (measured on the sludge settler ruler). The samples were obtained by 1) inserting the sample tube into the supernate to the desired sample location (upper samples first), 2) running the peristaltic pump (P-301) clockwise to draw the sample into the tube, 3) raising the sample tube out of the sludge settler, 4) placing the sample bottle

under the sample tube, and 5) running the pump (P-301) counter clockwise to discharge the sample into the bottle. The samples were weighed and the top, middle, and bottom supernate sample weights were 14.12, 14.03, and 14.46 grams, respectively.

### **B.6.6 Supernate Transfer**

Supernate was decanted by lowering the sample tube to within 2 inches of the solids layer and slowly pumping the supernate into the batch collection tank, C-301. As the liquid level came near the end of the sample tube, the sample tube was incrementally lowered until it was within a ½ inch of the solids level. Supernate removed in the process was 3954 mL, determined from observations of the liquid height in the sludge settler before and after decanting. The supernate was transferred from the batch collection tank to the supernate holding tank, C-302.

## **B.7 Second Water-Wash and Settling Test**

The Second Water Wash and Settling Test was performed using Procedure 3I-SOP-REC-F-40, Rev. 4. This procedure was started on May 26, 1998, and completed on June 1, 1998.

### **B.7.1 Inhibited Water Addition and Solids Resuspension**

The amount of inhibited water (0.01 M NaOH and 0.01 M NaNO<sub>2</sub>) to be added to obtain 4.3 wt% solids was determined to be 4552 grams. The inhibited water was weighed and added to the cold-chemical tank. We added 3456 grams of this water to the sludge settler.

The solids were resuspended and mixed for 3 minutes. Valve P-1-01-V01 was then opened to transfer the slurry to the sludge-receipt tank. The remaining water in the cold chemical tank was transferred to the sludge settler and circulated through the settler for 5 minutes to rinse out the column. This water was then transferred to the sludge-receipt tank. The agitator was turned on.

### **B.7.2 Agitate and Heat**

On May 26, 1998, the off-gas system was set for heating, and the heater in the sludge-receipt tank was turned on. While the slurry was heating, the agitator continued to operate. The slurry in the sludge-receipt tank was heated to 50°C and held at that temperature for 10 minutes while mixing with the agitator. Meanwhile, the temperature of the water in the circulating hot water bath was heated to 50°C.

### **B.7.3 Second Water-Wash Settling Test**

The slurry in the sludge-receipt tank was transferred to the settling column and the gravity settling test began. At the start of the settling test, the slurry volume was 5815 mL, corresponding to a height of 8 5/8 inches in the sludge settler.

During regular work hours, the solids/liquid interface was visually observed and recorded on data sheets. The entire settling process was also recorded on videotape (SPD-S107-07) so that the entire settling rate and solids/liquid interface could be documented. Electronic data were monitored and recorded every 10 minutes on a data disk (S107-2data.xls) automatically by the data-acquisition system. The settling test was terminated on June 1, 1998, after 139 hours. Approximately 479 grams of water evaporated during the test. The final total volume was 5483 mL, corresponding to a column height of

10 ¼ inches. The final volume of solids was 1351 mL, corresponding to a height of 30 ½ inches in the sludge settler.

#### **B.7.4 Axial Sampling**

Three samples (SPD-S107-33 through SPD-S107-35) of the supernate were taken at axial elevations of 12, 21, and 30 inches from the top of the sludge settler (measured on the sludge settler ruler). The samples were obtained by 1) inserting the sample tube into the supernate to the desired sample location (upper samples first), 2) running the peristaltic pump (P-301) clockwise to draw the sample into the tube, 3) raising the sample tube out of the sludge settler, 4) placing the sample bottle under the sample tube, and 5) running the pump (P-301) counter clockwise to discharge the sample into the bottle. The samples were weighed, and the top, middle, and bottom supernate sample weights were 14.12, 14.52, and 14.39 grams, respectively.

#### **B.7.5 Supernate Transfer**

Supernate was decanted by lowering the sample tube to within 2 inches of the solids layer and slowly pumping the supernate into the batch collection tank, C-301. As the liquid level came near the end of the sample tube, the sample tube was incrementally lowered until it was within a ½ inch of the solids level. Supernate removed in the process was 3980 mL, determined from observations of the liquid height in the sludge settler before and after decanting. The supernate was not transferred from the batch collection tank to the supernate holding tank, C-302.

### **B.8 Third Water Wash**

The Third Water Wash and Settling Test was performed using Procedure 3I-SOP-REC-F-41, Rev. 4. This procedure was started on June 1, 1998, and completed on June 17, 1998.

#### **B.8.1 Inhibited Water Addition and Solids Resuspension**

The amount of inhibited water (0.01 M NaOH and 0.01 M NaNO<sub>2</sub>) to be added to obtain 4.3 wt% solids was determined to be 4552 grams. The inhibited water was weighed and added to the cold chemical tank. We added 2066 grams of this water to the sludge settler.

The solids were resuspended and mixed for 2 minutes. Valve P-1-01-V01 was then opened to transfer the slurry to the sludge-receipt tank. The remaining water in the cold-chemical tank was transferred to the sludge settler and circulated through the settler for 5 minutes to rinse out the column. This water was then transferred to the sludge-receipt tank. The agitator was turned on.

#### **B.8.2 Agitate and Heat**

On June 1, 1998, the off-gas system was set for heating, and the heater in the sludge-receipt tank was turned on. While the slurry was heating, the agitator continued to operate. The slurry in the sludge-receipt tank was heated to 50°C and held at that temperature for 42 minutes while mixing with the agitator. Meanwhile, the temperature of the water in the circulating hot water bath was heated to 50°C.

### **B.8.3 Third Water Wash Settling Test**

The slurry in the sludge-receipt tank was transferred to the settling column, and the gravity settling test began. At the start of the settling test, the slurry volume was 5917 mL, corresponding to a height of 8 1/8 inches in the sludge settler.

During regular work hours, the solids/liquid interface was visually observed and recorded on data sheets. The entire settling process was also recorded on videotape (SPD-S107-08) so that the entire settling rate and solids/liquid interface could be documented. Electronic data were monitored and recorded every 10 minutes on a data disk (S107-2data.xls) automatically by the data-acquisition system. The settling test was terminated on June 9, 1998, after 190 hours. Approximately 430 grams of water evaporated during the test. The final total volume was 5534 mL, corresponding to a column height of 10 inches. The final volume of solids was 1427 mL, corresponding to a height of 30 1/8 inches in the sludge settler.

### **B.8.4 Axial Sampling**

Three samples (SPD-S107-36 through SPD-S107-38) of the supernate were taken at axial elevations of 11, 20, and 29 inches from the top of the sludge settler (measured on the sludge settler ruler). The samples were obtained by 1) inserting the sample tube into the supernate to the desired sample location (upper samples first), 2) running the peristaltic pump (P-301) clockwise to draw the sample into the tube, 3) raising the sample tube out of the sludge settler, 4) placing the sample bottle under the sample tube, and 5) running the pump (P-301) counter clockwise to discharge the sample into the bottle. The samples were weighed and the top, middle, and bottom supernate sample weights were 13.77, 13.61, and 13.45 grams, respectively.

### **B.8.5 Supernate Transfer**

Part of the supernate was decanted and pumped into the batch collection tank, C-301. We removed 2704 mL of supernate in the process, determined from observations of the liquid height in the sludge settler before and after decanting. The rest of the supernate was left in the settling column to be used for another settling test with a solids concentration of 8.7 wt%.

### **B.8.6 Solids Resuspension**

The solids in the sludge settler were then resuspended by 1) placing the sample tube approximately 2 inches below the liquid level and 2) circulating the slurry through the sample tube, pump P-202, and back through a port in the bottom of the sludge settler. The pump was run for 3 minutes, and then the sample tube was raised above the top of the slurry. The pump continued to run for another minute to clear the line and was then shut off. Immediately after this, the settling test began. At the start of the settling test, the slurry volume was 2830 mL, corresponding to a height of 23 1/4 inches in the sludge settler.

During regular work hours, the solids/liquid interface was visually observed and recorded on data sheets. The entire settling process was also recorded on videotape (SPD-S107-09), so that the entire settling rate and solids/liquid interface could be documented. Electronic data were monitored and recorded every 10 minutes on a data disk (S107-2data.xls) automatically by the data-acquisition system. The settling test was terminated on June 15, 1998, after 143 hours. Approximately 485 grams of water

evaporated during the test. The final total volume was 2346 mL, corresponding to a column height of 25 5/8 inches. The final volume of solids was 1427 mL, corresponding to a height of 30 1/8 inches in the sludge settler.

### **B.8.7 Axial Sampling**

Three samples (SPD-S107-39 through SPD-S107-41) of the supernate were taken at axial elevations of 27, 28, and 29 inches from the top of the sludge settler (measured on the sludge settler ruler). The samples were obtained by 1) inserting the sample tube into the supernate to the desired sample location (upper samples first), 2) running the peristaltic pump (P-301) clockwise to draw the sample into the tube, 3) raising the sample tube out of the sludge settler, 4) placing the sample bottle under the sample tube, and 5) running the pump (P-301) counter clockwise to discharge the sample into the bottle. The samples were weighed and the top, middle, and bottom supernate sample weights were 16.78, 16.00, and 16.21 grams, respectively.

### **B.8.8 Deionized Water Addition and Solids Resuspension**

We added 459 grams of deionized water to the settler. The solids in the sludge settler were then resuspended by 1) placing the sample tube approximately 2 inches below the liquid level and 2) circulating the slurry through the sample tube, pump P-202, and back through a port in the bottom of the sludge settler. When the slurry appeared uniform, valve P-1-01-V01 was opened, and the slurry was transferred to the sludge-receipt tank.

### **B.8.9 Slurry Sampling**

Four slurry samples (SPD-S107-42 through SPD-S107-45) were collected through sample port S-202. The sample weights were 18.54, 26.33, 14.29, and 7.57 grams, respectively. Approximately 25 grams of slurry were lost during the sampling process due to a spill. The solids/liquid interface heights in each of the sample bottles were compared after 24 hours, and it was found that all of the samples had the same heights relative to total volume.

Upon completion of the slurry sampling, the slurry was transferred back to the settler and allowed to settle over a period of 50 hours. Approximately 246 grams of water evaporated. At the end of this time, the supernate was decanted.

### **B.8.10 Supernate Transfer**

Supernate was decanted by lowering the sample tube to within 2 inches of the solids layer and slowly pumping the supernate into the batch-collection tank, C-301. As the liquid level came near the end of the sample tube, the sample tube was incrementally lowered until it was within 1/4 inch of the solids level. Supernate removed in the process was 1005 mL, determined from observations of the liquid height in the sludge settler before and after decanting.

## **B.9 Extended Caustic Leach**

The Extended Caustic Leach was performed using Procedure 3I-SOP-REC-F-42, Rev. 0. This procedure was started on June 17, 1998, and completed on July 6, 1998. A spill recovery plan (date June 23, 1998) was also performed during this time period.

### **B.9.1 Caustic Addition and Solids Resuspension**

The amount of caustic to be added to obtain a 5 wt% solution was calculated to be 4190 grams of 4.32 M NaOH. This molarity was used to create a 3 M solution when mixed with the sludge in the column. The caustic was placed in the cold-chemical tank and then transferred to the sludge settler on June 17, 1998.

The solids were resuspended and mixed for two minutes. Valve P-1-01-V01 was then opened to transfer the slurry to the sludge-receipt tank. The agitator was turned on, and the heater was set for 80°C.

### **B.9.2 Slurry Sampling**

At this time, a slurry sample was supposed to be taken through sample port S-202. While the slurry was being pumped through pump P-203, a hose split, and approximately 4027 grams of slurry were lost. All of the equipment was turned off, and the experiment was put into standby on June 17, 1998.

### **B.9.3 Recovery Plan**

On June 26, the recovery plan for the experiment began. The tubing on pump P-203 was replaced, and all other hoses were inspected for signs of wear. A shield was placed around the pump tubing to prevent leaks from spraying out beyond the catch basin. The dried slurry on the floor of the cell was cleaned up with rags, which were properly disposed of afterward. The agitator in the sludge-receipt tank was turned on for 10 minutes. At the end of this time, the slurry was transferred to the sludge settler to measure the volume of the remaining slurry. The total volume was 1504 mL, corresponding to a column height of 29  $\frac{3}{4}$  inches. The slurry was transferred back to the sludge-receipt tank, and the agitator was turned on. We added 218.75 grams of deionized water to the sludge-receipt tank to account for water that may have evaporated during the shutdown. The original test activities for the extended caustic leach were then resumed.

### **B.9.4 Slurry Sampling**

After the deionized water was mixed with the slurry, four slurry samples (SPD-S107-46 through SPD-S107-49) were collected through sample port S-202. The sample weights were 15.20, 16.00, 17.30, and 17.28 grams respectively. The slurry continued to be agitated and held at 80°C. After the slurry had been held at 80°C for 6  $\frac{1}{2}$  hours, another slurry sample (SPD-S107-50) with a weight of 15.62 grams was taken through sample port S-202. Another slurry sample (SPD-S107-51) was taken after 14 hours of heating. The sample weight was 22.40 grams.

### **B.9.5 Supernate Sampling**

On June 27, 1998, the circulating hot-water bath on the sludge settler was turned on and set to 80°C, and the heater in the sludge-receipt tank was turned off. The slurry in the sludge-receipt tank was then transferred to the sludge settler using the transfer pump, P-203. The slurry volume was 1376 mL, corresponding to a column height of 30  $\frac{3}{8}$  inches. The solids were allowed to settle to a level of 31  $\frac{3}{4}$  inches so that a supernate sample could be taken 22 hours into the extended leach test.

One sample (SPD-S107-52) of the supernate was taken at an axial elevation of 31 inches from the top of the sludge settler (measured on the sludge settler ruler). The sample was obtained by 1) inserting the sample tube into the supernate to the desired sample location (upper samples first), 2) running the peristaltic pump (P-301) clockwise to draw the sample into the tube, 3) raising the sample tube out of the sludge settler, 4) placing the sample bottle under the sample tube, and 5) running the pump (P-301) counter clockwise to discharge the sample into the bottle. The sample weight was 21.19 grams.

#### **B.9.6 Solids Resuspension and Deionized Water Addition**

The solids were resuspended and mixed for 1 minute. Valve P-1-01-V01 was then opened to transfer the slurry to the sludge-receipt tank. The agitator was turned on and the heater was set for 80°C. We added 149 grams of deionized water to the sludge-receipt tank to account for evaporation. Approximately 236 grams of water had evaporated since the beginning of the extended leach test.

#### **B.9.7 Slurry Sampling**

Two slurry samples (SPD-S107-53 and SPD-S107-54) were collected at 30 ½ and 38 ½ hours into the test, respectively. The sample weights were 18.23 and 26.47 grams, respectively.

The heater in the circulating hot-water bath was then turned on and set for 80°C, and the heater in the sludge-receipt tank was turned off. The slurry was transferred to the settler column by the transfer pump, P-203, and the solids were allowed to settle.

#### **B.9.8 Supernate Sampling**

The slurry volume upon transfer to the settler was 1223 mL, corresponding to a column height of 31 1/8 inches. The solids were allowed to settle to a level of 32 3/8 inches, and a supernate sample was taken 46 ½ hours into the extended-leach test.

The supernate sample (SPD-S107-55) was taken at an axial elevation of 31 ½ inches from the top of the sludge settler (measured on the sludge settler ruler). The sample was obtained by 1) inserting the sample tube into the supernate to the desired sample location (upper samples first), 2) running the peristaltic pump (P-301) clockwise to draw the sample into the tube, 3) raising the sample tube out of the sludge settler, 4) placing the sample bottle under the sample tube, and 5) running the pump (P-301) counter clockwise to discharge the sample into the bottle. The sample weight was 14.55 grams.

#### **B.9.9 Solids Resuspension and Deionized Water Addition**

The solids were resuspended and mixed for 2 minutes. Valve P-1-01-V01 was then opened to transfer the slurry to the sludge-receipt tank. The agitator was turned on and the heater was set for 80°C. We added 281 grams of deionized water to the sludge-receipt tank to account for evaporation. Approximately 250 grams of water had evaporated since the last water addition.

#### **B.9.10 Slurry Sampling**

Two slurry samples (SPD-S107-56 and SPD-S107-57) were collected at 54 ½ and 64 hours into the test, respectively. The sample weights were 23.33 and 20.84 grams, respectively.

The heater in the circulating hot-water bath was then turned on and set for 80°C, and the heater in the sludge-receipt tank was turned off. The slurry was transferred to the settler column by the transfer pump, P-203, and the solids were allowed to settle.

#### **B.9.11 Supernate Sampling**

The slurry volume upon transfer to the settler was 1274 mL, corresponding to a column height of 30 7/8 inches. The solids were allowed to settle to a level of 32 3/8 inches, and a supernate sample was taken 72 hours into the extended leach test.

The supernate sample (SPD-S107-58) was taken at an axial elevation of 31 ½ inches from the top of the sludge settler (measured on the sludge settler ruler). The sample was obtained by 1) inserting the sample tube into the supernate to the desired sample location (upper samples first), 2) running the peristaltic pump (P-301) clockwise to draw the sample into the tube, 3) raising the sample tube out of the sludge settler, 4) placing the sample bottle under the sample tube, and 5) running the pump (P-301) counter clockwise to discharge the sample into the bottle. The sample weight was 11.51 grams.

#### **B.9.12 Solids Resuspension and Deionized Water Addition**

The solids were resuspended and mixed for 1 minute. Valve P-1-01-V01 was then opened to transfer the slurry to the sludge-receipt tank. The agitator was turned on and the heater was set for 80°C. We added 102 grams of deionized water to the sludge-receipt tank to account for evaporation. Approximately 181 grams of water had evaporated since the last water addition.

#### **B.9.13 Supernate Sampling**

On June 30, 1998, the heater in the circulating hot water bath was turned on, and the heater in the sludge-receipt tank was turned off. The slurry in the sludge-receipt tank was then transferred to the sludge settler column when the hot water bath temperature had reached 80°C. The volume of the slurry was 1172 mL, corresponding to a column height of 31 3/8 inches. The solids were allowed to settle to a level of 32 ½ inches, and a supernate sample was taken 94 ½ hours into the extended-leach test.

The supernate sample (SPD-S107-59) was taken at an axial elevation of 32 inches from the top of the sludge settler (measured on the sludge settler ruler). The sample was obtained by 1) inserting the sample tube into the supernate to the desired sample location (upper samples first), 2) running the peristaltic pump (P-301) clockwise to draw the sample into the tube, 3) raising the sample tube out of the sludge settler, 4) placing the sample bottle under the sample tube, and 5) running the pump (P-301) counter clockwise to discharge the sample into the bottle. The sample weight was 11.82 grams.

#### **B.9.14 Solids Resuspension and Deionized Water Addition**

The solids were resuspended and mixed for two minutes. Valve P-1-01-V01 was then opened to transfer the slurry to the sludge-receipt tank. The agitator was turned on and the heater was set for 80°C. We added 204 grams of deionized water to the sludge-receipt tank to account for evaporation. Approximately 194 grams of water had evaporated since the last water addition.

### **B.9.15 Supernate Sampling**

On July 1, 1998, the heater in the circulating hot water bath was turned on, and the heater in the sludge-receipt tank was turned off. The slurry in the sludge-receipt tank was then transferred to the sludge settler column when the hot water bath temperature had reached 80°C. The volume of the slurry was 1198 mL, corresponding to a column height of 31 ¼ inches. The solids were allowed to settle to a level of 32 5/8 inches, and a supernate sample was taken 118 ½ hours into the extended leach test.

The supernate sample (SPD-S107-60) was taken at an axial elevation of 32 inches from the top of the sludge settler (measured on the sludge settler ruler). The sample was obtained by 1) inserting the sample tube into the supernate to the desired sample location (upper samples first), 2) running the peristaltic pump (P-301) clockwise to draw the sample into the tube, 3) raising the sample tube out of the sludge settler, 4) placing the sample bottle under the sample tube, and 5) running the pump (P-301) counter clockwise to discharge the sample into the bottle. The sample weight was 18.18 grams.

### **B.9.16 Solids Resuspension**

On July 1, 1998, the solids were resuspended and mixed for 1 minute. Valve P-1-01-V01 was then opened to transfer the slurry to the sludge-receipt tank. The agitator was turned on and the heater was set for 80°C. We added 178 grams of deionized water to the sludge-receipt tank to account for evaporation. Approximately 163 grams of water had evaporated since the last water addition. The slurry remained in the sludge-receipt tank until July 3, 1998, when it was transferred back to the sludge settler. At this point, the slurry volume was 1070 mL, corresponding to a column height of 31 7/8 inches. An additional 306 grams of deionized water were added to the settler to account for the approximately 305 grams of water that had evaporated since the last water addition.

### **B.9.17 Extended Caustic Leach Settling Test**

The solids in the settling column were resuspended, and the gravity settling test began at 166 hours. At the start of the settling test, the slurry volume was 1376 mL, corresponding to a height of 30 3/8 inches in the sludge settler.

During regular work hours, the solids/liquid interface was visually observed and recorded on data sheets. The entire settling process was also recorded on videotape (SPD-S107-10) so that the entire settling rate and solids/liquid interface could be documented. Electronic data were monitored and recorded every 10 minutes on a data disk (S107-2data.xls) automatically by the data-acquisition system. The settling test was terminated on July 6, 1998, at 236 hours. Approximately 434 grams of water evaporated during the test. The final total volume was 943 mL, corresponding to a column height of 32 ½ inches. The final volume of solids was 534 mL, corresponding to a height of 34 ½ inches in the sludge settler.

### **B.9.18 Axial Sampling**

At 237 hours, three samples (SPD-S107-61 through SPD-S107-63) of the supernate were taken at axial elevations of 33, 33 ½, and 34 inches from the top of the sludge settler (measured on the sludge settler ruler). The samples were obtained by 1) inserting the sample tube into the supernate to the desired sample location (upper samples first), 2) running the peristaltic pump (P-301) clockwise to draw the sample into the tube, 3) raising the sample tube out of the sludge settler, 4) placing the sample bottle

under the sample tube, and 5) running the pump (P-301) counter clockwise to discharge the sample into the bottle. The samples were weighed and the top, middle, and bottom supernate sample weights were 11.75, 10.22, and 13.58 grams, respectively.

### **B.9.19 Slurry Sampling**

At 237 hours, the solids were resuspended in the settler for 2 minutes until the mixture appeared uniform. Four slurry samples (SPD-S107-64 and SPD-S107-67) were then collected through the sample tube at an axial elevation of 35 inches in the sludge settler. The sample weights were 16.92, 17.28, 17.52 and 17.39 grams respectively.

## **Appendix C**

### **Chemical and Radiochemical Analysis**

## Battelle PNNL/RPG/Inorganic Analysis --- IC Report

### General Comments:

Six liquid samples were analyzed by ion chromatography (IC) for IC anions (fluoride, chloride, nitrite, bromide, nitrate, phosphate and sulfate). The samples were diluted 500 fold to ensure that the sample results were within the calibration range for each analyte. The sample results are reported below.

Sample	F <sup>-</sup> µg/mL	Cl <sup>-</sup> µg/mL	Br <sup>-</sup> µg/mL	NO <sub>2</sub> <sup>-</sup> µg/mL	NO <sub>3</sub> <sup>-</sup> µg/mL	PO <sub>4</sub> <sup>-3</sup> µg/mL	SO <sub>4</sub> <sup>-2</sup> µg/mL
98-4327	<125	560	150	10,200	15,500	530	450
98-4328	<125	200	130	3,380	5,170	900	<250
98-4329	<125	510	<125	1,120	1,900	530	<250
98-4330	<125	270	<125	570	450	310	<250
98-4331	<125	95	<125	580	<250	<250	<250
98-4332	<125	205	<125	590	<250	<250	<250

The sample chromatograms were examined to determine if oxalate was present in any of the samples. Although there was an unidentified peak occurring at a retention time of 8.4 minutes (later than the last quantified peak) for samples 98-4327, 98-4328 and 98-4329, this peak could not be oxalate (retention time 9.4 minutes). This conclusion was verified by comparing the chromatograms from the clients previous sample analysis, which did contain oxalate. In those samples, the oxalate peak occurred at the specified retention time.

There was an additional peak with a retention time of 1.45 minutes which overlapped the chloride peak. The intensity of this peak increased throughout the sample series until it was the dominant peak observed in the chromatogram. Thus, the quantitation of chloride in samples 98-4330, 4331 and 4332 is anticipated to have a large uncertainty. For the initial samples (98-4327, 4328 and 4329) the co-eluting peak was less than 20% of the chloride peak and the quantitation is predicted to be within ±10%.

### Q.C. Comments:

Following are results of quality control checks performed during IC analyses. In general, quality control checks met the requirements of the governing QA Plan, MCS-033.

Working Blank Spike: Since no matrix-matched laboratory control standard (LCS) was available, a blank spike was used for the LCS. The blank spike recoveries were between 96% and 106% for all analytes; well within the 80% to 120% acceptance criteria.

**Battelle PNNL/RPG/Inorganic Analysis ...  
ICPAES Analytical Report**

WO/Project: K87684/28966  
Client: K Brooks  
Impact Level: III

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ACL Nmbr(s): 98-004327 through 98-004340,  
98-4342 through 98-4344, and  
98-4843 through 98-4847  
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Client ID: "SPD-S107-006" through "SPD-S107-051",  
"SPD-2107-054" through "SPD-S107-057", and  
"SPD-S107-003" through "SPD-S107-066"

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ASR Nmbr(s): 4918, 4997  
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Total Samples: 23  
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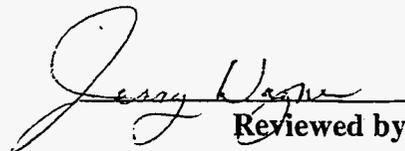
Procedure: PNL-ALO-211, "Determination of Elements by Inductively Coupled  
Argon Plasma Atomic Emission Spectrometry" (ICP-AES).

Analyst: DR Sanders

Analysis Date (Filename): 08/17/98 (A0471), 08/18/98 (A0472), 09/10/98 (A0477)

See ALO System File: "ICP-325-405-1" for traceability to Calibration,  
Quality Control, Verification, and Raw Data.

M&TE Number: ICPAES instrument -- WB73520  
Mettler AT400 Balance -- Ser.No. 360-06-01-029

 9-11-98  
Reviewed by

 9-14-98  
Concur

9/11/98

**Battelle PNNL/RPG/Inorganic Analysis ...**  
**ICPAES Analytical Report**

Seventeen radioactive aqueous samples were analyzed by ICPAES after processing by 325SAL using ALO-128 Acid Digestion procedure. Original sample size varied from about 5.0 to about 5.9 g-liquid. During processing, samples were diluted to 25 ml. Analytical dilution of 5-fold or 25 fold was also performed where needed. Measurement results reported have been corrected for all analytical dilutions.

Five radioactive sludge samples were also analyzed by ICPAES after processing by 325SAL using ALO-115 KOH fusion procedure. Sample size varied from about 0.07 to 0.12 grams. Samples were diluted to a final volume of 50 ml. Prior to analysis by ICPAES, each sample was further diluted by 2-fold. A process blank was prepared along with the samples. Sodium and Iron were present within EQL in the blank. Measurement results have been corrected for processing and other dilutions but have not been corrected for analytes present in the process blank. Sodium is known to be present in the fusion flux reagent. It is recommended that Sodium concentration in each sample be corrected for the amount of Sodium found in the process blank.

Analytes of interest include Al, Cr, Fe, Na, P, and Si. Other analytes of secondary importance include B, Bi, Ca, Pb, U, and Zr. Sodium and Aluminum concentrations were highest in the aqueous samples. Concentrations of analytes of interest ranged from just detectable to about 7,900 µg/ml Na. Uranium was not detected in any of the samples measured. Aluminum and Sodium were also the highest in concentration found in the sludge samples. Aluminum concentration in the sludge ranged from about 8 Wt% to a high of about 31 Wt%. Sodium ranged from about 9 Wt% to a high of about 34 Wt%.

Quality control checks performed for these sets of analyses include 5-fold serial dilution, analytical spikes (post spikes) excluding Uranium, linear range check, mid-range calibration check, process blanks. All quality control checks performed for analytes of interest were within MCS-033 tolerance limits with three exceptions. A post spike of Silicon for sample SPD-S107-006 resulted in an over recovery of 135%. All of the other multi-analyte spikes to this sample were recovered within tolerance limits. MCS-033 tolerance limit is 75% to 125% recoveries for all post spikes. The second exception is high concentration of Boron and Iron in the process blanks. Boron concentration in both process blanks amounted to approximately 50% of the Boron found in the samples. Iron concentration in one process blank was equivalent to about 55% or less of the Iron found in the samples. Iron concentration was below EQL for most samples. Sodium concentration in the process blank was within EQL but amounted to less than 5% of the very high concentration of Sodium present in the samples. As mentioned above both Sodium and Iron were within EQL and both analytes were greater than 5% of the concentration found in some of the samples.

9/11/98

## **Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Analytical Report**

See attached "ICPAES Data Report" for measurement results, detection limits, and etc. Analytes other than those requested are for information only. Please note bracketed values listed in the data report are within ten times instrument detection limit. Those measurement values have a potential uncertainty much greater than 15%.

### Comments:

- 1) "Final Results" have been corrected for all laboratory dilutions performed on the sample during processing and analysis unless specifically noted.
- 2) Detection limits (DL) shown are for acidified water unless specifically noted otherwise. Detection limits for other matrices may be determined if requested.
- 3) Routine precision and bias is typically  $\pm 15\%$  or better for samples in dilute, acidified water (e.g. 2% v/v HNO<sub>3</sub> or less) at analyte concentrations greater than ten times detection limit up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 mg/ml (0.5 per cent by weight).
- 4) Absolute precision, bias and detection limits may be determined on each sample if required by the client.
- 5) The maximum number of significant figures for all ICP measurements is 2.
- 6) To convert "WT%" to "mg/Kg" or " $\mu\text{g/g}$ ", multiply concentration value by 10,000.
- 7) To convert "mg/Kg" or " $\mu\text{g/g}$ " to "WT%", divide concentration value by 10,000.

**Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report** Page 1 of 3

Multiplier=	4.5	4.8	22.3	21.7	4.8
ALO#=	98-4327-PB	98-4327	98-4328 @5	98-4329 @5	98-4330
Client ID=	ProcessBlk	SPD-S107-006	SPD-S107-013	SPD-S107-021	SPD-S107-031
Run Date=	8/17/98	8/17/98	8/17/98	8/17/98	8/17/98
Det. Limit (ug/mL)	(Analyte)	ug/g	ug/g	ug/g	ug/g
0.025	Ag	--	--	--	--
0.060	Al	[0.59]	1,870	13,400	2,640
0.250	As	--	--	--	--
0.050	B	10.0	22.1	42.1	27.2
0.010	Ba	--	[0.074]	--	[0.099]
0.010	Be	--	--	--	--
0.100	Bi	--	[0.51]	--	--
0.250	Ca	[2.3]	[3.4]	[9.0]	[2.6]
0.015	Cd	--	--	--	--
0.200	Ce	--	--	--	--
0.050	Co	--	--	--	--
0.020	Cr	--	272	118	22.6
0.025	Cu	--	--	[0.74]	[1.6]
0.050	Dy	--	--	--	--
0.100	Eu	--	--	--	--
0.025	Fe	--	[0.55]	[2.4]	[5.0]
2.000	K	--	141	[47]	--
0.050	La	--	--	--	--
0.030	Li	--	3.41	[1.3]	[1.1]
0.100	Mg	--	--	--	--
0.050	Mn	--	--	--	--
0.050	Mo	--	5.81	[1.7]	--
0.150	Na	12.9	21,200	57,000	70,200
0.100	Nd	--	--	--	--
0.030	Ni	--	[0.70]	--	--
0.100	P	--	133	59.7	48.5
0.100	Pb	--	--	--	[3.5]
0.750	Pd	--	--	--	--
0.300	Rh	--	--	--	--
1.100	Ru	--	--	--	--
0.500	Sb	--	--	--	--
0.250	Se	--	--	--	--
0.500	Si	[4.8]	57.7	174	284
1.500	Sn	--	--	--	--
0.015	Sr	--	--	--	--
1.500	Te	--	--	--	--
1.000	Th	--	[7.2]	--	--
0.025	Ti	--	--	--	--
0.500	Tl	--	--	--	--
2.000	U	--	--	--	--
0.050	V	--	[0.66]	--	--
2.000	W	--	--	--	--
0.050	Y	--	--	--	--
0.050	Zn	--	[0.36]	[2.2]	[2.3]
0.050	Zr	--	--	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.  
 2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.  
 3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

**Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report** Page 2 of 3

Det. Limit (ug/mL)	Run Date= (Analyte)	Multiplier= ALO#= Client ID= SPD-S107-034	5.0 98-4331	4.9 98-4331-DUP SPD-S107-034 (dup)	5.0 98-4332 SPD-S107-037	22.0 98-4333 @5 SPD-S107-052	21.8 98-4334 @5 SPD-S107-055
		8/17/98	8/17/98	8/17/98	8/17/98	8/17/98	8/17/98
		ug/g	ug/g	ug/g	ug/g	ug/g	ug/g
0.025	Ag	--	--	--	--	--	--
0.060	Al	719	730	200	4,980	7,180	--
0.250	As	--	--	--	--	--	--
0.050	B	13.7	16.6	12.8	29.6	33.7	--
0.010	Ba	--	--	--	--	--	--
0.010	Be	--	--	--	--	--	--
0.100	Bi	--	--	--	--	--	--
0.250	Ca	[1.8]	[3.1]	[2.0]	--	--	--
0.015	Cd	--	--	--	--	--	--
0.200	Ce	--	--	--	--	--	--
0.050	Co	--	--	--	--	--	--
0.020	Cr	7.66	7.71	2.63	24.0	35.0	--
0.025	Cu	--	--	--	[0.71]	[0.79]	--
0.050	Dy	--	--	--	--	--	--
0.100	Eu	--	--	--	--	--	--
0.025	Fe	[0.22]	[0.21]	[0.14]	5.80	6.63	--
2.000	K	--	--	--	--	--	--
0.050	La	--	--	--	--	--	--
0.030	Li	--	--	--	--	--	--
0.100	Mg	--	--	--	--	--	--
0.050	Mn	--	--	--	--	--	--
0.050	Mo	--	--	--	--	--	--
0.150	Na	5,360	5,570	1,860	68,700	68,700	--
0.100	Nd	--	--	--	--	--	--
0.030	Ni	--	--	--	--	--	--
0.100	P	5.16	5.11	[2.9]	[7.2]	[8.2]	--
0.100	Pb	--	--	--	[2.3]	--	--
0.750	Pd	--	--	--	--	--	--
0.300	Rh	--	--	--	--	--	--
1.100	Ru	--	--	--	--	--	--
0.500	Sb	--	--	--	--	--	--
0.250	Se	--	--	--	--	--	--
0.500	Si	48.3	52.4	39.0	229	221	--
1.500	Sn	--	--	--	--	--	--
0.015	Sr	--	--	--	--	[0.42]	--
1.500	Te	--	--	--	--	--	--
1.000	Th	--	--	--	--	--	--
0.025	Ti	--	--	--	--	--	--
0.500	Tl	--	--	--	--	--	--
2.000	U	--	--	--	--	--	--
0.050	V	--	--	--	--	--	--
2.000	W	--	--	--	--	--	--
0.050	Y	--	--	--	--	--	--
0.050	Zn	--	--	--	--	--	--
0.050	Zr	--	--	--	--	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.  
 2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.  
 3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Det. Limit (ug/mL)	Multiplier= ALO#= Client ID= Run Date= (Analyte)	21.0 98-4335 @5 SPD-S107-058 8/17/98 ug/g	1.0 Na \$1000 PPM Cal Check Std. 8/17/98 (ug/mL)	1.0 Na \$500 PM Cal Check Std. 8/17/98 (ug/mL)		
0.025	Ag	--	--	--	--	--
0.060	Al	7,990	--	--	--	--
0.250	As	--	--	--	--	--
0.050	B	38.3	--	--	--	--
0.010	Ba	--	--	--	--	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	--	--	--	--
0.015	Cd	--	--	--	--	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	40.0	--	--	--	--
0.025	Cu	[0.70]	--	--	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	5.63	--	--	--	--
2.000	K	--	--	--	--	--
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	--	--	--	--
0.050	Mo	--	--	--	--	--
0.150	Na	63,400	994	506	--	--
0.100	Nd	--	--	--	--	--
0.030	Ni	--	--	--	--	--
0.100	P	[7.3]	--	--	--	--
0.100	Pb	--	--	--	--	--
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	223	--	--	--	--
1.500	Sn	--	--	--	--	--
0.015	Sr	[0.45]	--	--	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	[1.1]	--	--	--	--
0.050	Zr	--	--	--	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.  
 2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.  
 3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

# Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	Run Date= (Analyte)	Multiplier= 5.0 ALO#= 98-4336-PB Client ID= <u>Process Blank</u> 8/18/98	21.9 98-4336 @5 <u>SPD-S107-059</u> 8/18/98	22.0 98-4337 @5 <u>SPD-S107-060</u> 8/18/98	21.9 98-4337-DUP @5 <u>SPD-S107-060 (dup)</u> 8/18/98	21.1 98-4338 @5 <u>SPD-S107-062</u> 8/18/98
		ug/g	ug/g	ug/g	ug/g	ug/g
0.025	Ag	--	--	--	--	--
0.060	Al	[0.96]	9,270	9,150	8,910	13,200
0.250	As	--	--	--	--	--
0.050	B	13.9	31.3	23.8	24.0	28.2
0.010	Ba	[0.13]	--	--	--	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	[2.1]	--	[9.8]	[50]	[14]
0.015	Cd	--	--	--	--	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	--	47.4	48.8	47.8	80.6
0.025	Cu	--	[0.84]	[0.72]	[0.72]	[0.96]
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	1.33	6.36	[5.1]	[5.0]	[3.8]
2.000	K	--	--	--	--	--
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	--	--	--	--
0.050	Mo	--	--	--	[1.1]	[2.3]
0.150	Na	16.1	67,200	60,500	60,000	79,000
0.100	Nd	--	--	--	--	--
0.030	Ni	--	--	--	--	--
0.100	P	--	[8.5]	[8.0]	[8.9]	[15]
0.100	Pb	--	--	--	--	--
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	[5.6]	186	138	133	162
1.500	Sn	--	--	--	--	--
0.015	Sr	--	[0.68]	[0.66]	[0.67]	[0.80]
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	--	--	--	[1.4]	[1.5]
0.050	Zr	--	--	--	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.  
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# Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Multiplier=	22.5	21.9	21.8	22.1	22.6	
ALO#=#	98-4339 @5	98-4340 @5	98-4342 @5	98-4343 @5	98-4344 @5	
Client ID=#	SPD-S107-050	SPD-S107-051	SPD-S107-054	SPD-S107-056	SPD-S107-057	
Run Date=	8/18/98	8/18/98	8/18/98	8/18/98	8/18/98	
Det. Limit (ug/mL)	Run Date=	(Analyte)	ug/g	ug/g	ug/g	ug/g
0.025	Ag	--	--	--	--	--
0.060	Al	2,210	3,450	6,600	7,170	8,070
0.250	As	--	--	--	--	--
0.050	B	59.1	35.2	25.8	25.1	31.0
0.010	Ba	--	--	--	--	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	[20]	--	[22]	--	--
0.015	Cd	--	--	--	--	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	12.6	18.1	31.9	35.1	39.6
0.025	Cu	--	[0.58]	--	[0.60]	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	[3.7]	[4.1]	[3.2]	[2.6]	[2.4]
2.000	K	--	--	--	--	--
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	--	--	--	--
0.050	Mo	--	--	--	--	--
0.150	Na	60,900	64,400	68,800	67,600	68,100
0.100	Nd	--	--	--	--	--
0.030	Ni	--	--	--	--	--
0.100	P	[8.3]	[8.2]	[6.3]	[7.4]	[6.4]
0.100	Pb	--	--	--	--	--
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	323	152	110	[110]	[110]
1.500	Sn	--	--	--	--	--
0.015	Sr	--	--	--	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	--	--	--	--	--
0.050	Zr	--	--	--	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.  
 2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.  
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# Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	Multiplier= ALO#= Client ID= Run Date= (Analyte)	1.0	1.0			
		Na \$1000 PPM Check Std. 8/18/98 (ug/mL)	Na \$500 PPM Check Std. 8/18/98 (ug/mL)			
0.025	Ag	--	--	--	--	--
0.060	Al	--	--	--	--	--
0.250	As	--	--	--	--	--
0.050	B	--	--	--	--	--
0.010	Ba	--	--	--	--	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	[0.32]	--	--	--
0.015	Cd	--	--	--	--	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	--	--	--	--	--
0.025	Cu	--	--	--	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	--	--	--	--	--
2.000	K	--	--	--	--	--
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	--	--	--	--
0.050	Mo	--	--	--	--	--
0.150	Na	985	491	--	--	--
0.100	Nd	--	--	--	--	--
0.030	Ni	--	--	--	--	--
0.100	P	--	--	--	--	--
0.100	Pb	--	--	--	--	--
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	--	--	--	--	--
1.500	Sn	--	--	--	--	--
0.015	Sr	--	--	--	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	--	--	--	--	--
0.050	Zr	--	--	--	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.  
 2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.  
 3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

# Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	Run Date= (Analyte)	Multiplier= 1019.6 ALO#= 98-4843-PB @2 Client ID= <u>Process Blank</u> 9/10/98	865.1 98-4843 @2 <u>SPD-S107-003</u> 9/10/98	974.7 98-4844 @2 <u>SPD-S107-003-DUP</u> 9/10/98	983.3 98-4845 @2 <u>SPD-S107-044</u> 9/10/98	1464.1 98-4846 @2 <u>SPD-S107-048</u> 9/10/98
		ug/g	ug/g	ug/g	ug/g	ug/g
0.025	Ag	--	[70]	[74]	[220]	--
0.060	Al	[380]	285,000	281,000	314,000	77,500
0.250	As	--	--	--	--	--
0.050	B	--	[100]	[91]	[87]	1,940
0.010	Ba	[13]	[56]	[56]	116	[38]
0.010	Be	--	--	--	--	--
0.100	Bi	--	[89]	--	[180]	--
0.250	Ca	--	[2,100]	[1,300]	3,780	[1,800]
0.015	Cd	--	--	--	[18]	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	[94]
0.020	Cr	[32]	2,510	2,550	1,460	[250]
0.025	Cu	--	[41]	[41]	[65]	[92]
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	504	7,310	7,580	16,900	1,760
0.050	La	--	--	--	--	--
0.030	Li	--	[33]	[32]	[36]	--
0.100	Mg	--	[230]	[220]	[520]	[240]
0.050	Mn	[470]	1,010	1,010	1,810	808
0.050	Mo	--	--	[54]	--	--
0.150	Na	2,680	95,500	94,100	47,800	321,000
0.100	Nd	--	--	--	[150]	[150]
0.100	P	--	1,170	1,150	[540]	[420]
0.100	Pb	[110]	[92]	[110]	[200]	[190]
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	[670]	8,920	8,870	17,600	15,900
1.500	Sn	--	--	--	--	--
0.015	Sr	--	970	957	1,890	308
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	[70]	[89]	[150]	[54]
0.500	Tl	--	--	--	--	--
2.000	U	--	[16,000]	[16,000]	31,700	[3,300]
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	--	[76]	[81]	[92]	[82]
0.050	Zr	--	[130]	[120]	[400]	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.  
 2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.  
 3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

# Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	(Analyte)	ug/g				
	Multiplier=	978.5				
	ALO#=	98-4847 @2				
	Client ID=	SPD-S107-066				
	Run Date=	9/10/98				
0.025	Ag	[35]	--	--	--	--
0.060	Al	78,000	--	--	--	--
0.250	As	--	--	--	--	--
0.050	B	[97]	--	--	--	--
0.010	Ba	[80]	--	--	--	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	[2,000]	--	--	--	--
0.015	Cd	--	--	--	--	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	493	--	--	--	--
0.025	Cu	[30]	--	--	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	3,830	--	--	--	--
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	[120]	--	--	--	--
0.050	Mn	638	--	--	--	--
0.050	Mo	[74]	--	--	--	--
0.150	Na	335,000	--	--	--	--
0.100	Nd	--	--	--	--	--
0.100	P	[220]	--	--	--	--
0.100	Pb	--	--	--	--	--
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	8,930	--	--	--	--
1.500	Sn	--	--	--	--	--
0.015	Sr	305	--	--	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	[48]	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	[5,200]	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	[66]	--	--	--	--
0.050	Zr	--	--	--	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.  
 2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.  
 3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

**Battelle PNNL/RPG/Inorganic Analysis ...  
ICPAES Analytical Report**

**WO/Project:** K87684/28966  
**Client:** K Brooks

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**ACL Nmbr(s): 98-005150 through 98-005152, and 98-004340**  
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**Client ID: "SPD-S107-010" through "SPD-S107-040", and  
"SPD-S107-051"**  
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**ASR Nmbr(s): 4918.01**  
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**Total Samples: 4**  
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**Procedure:** PNL-ALO-211, "Determination of Elements by Inductively Coupled  
Argon Plasma Atomic Emission Spectrometry" (ICP-AES).

**Analyst:** DR Sanders

**Analysis Date (Filename):** 09/23/98 (A0485)

**See ALO System File: "ICP-325-405-1" for traceability to Calibration,  
Quality Control, Verification, and Raw Data.**

**M&TE Number:** ICPAES instrument -- WB73520  
Mettler AT400 Balance -- Ser.No. 360-06-01-029

*Jerry Wagner* 9-25-98  
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Reviewed by

*MW* 9/25/98  
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Concur

9/25/98

**Battelle PNNL/RPG/Inorganic Analysis ...  
ICPAES Analytical Report**

Four radioactive aqueous samples were analyzed by ICPAES after processing by 325SAL using ALO-128 Acid Digestion procedure. Original sample size varied from about 5.1 to about 5.9 g-liquid. During processing, samples were diluted to 25 ml. Analytical dilution of 5-fold or 25-fold was also performed where needed. Measurement results reported have been corrected for all processing and analytical dilutions. Measurements reported have not been corrected for analyte concentrations found in the process blank. Measurement results are reported in unit of  $\mu\text{g/g}$ . Density determination was not requested in the ASR.

Analytes of interest include Al, Cr, Fe, Na, P, and Si. Other analytes of secondary importance include B, Bi, Ca, Pb, U, and Zr. Sodium and Aluminum concentrations were highest in the aqueous samples. Concentrations of analytes of interest ranged from about 2,320 to 65,000  $\mu\text{g/g}$  Na and from about 256 to 8,650  $\mu\text{g/g}$  Al. Uranium was not detected in any of the samples measured.

Quality control checks performed for these analyses include 5-fold serial dilution, duplicate, analytical spikes (post spikes) including Uranium, linear range check, mid-range calibration check, and a process blank. All quality control checks performed for analytes of interest were within MCS-033 tolerance limits with one exception. The one exception is RPD (relative percent difference) in the duplicate sample for Boron. RPD for Boron was 23.5% MCS-033 limit is 20%. All other analyte concentrations above MDL in the sample were within 3% RPD. The relatively high RPD for Boron may have been due to contamination during sample preparation. Boron is found most everywhere, particularly if common Pyrex glass is used for sample preparation.

See attached "ICPAES Data Report" for measurement results, detection limits, and etc. Analytes other than those requested are for information only. Please note bracketed values listed in the data report are within ten times instrument detection limit. Those measurement values have a potential uncertainty much greater than 15%.

**Battelle PNNL/RPG/Inorganic Analysis ...  
ICPAES Analytical Report**

**Comments:**

- 1) **"Final Results" have been corrected for all laboratory dilutions performed on the sample during processing and analysis unless specifically noted.**
- 2) **Detection limits (DL) shown are for acidified water unless specifically noted otherwise. Detection limits for other matrices may be determined if requested.**
- 3) **Routine precision and bias is typically  $\pm 15\%$  or better for samples in dilute, acidified water (e.g. 2% v/v HNO<sub>3</sub> or less) at analyte concentrations greater than ten times detection limit up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 mg/ml (0.5 per cent by weight).**
- 4) **Absolute precision, bias and detection limits may be determined on each sample if required by the client.**
- 5) **The maximum number of significant figures for all ICP measurements is 2.**
- 6) **To convert "WT%" to "mg/Kg" or " $\mu\text{g/g}$ ", multiply concentration value by 10,000.**
- 7) **To convert "mg/Kg" or " $\mu\text{g/g}$ " to "WT%", divide concentration value by 10,000.**

**9/25/98**

# Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	Multiplier= ALO#= Client ID= Run Date= (Analyte)	4.9 98-5150-PB Process Blank 9/23/98 ug/g	21.3 98-4340 @5 SPD-S107-051 9/23/98 ug/g	4.9 98-5150 SPD-S107-010 9/23/98 ug/g	21.6 98-5151 @5 SPD-S107-024 9/23/98 ug/g	4.8 98-5152 SPD-S107-040 9/23/98 ug/g
0.025	Ag	-	-	-	-	-
0.060	Al	[1.3]	3,360	800	8,650	256
0.250	As	-	-	-	-	-
0.050	B	13.2	30.7	18.1	71.6	16.3
0.010	Ba	[0.093]	-	-	-	[0.063]
0.010	Be	-	-	-	-	-
0.100	Bi	-	-	-	-	-
0.250	Ca	-	[23]	-	-	-
0.015	Cd	-	-	-	-	-
0.200	Ce	-	-	-	-	-
0.050	Co	-	-	-	-	-
0.020	Cr	[0.16]	17.7	132	69.8	4.15
0.025	Cu	-	[0.64]	-	[1.0]	-
0.050	Dy	-	-	-	-	-
0.100	Eu	-	-	-	-	-
0.025	Fe	[0.99]	[4.8]	[1.1]	5.96	1.27
2.000	K	-	-	[67]	[60]	-
0.050	La	-	-	-	-	-
0.030	Li	-	-	1.58	[0.74]	-
0.100	Mg	-	-	-	-	-
0.050	Mn	-	-	-	-	-
0.050	Mo	-	-	2.72	-	-
0.150	Na	18.5	64,700	9,530	63,200	2,320
0.100	Nd	-	-	-	-	-
0.030	Ni	-	-	[0.43]	-	-
0.100	P	-	[8.5]	69.5	24.5	[4.3]
0.100	Pb	-	-	-	[3.8]	-
0.750	Pd	-	-	-	-	-
0.300	Rh	-	-	-	-	-
1.100	Ru	-	-	-	-	-
0.500	Sb	-	-	-	-	-
0.250	Se	-	-	-	-	-
0.500	Si	[21]	145	52.6	378	58.8
1.500	Sn	-	-	-	-	-
0.015	Sr	-	-	-	-	-
1.500	Te	-	-	-	-	-
1.000	Th	-	-	-	-	-
0.025	Ti	-	-	-	-	-
0.500	Tl	-	-	-	-	-
2.000	U	-	-	-	-	-
0.050	V	-	-	[0.38]	-	-
2.000	W	-	-	-	-	-
0.050	Y	-	-	-	-	-
0.050	Zn	[0.43]	-	[0.31]	[1.5]	[0.30]
0.050	Zr	-	-	-	-	-

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.  
 2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.  
 3) "-" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

# Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	Run Date= (Analyte)	Multiplier= ALO#= Client ID= Run Date= ug/g						
		4.8						
		98-5152-DUP						
		SPD-S107-040(dup)						
		9/23/98						
0.025	Ag	-	-	-	-	-	-	-
0.060	Al	255	-	-	-	-	-	-
0.250	As	-	-	-	-	-	-	-
0.050	B	12.8	-	-	-	-	-	-
0.010	Ba	-	-	-	-	-	-	-
0.010	Be	-	-	-	-	-	-	-
0.100	Bi	-	-	-	-	-	-	-
0.250	Ca	-	-	-	-	-	-	-
0.015	Cd	-	-	-	-	-	-	-
0.200	Ce	-	-	-	-	-	-	-
0.050	Co	-	-	-	-	-	-	-
0.020	Cr	4.16	-	-	-	-	-	-
0.025	Cu	-	-	-	-	-	-	-
0.050	Dy	-	-	-	-	-	-	-
0.100	Eu	-	-	-	-	-	-	-
0.025	Fe	1.24	-	-	-	-	-	-
2.000	K	-	-	-	-	-	-	-
0.050	La	-	-	-	-	-	-	-
0.030	Li	-	-	-	-	-	-	-
0.100	Mg	-	-	-	-	-	-	-
0.050	Mn	-	-	-	-	-	-	-
0.050	Mo	-	-	-	-	-	-	-
0.150	Na	2,300	-	-	-	-	-	-
0.100	Nd	-	-	-	-	-	-	-
0.030	Ni	-	-	-	-	-	-	-
0.100	P	[4.3]	-	-	-	-	-	-
0.100	Pb	-	-	-	-	-	-	-
0.750	Pd	-	-	-	-	-	-	-
0.300	Rh	-	-	-	-	-	-	-
1.100	Ru	-	-	-	-	-	-	-
0.500	Sb	-	-	-	-	-	-	-
0.250	Se	-	-	-	-	-	-	-
0.500	Si	57.5	-	-	-	-	-	-
1.500	Sn	-	-	-	-	-	-	-
0.015	Sr	-	-	-	-	-	-	-
1.500	Te	-	-	-	-	-	-	-
1.000	Th	-	-	-	-	-	-	-
0.025	Ti	-	-	-	-	-	-	-
0.500	Tl	-	-	-	-	-	-	-
2.000	U	-	-	-	-	-	-	-
0.050	V	-	-	-	-	-	-	-
2.000	W	-	-	-	-	-	-	-
0.050	Y	-	-	-	-	-	-	-
0.050	Zn	[0.33]	-	-	-	-	-	-
0.050	Zr	-	-	-	-	-	-	-

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.  
 2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.  
 3) "-" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Date September 15, 1998

To K.P. Brooks

From J. J. Wagner

Subject "Free Hydroxide" determination, ASR 4918

Analysis of "free hydroxide" was performed on samples **SPD-S107-006** through **SPD-S107-037**, **SPD-S107-062**, and **SPD-S107-050**; ACL# 98-004327 through 98-004332, 98-004338, and 98-004339. Sample volumes were transferred directly from original sample material into approximately 10 ml of quartz distilled water and immediately titrated. Samples were titrated using a total of 4ml of titrant. No attempt was made to estimate density of original sample material. Manual pipettes, which were verified before use, were used to measure sample material.

A Mettler, model DL21, serial number L885377 instrument was used. The titrations were performed in Bldg. 325, Lab 511. Procedure PCS-TP-511-3 was used to titrate the samples on the following dates: 9-1-98, 9-2-98.

A QC check was performed using NIST traceable NaOH to demonstrate repeatability and accuracy of free-OH measurements. Volumes titrated were 1.00ml aliquots of 0.1014N NaOH. Accuracy of measurement results agreed within 1.6% of the "true value" for aliquots tested.

Samples were titrated using 0.075 to 0.20ml volumes of original sample material. Each sample aliquot was placed in a plastic scintillation vial and 10.0 ml of quartz distilled water was added plus a magnetic stir bar. Each sample was stirred with a magnetic stirrer and titrated using 0.1037N HCl. Normality of the titrant (0.1037 N HCl) was prepared and verified by R. G. Swoboda on 7-10-98. Titrant is traceable to NIST. The apparent "free hydroxide" is calculated based upon the first equivalence point on the titration curve. The molar concentration obtained is listed in the attached table labeled "Free OH<sup>-</sup> (mols)". Concentration of free OH<sup>-</sup> varied from about 0.07 to about 2.9 mols OH<sup>-</sup>.

In a previous titration of caustic Tank sludge samples, a spiked sample was prepared and analyzed to verify that the free OH<sup>-</sup> normally was associated with the first equivalence point value. The first equivalence point measured for the spiked sample did in fact, occur at essentially the same pH as the non-spike sample (pH ~9.8 and 10.0 respectively). Recovery of the added spike (1 ml of 0.1014N NaOH, NIST traceable) was 100.4%. pH of the 1<sup>st</sup> equivalence point is highly dependent on other concomitants in the sample. The first equivalence point for pure NaOH occurs between about pH 6.5 and pH 6.9.

The formula used for calculating the "free OH<sup>-</sup>" concentration follows.

$$DF * (V_{\text{titrant}} * N_{\text{titrant}}) / V_{\text{sample}}$$

$N_{\text{titrant}} = 0.1037$  (Normality of HCl)

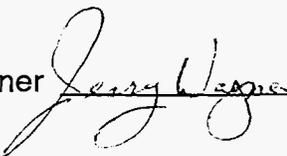
$DF = 1$  (dilution factor)

$V_{\text{titrant}} =$  volume (ml) of titrant (1<sup>st</sup> Equiv. Point)

$V_{\text{sample}} =$  aliquot volume (ml) of (original or diluted sample used)

See attached table for summary results and titration curves (pH vs. volume of titrant and the derivative-pH/ml vs. volume of titrant). Titration printouts also include raw data of running total of titrant volume(ml), incremental titrant added(ml), signal(pH), signal change(pH), and derivative(pH/ml).

JJ Wagner

 9-15-98

Concur

 9/15/98

K. Brooks ASR# 4918

[Cr] [Al] [Na] free OH-  
(ug/ml) (ug/ml) (ug/ml) (mols)

7	--	--	--	0.1023
5	272	1870	21200	0.246
6	118	13400	57000	1.982
7	143	15600	70200	2.863
7	--	--	--	0.1030
8	22.6	2640	17500	0.685
9	7.66	719	5360	0.183
10	2.63	200	1860	0.071
16	80.6	13200	79000	2.837
17	12.6	2210	60900	2.948

ALO#	Client ID
QC Check	LCS= 0.1014M Na
98-4327	SPD-S107-006
98-4328	SPD-S107-013
98-4329	SPD-S107-021
QC Check	LCS= 0.1014M Na
98-4330	SPD-S107-031
98-4331	SPD-S107-034
98-4332	SPD-S107-037
98-4338	SPD-S107-062
98-4339	SPD-S107-050

Titrant for (N(HCl))= .1037		Titrant for (N(HCl))= .1037		Titrant for (N(HCl))= .1037		Titrant for (N(HCl))= .1037		Titrant for (N(HCl))= .1037		Titrant for (N(HCl))= .1037		Titrant for (N(HCl))= .1037		
1st Equiv. Pt.	2nd Equiv. Pt.	3rd Equiv. Pt.	4th Equiv. Pt.	5th Equiv. Pt.	6th Equiv. Pt.	7th Equiv. Pt.	vol. (ml)	pH	vol. (ml)	pH	vol. (ml)	pH	vol. (ml)	pH
1.974	6.7	0.264	7.8	0.211	5.1	--	--	--	--	--	--	--	--	--
0.475	9.9	0.759	6.8	0.228	4.6	0.213	4.3	0.145	4.3	0.283	3.8	0.200	4.1	
1.911	10.4	0.859	7.0	--	--	--	--	--	--	--	--	--	--	
2.761	10.4	--	--	0.141	4.5	0.203	4.3	0.212	3.8	--	--	--	--	
1.986	6.9	0.295	6.7	0.163	3.9	--	--	--	--	--	--	--	--	
1.321	10.1	0.085	6.7	--	--	--	--	--	--	--	--	--	--	
0.354	9.6	--	--	0.350	5.3	0.570	3.6	--	--	--	--	--	--	
0.137	6.6	0.905	7.9	0.338	3.3	--	--	--	--	--	--	--	--	
2.052	10.5	0.170	6.4	--	--	--	--	--	--	--	--	--	--	
2.843	9.6	--	--	--	--	--	--	--	--	--	--	--	--	

Titration performed in 325 Bldg. Lab 511 using a Mettler model DL21 titrator, Ser. No. L885377.

Analyzed: 9-1-98 & 9-2-98 Analyst: J.J. Wagner

Jerry Wagner *Jerry Wagner* 9-15-98 Date Signature

Concur *MW* 9/15/98 Date Signature

File/LB

Date September 9, 1998

 To KP Brooks  
 From MW Urie *MW Urie*  
 Subject TOC/TIC/TC by Hot Persulfate for Samples  
Submitted under ASR-4997

The analysis of these samples was done by the hot persulfate wet oxidation method, PNL-ALO-381, rev. 1. The work was performed at Impact Level II, according to ASR 4997, dated April 20, 1998. The hot persulfate method uses acid decomposition for TIC and acidic potassium persulfate oxidation at 92-95 °C for TOC, all on the same weighed sample, with TC being the sum of the TIC and TOC.

The samples were analyzed on August 28, 1998, and the table below shows the results, rounded to two to three significant figures. The full results are located on the attached spreadsheet, with the supporting raw data located on data sheets and review report spreadsheets and on file in the Radiochemical Processing Group's Laboratory Support Office under Project 28966 Files 98-04843/04844. The TIC and TOC standard is calcium carbonate and glucose, respectively. The standard materials were used in solid form for system calibration standards as well as matrix spikes. TIC and TOC percent recovery are determined using the appropriate standard (i.e., calcium carbonate for TIC or glucose for TOC). All sample results are corrected for average percent recovery of system calibration standards and are also corrected for contribution from the blank.

ALO Number	Sample ID	Wt (g)	TIC (µg/g)	TIC RPD	TOC (µg/g)	TOC RPD	TC (µg/g)	TC RPD
98-04843	SPD-S107-003	0.1004	6845	8	2675	1	9520	6
	SPD-S107-003 Dup	0.0981	7450		2643		10093	
98-04844	SPD-S107-044	0.0604	4706	45	2001	26	6707	40
	SPD-S107-044 Dup	0.0595	7413		2604		10017	
98-04844 Spike	SPD-S107-044 Spike	0.0509	108%		106%		107%	

**QC Narrative:** QC for the method involved calibration blanks, system calibration standards, sample duplicates, and one matrix spike for the batch. The average of the QC system calibration standards was within acceptance criteria at 96.3% recovery for TIC and 96.8% recovery for TOC. For TIC, the standards ranged from 88% to 111% recovery; a significantly wider range than typically obtained. The calibration blanks were acceptable, averaging 16 µgC for TIC and 51 µgC for TOC.

The accuracy of the TIC and TOC measurements can be estimated by the recovery results from the matrix spike. The matrix spike was prepared by adding solid calcium carbonate and glucose spikes to a pre-weighed aliquot of sample 98-04844. The matrix spike recovery for both TIC and TOC were within acceptance criteria, being 108% and 106%, respectively. The precision, estimated by the RPD (Relative Percent Difference) between duplicates, met acceptance criteria (i.e., RPD <20%) for TIC and TOC analyses only for sample 98-04843. Significant sample heterogeneity was observed in both samples, making representative sub-sampling very difficult.

**Problems:** No significant problems were encountered for these samples.

Concur by:



Date:

9-9-98

 Files: C120-P-701a.doc, C120-P-701.xls  
 System File: TOC082898

 Sequence Number: 120  
 ASR Number: 4997



**Battelle Pacific Northwest Laboratory  
Radiochemical Processing Group-325 Building  
Radioanalytical Applications Team**

98-4327  
9/11/98

Client : Brooks      Wp#: K87684

Cognizant Scientist: C Soderqvist

Date : 9/14/98

Concur : Richard T. Ross

Date : 9/15/98

**Measured Activities (uCi/g)**

ALO ID Client ID	Gross Alpha Error %	Pu-239/240 Error %	Am-241/ Pu-238 Error %	Cm-243/244 Error %	Sum of Individual Alphas
<b>Aqueous Samples:</b>					
98-4327PB Process Blank	<2.E-5	1.25E-6 9%	3.15E-6 5%	8.57E-7 11%	5.26E-6
98-4327 SPD-S107-006	6.09E-5 13%	6.25E-5 6%	2.18E-5 11%	3.72E-6 32%	8.80E-5
98-4328 SPD-S107-013	7.00E-5 12%	4.84E-5 6%	3.62E-5 7%	6.84E-6 18%	9.14E-5
98-4329 SPD-S107-021	5.92E-5 13%	9.34E-5 4%	1.50E-5 13%	<4.E-6	1.08E-4
98-4330 SPD-S107-031	3.87E-5 18%	7.13E-6 36%	1.30E-5 33%	4.54E-6 30%	2.47E-5
98-4330 Rep SPD-S107-031	-	1.04E-5 16%	2.71E-5 9%	5.57E-6 22%	4.31E-5
RPD		37%	70%	20%	
98-4331 SPD-S107-034	2.10E-5 28%	3.68E-6 44%	1.07E-5 20%	7.02E-6 23%	2.14E-5
98-4331 DUP SPD-S107-034	2.37E-5 31%	1.16E-5 22%	1.79E-5 18%	<5.E-6	2.95E-5
RPD	12%	104%	50%		
98-4332 SPD-S107-037	8.08E-5 13%	3.29E-5 13%	3.58E-5 11%	<3.E-6	6.87E-5
98-4332-Rep SPD-S107-037	9.16E-5 12%	-	-	-	
RPD	13%				

Client : Brooks      Wp#: K87684

Measured Activities (uCi/g)

ALO ID Client ID	Gross Alpha Error %	Pu-239/240 Error %	Am-241/ Pu-238 Error %	Cm-243/244 Error %	Sum of Individual Alphas
<b>Fusion Samples:</b>					
98-4843-PB Process Blank	5.47E-3	2.17E-3 3%	4.66E-03 2%	1.35E-3 3%	8.18E-3
98-4843 SPD-S107-003	5.19E-1	7.42E-1 2%	2.55E-1 2%	2.07E-3 29%	9.99E-1
98-4844 SPD-S107-003-Dup	5.63E-1	7.36E-1 2%	2.68E-1 3%	2.24E-3 34%	1.01E+0
RPD	33%	1%	5%	8%	
98-4845 SPD-S107-044	1.72E+0	1.58E+0 2%	5.58E-1 2%	5.16E-3 18%	2.14E+0
Standard	108%	101%			
Sample Spike	90%	85%			
Blank	<1.E-5	<5.E-8	<5.E-8	<4.E-8	

The gross alpha results for the fusion samples are biased low from solids loading. Use the sum of the individual alpha emitters instead (far right column).

