

Characterization, Washing, Leaching, and Filtration of C-104 Sludge

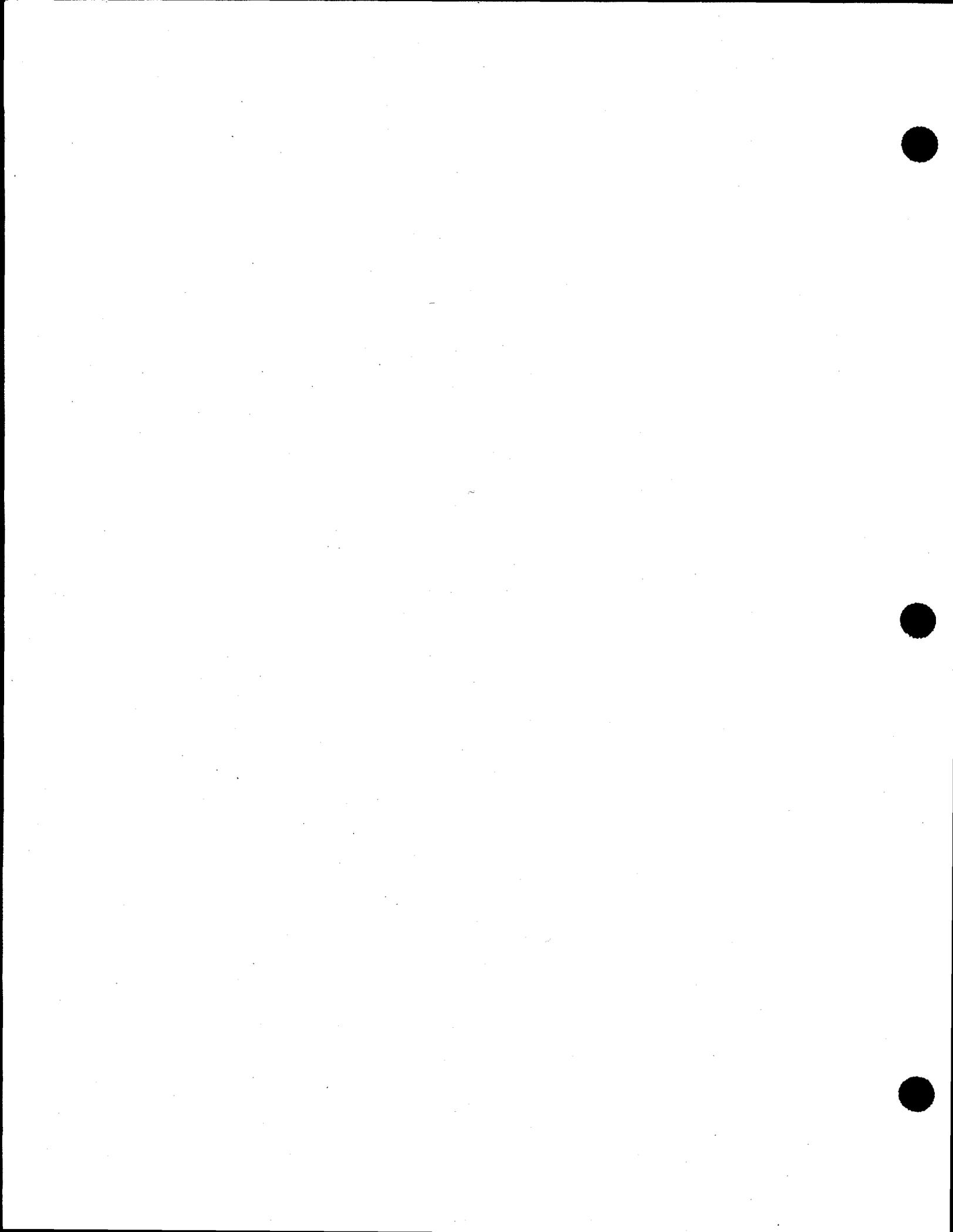
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Summary

Approximately 1400 g of wet Hanford Tank C-104 Sludge was evaluated by Battelle for the high-level waste (HLW) pretreatment processes of ultrafiltration, dilute caustic washing, and elevated-temperature caustic leaching. The filterability of diluted C-104 sludge was measured with a 0.1- μm sintered metal Mott filter using a 24-inch-long, single-element, crossflow filtration system (cells unit filter [CUF]). While the filtrate was being recirculated prior to washing and leaching, a 6.9 wt% solids^(a) slurry was evaluated with a matrix of seven 1-hour conditions of varying trans-membrane pressure (30 to 70 psid) and axial velocity (9 to 15 ft/s). The filtrate flux and backpulse efficiency were determined for each condition. The slurry was concentrated to 23 wt% solids, a second matrix of six 1-hour conditions was performed, and data analogous to that recorded in the first matrix were obtained.