Battelle Pacific Northwest Laboratory  
Radiochemical Processing Group-325 Building  
Radioanalytical Applications Team

98-4327  
9/11/98

Client : Brooks      Wp#: K87684

Cognizant Scientist: C. Soderqvist

Date : 9-11-98

Concur : J. K. Sudej

Date : 9/11/98

Measured Activities (uCi/g)

<u>ALO ID</u>	<u>Sr-90</u>
<u>Client ID</u>	<u>Error %</u>
<b>Aqueous Samples:</b>	
98-4327PB	1.11E-2
Process Blank	3%
98-4327	< 3E-2
SPD-S107-006	
98-4328	4.57E-3
SPD-S107-013	59%
98-4329	8.70E-3
SPD-S107-021	31%
98-4330	4.13E-3
SPD-S107-031	6%
98-4330 Rep	4.57E-3
SPD-S107-034	5%
RPD	10%
98-4331	2.87E-3
SPD-S107-034	7%
98-4331 DUP	3.36E-3
SPD-S107-034	6%
RPD	16%
98-4332	2.57E-3
SPD-S107-037	7%

Client : Brooks      Wp#: K87684

Measured Activities (uCi/g)

<u>ALO ID</u>	<u>Sr-90</u>
<u>Client ID</u>	<u>Error %</u>
<b>Fusion Samples:</b>	
98-4843-PB	4.10E-1
Process Blank	3%
98-4843	3.77E+2
SPD-S107-003	3%
98-4844	3.98E+2
SPD-S107-003-Dup	3%
RPD	5%
98-4845	8.37E+2
SPD-S107-044	3%
Standard	90%
Sample Spike	88%
Blank	< 3E-4

## Radiochemistry

### S107 Samples

Aqueous decants (SPD-S107-006, -013, -021, -031, -034, and -037) and fusions (SPD-S107-003 and -044) of the S107 slurry samples were analyzed for gamma, alpha, and  $^{90}\text{Sr}$  activity. The aqueous samples were aliquoted in the hot cells with a process blank; the slurries were fused in the hot cells with a prep blank following the fusion procedure PNL-ALO-115. Radiochemistry results are presented on the attached table in  $\mu\text{Ci/g}$  for both types of samples since the liquids were aliquoted on a weight basis and specific gravity data was not provided.

### Gamma Energy Analysis

Direct aliquots of the aqueous samples and fusions of the slurry samples were counted directly for gamma emitters (procedure PNL-ALO-450). The dominant gamma emitter is  $^{137}\text{Cs}$ , with smaller amounts of  $^{60}\text{Co}$ ,  $^{154}\text{Eu}$ ,  $^{155}\text{Eu}$  and  $^{241}\text{Am}$ . Since the samples were counted as received with no further preparation, no spikes or duplicates were prepared in the laboratory; however, duplicate samples were prepped in the hot cells in both cases. The duplicate results agree well except for  $^{60}\text{Co}$  in sample SPD-S107-003. The reason for this difference is not understood. Weak activities were seen in the hot cell blanks; however, the activities are negligible relative to the samples.

### Alpha Analysis

The samples were analyzed for alpha emitters by first counting dried aliquots for gross alpha emission (procedure PNL-ALO-420, 421), then mounting aliquots for alpha spectroscopy to identify and measure individual alpha emitters (procedure PNL-ALO-496, 469). Ideally, the sum of the individual alpha emitters equals the gross alpha result, but mass loading from dissolved solids in the samples often causes the gross alpha results to be low. Fusion preparations, particularly, tend to be about 40% low. For the S107 samples, the sum of the individual alpha emitters (far right column on the attached report) is a better estimate of the total alpha activity than the gross alpha result.

The aqueous samples have relatively little alpha emission (20 to 100 pCi per gram). Because the alpha count rate is so low, the gross alpha results have high counting error. (The high beta count rate from  $^{137}\text{Cs}$  limited the amount of sample we could count for gross alpha.) The alpha spectroscopy results had much longer counting times and somewhat smaller counting errors.

The gross alpha results for the aqueous samples agree reasonably well with the sum of the individual alpha emitters, considering the large counting errors. The hot cell duplicates and the lab replicates agree poorly, but the counting error is very high for these samples. The hot cell blank has easily detectable alpha—5 pCi per gram, compared to 20 pCi per gram for the lowest sample.

The fusion samples have  $10^5$  times more alpha than the aqueous samples, and the counting errors are small. Solids loading on the gross alpha counting mounts (from the fusion flux) caused low results, so the sum of the individual alpha emitters should be used instead of the gross alpha result. The fusion blank has easily detectable alpha, but the samples are high enough that the blank is inconsequential. The duplicates agree well inside expected uncertainty.

The matrix spike and reagent spike gave good results. (The two spikes were processed in a hood, not a hot cell, and are unaffected by the hot cell blank. Unlike the aqueous samples, the spikes had adequate activity for good counting statistics.) The lab blank had no detectable alpha activity.

### Strontium-90 Analysis

The samples were analyzed for  $^{90}\text{Sr}$  by chemical separation of strontium followed by beta counting (procedures PNL-ALO-476 and 484). The aqueous samples had far more  $^{137}\text{Cs}$  than  $^{90}\text{Sr}$ —in fact,  $^{90}\text{Sr}$  was barely detectable in two samples and not detectable in a third. (Aliquots were calculated from the gross beta emission, which turned out to be essentially all  $^{137}\text{Cs}$ .) The hot cell blank had more  $^{90}\text{Sr}$  than any of the aqueous samples. The hot cell duplicates and the lab replicates agree reasonably well. The lab blank had no detectable  $^{90}\text{Sr}$ . The matrix spike and reagent spike gave good results.

The fusions have more  $^{90}\text{Sr}$  than  $^{137}\text{Cs}$ . The hot cell fusion blank has easily detectable  $^{90}\text{Sr}$ , but is negligible compared to the samples. The duplicate results agree well.

Richard T. R. → 9/15/98  
J.R. Hammond 9/15/98

Battelle Pacific Northwest Laboratory  
 Radiochemical Processing Group-325 Building  
 Radioanalytical Applications Team

98-4327  
 9/4/98

Client : Brooks  
 Wp#: K87684

Cognizant Scientist: J.R. Greenwood

Date : 9/4/98

Concur : T. Trang-le

Date : 9/4/98

Measured Activities (uCi/g)

ALO ID Client ID	Co-60 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
<b>Aqueous Samples:</b>					
98-4327PB Process Blank	<5.E-6	1.11E-4 4%	<2.E-5	<3.E-5	<3.E-5
98-4327 SPD-S107-006	<1.E-4	1.40E+1 2%	<4.E-4	<8.E-3	<8.E-3
98-4328 SPD-S107-013	<5.E-5	4.58E+0 2%	<2.E-4	<4.E-3	<4.E-3
98-4329 SPD-S107-021	<1.E-4	2.85E+0 2%	<3.E-4	<3.E-3	<3.E-3
98-4330 SPD-S107-031	3.95E-4 3%	4.01E-1 2%	<4.E-5	<4.E-4	<4.E-4
98-4331 SPD-S107-034	<2.E-5	1.64E-1 2%	<4.E-5	<3.E-4	<3.E-4
98-4331 DUP SPD-S107-034	<2.E-5	1.68E-1 2%	<4.E-5	<3.E-4	<3.E-4
RPD		2%			
98-4332 SPD-S107-037	<2.E-5	1.37E-1 2%	<4.E-5	<3.E-4	<3.E-4
<b>Fusion Samples:</b>					
98-4843-PB Process Blank	<6.E-4	1.36E-1 2%	<2.E-3	<2.E-3	1.79E-3 40%
98-4843 SPD-S107-003	4.41E-1 2%	1.02E+2 2%	8.84E-2 5%	<8.E-2	2.20E-1 12%
98-4844 SPD-S107-003-Dup	5.14E-2 3%	1.03E+2 2%	8.79E-2 5%	6.43E-2 22%	2.67E-1 11%
RPD	158%	1%	1%		19%
98-4845 SPD-S107-044	5.84E-2 3%	8.46E+1 2%	2.52E-1 3%	1.49E-1 12%	5.27E-1 8%

## Radiochemistry

### S107 Samples

Aqueous decants (SPD-S107-006, -013, -021, -031, -034, and -037) and fusions (SPD-S107-003 and -044) of the S107 slurry samples were analyzed for gamma, alpha, and  $^{90}\text{Sr}$  activity. The aqueous samples were aliquoted in the hot cells with a process blank; the slurries were fused in the hot cells with a prep blank following the fusion procedure PNL-ALO-115. Radiochemistry results are presented on the attached table in  $\mu\text{Ci/g}$  for both types of samples since the liquids were aliquoted on a weight basis and specific gravity data was not provided.

### Gamma Energy Analysis

The samples were directly counted for gamma emitters according to procedure PNL-ALO-450. The dominant gamma emitter is  $^{137}\text{Cs}$ , with smaller amounts of  $^{60}\text{Co}$ ,  $^{154}\text{Eu}$ ,  $^{155}\text{Eu}$  and  $^{241}\text{Am}$ . Since the samples were counted as received with no further preparation, no spikes or duplicates were prepared in the laboratory; however, duplicate samples were prepped in the hot cells in both cases. The duplicate results are in good agreement except for the  $^{60}\text{Co}$  seen in sample SPD-S107-003. The reason for this difference is not understood. Weak activities were seen in the hot cell blanks; however, the activities are negligible relative to the samples.

*J. R. Howard*

9-4-98

## **Appendix D**

### **Radioactive Colloids Laboratory Analyses**

# Particle Size Analysis

S-107 REPS DUP

Date: 09/21/98 Meas #: 00033

Time: 08:14 Pres #: 01

S-107:REPS DUP 10 min  
40 ml/s, in 0.53 M NaOH/0.1 M NaNO3

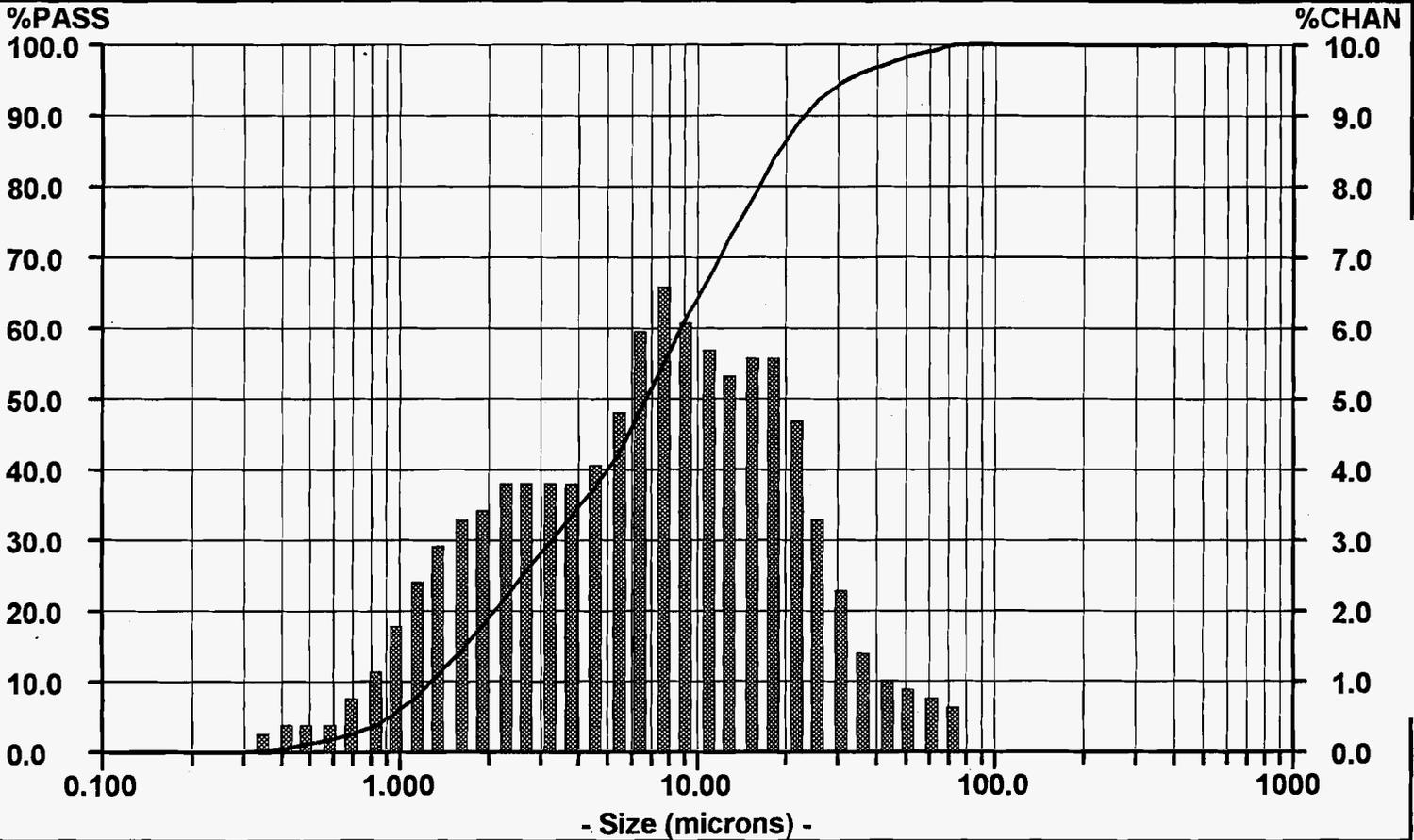
### Summary

mv = 10.35  
mn = 0.629  
ma = 3.222  
cs = 1.862  
sd = 8.343

### Percentiles

10% = 1.284 60% = 8.833  
20% = 2.114 70% = 11.98  
30% = 3.301 80% = 16.30  
40% = 5.029 90% = 23.05  
50% = 6.766 95% = 31.94

Dia	Vol%	Width
18.24	33%	18.26
6.511	38%	4.896
1.658	29%	1.742



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	61.62	6.28						
592.0	100.00	0.00	7.778	55.34	6.61						
497.8	100.00	0.00	6.541	48.73	6.04						
418.6	100.00	0.00	5.500	42.69	4.95						
352.0	100.00	0.00	4.625	37.74	4.12						
296.0	100.00	0.00	3.889	33.62	3.82						
248.9	100.00	0.00	3.270	29.80	3.87						
209.3	100.00	0.00	2.750	25.93	3.93						
176.0	100.00	0.00	2.312	22.00	3.80						
148.0	100.00	0.00	1.945	18.20	3.58						
124.5	100.00	0.00	1.635	14.62	3.36						
104.7	100.00	0.00	1.375	11.26	3.07						
88.00	100.00	0.00	1.156	8.19	2.53						
74.00	100.00	0.78	0.972	5.66	1.83						
62.23	99.22	0.82	0.818	3.83	1.22						
52.33	98.40	0.94	0.688	2.61	0.81						
44.00	97.46	1.16	0.578	1.80	0.57						
37.00	96.30	1.57	0.486	1.23	0.45						
31.11	94.73	2.31	0.409	0.78	0.40						
26.16	92.42	3.49	0.344	0.38	0.38						
22.00	88.93	4.82	0.289	0.00	0.00						
18.50	84.11	5.64	0.243	0.00	0.00						
15.56	78.47	5.66	0.204	0.00	0.00						
13.08	72.81	5.49	0.172	0.00	0.00						
11.00	67.32	5.70	0.145	0.00	0.00						

# Particle Size Analysis

S-107 REPS DUP

Date: 09/21/98 Meas #: 00033

Time: 08:14 Pres #: 01

S-107:REPS DUP | Omin  
40 ml/s, in 0.53 M NaOH/0.1 M NaNO3

**Summary**

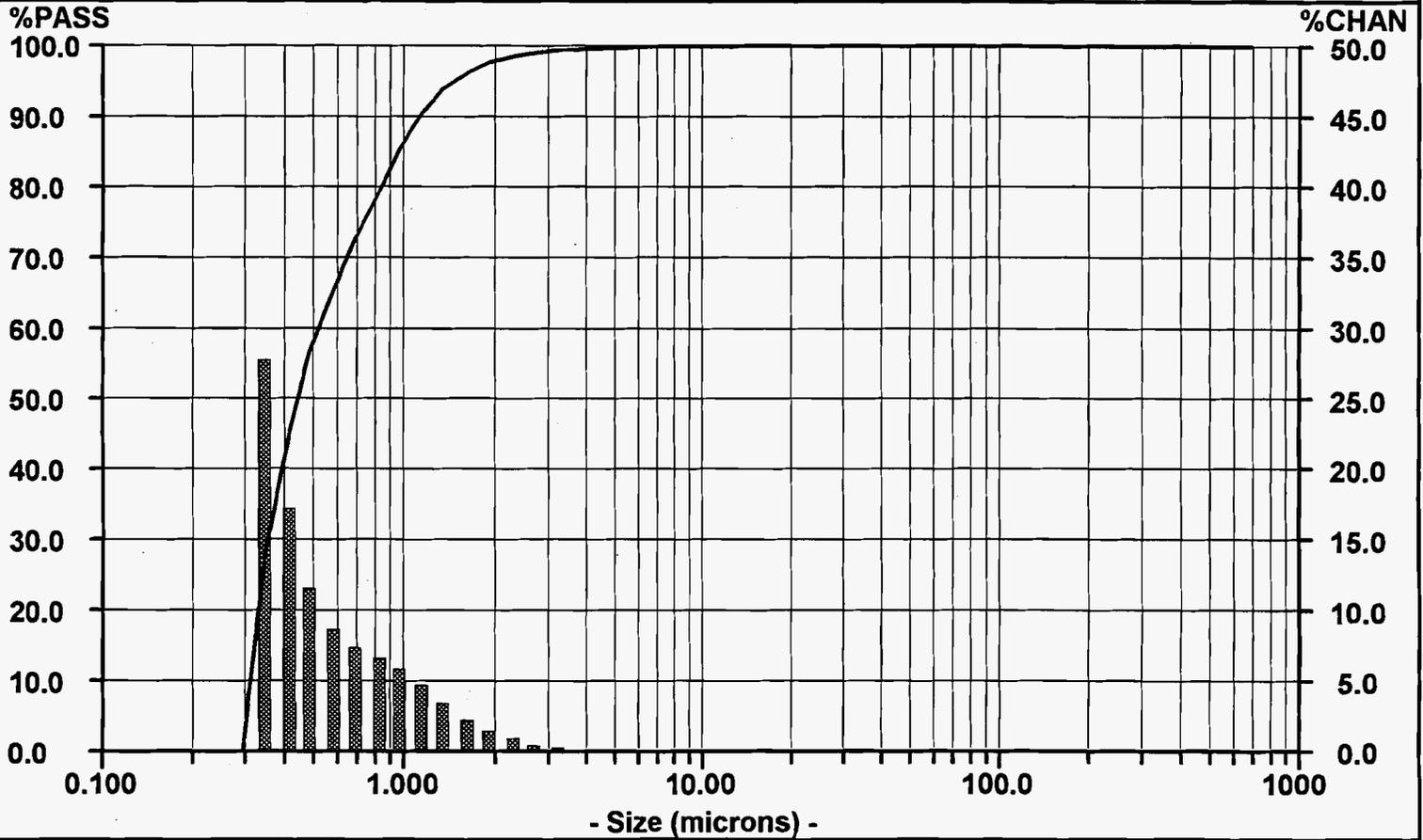
mv = 10.35  
mn = 0.629  
ma = 3.222  
cs = 1.862  
sd = 0.303

**Percentiles**

10% = 0.311 60% = 0.516  
20% = 0.328 70% = 0.639  
30% = 0.350 80% = 0.827  
40% = 0.384 90% = 1.138  
50% = 0.436 95% = 1.481

**Dia Vol% Width**

0.436 100% 0.606



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	99.98	0.02						
592.0	100.00	0.00	7.778	99.96	0.04						
497.8	100.00	0.00	6.541	99.92	0.06						
418.6	100.00	0.00	5.500	99.86	0.09						
352.0	100.00	0.00	4.625	99.77	0.12						
296.0	100.00	0.00	3.889	99.65	0.19						
248.9	100.00	0.00	3.270	99.46	0.33						
209.3	100.00	0.00	2.750	99.13	0.66						
176.0	100.00	0.00	2.312	98.57	0.91						
148.0	100.00	0.00	1.945	97.66	1.45						
124.5	100.00	0.00	1.635	96.21	2.28						
104.7	100.00	0.00	1.375	93.93	3.51						
88.00	100.00	0.00	1.156	90.42	4.86						
74.00	100.00	0.00	0.972	85.56	5.90						
62.23	100.00	0.00	0.818	79.66	6.62						
52.33	100.00	0.00	0.688	73.04	7.42						
44.00	100.00	0.00	0.578	65.62	8.77						
37.00	100.00	0.00	0.486	56.85	11.61						
31.11	100.00	0.00	0.409	45.24	17.37						
26.16	100.00	0.00	0.344	27.87	27.87						
22.00	100.00	0.00	0.289	0.00	0.00						
18.50	100.00	0.00	0.243	0.00	0.00						
15.56	100.00	0.00	0.204	0.00	0.00						
13.08	100.00	0.01	0.172	0.00	0.00						
11.00	99.99	0.01	0.145	0.00	0.00						

# Particle Size Analysis

S-107 REPS DUP

Date: 09/21/98 Meas #: 00035

Time: 08:24 Pres #: 01

S-107:REPS DUP 20 min  
60 ml/s, in 0.53 M NaOH/0.1 M NaNO3

**Summary**

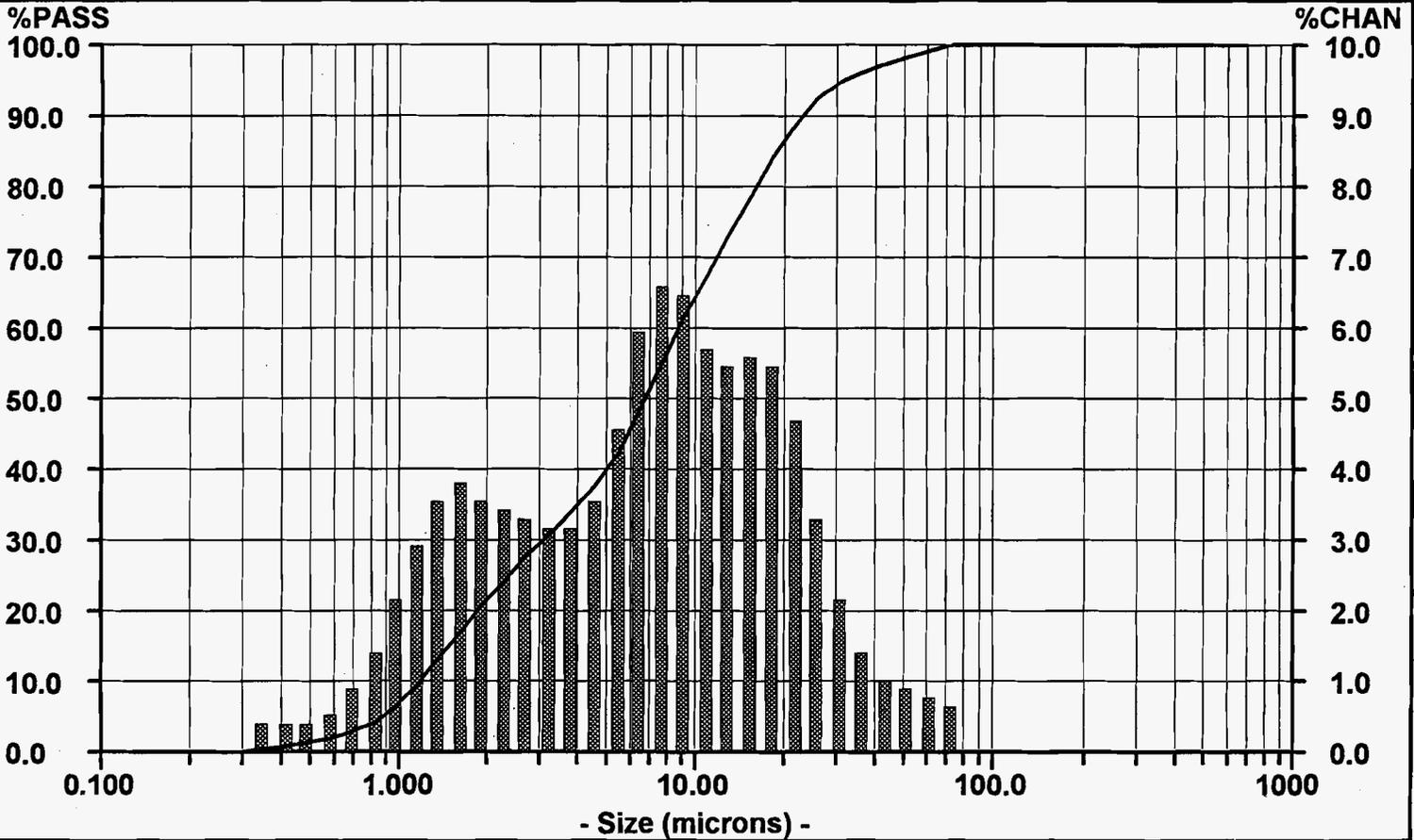
mv = 10.27  
mn = 0.626  
ma = 3.046  
cs = 1.970  
sd = 8.380

**Percentiles**

10% = 1.178 60% = 8.836  
20% = 1.874 70% = 11.88  
30% = 3.122 80% = 16.17  
40% = 5.072 90% = 22.91  
50% = 6.828 95% = 31.84

**Dia Vol% Width**

18.18 32% 18.28  
6.409 40% 5.235  
1.409 28% 1.368



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	61.67	6.53						
592.0	100.00	0.00	7.778	55.14	6.79						
497.8	100.00	0.00	6.541	48.35	6.01						
418.6	100.00	0.00	5.500	42.34	4.66						
352.0	100.00	0.00	4.625	37.68	3.63						
296.0	100.00	0.00	3.889	34.05	3.20						
248.9	100.00	0.00	3.270	30.85	3.20						
209.3	100.00	0.00	2.750	27.65	3.36						
176.0	100.00	0.00	2.312	24.29	3.52						
148.0	100.00	0.00	1.945	20.77	3.67						
124.5	100.00	0.00	1.635	17.10	3.80						
104.7	100.00	0.00	1.375	13.30	3.68						
88.00	100.00	0.00	1.156	9.62	3.09						
74.00	100.00	0.79	0.972	6.53	2.20						
62.23	99.21	0.83	0.818	4.33	1.42						
52.33	98.38	0.94	0.688	2.91	0.92						
44.00	97.44	1.14	0.578	1.99	0.64						
37.00	96.30	1.53	0.486	1.35	0.50						
31.11	94.77	2.26	0.409	0.85	0.44						
26.16	92.51	3.43	0.344	0.41	0.41						
22.00	89.08	4.76	0.289	0.00	0.00						
18.50	84.32	5.59	0.243	0.00	0.00						
15.56	78.73	5.65	0.204	0.00	0.00						
13.08	73.08	5.55	0.172	0.00	0.00						
11.00	67.53	5.86	0.145	0.00	0.00						

# Particle Size Analysis

S-107 REPS DUP

Date: 09/21/98 Meas #: 00035  
Time: 08:24 Pres #: 01

S-107:REPS DUP *20 min*  
60 ml/s, in 0.53 M NaOH/0.1 M NaNO3

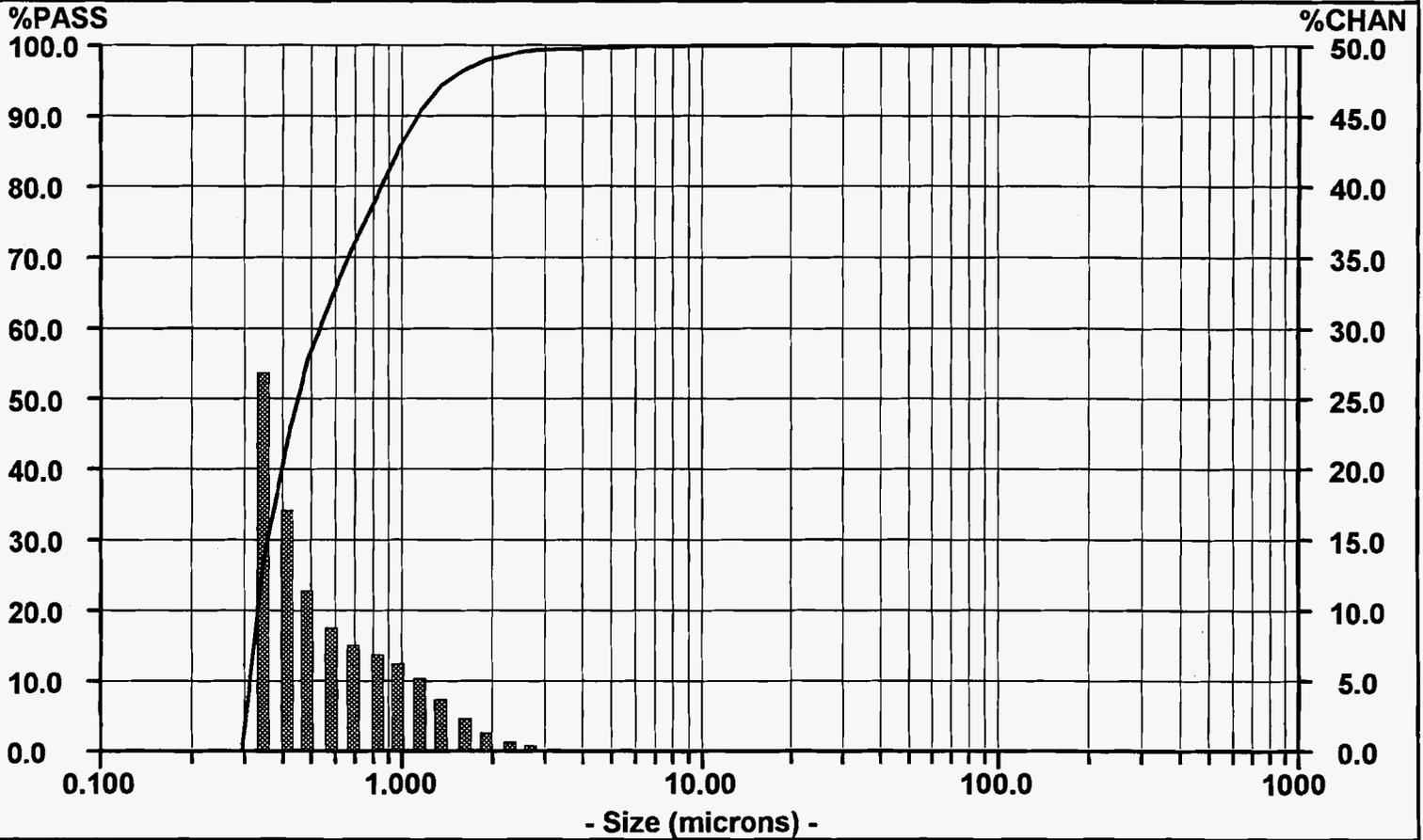
**Summary**

mv = 10.27  
mn = 0.626  
ma = 3.046  
cs = 1.970  
sd = 0.307

**Percentiles**

10% = 0.311 60% = 0.527  
20% = 0.330 70% = 0.654  
30% = 0.353 80% = 0.840  
40% = 0.388 90% = 1.128  
50% = 0.444 95% = 1.422