The low-solids-concentration matrix produced filtrate flux rates that ranged from 0.038 to 0.083 gpm/ft². The high-solids-concentration matrix produced filtrate flux rates that ranged from 0.0095 to 0.0172 gpm/ft². In both cases, the optimum filtrate flux was at the highest axial velocity (15 ft/s) and transmembrane pressure had little effect. Nearly all of the measured filtrate fluxes were more than an order of magnitude greater than the required plant flux for C-104 of 0.00126 gpm/ft². In both matrices, the filtrate flux appeared to be proportional to axial velocity, and the permeability appeared to be inversely proportional to the trans-membrane pressure. The first test condition was repeated as the last test condition for each matrix. In both cases, there was a significant decrease in filtrate flux, indicating some filter fouling during the test matrix that could not be removed by backpulsing alone, although the backpulse number and duration were not optimized.

Following testing of these two matrices, the material was washed within the CUF by continuously adding approximately 5 L of 0.01-M NaOH and then removing it through the filter as permeate. The purpose of this washing step with 0.01-M NaOH was to remove water-soluble components that might inhibit dissolution of salts during caustic leaching, while avoiding peptization of the solids that occurs at a pH below 12. After washing the sludge with dilute caustic, it was combined with 3-M caustic, and the slurry was leached in a stainless steel vessel at 85°C for 8 hours. This leaching was followed by two 0.01-M caustic washes, each conducted in a stainless steel vessel to dilute remaining analytes from the interstitial liquids. Each rinse was performed at 85°C for 8 hours. Permeate from each of these process steps was removed using the crossflow filter system. Samples of the permeate from each slurry-washing activity and all intermediate process steps were taken and analyzed for chemical and radiochemical constituents. The fraction of each component removed was calculated. Key results are presented in Table S.1.

(a) Solids concentrations are generally reported on an insoluble solids basis. This is done by mathematically subtracting out the dissolved solids from the sample.

The primary components in the initial tank sludge were sodium and aluminum. With these two constituents removed during the washing and leaching steps, the primary components in the final sludge were Th, U, and Zr. These became the limiting species in the HLW glass.

The rheological properties of the C-104 slurries were determined with a viscometer according to procedure BNFL-TP-29953-010. The initial 6.9-wt% material exhibited Newtonian behavior with a viscosity between 2–4 cP. The water-washed slurry that had a measured solids concentration of 24 wt% was found to be yield pseudoplastic and thixotropic. With the initial increase in shear, the viscosity of the slurry at 100 s⁻¹ was 300–360 cP. During the decrease in shear, the viscosity of the slurry at 100 s⁻¹ was 180–240 cP. The final slurry samples with a solids concentration of ~20 wt % solids also exhibited yield pseudoplastic behavior with a viscosity at 100 s⁻¹ of 30 to 40 cP.

Table S.1. Solubility of C-104 Sludge Key Components in 0.01 M NaOH and 3 M NaOH

Component	Fraction Removed in Water Washes (%)	Fraction Removed in Caustic Leaches (%)	Fraction in Solids Residue (%)
Al	2.4	90.8	6.8
Cr	7.0	43.5	49.5
Fe	0.01	0.08	99.9
Na	91.1	N/A	4.6
P	18.9	52.2	29
Th	<0.1	<0.4	>99.5
U	0.6	<1.4	>98
Zr	0.01	0.014	99.98

N/A = Not Analyzed. It is difficult to measure the small amount Na removed with large Na additions.

The initial C-104 sludge had a bi-modal particle size distribution centered at 1.6 and 22 microns with a significant quantity of sub-micron particles. During the course of pumping, washing, and caustic leaching, the large particles or agglomerates were broken up, and the smaller particles were dissolved, creating a single distribution at approximately 1 micron.

Flow-induced shear or sonication did not easily break the particle agglomerates in the initial sample. The PSD of the final slurry was much more easily shifted to smaller particle sizes by shearing. Whether this is just an effect of grinding during pumping or whether it is a chemical effect of washing and leaching is not clear.