**Dia Vol% Width**  
0.444 100% 0.613



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	99.98	0.02						
592.0	100.00	0.00	7.778	99.96	0.04						
497.8	100.00	0.00	6.541	99.92	0.06						
418.6	100.00	0.00	5.500	99.86	0.07						
352.0	100.00	0.00	4.625	99.79	0.10						
296.0	100.00	0.00	3.889	99.69	0.14						
248.9	100.00	0.00	3.270	99.55	0.24						
209.3	100.00	0.00	2.750	99.31	0.43						
176.0	100.00	0.00	2.312	98.88	0.76						
148.0	100.00	0.00	1.945	98.12	1.33						
124.5	100.00	0.00	1.635	96.79	2.32						
104.7	100.00	0.00	1.375	94.47	3.77						
88.00	100.00	0.00	1.156	90.70	5.33						
74.00	100.00	0.00	0.972	85.37	6.37						
62.23	100.00	0.00	0.818	79.00	6.92						
52.33	100.00	0.00	0.688	72.08	7.56						
44.00	100.00	0.00	0.578	64.52	8.83						
37.00	100.00	0.00	0.486	55.69	11.58						
31.11	100.00	0.00	0.409	44.11	17.14						
26.16	100.00	0.00	0.344	26.97	26.97						
22.00	100.00	0.00	0.289	0.00	0.00						
18.50	100.00	0.00	0.243	0.00	0.00						
15.56	100.00	0.00	0.204	0.00	0.00						
13.08	100.00	0.01	0.172	0.00	0.00						
11.00	99.99	0.01	0.145	0.00	0.00						

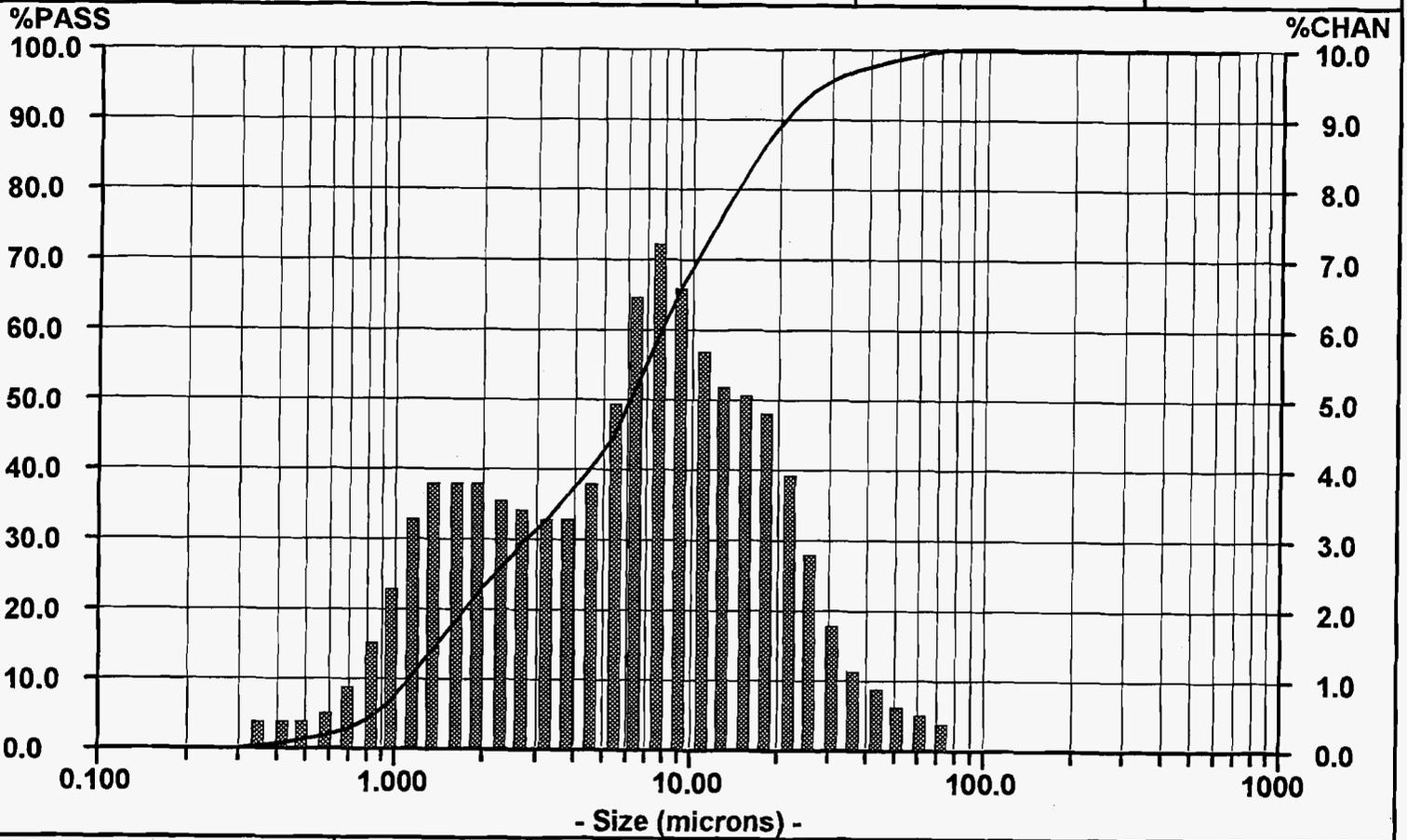
# Particle Size Analysis

S-107 REPS DUP

Date: 09/21/98 Meas #: 00038  
Time: 08:41 Pres #: 01

S-107:REPS DUP  
40 ml/s, in 0.53 M NaOH/0.1 M NaNO3  
Sonication #2 @ 40 W-90 sec

Summary	Percentiles		Dia	Vol%	Width
mv = 9.240	10% = 1.124	60% = 7.905	8.928	70%	14.99
mn = 0.635	20% = 1.749	70% = 10.36	1.390	30%	1.369
ma = 2.867	30% = 2.806	80% = 14.41			
cs = 2.093	40% = 4.547	90% = 20.79			
sd = 7.528	50% = 6.219	95% = 28.25			



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	66.12	6.77						
592.0	100.00	0.00	7.778	59.35	7.32						
497.8	100.00	0.00	6.541	52.03	6.55						
418.6	100.00	0.00	5.500	45.48	5.06						
352.0	100.00	0.00	4.625	40.42	3.92						
296.0	100.00	0.00	3.889	36.50	3.45						
248.9	100.00	0.00	3.270	33.05	3.45						
209.3	100.00	0.00	2.750	29.60	3.59						
176.0	100.00	0.00	2.312	26.01	3.69						
148.0	100.00	0.00	1.945	22.32	3.80						
124.5	100.00	0.00	1.635	18.52	3.96						
104.7	100.00	0.00	1.375	14.56	3.95						
88.0	100.00	0.00	1.156	10.61	3.43						
74.00	100.00	0.56	0.972	7.18	2.49						
62.23	99.44	0.61	0.818	4.69	1.60						
52.33	98.83	0.73	0.688	3.09	1.02						
44.00	98.10	0.91	0.578	2.07	0.69						
37.00	97.19	1.25	0.486	1.38	0.52						
31.11	95.94	1.86	0.409	0.86	0.45						
26.16	94.08	2.87	0.344	0.41	0.41						
22.00	91.21	4.05	0.289	0.00	0.00						
18.50	87.16	4.90	0.243	0.00	0.00						
15.56	82.26	5.12	0.204	0.00	0.00						
13.08	77.14	5.23	0.172	0.00	0.00						
11.00	71.91	5.79	0.145	0.00	0.00						

# Particle Size Analysis

S-107 REPS DUP

Date: 09/21/98 Meas #: 00038  
Time: 08:41 Pres #: 01

S-107:REPS DUP  
40 ml/s, in 0.53 M NaOH/0.1 M NaNO3  
Sonication #2 @ 40 W-90 sec

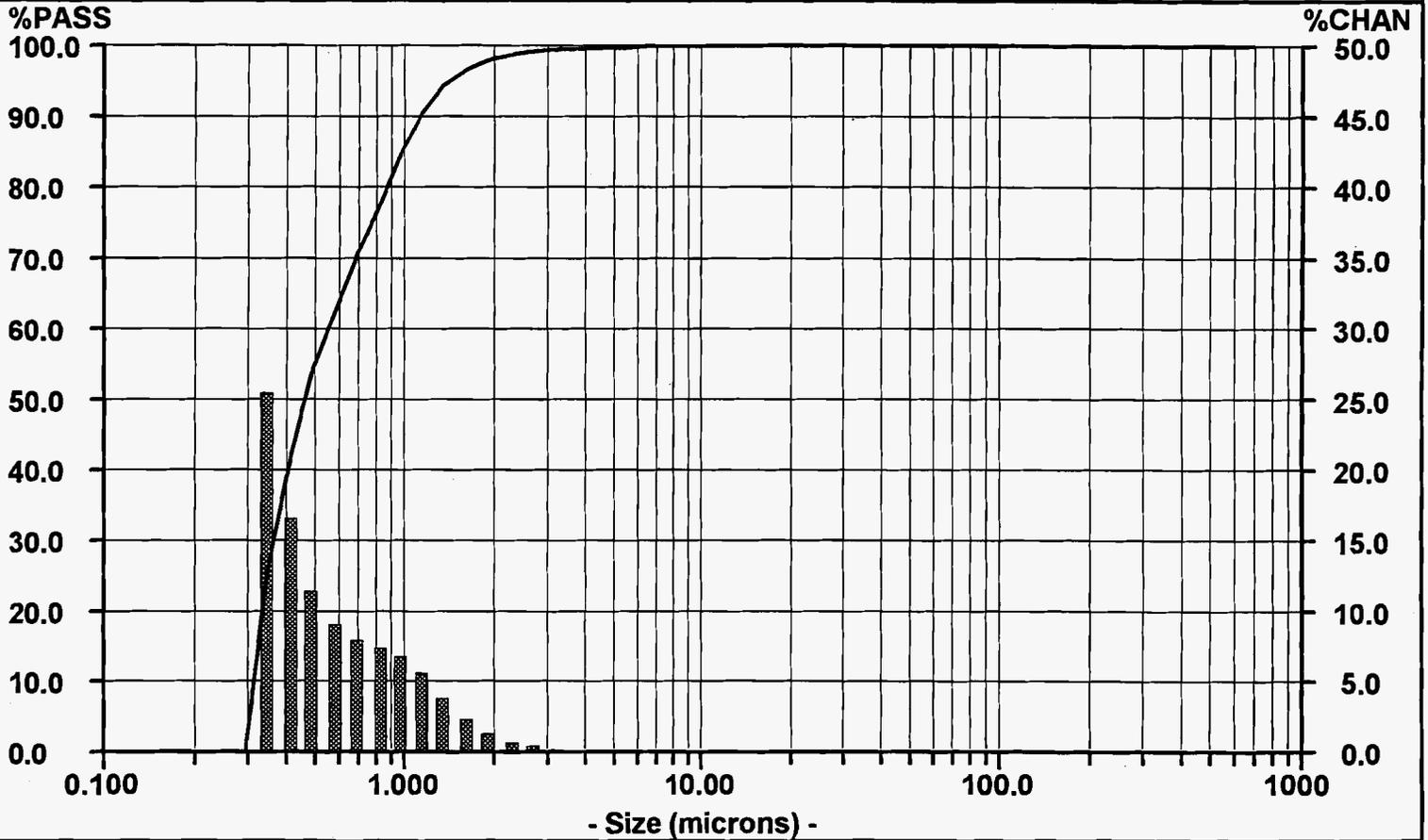
**Summary**

mv = 9.240  
mn = 0.635  
ma = 2.867  
cs = 2.093  
sd = 0.311

**Percentiles**

10% = 0.312 60% = 0.547  
20% = 0.332 70% = 0.676  
30% = 0.357 80% = 0.855  
40% = 0.397 90% = 1.131  
50% = 0.457 95% = 1.420

**Dia Vol% Width**  
0.457 100% 0.623



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	99.98	0.02						
592.0	100.00	0.00	7.778	99.96	0.04						
497.8	100.00	0.00	6.541	99.92	0.06						
418.6	100.00	0.00	5.500	99.86	0.08						
352.0	100.00	0.00	4.625	99.78	0.10						
296.0	100.00	0.00	3.889	99.68	0.15						
248.9	100.00	0.00	3.270	99.53	0.25						
209.3	100.00	0.00	2.750	99.28	0.44						
176.0	100.00	0.00	2.312	98.84	0.76						
148.0	100.00	0.00	1.945	98.08	1.31						
124.5	100.00	0.00	1.635	96.77	2.30						
104.7	100.00	0.00	1.375	94.47	3.85						
88.00	100.00	0.00	1.156	90.62	5.62						
74.00	100.00	0.00	0.972	85.00	6.85						
62.23	100.00	0.00	0.818	78.15	7.41						
52.33	100.00	0.00	0.688	70.74	7.97						
44.00	100.00	0.00	0.578	62.77	9.05						
37.00	100.00	0.00	0.486	53.72	11.45						
31.11	100.00	0.00	0.409	42.27	16.67						
26.16	100.00	0.00	0.344	25.60	25.60						
22.00	100.00	0.00	0.289	0.00	0.00						
18.50	100.00	0.00	0.243	0.00	0.00						
15.56	100.00	0.00	0.204	0.00	0.00						
13.08	100.00	0.01	0.172	0.00	0.00						
11.00	99.99	0.01	0.145	0.00	0.00						

# Particle Size Analysis

S-107 SCLPS DUP

Date: 09/21/98 Meas #: 00049

Time: 13:20 Pres #: 01

S-107:SCLPS DUP 10 min  
40 ml/s, in 1.75 M NaOH/0.1 M NaNO3

### Summary

mv = 7.475  
mn = 0.815  
ma = 2.877  
cs = 2.085  
sd = 5.792

### Percentiles

10% = 1.206 60% = 6.529  
20% = 1.779 70% = 8.493  
30% = 2.604 80% = 11.42  
40% = 3.656 90% = 16.76  
50% = 4.968 95% = 22.53

### Dia Vol% Width

6.345 82% 11.94  
1.150 18% 0.700

%PASS

100.0

90.0

80.0

70.0

60.0

50.0

40.0

30.0

20.0

10.0

0.0

0.100

1.000

10.00

100.0

1000

- Size (microns) -

%CHAN

10.0

9.0

8.0

7.0

6.0

5.0

4.0

3.0

2.0

1.0

0.0

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	73.08	6.37						
592.0	100.00	0.00	7.778	66.71	6.64						
497.8	100.00	0.00	6.541	60.07	6.47						
418.6	100.00	0.00	5.500	53.60	6.03						
352.0	100.00	0.00	4.625	47.57	5.63						
296.0	100.00	0.00	3.889	41.94	5.34						
248.9	100.00	0.00	3.270	36.60	5.07						
209.3	100.00	0.00	2.750	31.53	4.74						
176.0	100.00	0.00	2.312	26.79	4.50						
148.0	100.00	0.00	1.945	22.29	4.47						
124.5	100.00	0.00	1.636	17.82	4.53						
104.7	100.00	0.00	1.375	13.29	4.27						
88.00	100.00	0.00	1.156	9.02	3.38						
74.00	100.00	0.00	0.972	5.64	2.22						
62.23	100.00	0.33	0.818	3.42	1.32						
52.33	99.67	0.42	0.688	2.10	0.80						
44.00	99.25	0.58	0.578	1.30	0.54						
37.00	98.67	0.83	0.486	0.76	0.42						
31.11	97.84	1.25	0.409	0.34	0.34						
26.16	96.59	1.89	0.344	0.00	0.00						
22.00	94.70	2.73	0.289	0.00	0.00						
18.50	91.97	3.63	0.243	0.00	0.00						
15.56	88.34	4.40	0.204	0.00	0.00						
13.08	83.94	5.09	0.172	0.00	0.00						
11.00	78.85	5.77	0.145	0.00	0.00						

# Particle Size Analysis

S-107 SCLPS DUP

Date: 09/21/98 Meas #: 00049

Time: 13:20 Pres #: 01

S-107:SCLPS DUP 10 min  
40 ml/s, in 1.75 M NaOH/0.1 M NaNO3

### Summary

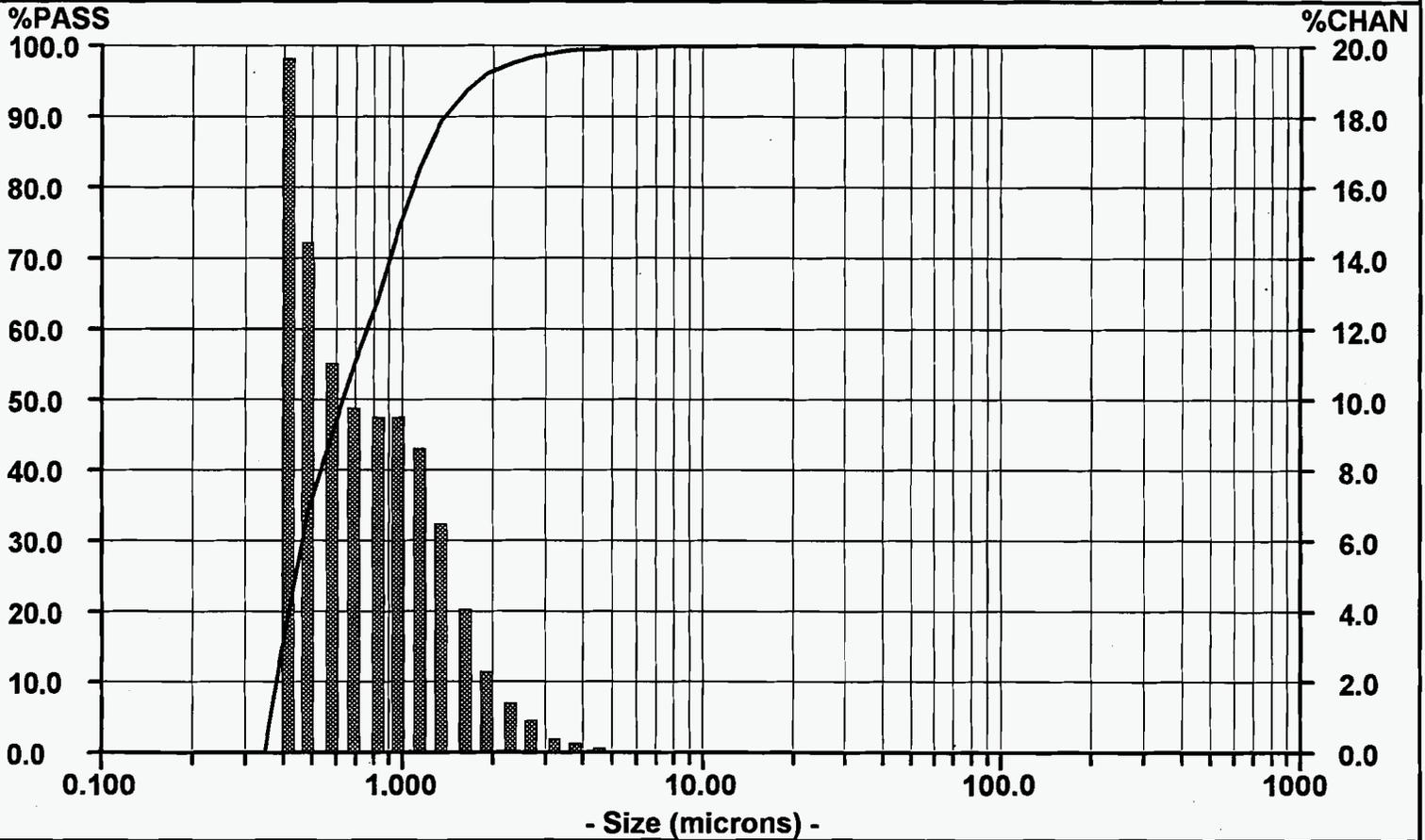
mv = 7.475  
mn = 0.815  
ma = 2.877  
cs = 2.085  
sd = 0.393

### Percentiles

10% = 0.379 60% = 0.750  
20% = 0.410 70% = 0.898  
30% = 0.459 80% = 1.084  
40% = 0.529 90% = 1.395  
50% = 0.626 95% = 1.773

### Dia Vol% Width

0.626 100% 0.785



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	99.97	0.03						
592.0	100.00	0.00	7.778	99.94	0.06						
497.8	100.00	0.00	6.541	99.88	0.09						
418.6	100.00	0.00	5.500	99.79	0.14						
352.0	100.00	0.00	4.625	99.65	0.23						
296.0	100.00	0.00	3.889	99.42	0.36						
248.9	100.00	0.00	3.270	99.06	0.58						
209.3	100.00	0.00	2.750	98.48	0.91						
176.0	100.00	0.00	2.312	97.57	1.45						
148.0	100.00	0.00	1.945	96.12	2.42						
124.5	100.00	0.00	1.635	93.70	4.12						
104.7	100.00	0.00	1.375	89.58	6.53						
88.00	100.00	0.00	1.156	83.05	8.70						
74.00	100.00	0.00	0.972	74.35	9.59						
62.23	100.00	0.00	0.818	64.76	9.59						
52.33	100.00	0.00	0.688	55.17	9.81						
44.00	100.00	0.00	0.578	45.36	11.12						
37.00	100.00	0.00	0.486	34.24	14.51						
31.11	100.00	0.00	0.409	19.73	19.73						
26.16	100.00	0.00	0.344	0.00	0.00						
22.00	100.00	0.00	0.289	0.00	0.00						
18.50	100.00	0.00	0.243	0.00	0.00						
15.66	100.00	0.00	0.204	0.00	0.00						
13.08	100.00	0.01	0.172	0.00	0.00						
11.00	99.99	0.02	0.145	0.00	0.00						

# Particle Size Analysis

S-107 SCLPS DUP

Date: 09/21/98 Meas #: 00051

Time: 13:30 Pres #: 01

S-107:SCLPS DUP 20 min  
60 ml/s, in 1.75 M NaOH/0.1 M NaNO3

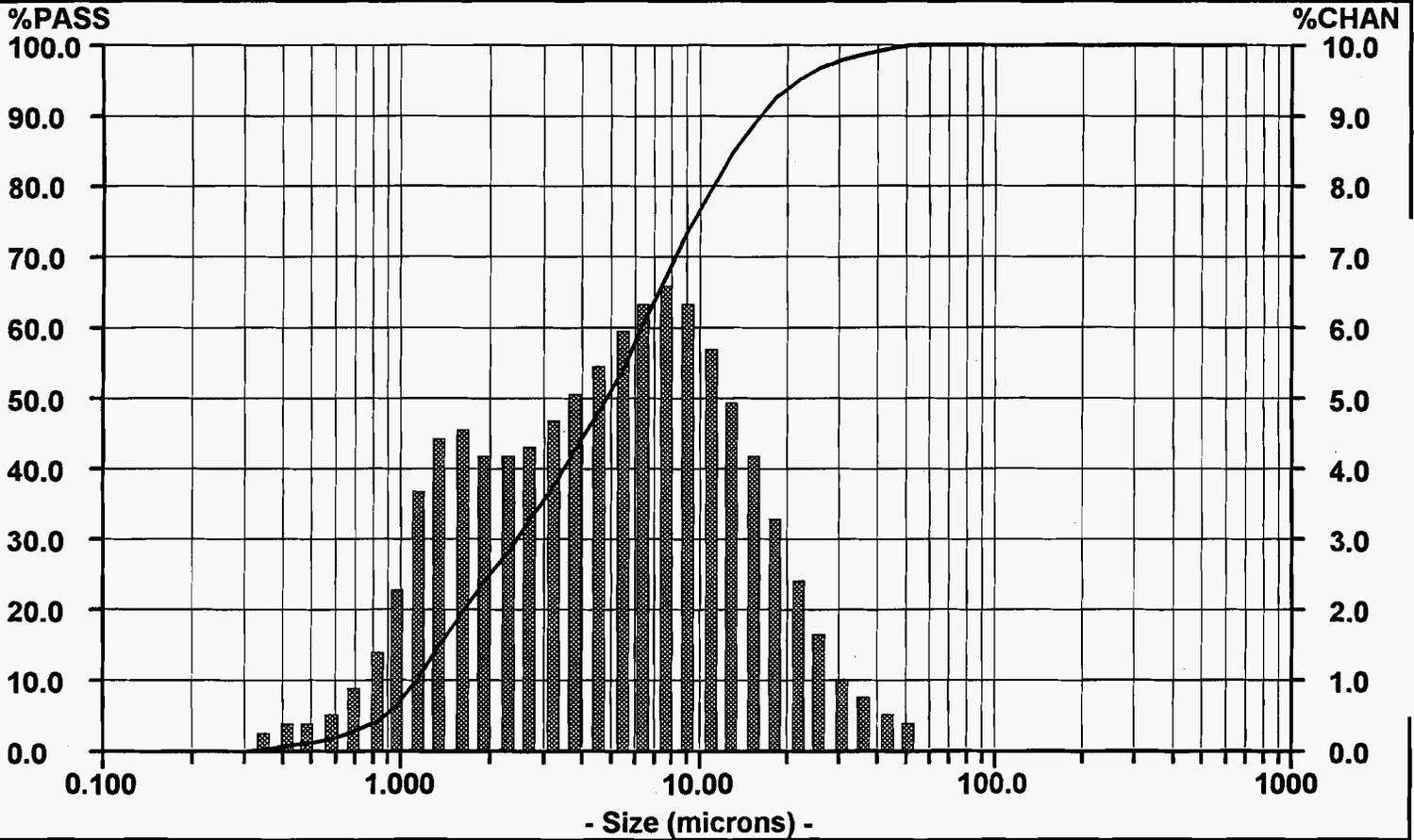
### Summary

mv = 7.197  
mn = 0.667  
ma = 2.677  
cs = 2.241  
sd = 5.633

### Percentiles

10% = 1.133 60% = 6.398  
20% = 1.653 70% = 8.298  
30% = 2.467 80% = 11.08  
40% = 3.545 90% = 16.16  
50% = 4.858 95% = 21.70

Dia	Vol%	Width
6.749	76%	11.60
1.229	24%	0.885



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	73.98	6.42						
592.0	100.00	0.00	7.778	67.56	6.71						
497.8	100.00	0.00	6.541	60.85	6.49						
418.6	100.00	0.00	5.500	54.36	6.01						
352.0	100.00	0.00	4.625	48.35	5.54						
296.0	100.00	0.00	3.889	42.81	5.17						
248.9	100.00	0.00	3.270	37.64	4.81						
209.3	100.00	0.00	2.750	32.83	4.45						
176.0	100.00	0.00	2.312	28.38	4.27						
148.0	100.00	0.00	1.945	24.11	4.38						
124.5	100.00	0.00	1.635	19.73	4.66						
104.7	100.00	0.00	1.375	15.07	4.58						
88.00	100.00	0.00	1.156	10.49	3.72						
74.00	100.00	0.00	0.972	6.77	2.48						
62.23	100.00	0.00	0.818	4.29	1.48						
52.33	100.00	0.54	0.688	2.81	0.91						
44.00	99.46	0.62	0.578	1.90	0.62						
37.00	98.84	0.80	0.486	1.28	0.47						
31.11	98.04	1.14	0.409	0.81	0.42						
26.16	96.90	1.72	0.344	0.39	0.39						
22.00	95.18	2.54	0.289	0.00	0.00						
18.50	92.64	3.47	0.243	0.00	0.00						
15.56	89.17	4.32	0.204	0.00	0.00						
13.08	84.85	5.07	0.172	0.00	0.00						
11.00	79.78	5.80	0.145	0.00	0.00						

# Particle Size Analysis

S-107 SCLPS DUP

Date: 09/21/98 Meas #: 00051  
Time: 13:30 Pres #: 01

SCLPS DUP 20 min  
in 1.75 M NaOH/0.1 M NaNO3

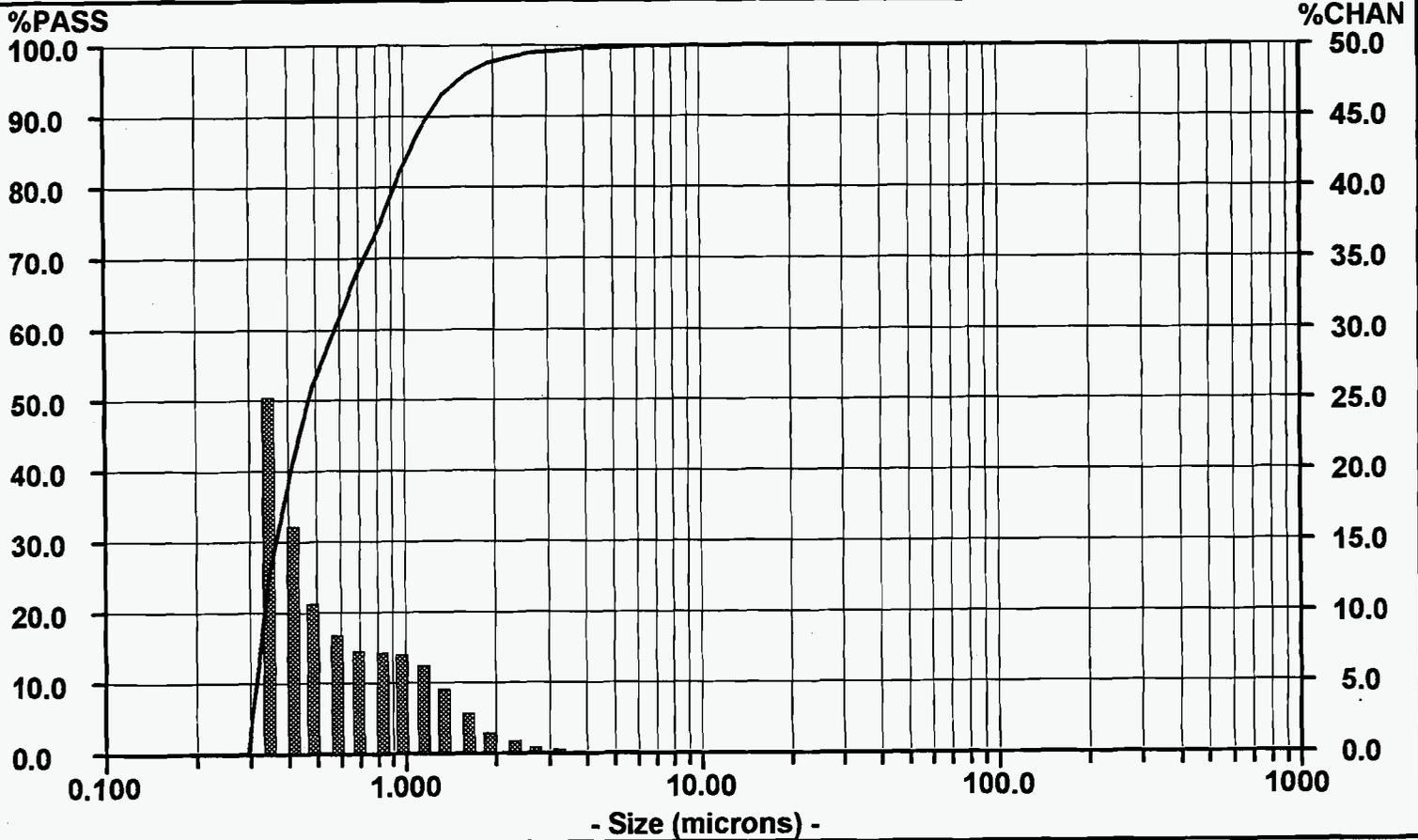
### Summary

mv = 7.197  
mn = 0.667  
ma = 2.677  
cs = 2.241  
sd = 0.347

### Percentiles

10% = 0.312 60% = 0.570  
20% = 0.332 70% = 0.722  
30% = 0.358 80% = 0.920  
40% = 0.401 90% = 1.212  
50% = 0.468 95% = 1.522