Terms and Abbreviations

AEA	alpha energy analysis
BNFL	BNFL, Inc; subsidiary of British Nuclear Fuels, Ltd.
CUF	cells unit filter
DF	decontamination factor
DI	deionized water
GEA	gamma energy analysis
HLRF	High Level Radiochemistry Facility
HLW	high-level waste
IC	ion chromatography
ICP-AES	inductively coupled plasma-atomic emission spectroscopy
ICP-MS	inductively coupled plasma-mass spectrometry
LRB	laboratory record book
MSE	mean squared error
PID	proportional-integral-derivative controller
PMG	precious metals group
PSD	particle size distribution
RPD	relative percent difference
RPL	Radiochemical Processing Laboratory
RPP-WTP	River Protection Project Waste Treatment Plant
SAL	Shielded Analytical Laboratory
TMP	trans-membrane pressure
TIC	total inorganic carbon
TOC	total organic carbon
TRU	transuranic

Units

°C	degrees Celsius
g	gram
g/mL	gram per milliliter
µg/g - µg/mL	microgram per gram/microgram per milliliter
µCi/g - µCi/mL	microcurie per gram/microcurie per milliliter
mL	milliliter
mmole/mL	millimole per milliliter
nCi/g	nanocurie per gram
pCi/g	picocurie per gram
Vol%	volume percent
Wt%	weight percent
M	molarity

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1.0 Introduction

The River Protection Project Waste Treatment Plant (RPP-WTP) flowsheets developed by British Nuclear Fuels Limited (BNFL) Inc. plan to use caustic leaching and/or water washing for pretreatment of the Envelope D Hanford sludge before high-level waste (HLW) vitrification (DOE-RL 1996). These pretreatment steps reduce the quantity of HLW generated by removing components such as Al, Cr, Na, and P that are soluble either in water or high-temperature caustic and often limit the waste loading in the glass.

The RPP-WTP flowsheets also use crossflow filtration to separate the leach and wash solutions from the solids between each step. Unlike traditional dead-end filtration, which has a declining rate caused by the growth of a filter cake on the surface of the filter medium, in crossflow filtration, the filter cake is swept away by the fluid flowing across it. This filtration method is especially beneficial when there are very fine particles and when system simplicity is required.

The first objective of this work was to test crossflow filtration using actual Envelope D Hanford tank waste (C-104) in a modified cells unit filter (CUF) filtration rig fabricated at Battelle. Similar to the studies with supernatants, the permeability of the diluted C-104 sludge through a single-element 0.1- μm Mott filter was evaluated as a function of trans-membrane pressure, axial velocity, and time for both high and low solids concentrations (Brooks et al. 1999; Hallen et al. 2000). The radioactive tests with the single-element CUF unit will provide information for equipment-performance evaluation and a design basis for a scaled process.

The second object of this work was to evaluate washing and leaching characteristics of the C-104 sludge. The slurried feed was de-watered and then washed multiple times with 0.01 M NaOH to determine the concentration of water-soluble components. It was subsequently leached with NaOH at elevated temperatures to determine the concentration of caustic-soluble components. The chemical and radiochemical composition of the filtrate and the final leached solids was measured to determine the efficiency of the filtration, leaching, and washing processes.

This report describes the test apparatus, the experimental approach, the results of the tests, and the chemical and radiochemical analysis of the sludge from Tank C-104 and filtrates generated during the washing and caustic-leaching steps.^(a) This report also provides a means of transmitting to BNFL the completed test instruction and raw filtration and analytical data.

(a) The results presented in this report are based on work conducted under Test Plan TP-29953-047, test instructions TP-29953-029 and -051, and Procedure TP-29953-020. Some data are recorded in Laboratory Record Book (LRB) #13745. Conditions for conducting these tests were given in the "C-104 and C-106 Caustic Leach Test Specification," TSP-W375-99-00005, Rev 0.

2.0 Test Conditions

Small-scale radioactive crossflow filtration, water-washing, and caustic-leaching tests using slurry samples from Tank C-104 were conducted from 8/24/99 through 8/28/99. The work was performed in the High Level Radiochemistry Facility (HLRF) hot cells located in Radiochemical Processing Laboratory (RPL) facilities.