Dia Vol% Width  
0.468 100% 0.693



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	99.98	0.02						
592.0	100.00	0.00	7.778	99.96	0.04						
497.8	100.00	0.00	6.541	99.92	0.06						
418.6	100.00	0.00	5.500	99.86	0.09						
352.0	100.00	0.00	4.625	99.77	0.15						
296.0	100.00	0.00	3.889	99.62	0.23						
248.9	100.00	0.00	3.270	99.39	0.36						
209.3	100.00	0.00	2.750	99.03	0.56						
176.0	100.00	0.00	2.312	98.47	0.91						
148.0	100.00	0.00	1.945	97.56	1.57						
124.5	100.00	0.00	1.635	95.99	2.81						
104.7	100.00	0.00	1.375	93.18	4.63						
88.00	100.00	0.00	1.156	88.55	6.33						
74.00	100.00	0.00	0.972	82.22	7.09						
62.23	100.00	0.00	0.818	75.13	7.11						
52.33	100.00	0.00	0.688	68.02	7.38						
44.00	100.00	0.00	0.578	60.64	8.44						
37.00	100.00	0.00	0.486	52.20	10.74						
31.11	100.00	0.00	0.409	41.46	16.15						
26.16	100.00	0.00	0.344	25.31	25.31						
22.00	100.00	0.00	0.289	0.00	0.00						
18.50	100.00	0.00	0.243	0.00	0.00						
15.56	100.00	0.00	0.204	0.00	0.00						
13.08	100.00	0.01	0.172	0.00	0.00						
11.00	99.99	0.01	0.145	0.00	0.00						

# Particle Size Analysis

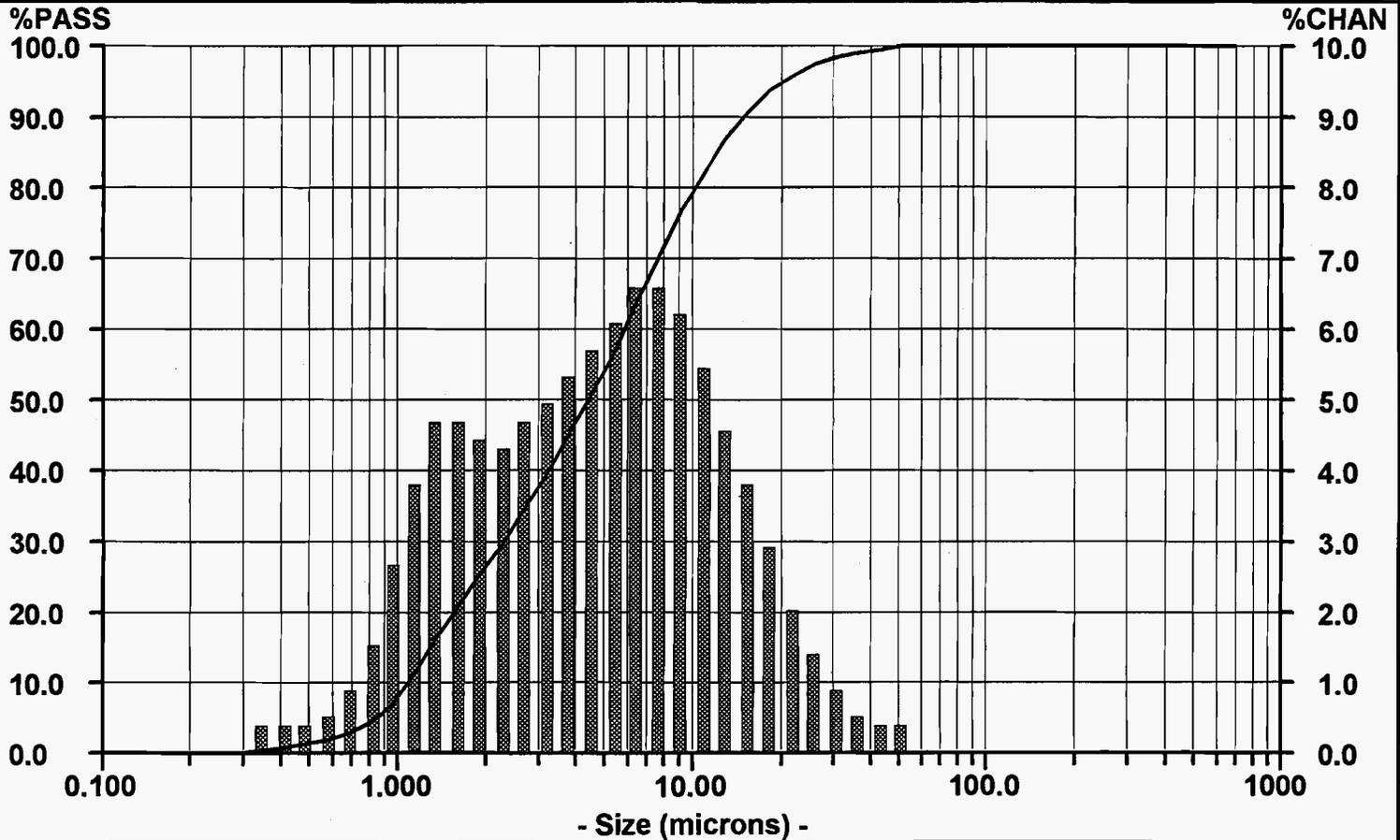
S-107 SCLPS DUP

Date: 09/21/98 Meas #: 00054

Time: 13:46 Pres #: 01

S-107:SCLPS DUP  
40 ml/s, in 1.75 M NaOH/0.1 M NaNO3  
Sonification #2 @40 W-90 sec

Summary	Percentiles		Dia	Vol%	Width
mv = 6.696	10% = 1.095	60% = 5.936	6.376	74%	10.68
mn = 0.665	20% = 1.578	70% = 7.688	1.218	26%	0.888
ma = 2.553	30% = 2.315	80% = 10.21			
cs = 2.350	40% = 3.296	90% = 14.95			
sd = 5.158	50% = 4.499	95% = 20.16			



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	76.77	6.32						
592.0	100.00	0.00	7.778	70.45	6.70						
497.8	100.00	0.00	6.541	63.75	6.62						
418.6	100.00	0.00	5.500	57.13	6.18						
352.0	100.00	0.00	4.625	50.95	5.76						
296.0	100.00	0.00	3.889	45.19	5.43						
248.9	100.00	0.00	3.270	39.76	5.09						
209.3	100.00	0.00	2.750	34.67	4.70						
176.0	100.00	0.00	2.312	29.97	4.46						
148.0	100.00	0.00	1.945	25.51	4.53						
124.5	100.00	0.00	1.635	20.98	4.82						
104.7	100.00	0.00	1.375	16.16	4.80						
88.00	100.00	0.00	1.156	11.36	3.99						
74.00	100.00	0.00	0.972	7.37	2.70						
62.23	100.00	0.00	0.818	4.67	1.63						
52.33	100.00	0.42	0.688	3.04	1.00						
44.00	99.58	0.50	0.578	2.04	0.67						
37.00	99.08	0.67	0.486	1.37	0.51						
31.11	98.41	0.96	0.409	0.86	0.45						
26.16	97.45	1.46	0.344	0.41	0.41						
22.00	95.99	2.17	0.289	0.00	0.00						
18.50	93.82	3.01	0.243	0.00	0.00						
15.56	90.81	3.84	0.204	0.00	0.00						
13.08	86.97	4.67	0.172	0.00	0.00						
11.00	82.30	5.53	0.145	0.00	0.00						

# Particle Size Analysis

S-107 SCLPS DUP

Date: 09/21/98 Meas #: 00054

Time: 13:46 Pres #: 01

S-107:SCLPS DUP

40 ml/s, in 1.75 M NaOH/0.1 M NaNO3

Sonification #2 @40 W-90 sec

### Summary

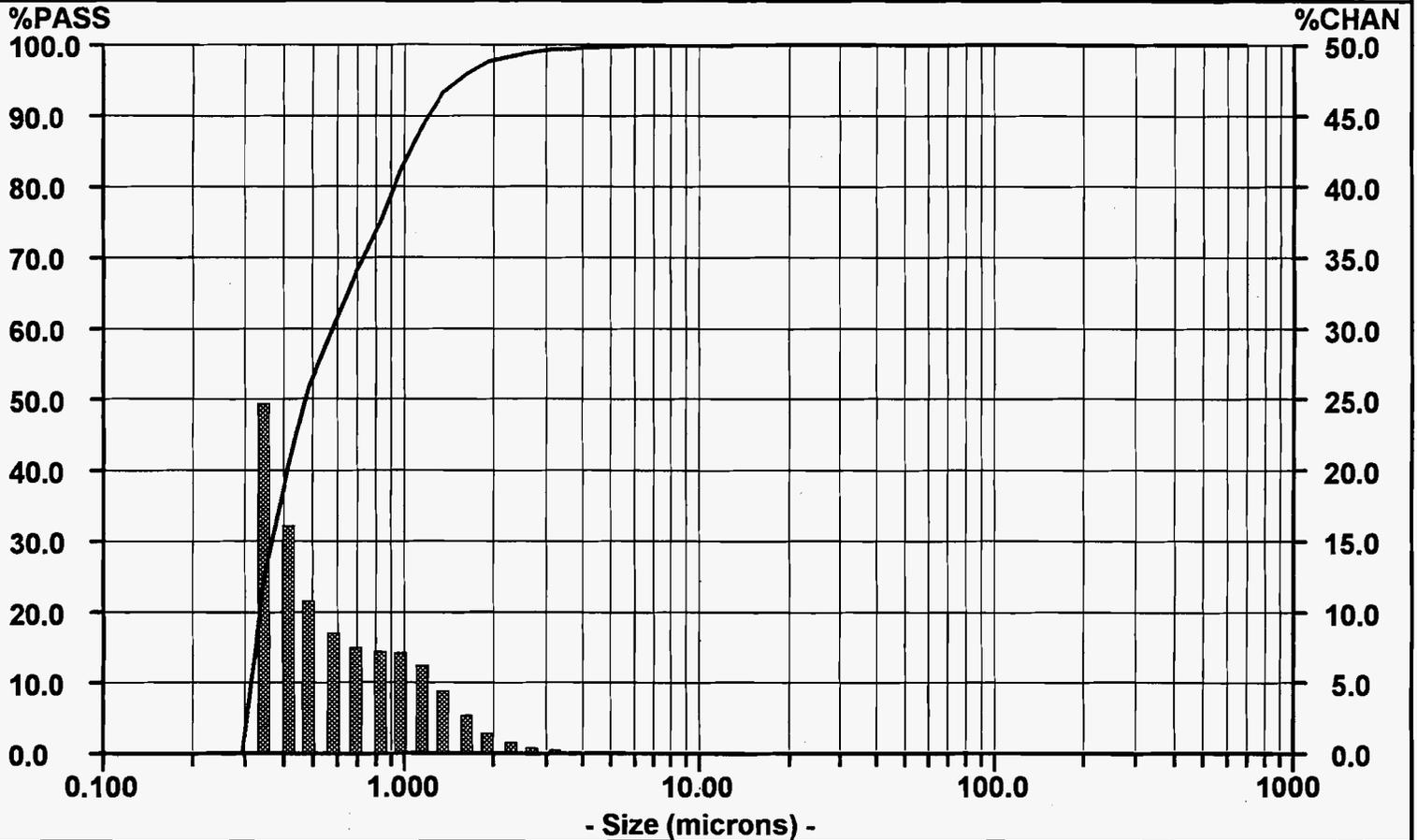
mv = 6.696  
 mn = 0.665  
 ma = 2.553  
 cs = 2.350  
 sd = 0.342

### Percentiles

10% = 0.313 60% = 0.573  
 20% = 0.333 70% = 0.721  
 30% = 0.360 80% = 0.915  
 40% = 0.404 90% = 1.201  
 50% = 0.470 95% = 1.506

### Dia Vol% Width

0.470 100% 0.685



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	99.98	0.02						
592.0	100.00	0.00	7.778	99.96	0.03						
497.8	100.00	0.00	6.541	99.93	0.06						
418.6	100.00	0.00	5.500	99.87	0.09						
352.0	100.00	0.00	4.625	99.78	0.14						
296.0	100.00	0.00	3.889	99.64	0.23						
248.9	100.00	0.00	3.270	99.41	0.36						
209.3	100.00	0.00	2.750	99.05	0.55						
176.0	100.00	0.00	2.312	98.50	0.89						
148.0	100.00	0.00	1.945	97.61	1.51						
124.5	100.00	0.00	1.635	96.10	2.71						
104.7	100.00	0.00	1.375	93.39	4.53						
88.00	100.00	0.00	1.156	88.86	6.34						
74.00	100.00	0.00	0.972	82.52	7.20						
62.23	100.00	0.00	0.818	75.32	7.32						
52.33	100.00	0.00	0.688	68.00	7.57						
44.00	100.00	0.00	0.578	60.43	8.52						
37.00	100.00	0.00	0.486	51.91	10.89						
31.11	100.00	0.00	0.409	41.02	16.16						
26.16	100.00	0.00	0.344	24.86	24.86						
22.00	100.00	0.00	0.289	0.00	0.00						
18.50	100.00	0.00	0.243	0.00	0.00						
15.56	100.00	0.00	0.204	0.00	0.00						
13.08	100.00	0.01	0.172	0.00	0.00						
11.00	99.99	0.01	0.145	0.00	0.00						

# Particle Size Analysis

S-107 TWPS2

Date: 09/22/98 Meas #: 00086  
Time: 10:13 Pres #: 01

S-107:TWPS2 10min  
40 ml/s, In 0.46 M NaOH/0.1MNaNO3

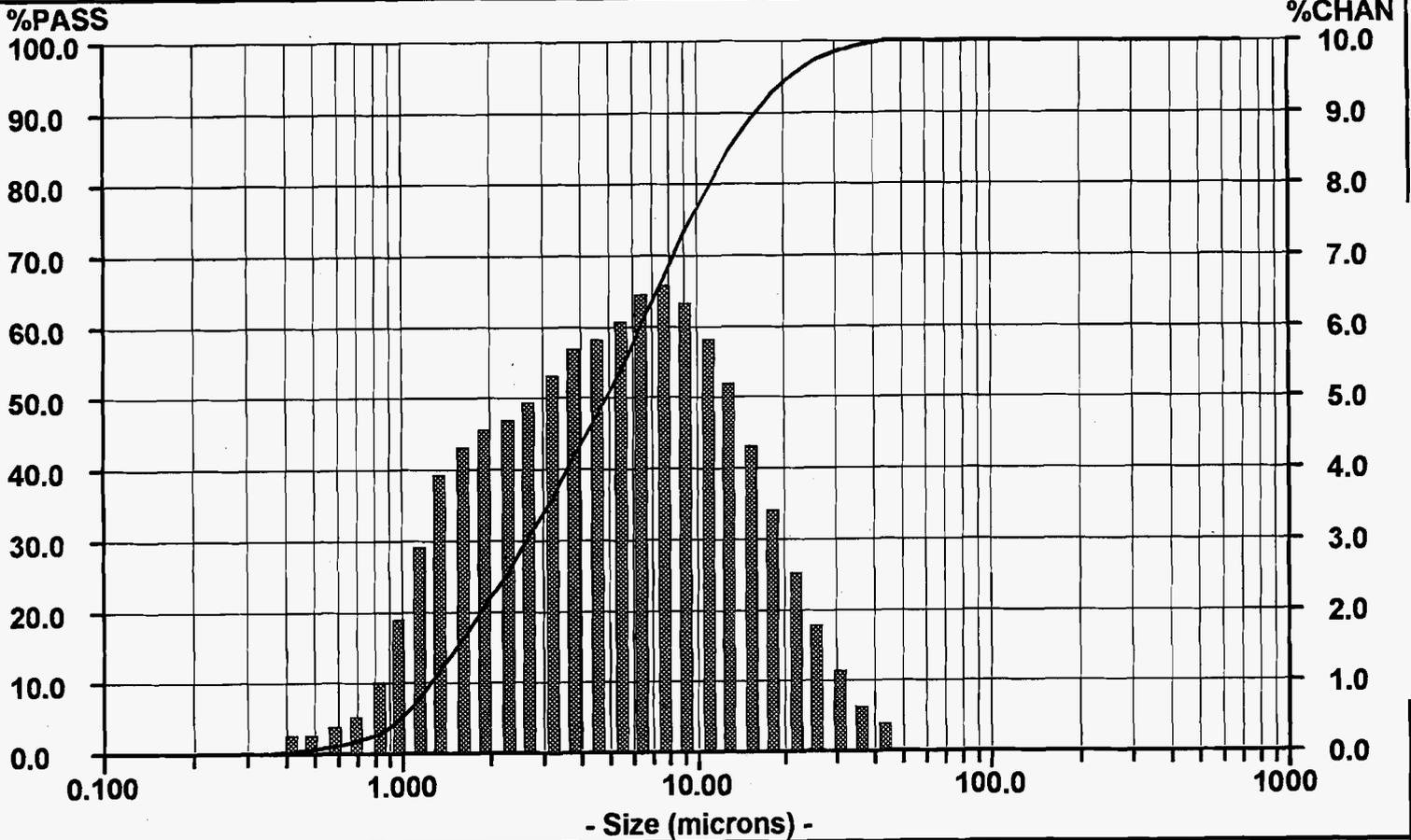
### Summary

mv = 7.103  
mn = 0.831  
ma = 2.958  
cs = 2.028  
sd = 5.522

### Percentiles

10% = 1.272 60% = 6.441  
20% = 1.873 70% = 8.347  
30% = 2.675 80% = 11.09  
40% = 3.664 90% = 16.00  
50% = 4.913 95% = 21.01

Dia	Vol%	Width
4.913	100%	11.04



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	73.81	6.49						
592.0	100.00	0.00	7.778	67.32	6.72						
497.8	100.00	0.00	6.541	60.60	6.53						
418.6	100.00	0.00	5.500	54.07	6.18						
352.0	100.00	0.00	4.625	47.89	5.90						
296.0	100.00	0.00	3.889	41.99	5.70						
248.9	100.00	0.00	3.270	36.29	5.45						
209.3	100.00	0.00	2.750	30.84	5.09						
176.0	100.00	0.00	2.312	25.75	4.75						
148.0	100.00	0.00	1.945	21.00	4.60						
124.5	100.00	0.00	1.635	16.40	4.49						
104.7	100.00	0.00	1.375	11.91	4.02						
88.00	100.00	0.00	1.156	7.89	3.02						
74.00	100.00	0.00	0.972	4.87	1.90						
62.23	100.00	0.00	0.818	2.97	1.11						
52.33	100.00	0.00	0.688	1.86	0.68						
44.00	100.00	0.49	0.578	1.18	0.47						
37.00	99.51	0.79	0.486	0.71	0.38						
31.11	98.72	1.23	0.409	0.33	0.33						
26.16	97.49	1.86	0.344	0.00	0.00						
22.00	95.63	2.67	0.289	0.00	0.00						
18.50	92.96	3.58	0.243	0.00	0.00						
15.56	89.38	4.43	0.204	0.00	0.00						
13.08	84.95	5.21	0.172	0.00	0.00						
11.00	79.74	5.93	0.145	0.00	0.00						

# Particle Size Analysis

S-107 TWPS2

Date: 09/22/98 Meas #: 00086

Time: 10:13 Pres #: 01

S-107:TWPS2 10 min  
40 ml/s, in 0.46 M NaOH/0.1MNaNO3

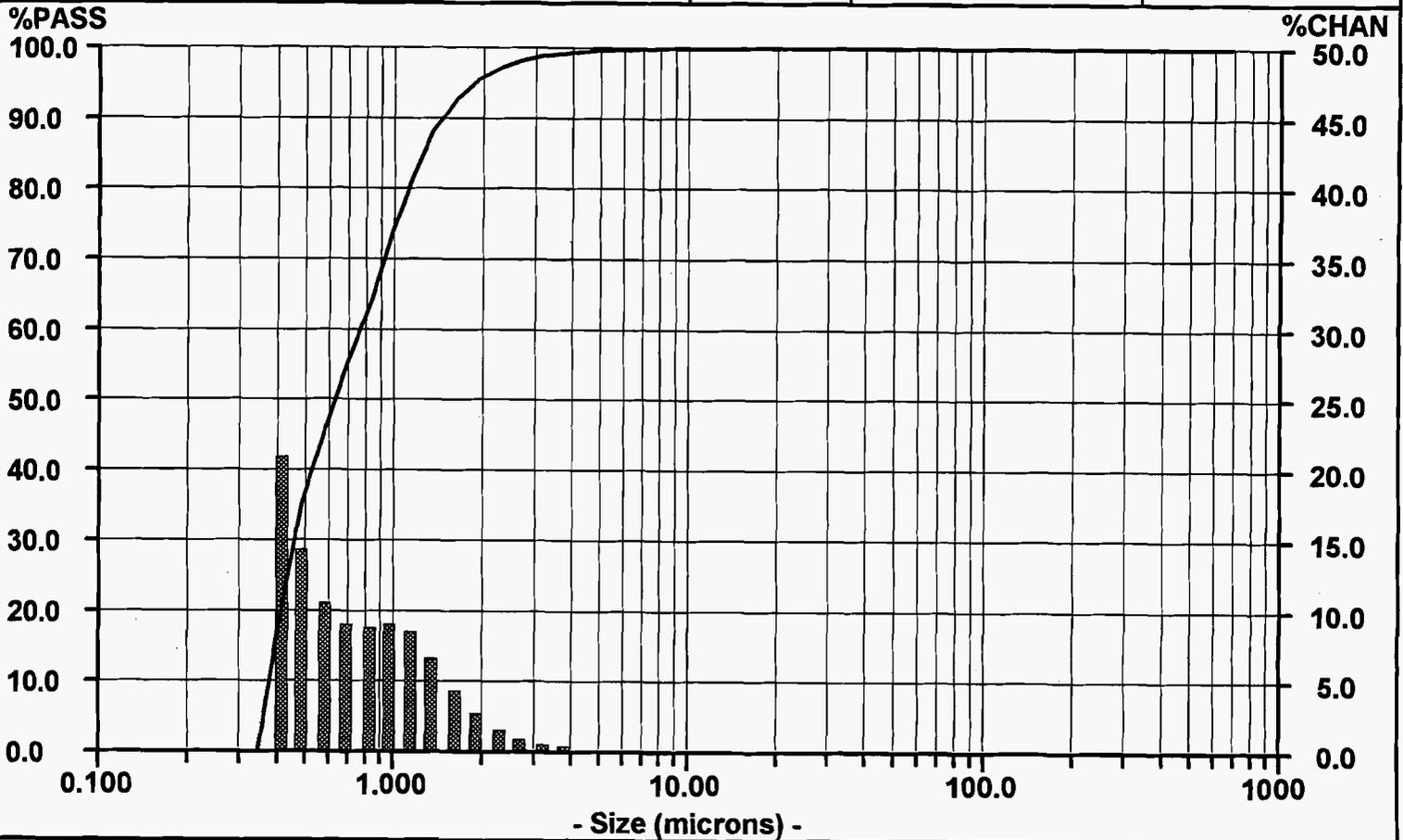
### Summary

mv = 7.103  
mn = 0.831  
ma = 2.958  
cs = 2.028  
sd = 0.416

### Percentiles

10% = 0.377 60% = 0.756  
20% = 0.406 70% = 0.917  
30% = 0.451 80% = 1.118  
40% = 0.521 90% = 1.457  
50% = 0.622 95% = 1.871

Dia	Vol%	Width
1.064	45%	0.849
0.438	55%	0.208



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	99.96	0.04						
592.0	100.00	0.00	7.778	99.92	0.06						
497.8	100.00	0.00	6.541	99.86	0.10						
418.6	100.00	0.00	5.500	99.76	0.16						
352.0	100.00	0.00	4.625	99.60	0.26						
296.0	100.00	0.00	3.889	99.34	0.42						
248.9	100.00	0.00	3.270	98.92	0.68						
209.3	100.00	0.00	2.750	98.24	1.07						
176.0	100.00	0.00	2.312	97.17	1.67						
148.0	100.00	0.00	1.945	95.50	2.73						
124.5	100.00	0.00	1.635	92.77	4.48						
104.7	100.00	0.00	1.375	88.29	6.74						
88.00	100.00	0.00	1.156	81.55	8.52						
74.00	100.00	0.00	0.972	73.03	9.00						
62.23	100.00	0.00	0.818	64.03	8.84						
52.33	100.00	0.00	0.688	55.19	9.14						
44.00	100.00	0.00	0.578	46.05	10.61						
37.00	100.00	0.00	0.486	35.44	14.40						
31.11	100.00	0.00	0.409	21.04	21.04						
26.16	100.00	0.00	0.344	0.00	0.00						
22.00	100.00	0.00	0.289	0.00	0.00						
18.50	100.00	0.00	0.243	0.00	0.00						
15.56	100.00	0.01	0.204	0.00	0.00						
13.08	99.99	0.01	0.172	0.00	0.00						
11.00	99.98	0.02	0.145	0.00	0.00						

# Particle Size Analysis

S-107 TWPS2

Date: 09/22/98 Meas #: 00087

Time: 10:19 Pres #: 01

S-107:TWPS2 20 min  
 40 mts, In 0.46 M NaOH/0.1MNaNO3  
 60 ml/s

**Summary**

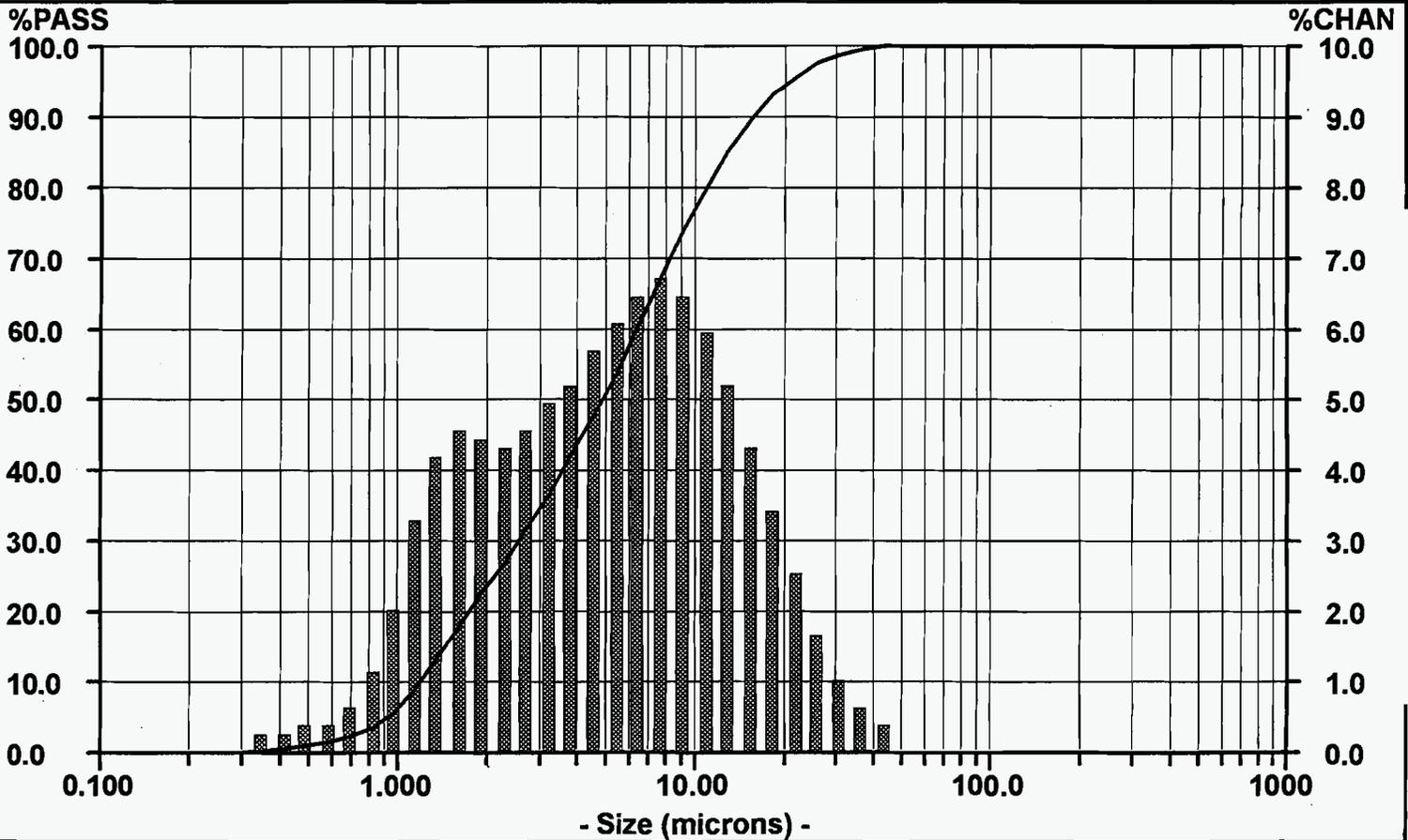
mv = 6.999  
 mn = 0.676  
 ma = 2.785  
 cs = 2.154  
 sd = 5.495

**Percentiles**

10% = 1.199 60% = 6.432  
 20% = 1.758 70% = 8.305  
 30% = 2.579 80% = 10.98  
 40% = 3.630 90% = 15.74  
 50% = 4.910 95% = 20.60

**Dia Vol% Width**

6.652 77% 11.18  
 1.265 23% 0.882



- Size (microns) -

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	74.05	6.57						
592.0	100.00	0.00	7.778	67.48	6.82						
497.8	100.00	0.00	6.541	60.66	6.58						
418.6	100.00	0.00	5.500	54.08	6.15						
352.0	100.00	0.00	4.625	47.93	5.74						
296.0	100.00	0.00	3.889	42.19	5.39						
248.9	100.00	0.00	3.270	36.80	5.03						
209.3	100.00	0.00	2.750	31.77	4.68						
176.0	100.00	0.00	2.312	27.09	4.47						
148.0	100.00	0.00	1.945	22.62	4.52						
124.5	100.00	0.00	1.635	18.10	4.63						
104.7	100.00	0.00	1.375	13.47	4.32						
88.00	100.00	0.00	1.156	9.15	3.33						
74.00	100.00	0.00	0.972	5.82	2.12						
62.23	100.00	0.00	0.818	3.70	1.24						
52.33	100.00	0.00	0.688	2.46	0.76						
44.00	100.00	0.44	0.578	1.70	0.53						
37.00	99.56	0.73	0.486	1.17	0.42						
31.11	98.83	1.16	0.409	0.75	0.38						
26.16	97.67	1.78	0.344	0.37	0.37						
22.00	95.89	2.60	0.289	0.00	0.00						
18.50	93.29	3.54	0.243	0.00	0.00						
15.56	89.75	4.44	0.204	0.00	0.00						
13.08	85.31	5.26	0.172	0.00	0.00						
11.00	80.05	6.00	0.145	0.00	0.00						