2.1 Test Material Preparation

Battelle received samples from Tank 241-C-104 from Hanford's 222-S laboratory on March 3, 1999. This material was received in 14 glass jars. Figure 2.1 lists the sample numbers along with the mass of material recovered from each jar. The material in the jars was transferred to a stainless steel mixing vessel equipped with a motorized impeller. Before being used, all components of the mixing vessel were rinsed with methanol and then baked in an evaporation oven at 102°C for 12 h. Materials in the vessel were mixed for 1 h and 20 min before collecting subsamples. The materials were actively mixed while subsamples were collected through a 1.9-cm (.75-in.) ball valve located on the bottom of the vessel. The hot-cell temperature during the mixing process was 34°C.

The first three subsamples (C-104 COMP A, B, and GL) were collected and allowed to settle. After approximately 10 days, the volume of settled solids in these three samples was measured to determine the effectiveness of the sub-sampling technique at collecting samples with representative solids/liquid ratios. It was assumed that after 1 h of active mixing, the settled solids were effectively homogenized. The three subsamples contained 88.9, 89.2, and 89.9 vol% settled solids. Given that these results are within 1 vol%, the sampling technique was determined to provide representative subsamples.

The remaining material in the mixing vessel was then collected in three glass jars during active mixing. These jars were labeled C-104 COMP C, C-104 COMP D, and C-104 COMP E. A significant amount of material remained in the mixing vessel because of a flat bottom. The vessel was rinsed with two 50-mL aliquots of 0.01 M NaOH, and the rinse solution was collected in a jar labeled C-104 RIN. The vessel was then rinsed again with another two 50-mL aliquots of 0.01 M NaOH, and the rinse solution was collected in a jar labeled C-104 RIN 2. The material in the jars labeled C-104 COMP A, C-104 COMP B, and C-104 COMP E was used for "as-received" regulatory analysis. The material in the jar labeled C-104 COMP GL was used for the solubility versus temperature, and small-scale caustic and water-washing tests. CUF process testing utilized the material in the jars labeled C-104 COMP C, C-104 COMP D, C-104 RIN, and C-104 RIN2.