# Particle Size Analysis

S-107 TWPS2

Date: 09/22/98 Meas #: 00087  
Time: 10:19 Pres #: 01

S-107:TWPS2 26 min  
40 ml/s, In 0.46 M NaOH/0.1MNaNO3

*Irregular 60ml/s*

### Summary

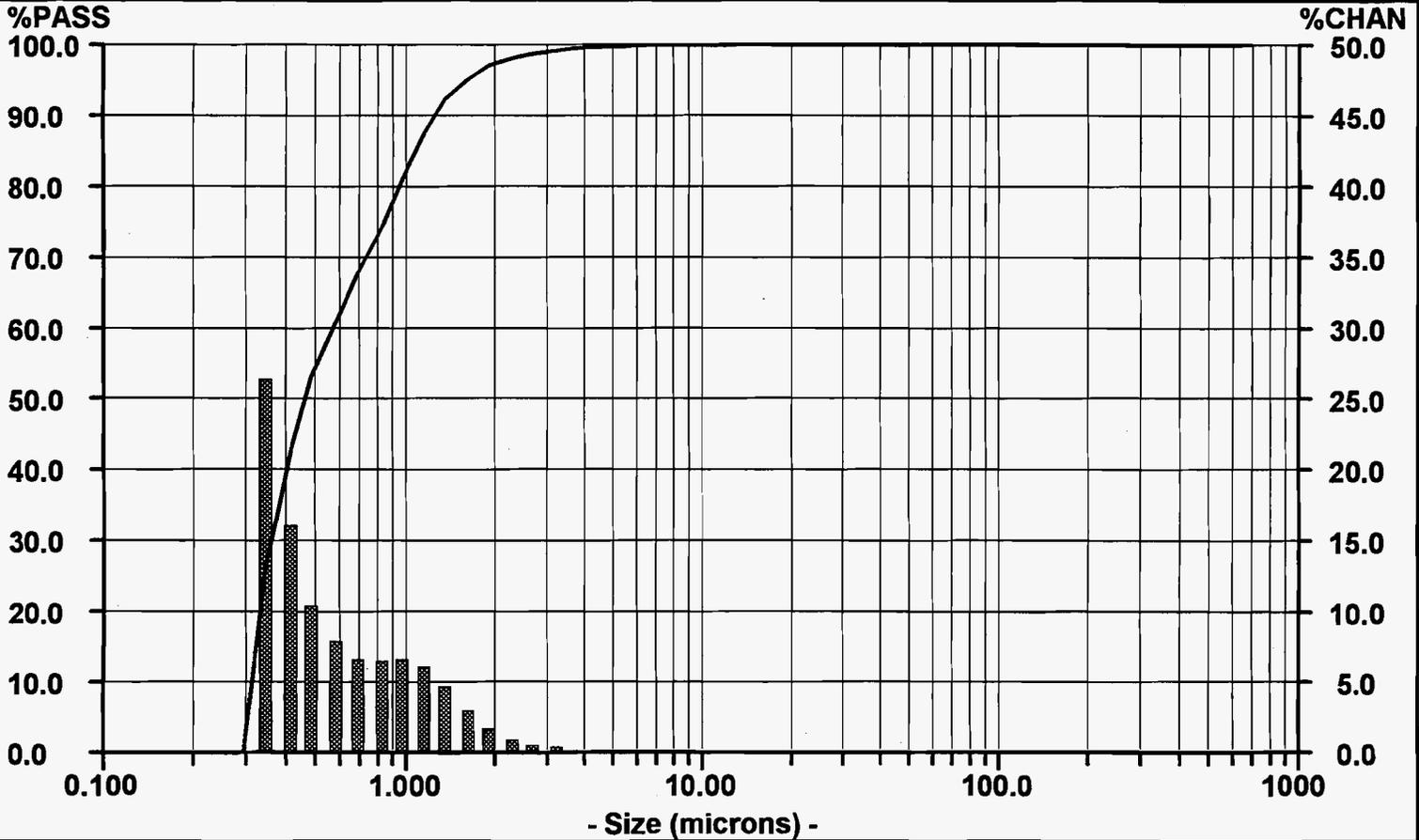
mv = 6.999  
mn = 0.676  
ma = 2.786  
cs = 2.154  
sd = 0.362

### Percentiles

10% = 0.311 60% = 0.563  
20% = 0.330 70% = 0.725  
30% = 0.354 80% = 0.941  
40% = 0.394 90% = 1.256  
50% = 0.459 95% = 1.592

### Dia Vol% Width

1.046 32% 0.791  
0.368 68% 0.214



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	99.98	0.02						
592.0	100.00	0.00	7.778	99.96	0.04						
497.8	100.00	0.00	6.641	99.92	0.07						
418.6	100.00	0.00	5.500	99.85	0.11						
352.0	100.00	0.00	4.625	99.74	0.17						
296.0	100.00	0.00	3.889	99.57	0.27						
248.9	100.00	0.00	3.270	99.30	0.42						
209.3	100.00	0.00	2.750	98.88	0.65						
176.0	100.00	0.00	2.312	98.23	1.05						
148.0	100.00	0.00	1.945	97.18	1.78						
124.5	100.00	0.00	1.635	95.40	3.08						
104.7	100.00	0.00	1.375	92.32	4.82						
88.00	100.00	0.00	1.156	87.50	6.26						
74.00	100.00	0.00	0.972	81.24	6.68						
62.23	100.00	0.00	0.818	74.56	6.58						
52.33	100.00	0.00	0.688	67.98	6.80						
44.00	100.00	0.00	0.578	61.18	7.97						
37.00	100.00	0.00	0.486	53.21	10.59						
31.11	100.00	0.00	0.409	42.62	16.13						
26.16	100.00	0.00	0.344	26.49	26.49						
22.00	100.00	0.00	0.289	0.00	0.00						
18.50	100.00	0.00	0.243	0.00	0.00						
15.56	100.00	0.00	0.204	0.00	0.00						
13.08	100.00	0.01	0.172	0.00	0.00						
11.00	99.99	0.01	0.145	0.00	0.00						

# Particle Size Analysis

S-107 TWPS2

Date: 09/22/98 Meas #: 00089

Time: 10:34 Pres #: 01

S-107:TWPS2

40 ml/s, In 0.46 M NaOH/0.1MNaNO3

Sonication #2 @40W

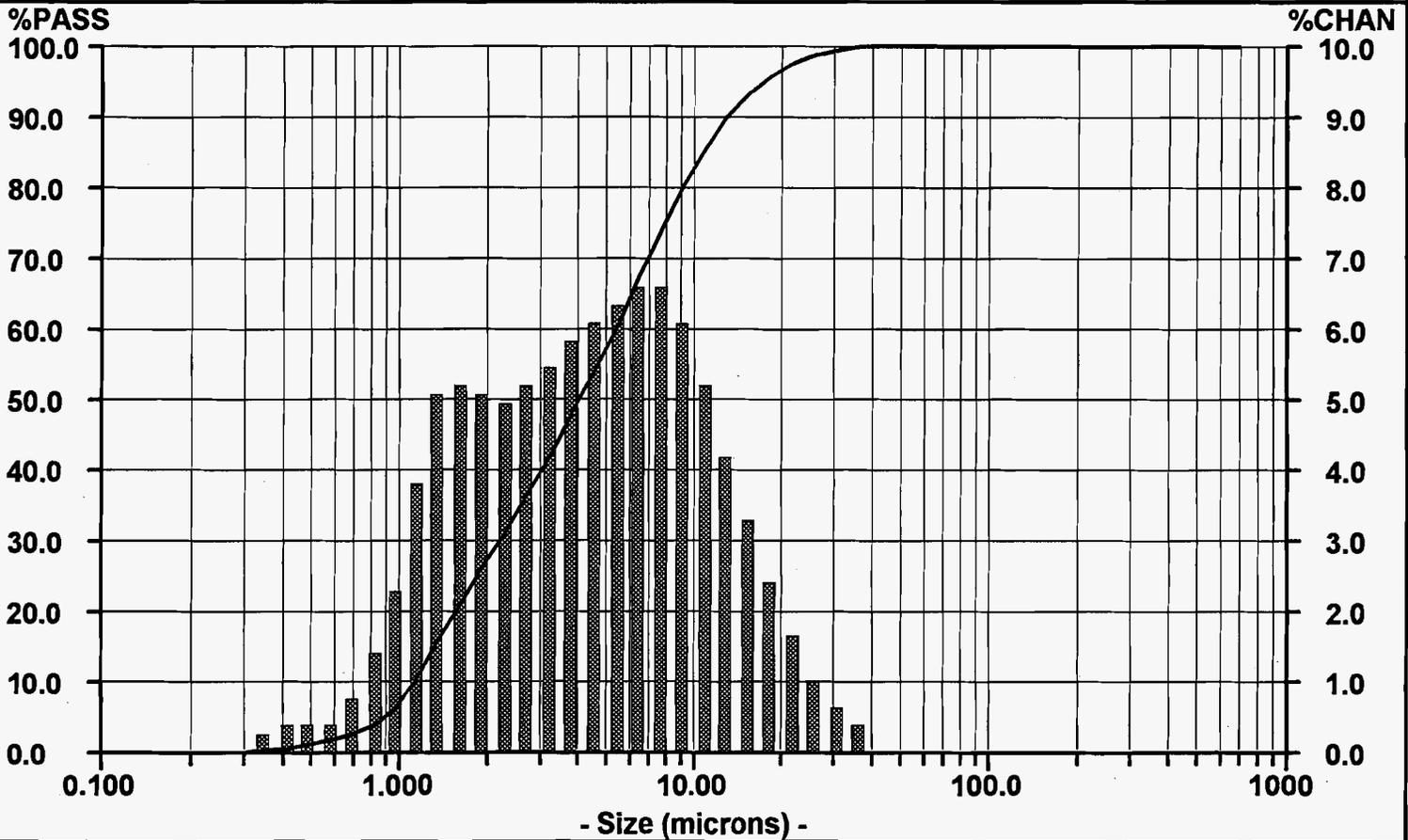
### Summary

mv = 5.903  
 mn = 0.691  
 ma = 2.524  
 cs = 2.377  
 sd = 4.506

### Percentiles

10% = 1.133 60% = 5.409  
 20% = 1.582 70% = 6.997  
 30% = 2.217 80% = 9.176  
 40% = 3.072 90% = 13.10  
 50% = 4.120 95% = 17.29

Dia	Vol%	Width
5.862	74%	9.250
1.266	26%	0.863



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	80.28	6.20						
592.0	100.00	0.00	7.778	74.08	6.71						
497.8	100.00	0.00	6.541	67.37	6.73						
418.6	100.00	0.00	5.500	60.64	6.48						
352.0	100.00	0.00	4.625	54.16	6.18						
296.0	100.00	0.00	3.889	47.98	5.91						
248.9	100.00	0.00	3.270	42.07	5.59						
209.3	100.00	0.00	2.750	36.48	5.25						
176.0	100.00	0.00	2.312	31.23	5.05						
148.0	100.00	0.00	1.945	26.18	5.16						
124.5	100.00	0.00	1.635	21.02	5.39						
104.7	100.00	0.00	1.375	15.63	5.11						
88.00	100.00	0.00	1.156	10.52	3.95						
74.00	100.00	0.00	0.972	6.57	2.49						
62.23	100.00	0.00	0.818	4.08	1.42						
52.33	100.00	0.00	0.688	2.66	0.85						
44.00	100.00	0.00	0.578	1.81	0.57						
37.00	100.00	0.45	0.486	1.24	0.45						
31.11	99.55	0.73	0.409	0.79	0.41						
26.16	98.82	1.16	0.344	0.38	0.38						
22.00	97.66	1.76	0.289	0.00	0.00						
18.50	95.90	2.53	0.243	0.00	0.00						
15.56	93.37	3.40	0.204	0.00	0.00						
13.08	89.97	4.35	0.172	0.00	0.00						
11.00	85.62	5.34	0.145	0.00	0.00						

# Particle Size Analysis

S-107 TWPS2

Date: 09/22/98 Meas #: 00089  
Time: 10:34 Pres #: 01

S-107:TWPS2

40 ml/s, In 0.46 M NaOH/0.1MNaNO3  
Sonication #2 @40W

### Summary

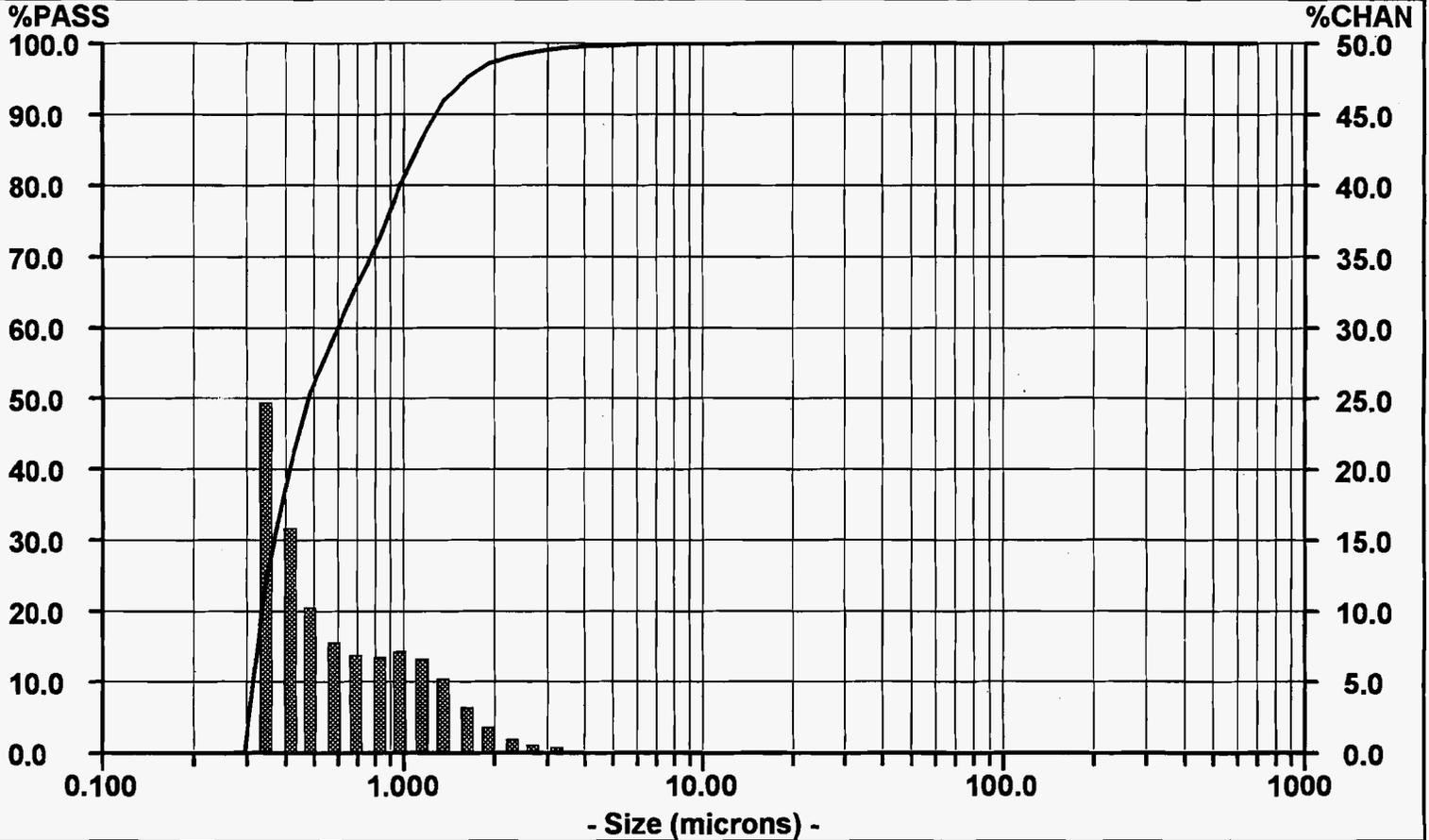
mv = 5.903  
mn = 0.691  
ma = 2.524  
cs = 2.377  
sd = 0.374

### Percentiles

10% = 0.313 60% = 0.593  
20% = 0.333 70% = 0.762  
30% = 0.360 80% = 0.972  
40% = 0.405 90% = 1.277  
50% = 0.476 95% = 1.606

### Dia Vol% Width

1.046 34% 0.771  
0.371 66% 0.218



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	99.98	0.02						
592.0	100.00	0.00	7.778	99.96	0.04						
497.8	100.00	0.00	6.541	99.92	0.06						
418.6	100.00	0.00	5.500	99.86	0.10						
352.0	100.00	0.00	4.625	99.76	0.17						
296.0	100.00	0.00	3.889	99.59	0.27						
248.9	100.00	0.00	3.270	99.32	0.42						
209.3	100.00	0.00	2.750	98.90	0.67						
176.0	100.00	0.00	2.312	98.23	1.08						
148.0	100.00	0.00	1.945	97.15	1.86						
124.5	100.00	0.00	1.635	95.29	3.27						
104.7	100.00	0.00	1.375	92.02	5.21						
88.00	100.00	0.00	1.156	86.81	6.79						
74.00	100.00	0.00	0.972	80.02	7.18						
62.23	100.00	0.00	0.818	72.84	6.89						
52.33	100.00	0.00	0.688	65.95	6.95						
44.00	100.00	0.00	0.578	59.00	7.83						
37.00	100.00	0.00	0.486	51.17	10.38						
31.11	100.00	0.00	0.409	40.79	15.91						
26.16	100.00	0.00	0.344	24.88	24.88						
22.00	100.00	0.00	0.289	0.00	0.00						
18.50	100.00	0.00	0.243	0.00	0.00						
15.56	100.00	0.00	0.204	0.00	0.00						
13.08	100.00	0.01	0.172	0.00	0.00						
11.00	99.99	0.01	0.145	0.00	0.00						

# Particle Size Analysis

S-107 IECPS DUP

Date: 09/21/98 Meas #: 00064

Time: 15:14 Pres #: 01

S-107:IECPS DUP 10 min  
40 ml/s, in 1.96 M NaOH/0.1 M NaNO3

**Summary**

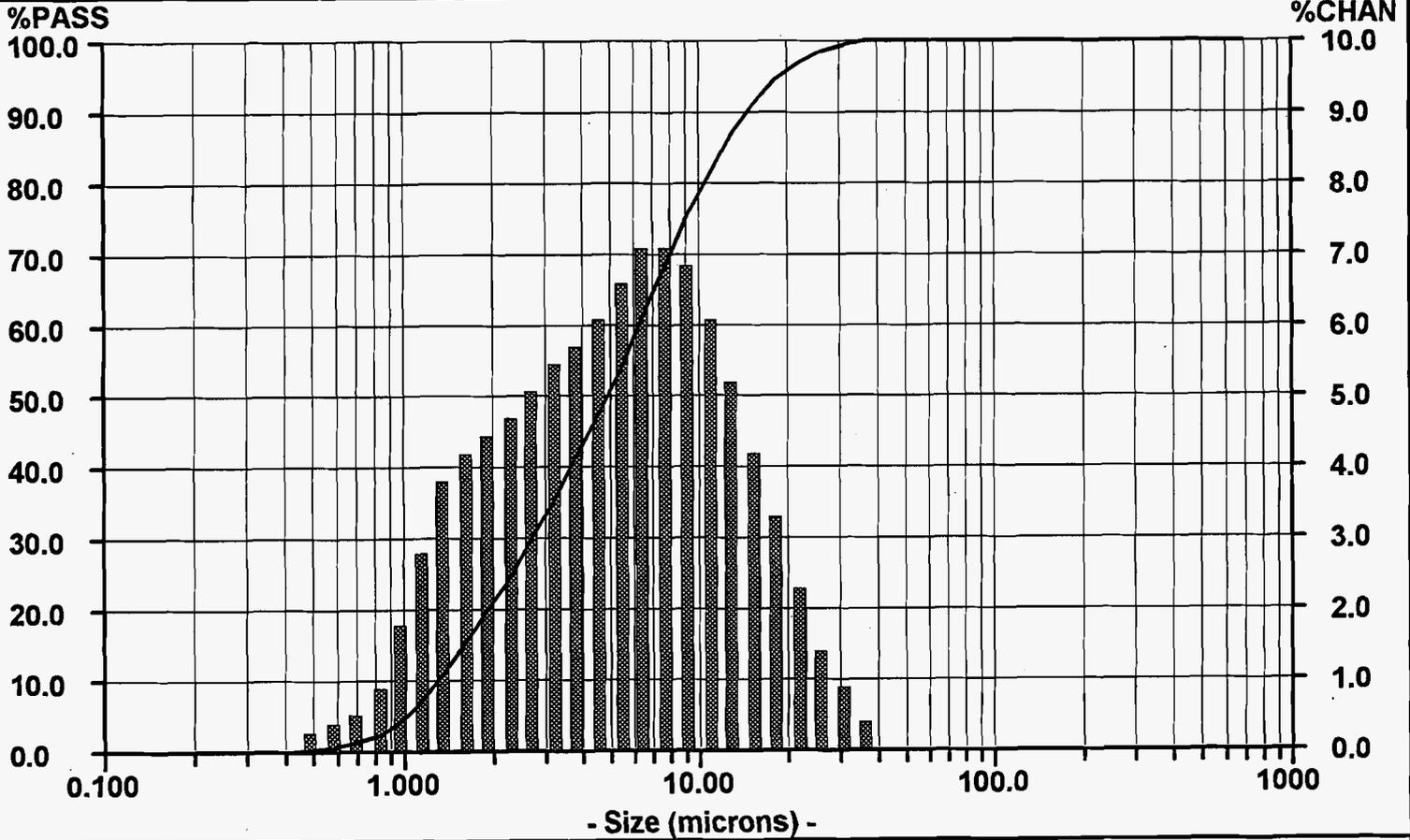
mv = 6.710  
mn = 0.986  
ma = 3.060  
cs = 1.961  
sd = 5.085

**Percentiles**

10% = 1.311 60% = 6.330  
20% = 1.939 70% = 8.040  
30% = 2.749 80% = 10.46  
40% = 3.725 90% = 14.72  
50% = 4.926 95% = 18.88

**Dia Vol% Width**

4.926 100% 10.17



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	75.56	6.91						
592.0	100.00	0.00	7.778	68.65	7.28						
497.8	100.00	0.00	6.541	61.37	7.10						
418.6	100.00	0.00	5.500	54.27	6.61						
352.0	100.00	0.00	4.625	47.66	6.17						
296.0	100.00	0.00	3.889	41.49	5.89						
248.9	100.00	0.00	3.270	35.60	5.59						
209.3	100.00	0.00	2.750	30.01	5.17						
176.0	100.00	0.00	2.312	24.84	4.76						
148.0	100.00	0.00	1.945	20.08	4.53						
124.5	100.00	0.00	1.635	15.55	4.39						
104.7	100.00	0.00	1.375	11.16	3.94						
88.00	100.00	0.00	1.156	7.22	2.97						
74.00	100.00	0.00	0.972	4.25	1.85						
62.23	100.00	0.00	0.818	2.40	1.05						
52.33	100.00	0.00	0.688	1.35	0.62						
44.00	100.00	0.00	0.578	0.73	0.42						
37.00	100.00	0.52	0.486	0.31	0.31						
31.11	99.48	0.90	0.409	0.00	0.00						
26.16	98.58	1.52	0.344	0.00	0.00						
22.00	97.06	2.38	0.289	0.00	0.00						
18.50	94.68	3.38	0.243	0.00	0.00						
15.56	91.30	4.35	0.204	0.00	0.00						
13.08	86.95	5.25	0.172	0.00	0.00						
11.00	81.70	6.14	0.145	0.00	0.00						

# Particle Size Analysis

S-107 IECPS DUP

Date: 09/21/98 Meas #: 00064  
Time: 15:14 Pres #: 01

S-107:IECPS DUP 10 min  
40 ml/s, in 1.96 M NaOH/0.1 M NaNO3

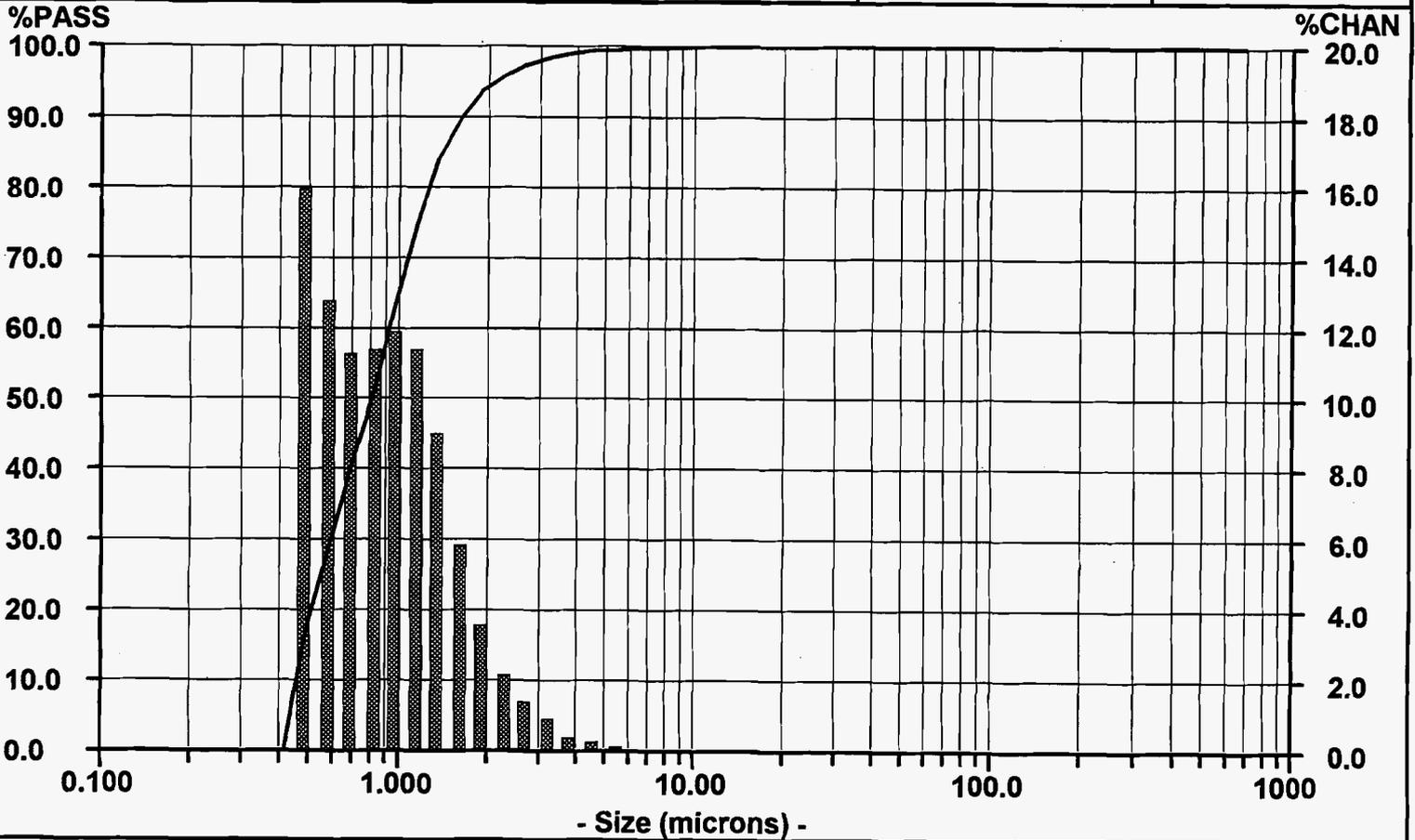
### Summary

mv = 6.710  
mn = 0.986  
ma = 3.060  
cs = 1.961  
sd = 0.443

### Percentiles

10% = 0.459 60% = 0.922  
20% = 0.510 70% = 1.067  
30% = 0.587 80% = 1.262  
40% = 0.685 90% = 1.631  
50% = 0.797 95% = 2.120

Dia	Vol%	Width
0.984	71%	0.868
0.479	29%	0.106



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.260	99.95	0.05						
592.0	100.00	0.00	7.778	99.90	0.09						
497.8	100.00	0.00	6.541	99.81	0.16						
418.6	100.00	0.00	5.500	99.66	0.24						
352.0	100.00	0.00	4.625	99.42	0.37						
296.0	100.00	0.00	3.889	99.05	0.60						
248.9	100.00	0.00	3.270	98.45	0.95						
209.3	100.00	0.00	2.750	97.50	1.48						
176.0	100.00	0.00	2.312	96.02	2.29						
148.0	100.00	0.00	1.945	93.73	3.66						
124.5	100.00	0.00	1.635	90.07	5.97						
104.7	100.00	0.00	1.375	84.10	9.01						
88.00	100.00	0.00	1.156	75.09	11.43						
74.00	100.00	0.00	0.972	63.66	11.95						
62.23	100.00	0.00	0.818	51.71	11.41						
52.33	100.00	0.00	0.688	40.30	11.36						
44.00	100.00	0.00	0.578	28.94	12.93						
37.00	100.00	0.00	0.486	16.01	16.01						
31.11	100.00	0.00	0.409	0.00	0.00						
26.16	100.00	0.00	0.344	0.00	0.00						
22.00	100.00	0.00	0.289	0.00	0.00						
18.50	100.00	0.00	0.243	0.00	0.00						
15.56	100.00	0.01	0.204	0.00	0.00						
13.08	99.99	0.01	0.172	0.00	0.00						
11.00	99.98	0.03	0.145	0.00	0.00						

# Particle Size Analysis

S-107 IECPS DUP

Date: 09/21/98 Meas #: 00067

Time: 15:24 Pres #: 01

S-107:IECPS DUP *20 min*  
60 ml/s, in 1.96 M NaOH/0.1 M NaNO3

**Summary**

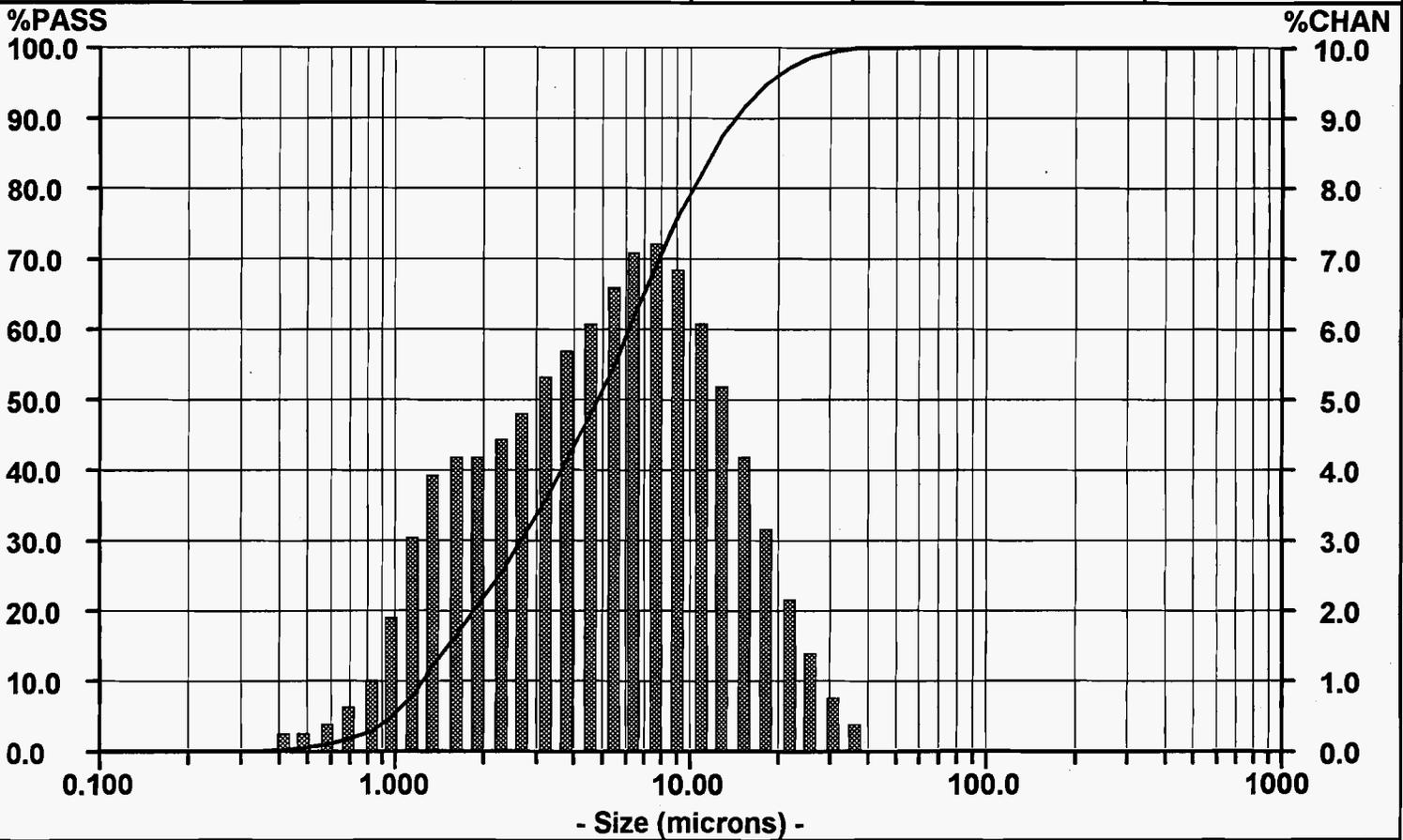
mv = 6.583  
mn = 0.837  
ma = 2.927  
cs = 2.050  
sd = 5.000

**Percentiles**

10% = 1.254 60% = 6.255  
20% = 1.871 70% = 7.932  
30% = 2.704 80% = 10.28  
40% = 3.693 90% = 14.39  
50% = 4.877 95% = 18.41

**Dia Vol% Width**

4.877 100% 10.00



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	76.16	6.96						
592.0	100.00	0.00	7.778	69.20	7.32						
497.8	100.00	0.00	6.541	61.88	7.18						
418.6	100.00	0.00	5.500	54.70	6.69						
352.0	100.00	0.00	4.625	48.01	6.22						
296.0	100.00	0.00	3.889	41.79	5.85						
248.9	100.00	0.00	3.270	35.94	5.44						
209.3	100.00	0.00	2.750	30.50	4.96						
176.0	100.00	0.00	2.312	25.54	4.55						
148.0	100.00	0.00	1.945	20.99	4.39						
124.5	100.00	0.00	1.635	16.60	4.37						
104.7	100.00	0.00	1.375	12.23	4.04						
88.00	100.00	0.00	1.156	8.19	3.13						
74.00	100.00	0.00	0.972	5.06	2.01						
62.23	100.00	0.00	0.818	3.05	1.18						
52.33	100.00	0.00	0.688	1.87	0.71						
44.00	100.00	0.00	0.578	1.16	0.48						
37.00	100.00	0.45	0.486	0.68	0.37						
31.11	99.55	0.81	0.409	0.31	0.31						
26.16	98.74	1.41	0.344	0.00	0.00						
22.00	97.33	2.25	0.289	0.00	0.00						
18.50	95.08	3.26	0.243	0.00	0.00						
15.56	91.82	4.27	0.204	0.00	0.00						
13.08	87.55	5.23	0.172	0.00	0.00						
11.00	82.32	6.16	0.145	0.00	0.00						

# Particle Size Analysis

S-107 IECPS DUP

Date: 09/21/98 Meas #: 00067  
Time: 15:24 Pres #: 01

S-107:IECPS DUP *20min*  
60 ml/s, in 1.96 M NaOH/0.1 M NaNO3

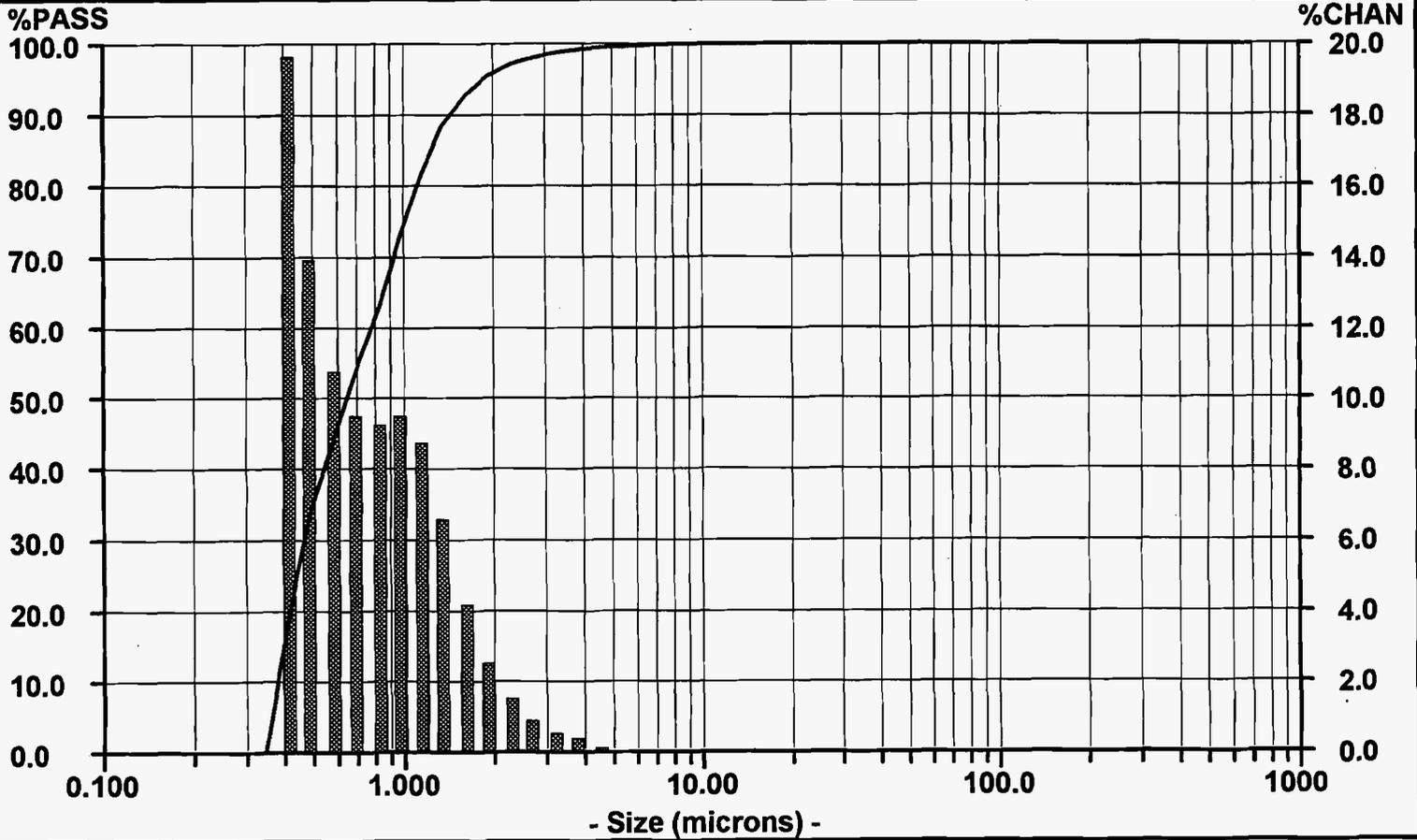
### Summary

mv = 6.583  
mn = 0.837  
ma = 2.927  
cs = 2.060  
sd = 0.409

### Percentiles

10% = 0.379 60% = 0.767  
20% = 0.410 70% = 0.920  
30% = 0.461 80% = 1.113  
40% = 0.535 90% = 1.442  
50% = 0.637 95% = 1.860

Dia	Vol%	Width
1.049	46%	0.825
0.444	54%	0.213



- Size (microns) -

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	99.97	0.04						
592.0	100.00	0.00	7.778	99.93	0.07						
497.8	100.00	0.00	6.541	99.86	0.11						
418.6	100.00	0.00	5.500	99.75	0.18						
352.0	100.00	0.00	4.625	99.57	0.27						
296.0	100.00	0.00	3.889	99.30	0.43						
248.9	100.00	0.00	3.270	98.87	0.68						
209.3	100.00	0.00	2.750	98.19	1.04						
176.0	100.00	0.00	2.312	97.15	1.60						
148.0	100.00	0.00	1.945	95.55	2.60						
124.5	100.00	0.00	1.635	92.95	4.36						
104.7	100.00	0.00	1.375	88.59	6.76						
88.00	100.00	0.00	1.156	81.83	8.82						
74.00	100.00	0.00	0.972	73.01	9.51						
62.23	100.00	0.00	0.818	63.50	9.39						
52.33	100.00	0.00	0.688	54.11	9.53						
44.00	100.00	0.00	0.578	44.58	10.83						
37.00	100.00	0.00	0.486	33.75	14.00						
31.11	100.00	0.00	0.409	19.75	19.75						
26.16	100.00	0.00	0.344	0.00	0.00						
22.00	100.00	0.00	0.289	0.00	0.00						
18.50	100.00	0.00	0.243	0.00	0.00						
15.56	100.00	0.00	0.204	0.00	0.00						
13.08	100.00	0.01	0.172	0.00	0.00						
11.00	99.99	0.02	0.145	0.00	0.00						

# Particle Size Analysis

S-107 IECPS DUP

Date: 09/21/98 Meas #: 00069  
Time: 15:36 Pres #: 01

S-107:IECPS DUP  
40 ml/s, in 1.96 M NaOH/0.1 M NaNO3  
Sonication #2 @40 W-90 sec

### Summary

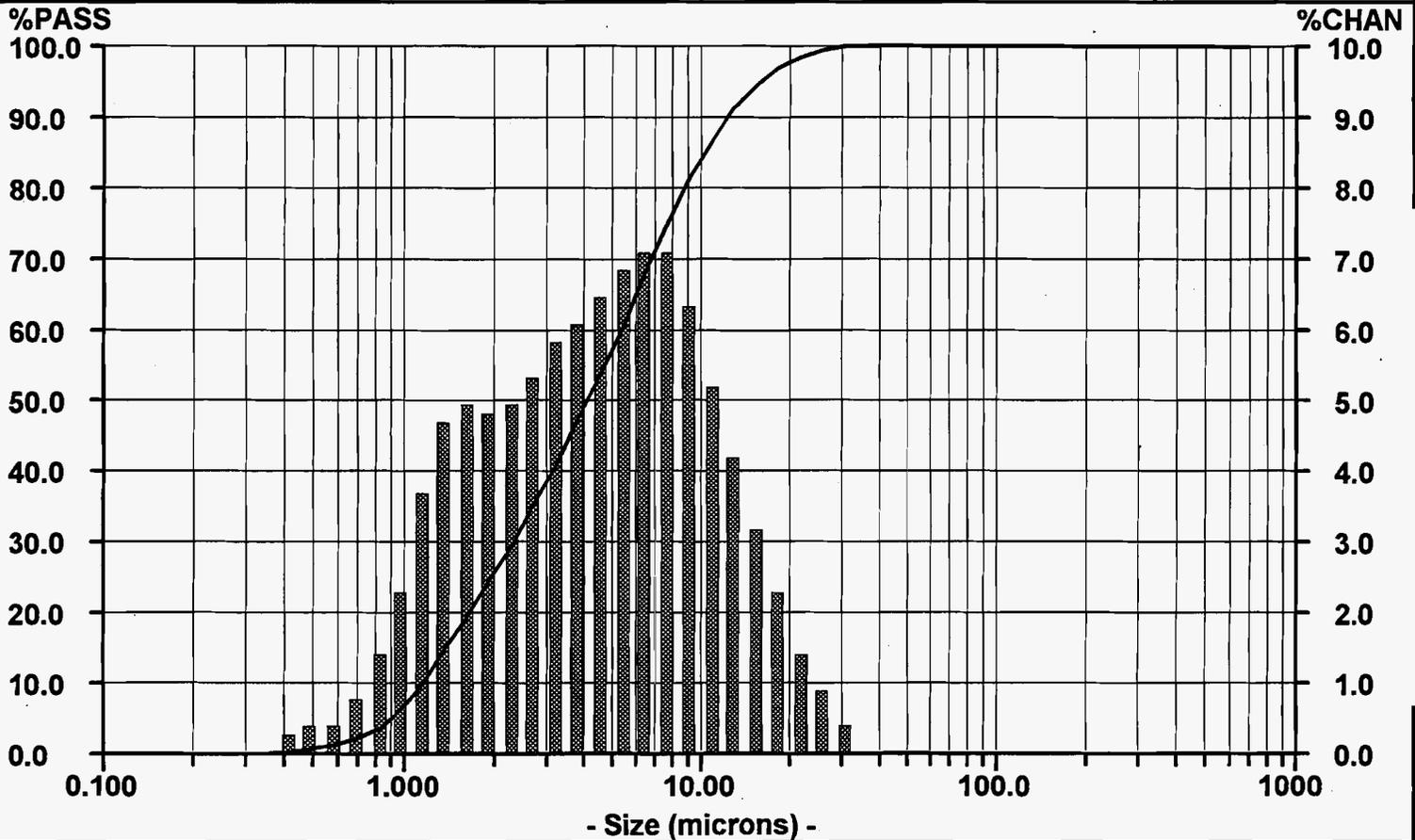
mv = 5.691  
mn = 0.828  
ma = 2.631  
cs = 2.281  
sd = 4.258

### Percentiles

10% = 1.161 60% = 5.392  
20% = 1.656 70% = 6.857  
30% = 2.340 80% = 8.842  
40% = 3.174 90% = 12.38  
50% = 4.176 95% = 16.00

### Dia Vol% Width

5.368 80% 8.531  
1.153 20% 0.691



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	81.59	6.44						
592.0	100.00	0.00	7.778	75.15	7.12						
497.8	100.00	0.00	6.541	68.03	7.21						
418.6	100.00	0.00	5.500	60.82	6.91						
352.0	100.00	0.00	4.625	53.91	6.56						
296.0	100.00	0.00	3.889	47.35	6.29						
248.9	100.00	0.00	3.270	41.06	5.95						
209.3	100.00	0.00	2.750	35.11	5.47						
176.0	100.00	0.00	2.312	29.64	5.06						
148.0	100.00	0.00	1.945	24.58	4.93						
124.5	100.00	0.00	1.635	19.65	5.00						
104.7	100.00	0.00	1.375	14.65	4.75						
88.00	100.00	0.00	1.156	9.90	3.78						
74.00	100.00	0.00	0.972	6.12	2.46						
62.23	100.00	0.00	0.818	3.66	1.44						
52.33	100.00	0.00	0.688	2.22	0.86						
44.00	100.00	0.00	0.578	1.36	0.57						
37.00	100.00	0.00	0.486	0.79	0.44						
31.11	100.00	0.53	0.409	0.35	0.35						
26.16	99.47	0.95	0.344	0.00	0.00						
22.00	98.52	1.57	0.289	0.00	0.00						
18.50	96.95	2.38	0.243	0.00	0.00						
15.56	94.57	3.29	0.204	0.00	0.00						
13.08	91.28	4.30	0.172	0.00	0.00						
11.00	86.98	5.39	0.145	0.00	0.00						

# Particle Size Analysis

S-107 IECPS DUP

Date: 09/21/98 Meas #: 00069  
Time: 15:36 Pres #: 01

S-107:IECPS DUP  
40 ml/s, in 1.96 M NaOH/0.1 M NaNO3  
Sonication #2 @40 W-90 sec

### Summary

mv = 5.691  
mn = 0.828  
ma = 2.631  
cs = 2.281  
sd = 0.399

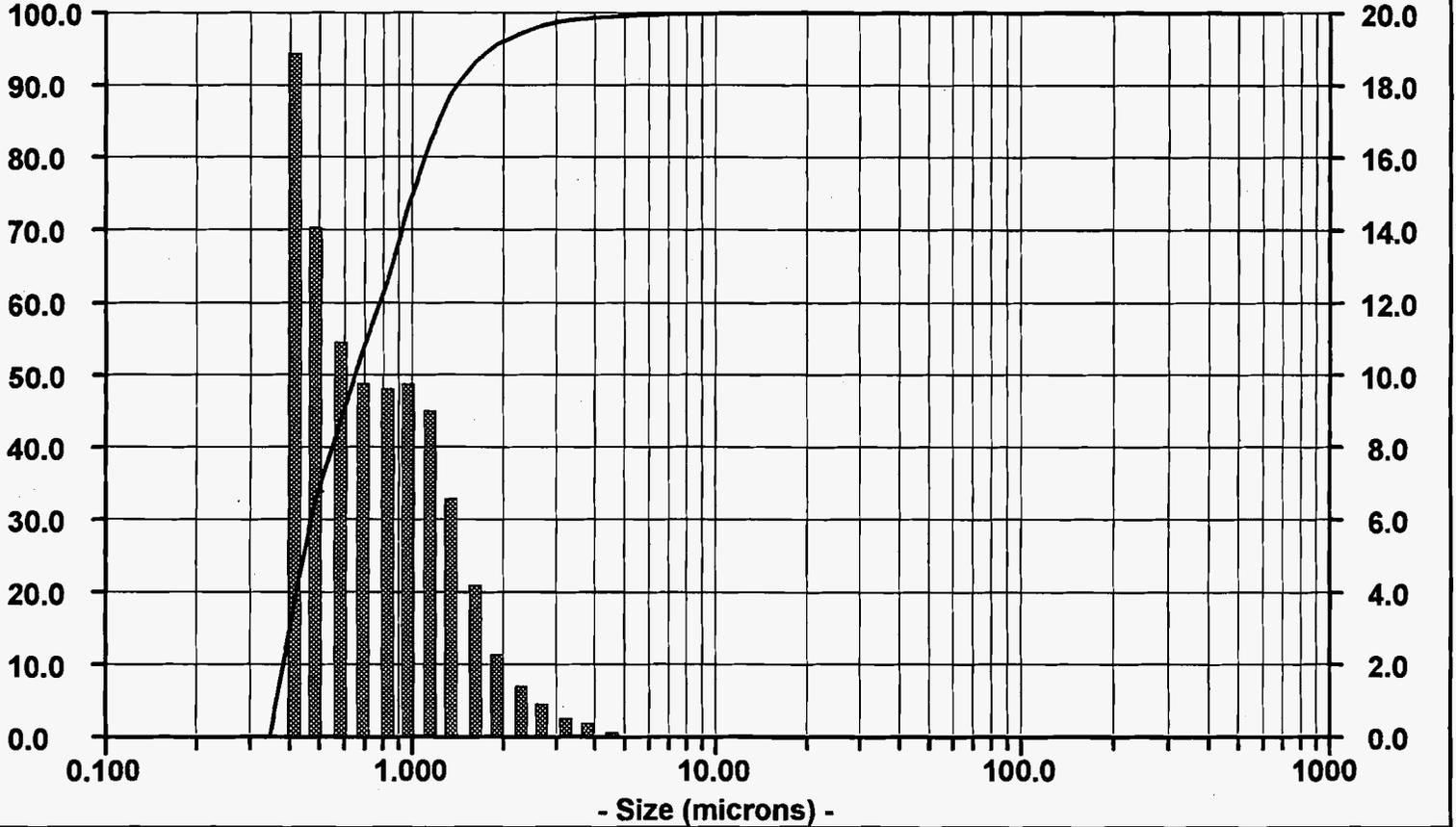
### Percentiles

10% = 0.380 60% = 0.769  
20% = 0.414 70% = 0.916  
30% = 0.466 80% = 1.100  
40% = 0.541 90% = 1.415  
50% = 0.642 95% = 1.809

### Dia Vol% Width

1.036 46% 0.786  
0.447 54% 0.215

%PASS



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	99.98	0.03						
592.0	100.00	0.00	7.778	99.95	0.06						
497.8	100.00	0.00	6.541	99.89	0.10						
418.6	100.00	0.00	5.500	99.79	0.15						
352.0	100.00	0.00	4.625	99.64	0.25						
296.0	100.00	0.00	3.889	99.39	0.40						
248.9	100.00	0.00	3.270	98.99	0.63						
209.3	100.00	0.00	2.750	98.36	0.97						
176.0	100.00	0.00	2.312	97.39	1.51						
148.0	100.00	0.00	1.945	95.88	2.48						
124.5	100.00	0.00	1.635	93.40	4.23						
104.7	100.00	0.00	1.375	89.17	6.75						
88.00	100.00	0.00	1.156	82.42	9.05						
74.00	100.00	0.00	0.972	73.37	9.88						
62.23	100.00	0.00	0.818	63.49	9.73						
52.33	100.00	0.00	0.688	53.76	9.80						
44.00	100.00	0.00	0.578	43.96	10.91						
37.00	100.00	0.00	0.486	33.05	14.14						
31.11	100.00	0.00	0.409	18.91	18.91						
26.16	100.00	0.00	0.344	0.00	0.00						
22.00	100.00	0.00	0.289	0.00	0.00						
18.50	100.00	0.00	0.243	0.00	0.00						
15.56	100.00	0.00	0.204	0.00	0.00						
13.08	100.00	0.01	0.172	0.00	0.00						
11.00	99.99	0.01	0.145	0.00	0.00						

# Particle Size Analysis

S-107 FSPS DUP

Date: 09/21/98 Meas #: 00074

Time: 16:03 Pres #: 01

S-107:FSPS *10 min*  
40 ml/s, in 1.96 M NaOH/0.1 M NaNO3

### Summary

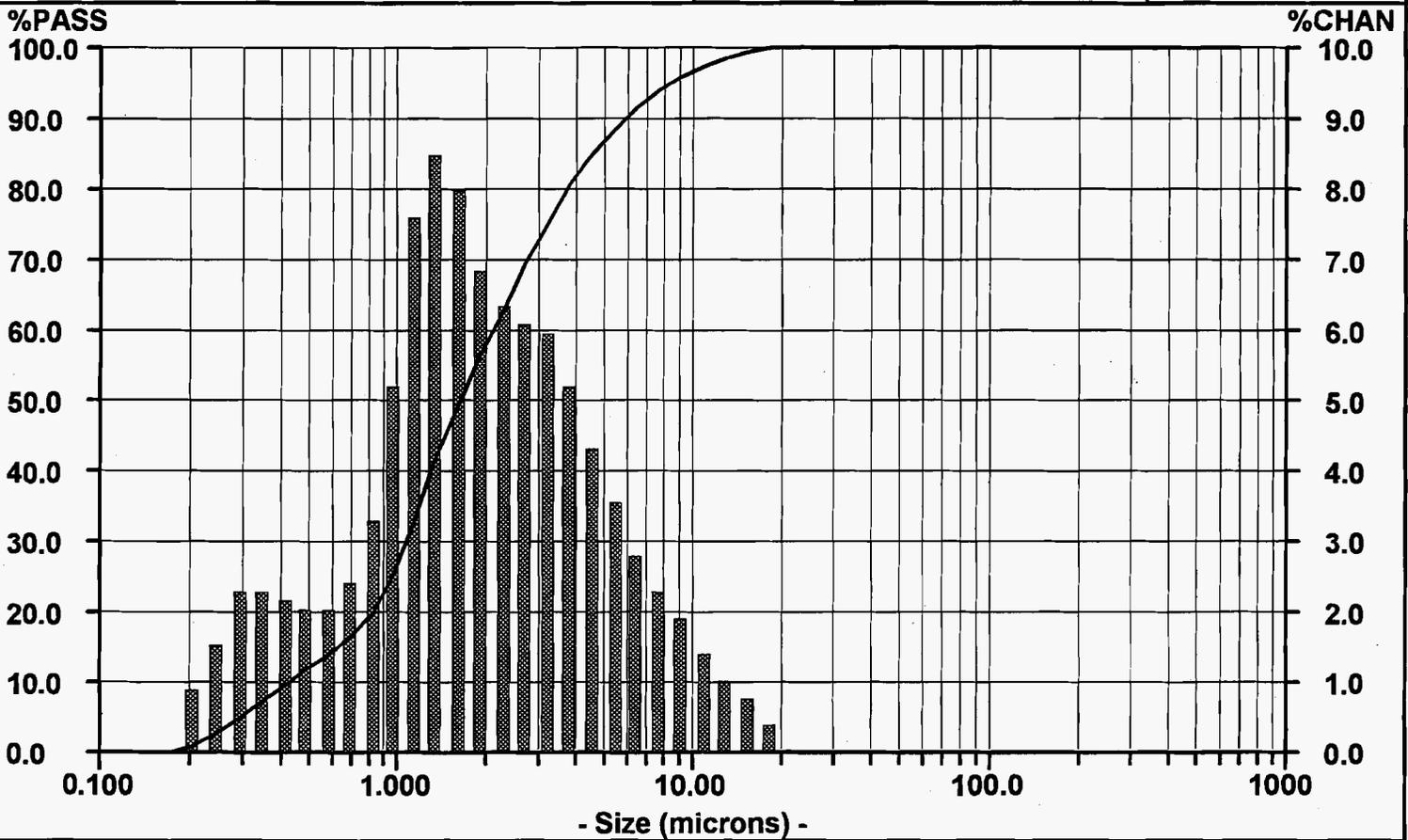
mv = 2.615  
mn = 0.311  
ma = 1.059  
cs = 5.665  
sd = 1.874

### Percentiles

10% = 0.418 60% = 2.126  
20% = 0.817 70% = 2.797  
30% = 1.085 80% = 3.796  
40% = 1.331 90% = 5.907  
50% = 1.649 95% = 8.442

### Dia Vol% Width

1.857 90% 3.779  
0.286 10% 0.145



- Size (microns) -

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	95.95	1.90						
592.0	100.00	0.00	7.778	94.05	2.38						
497.8	100.00	0.00	6.541	91.67	2.95						
418.6	100.00	0.00	5.500	88.72	3.61						
352.0	100.00	0.00	4.625	85.11	4.41						
296.0	100.00	0.00	3.889	80.70	5.30						
248.9	100.00	0.00	3.270	75.40	6.00						
209.3	100.00	0.00	2.750	69.40	6.30						
176.0	100.00	0.00	2.312	63.10	6.45						
148.0	100.00	0.00	1.945	56.65	6.99						
124.5	100.00	0.00	1.635	49.66	8.03						
104.7	100.00	0.00	1.375	41.63	8.63						
88.00	100.00	0.00	1.156	33.00	7.61						
74.00	100.00	0.00	0.972	25.39	5.37						
62.23	100.00	0.00	0.818	20.02	3.49						
52.33	100.00	0.00	0.688	16.53	2.51						
44.00	100.00	0.00	0.578	14.02	2.15						
37.00	100.00	0.00	0.486	11.87	2.13						
31.11	100.00	0.00	0.409	9.74	2.28						
26.16	100.00	0.00	0.344	7.46	2.44						
22.00	100.00	0.00	0.289	5.02	2.32						
18.50	100.00	0.53	0.243	2.70	1.70						
15.56	99.47	0.85	0.204	1.00	1.00						
13.08	98.62	1.17	0.172	0.00	0.00						
11.00	97.45	1.50	0.145	0.00	0.00						

# Particle Size Analysis

S-107 FSPS DUP

Date: 09/21/98 Meas #: 00074

Time: 16:03 Pres #: 01

S-107:FSPS 10 min  
40 ml/s, in 1.96 M NaOH/0.1 M NaNO3

**Summary**

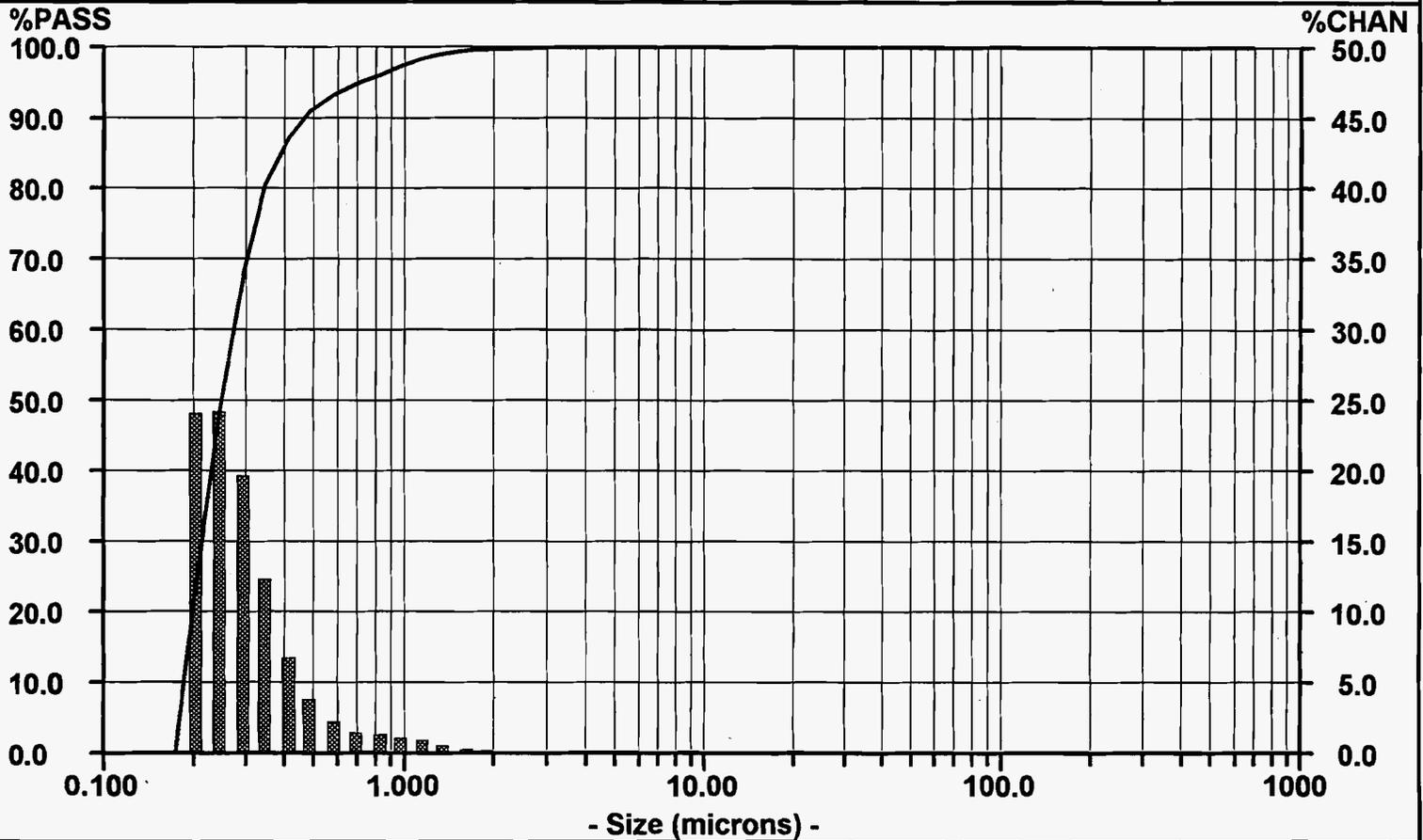
mv = 2.615  
mn = 0.311  
ma = 1.059  
cs = 5.665  
sd = 0.088

**Percentiles**

10% = 0.187 60% = 0.267  
20% = 0.199 70% = 0.296  
30% = 0.212 80% = 0.341  
40% = 0.228 90% = 0.458  
50% = 0.246 95% = 0.689

**Dia Vol% Width**

0.246 100% 0.177



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	100.00	0.00						
592.0	100.00	0.00	7.778	100.00	0.00						
497.8	100.00	0.00	6.541	100.00	0.00						
418.6	100.00	0.00	5.500	100.00	0.00						
352.0	100.00	0.00	4.625	100.00	0.01						
296.0	100.00	0.00	3.889	99.99	0.02						
248.9	100.00	0.00	3.270	99.97	0.04						
209.3	100.00	0.00	2.750	99.93	0.06						
176.0	100.00	0.00	2.312	99.87	0.11						
148.0	100.00	0.00	1.945	99.76	0.20						
124.5	100.00	0.00	1.635	99.56	0.38						
104.7	100.00	0.00	1.375	99.18	0.68						
88.00	100.00	0.00	1.156	98.50	1.01						
74.00	100.00	0.00	0.972	97.49	1.20						
62.23	100.00	0.00	0.818	96.29	1.31						
52.33	100.00	0.00	0.688	94.98	1.69						
44.00	100.00	0.00	0.578	93.39	2.28						
37.00	100.00	0.00	0.486	91.11	3.80						
31.11	100.00	0.00	0.409	87.31	6.84						
26.16	100.00	0.00	0.344	80.47	12.35						
22.00	100.00	0.00	0.289	68.12	19.71						
18.50	100.00	0.00	0.243	48.41	24.23						
15.56	100.00	0.00	0.204	24.18	24.18						
13.08	100.00	0.00	0.172	0.00	0.00						
11.00	100.00	0.00	0.145	0.00	0.00						

# Particle Size Analysis

S-107 FSPS DUP

Date: 09/21/98 Meas #: 00076

Time: 16:13 Pres #: 01

S-107:FSPS  
 48 ml/s, in 1.96 M NaOH/0.1 M NaNO3  
 60 ml/s  
 20 min

### Summary

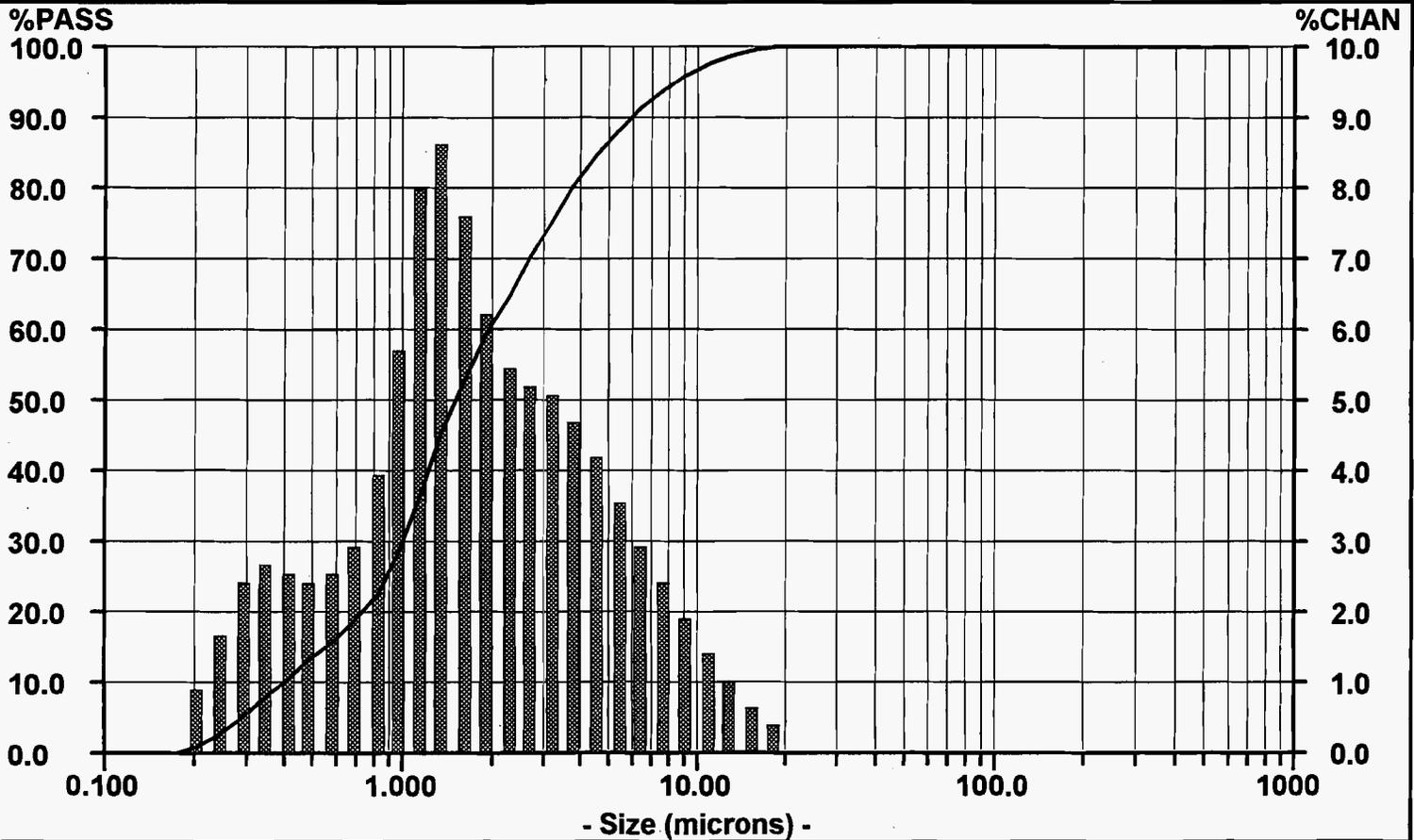
mv = 2.543  
 mn = 0.314  
 ma = 0.999  
 cs = 6.005  
 sd = 1.947

### Percentiles

10% = 0.390 60% = 1.970  
 20% = 0.727 70% = 2.715  
 30% = 1.001 80% = 3.826  
 40% = 1.232 90% = 5.973  
 50% = 1.514 95% = 8.388

### Dia Vol% Width

1.726 89% 3.945  
 0.290 11% 0.144



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	96.08	2.01						
592.0	100.00	0.00	7.778	94.07	2.52						
497.8	100.00	0.00	6.541	91.55	3.09						
418.6	100.00	0.00	5.500	88.46	3.69						
352.0	100.00	0.00	4.625	84.77	4.32						
296.0	100.00	0.00	3.889	80.45	4.87						
248.9	100.00	0.00	3.270	75.58	5.19						
209.3	100.00	0.00	2.750	70.39	5.29						
176.0	100.00	0.00	2.312	66.10	5.52						
148.0	100.00	0.00	1.945	59.58	6.32						
124.5	100.00	0.00	1.635	53.26	7.72						
104.7	100.00	0.00	1.375	45.54	8.76						
88.00	100.00	0.00	1.156	36.78	8.01						
74.00	100.00	0.00	0.972	28.77	5.89						
62.23	100.00	0.00	0.818	22.88	4.02						
52.33	100.00	0.00	0.688	18.86	3.00						
44.00	100.00	0.00	0.578	15.86	2.61						
37.00	100.00	0.00	0.486	13.25	2.55						
31.11	100.00	0.00	0.409	10.70	2.65						
26.16	100.00	0.00	0.344	8.05	2.75						
22.00	100.00	0.00	0.289	5.30	2.52						
18.50	100.00	0.43	0.243	2.78	1.78						
15.56	99.57	0.78	0.204	1.00	1.00						
13.08	98.79	1.15	0.172	0.00	0.00						
11.00	97.64	1.56	0.145	0.00	0.00						

# Particle Size Analysis

S-107 FSPS DUP

Date: 09/21/98 Meas #: 00076  
Time: 16:13 Pres #: 01

S-107:FSPS *20 min*  
*40 ml/s, in 1.96 M NaOH/0.1 M NaNO3*  
*60 m/s*

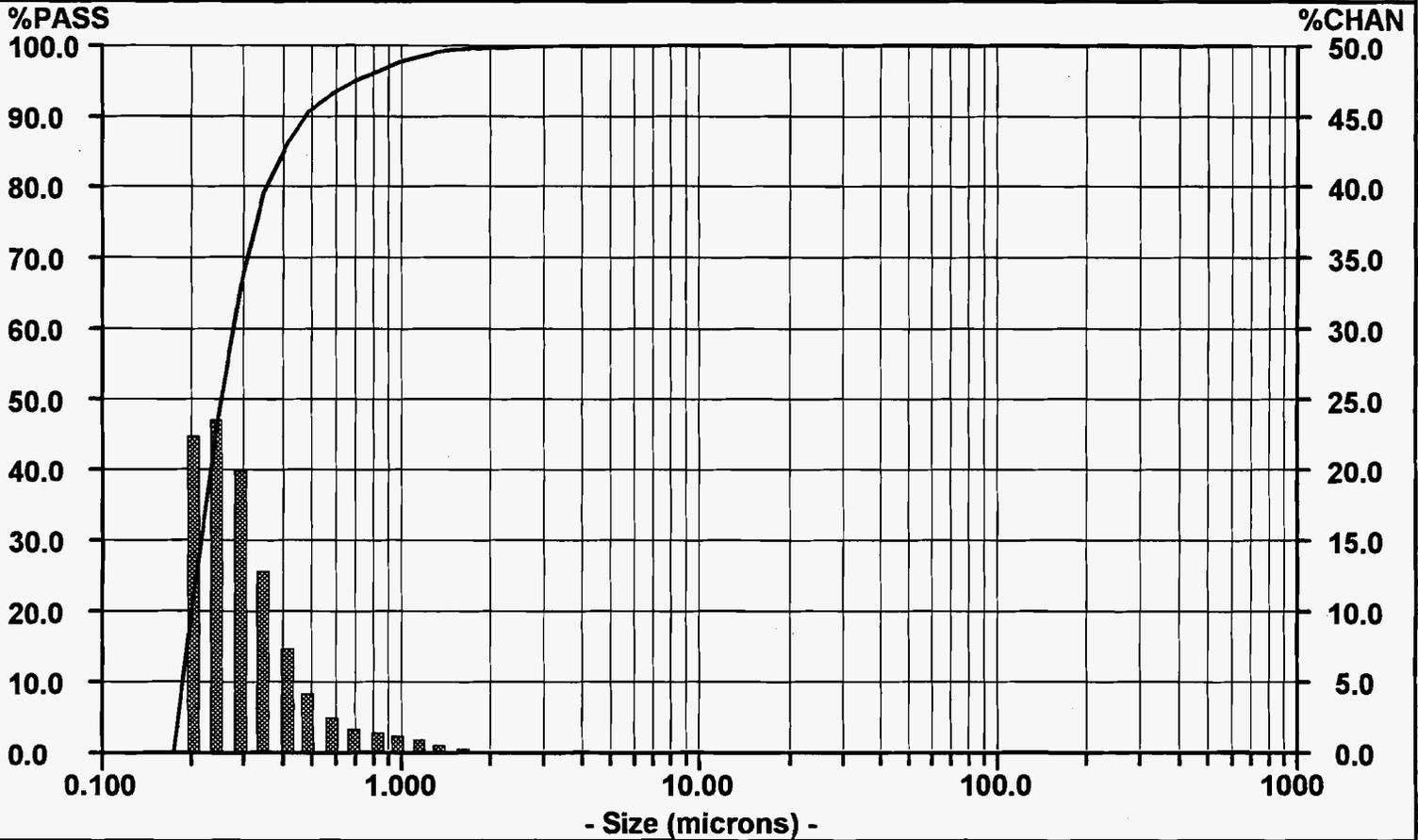
### Summary

mv = 2.543  
mn = 0.314  
ma = 0.999  
cs = 6.005  
sd = 0.093

### Percentiles

10% = 0.188 60% = 0.273  
20% = 0.201 70% = 0.302  
30% = 0.215 80% = 0.350  
40% = 0.232 90% = 0.470  
50% = 0.251 95% = 0.685

Dia	Vol%	Width
0.251	100%	0.185



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	100.00	0.00						
592.0	100.00	0.00	7.778	100.00	0.00						
497.8	100.00	0.00	6.541	100.00	0.00						
418.6	100.00	0.00	5.500	100.00	0.00						
352.0	100.00	0.00	4.625	100.00	0.01						
296.0	100.00	0.00	3.889	99.99	0.02						
248.9	100.00	0.00	3.270	99.97	0.03						
209.3	100.00	0.00	2.750	99.94	0.05						
176.0	100.00	0.00	2.312	99.89	0.09						
148.0	100.00	0.00	1.945	99.80	0.16						
124.5	100.00	0.00	1.635	99.64	0.34						
104.7	100.00	0.00	1.375	99.30	0.64						
88.00	100.00	0.00	1.156	98.66	0.99						
74.00	100.00	0.00	0.972	97.67	1.22						
62.23	100.00	0.00	0.818	96.45	1.40						
52.33	100.00	0.00	0.688	95.05	1.77						
44.00	100.00	0.00	0.578	93.28	2.58						
37.00	100.00	0.00	0.486	90.70	4.23						
31.11	100.00	0.00	0.409	86.47	7.40						
26.16	100.00	0.00	0.344	79.07	12.96						
22.00	100.00	0.00	0.289	66.11	19.94						
18.50	100.00	0.00	0.243	46.17	23.65						
15.56	100.00	0.00	0.204	22.52	22.52						
13.08	100.00	0.00	0.172	0.00	0.00						
11.00	100.00	0.00	0.145	0.00	0.00						

# Particle Size Analysis

S-107 FSPS

Date: 09/21/98 Meas #: 00078

Time: 16:25 Pres #: 01

S-107:FSPS

40 ml/s, in 1.96 M NaOH/0.1 M NaNO3

Sonication #2 @40 W-90sec

### Summary

mv = 2.234  
mn = 0.321  
ma = 0.969  
cs = 6.194  
sd = 1.677

### Percentiles

10% = 0.390 60% = 1.791  
20% = 0.704 70% = 2.434  
30% = 0.962 80% = 3.393  
40% = 1.173 90% = 5.096  
50% = 1.417 95% = 6.917

Dia Vol% Width

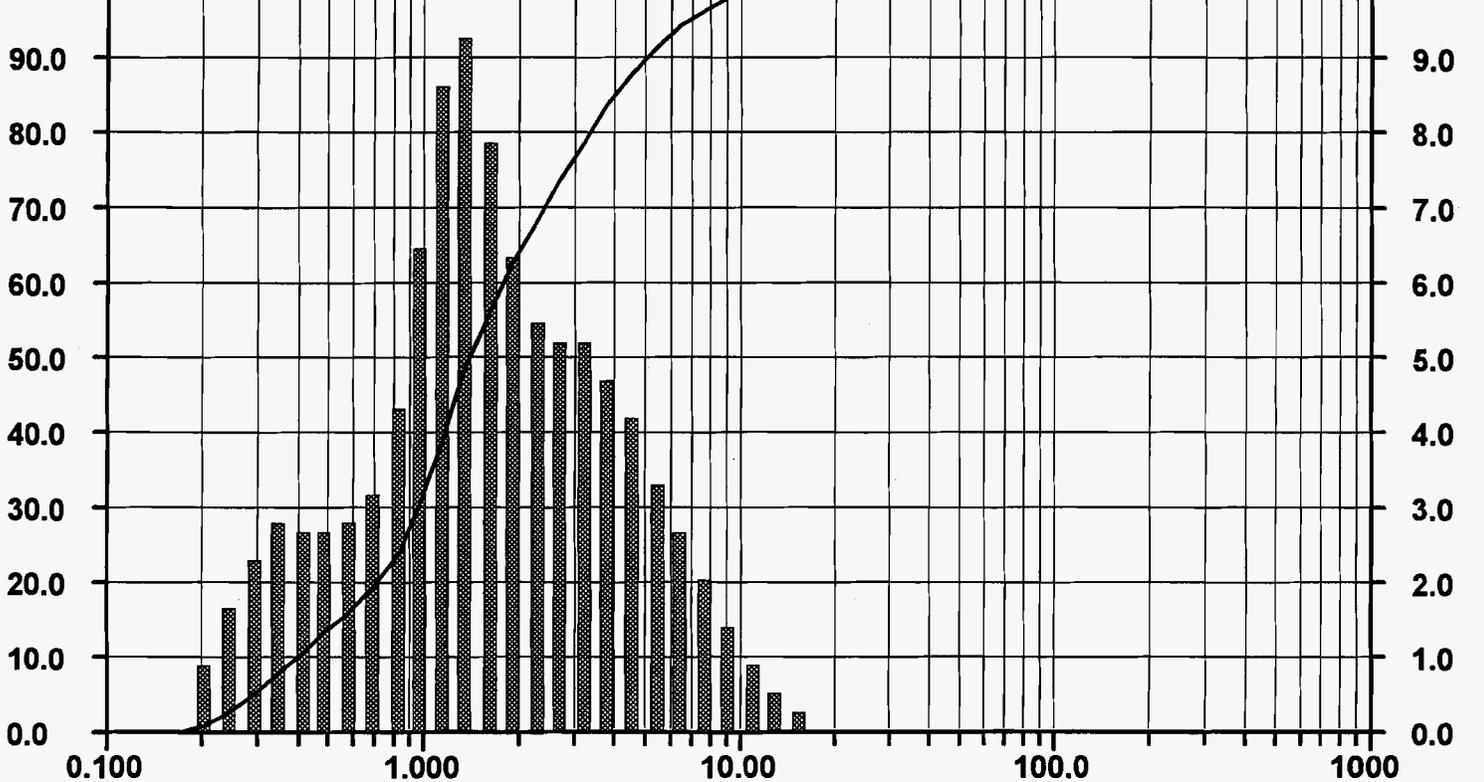
1.592 89% 3.365  
0.293 11% 0.144

%PASS

100.0

%CHAN

10.0



- Size (microns) -

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	97.92	1.64						
592.0	100.00	0.00	7.778	96.38	2.13						
497.8	100.00	0.00	6.541	94.25	2.79						
418.6	100.00	0.00	5.500	91.46	3.49						
352.0	100.00	0.00	4.625	87.97	4.20						
296.0	100.00	0.00	3.889	83.77	4.83						
248.9	100.00	0.00	3.270	78.94	5.20						
209.3	100.00	0.00	2.750	73.74	5.31						
176.0	100.00	0.00	2.312	68.43	5.54						
148.0	100.00	0.00	1.945	62.89	6.40						
124.5	100.00	0.00	1.635	56.49	7.98						
104.7	100.00	0.00	1.375	48.51	9.31						
88.00	100.00	0.00	1.156	39.20	8.75						
74.00	100.00	0.00	0.972	30.45	6.63						
62.23	100.00	0.00	0.818	23.92	4.44						
52.33	100.00	0.00	0.688	19.48	3.27						
44.00	100.00	0.00	0.578	16.21	2.80						
37.00	100.00	0.00	0.486	13.41	2.70						
31.11	100.00	0.00	0.409	10.71	2.76						
26.16	100.00	0.00	0.344	7.95	2.80						
22.00	100.00	0.00	0.289	5.15	2.49						
18.50	100.00	0.00	0.243	2.66	1.71						
15.56	100.00	0.37	0.204	0.95	0.95						
13.08	99.63	0.66	0.172	0.00	0.00						
11.00	98.97	1.05	0.145	0.00	0.00						

# Particle Size Analysis

S-107 FSPS

Date: 09/21/98 Meas #: 00078

Time: 16:25 Pres #: 01

S-107:FSPS

40 ml/s, in 1.96 M NaOH/0.1 M NaNO3

Sonication #2 @40 W-90sec

### Summary

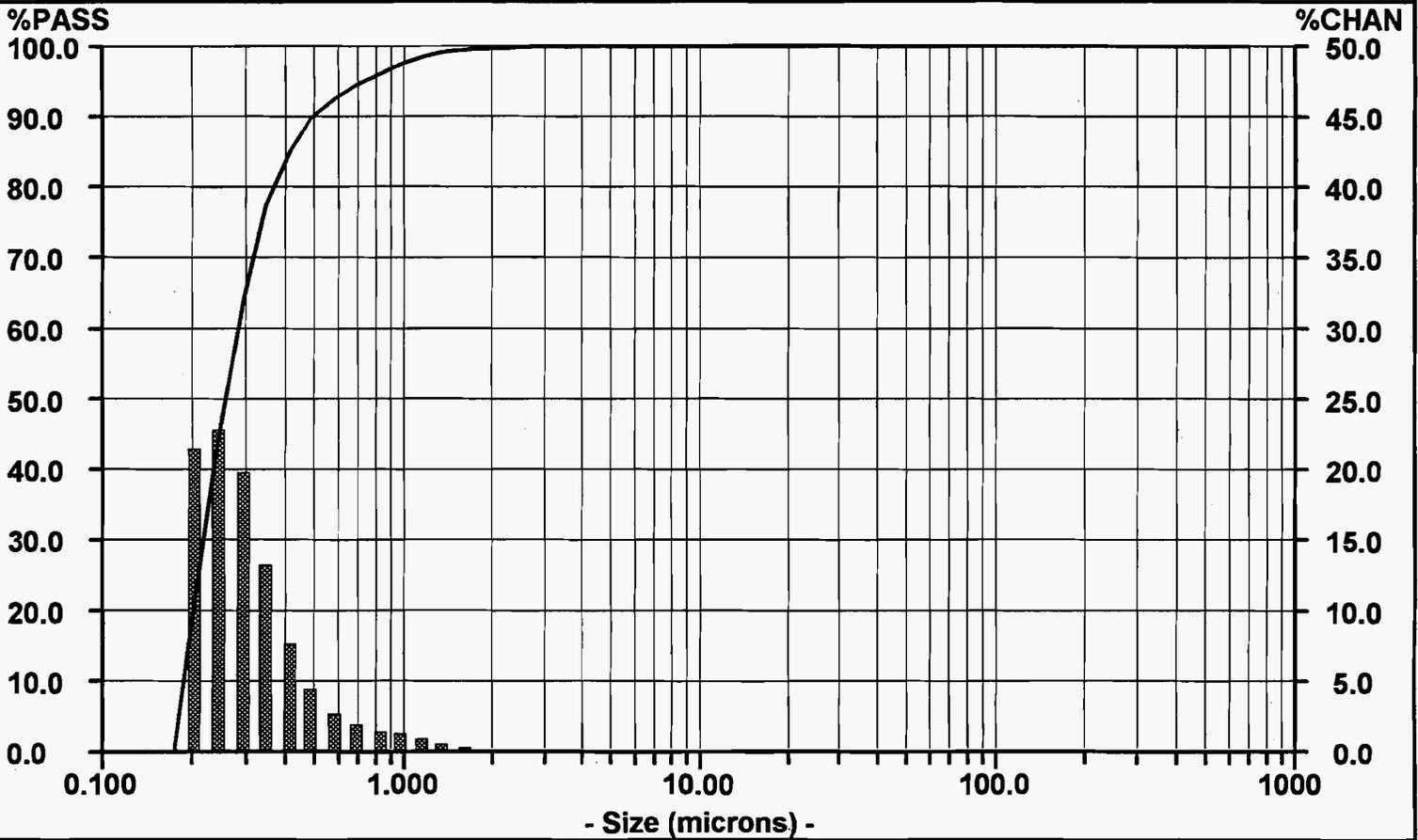
mv = 2.234  
mn = 0.321  
ma = 0.969  
cs = 6.194  
sd = 0.098

### Percentiles

10% = 0.189 60% = 0.277  
20% = 0.202 70% = 0.309  
30% = 0.217 80% = 0.359  
40% = 0.235 90% = 0.490  
50% = 0.254 95% = 0.718

### Dia Vol% Width

0.254 100% 0.196

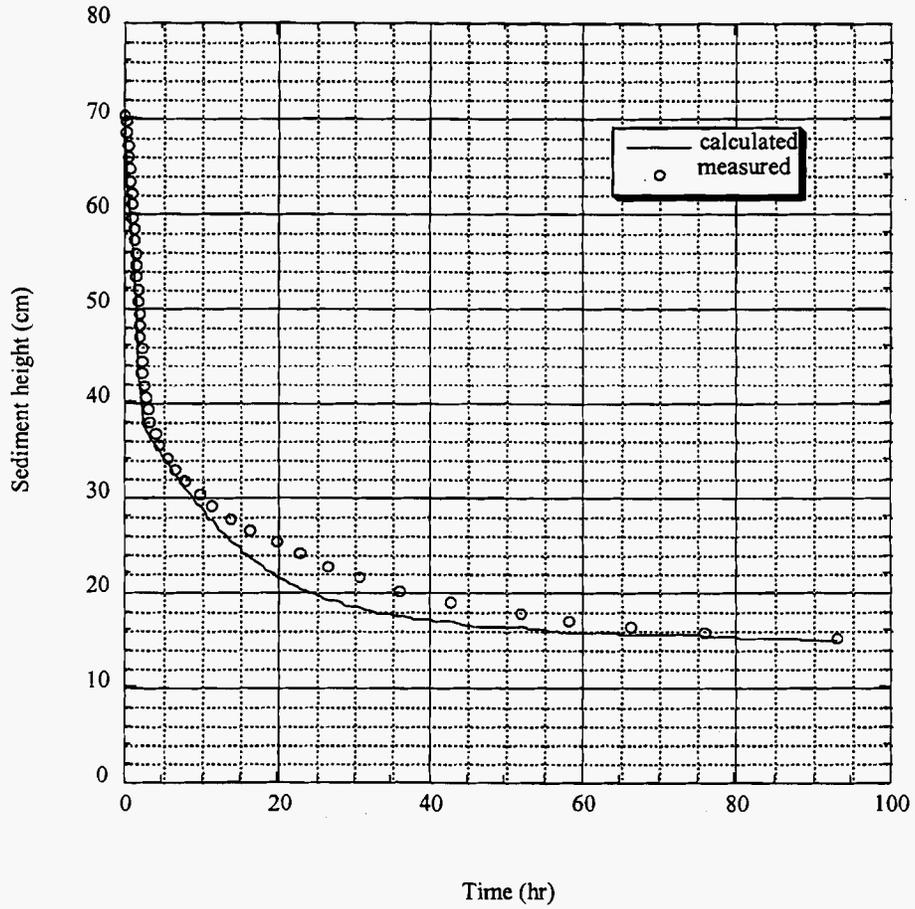


SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	100.00	0.00						
592.0	100.00	0.00	7.778	100.00	0.00						
497.8	100.00	0.00	6.541	100.00	0.00						
418.6	100.00	0.00	5.500	100.00	0.00						
352.0	100.00	0.00	4.625	100.00	0.01						
296.0	100.00	0.00	3.889	99.99	0.02						
248.9	100.00	0.00	3.270	99.97	0.03						
209.3	100.00	0.00	2.750	99.94	0.05						
176.0	100.00	0.00	2.312	99.89	0.09						
148.0	100.00	0.00	1.945	99.80	0.17						
124.5	100.00	0.00	1.635	99.63	0.35						
104.7	100.00	0.00	1.375	99.28	0.69						
88.00	100.00	0.00	1.156	98.59	1.09						
74.00	100.00	0.00	0.972	97.50	1.36						
62.23	100.00	0.00	0.818	96.14	1.56						
52.33	100.00	0.00	0.688	94.58	1.94						
44.00	100.00	0.00	0.578	92.64	2.79						
37.00	100.00	0.00	0.486	89.85	4.52						
31.11	100.00	0.00	0.409	85.33	7.76						
26.16	100.00	0.00	0.344	77.57	13.29						
22.00	100.00	0.00	0.289	64.28	19.85						
18.50	100.00	0.00	0.243	44.43	22.88						
15.56	100.00	0.00	0.204	21.55	21.55						
13.08	100.00	0.00	0.172	0.00	0.00						
11.00	100.00	0.00	0.145	0.00	0.00						

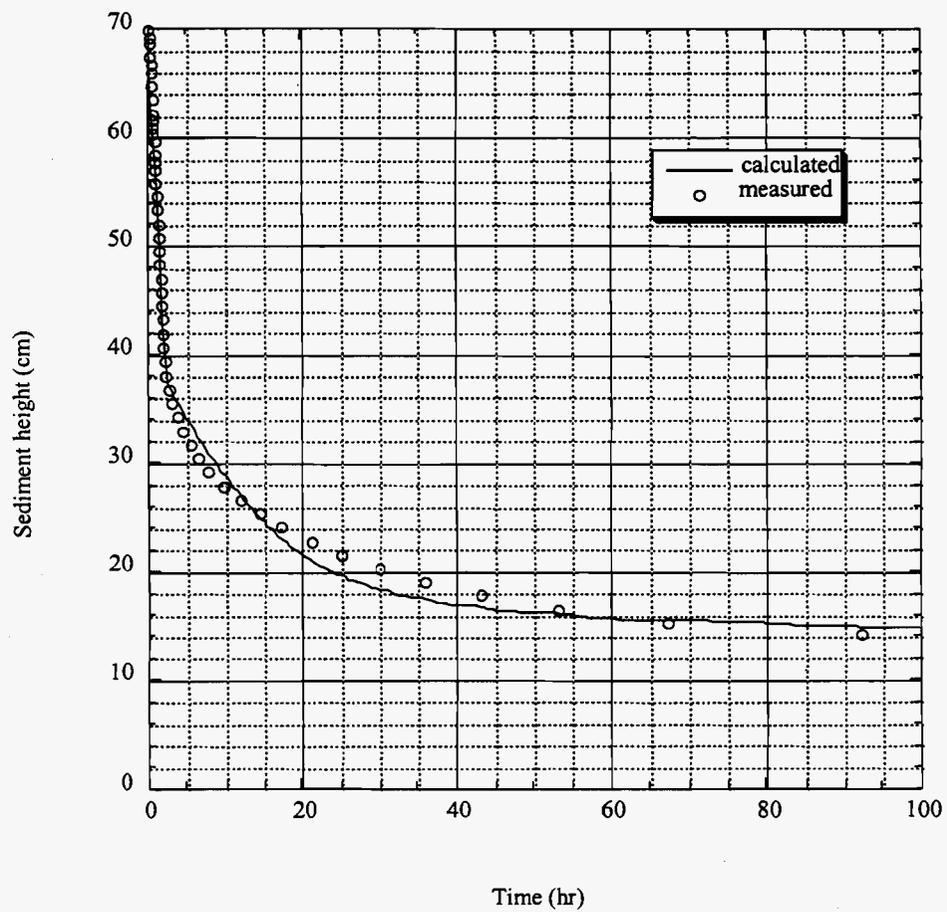
## **Appendix E**

### **Additional Figures from Theoretical Analysis**

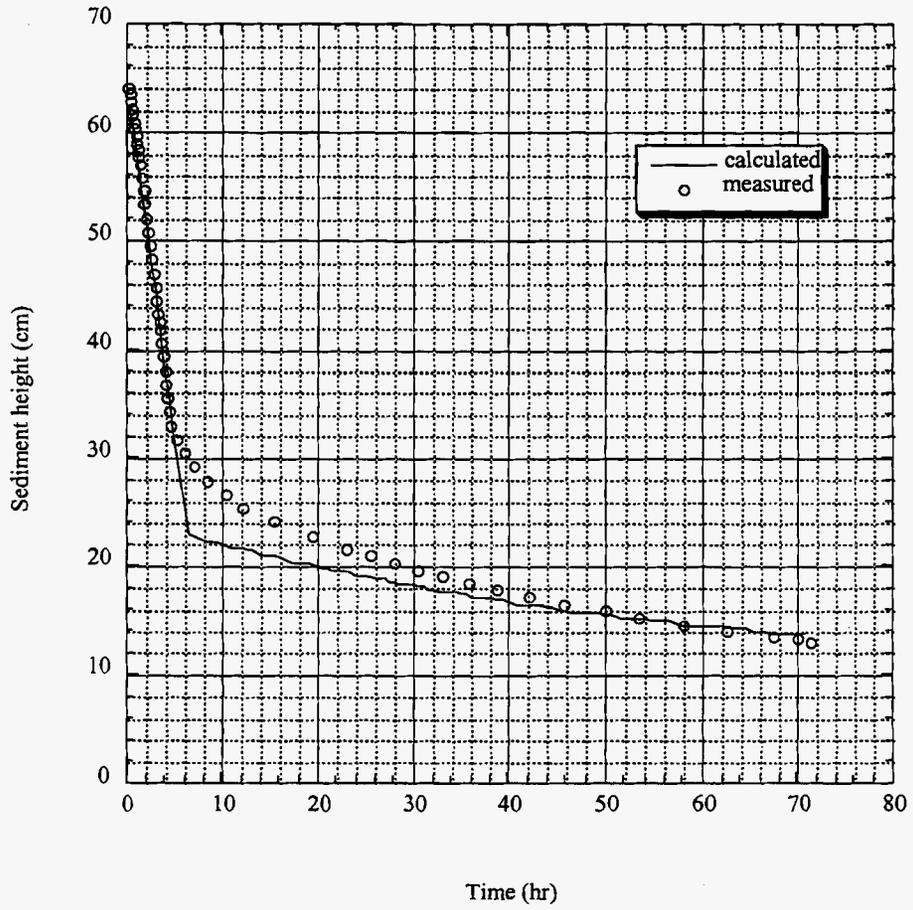
Third Water Wash (5 wt%)



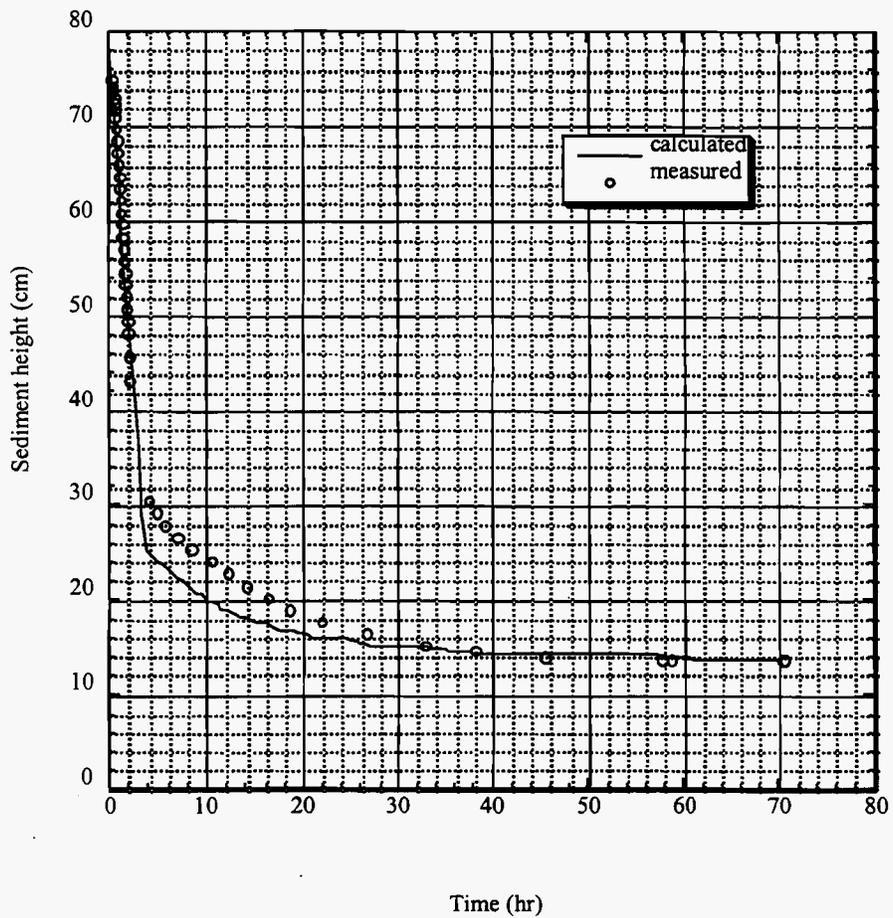
### Second Water Wash



Second Caustic Leach (Part 2)



Second Retrieval Wash



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