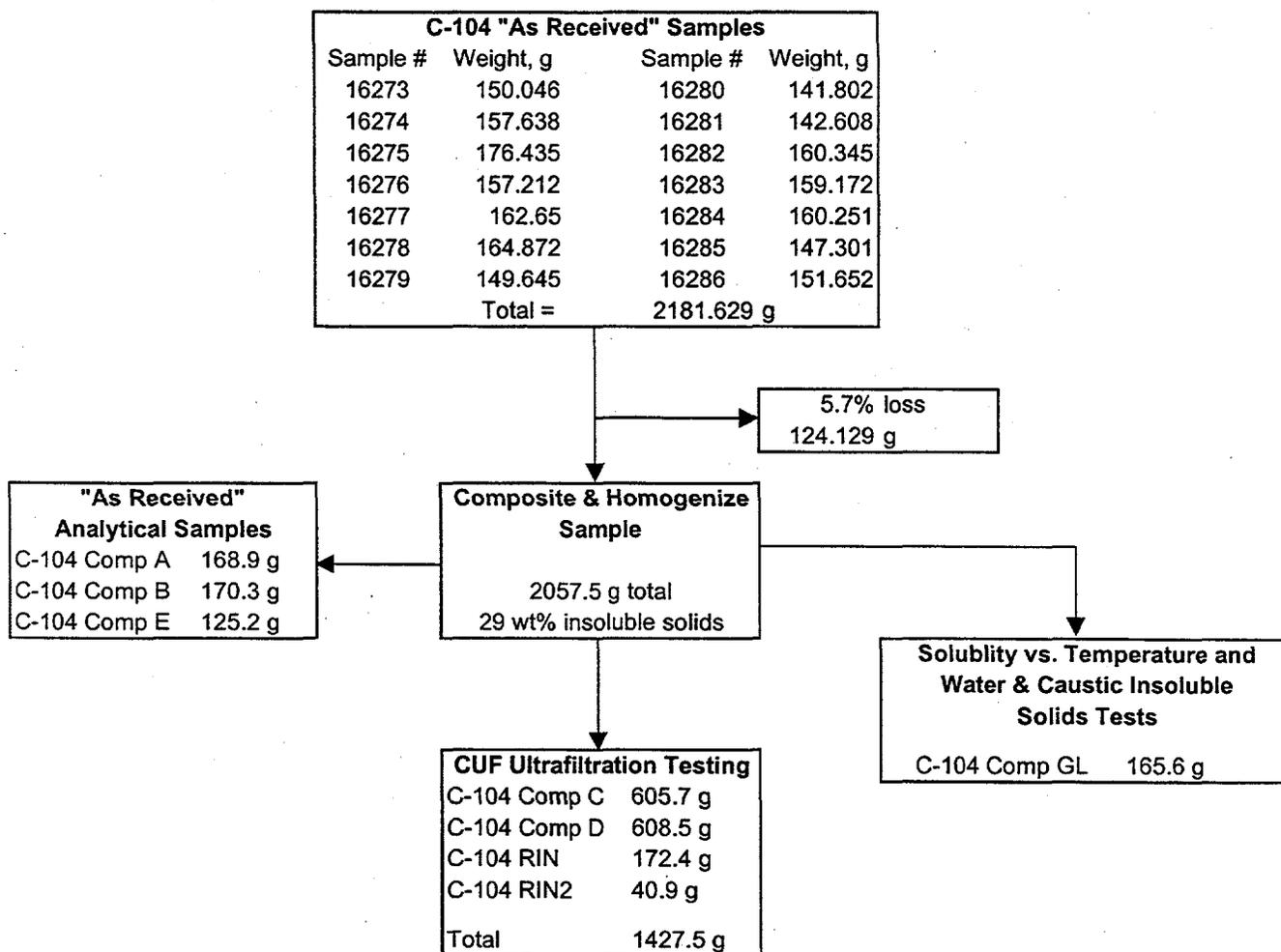


Figure 2.1. Sample Flow Diagram for the C-104 Sludge Before Crossflow Filtration Testing

2.2 Overview of Testing

For the C-104 crossflow filtration experiments, measurements of filtrate flux as a function of trans-membrane pressure (TMP) and centerline crossflow velocity were recorded for both a dilute and a concentrated slurry. The dilute feed was 6.9 wt% solids,^(a) and the concentrated feed was 23 wt% solids. The filtrate was recycled back into the feed tank to maintain the steady-state solids concentration for testing. Each condition was run for 60 min with data taken every 10 min. The system was backpulsed at

(a) Solids concentrations are generally reported on an insoluble solids basis. This is done by mathematically subtracting out the dissolved solids from the sample. If solids are reported on a different basis, they will be delineated as such.

least twice between each condition, but not backpulsed at any time during the condition. The slurry temperature was maintained at $25 \pm 5^\circ\text{C}$ for all filtrate rate testing.

The filtration test conditions are based on an empirically derived matrix to determine the optimum de-watering conditions for the feed slurry. A 5-point matrix around the center-point at 50 psid and 12.2 ft/s tests the conditions of TMP (30 psid, 50 psid, 70 psid) and velocity (9.1 ft/s, 12.2 ft/s, 15.2 ft/s). It is hypothesized that the subsurface filter fouling (that cannot be removed by simple backpulsing) may influence the selection of optimum de-watering conditions as each test is conducted. To incorporate error introduced by the subsurface filter fouling, and to account for such errors in selecting an optimum de-watering condition, the center-point was used for the initial testing conditions and repeated for the final testing conditions.

Following the filtration tests, the slurry was washed at 25°C in the CUF system. This was accomplished through seven consecutive additions of 0.01 M NaOH washing solution to the CUF slurry feed tank, followed by the removal of an equal amount of filtrate using the crossflow filter. The filtrate from these washes was collected in three separate containers, and each was sampled for analysis.

The slurry was then put through a caustic leaching process. The slurry was transferred from the CUF to a separate leaching container, combined with 3.0-M NaOH, heated to 85°C , and held with steady mixing for 8 h. The leached slurry was then transferred back into the CUF, and the slurry was de-watered. Following the leaching step, two 8-hour, 0.01-M NaOH wash cycles were performed at 85°C to reduce the dissolved solids contained in the interstitial liquid.

The final washed sludge was transferred into a storage container for additional HLW pretreatment and vitrification tests. The available solids and the minimum operating volume of the CUF (800 to 900 mL) limited the maximum solids concentration that could be attained in the CUF. All wash solutions and four slurry samples were analyzed for chemical and radiochemical constituents. Additional samples were obtained to determine rheological properties and to verify by in-cell centrifugation the approximate solids loading at specified points. These samples were added back to the system after completion of the procedure when possible.

2.3 Testing Apparatus

Crossflow filtration testing of the feed was conducted on a Battelle-modified CUF, with the following specifications:

- single-tube filter module, 24"-long tube; 3/8" ID
- 0.1- μm Mott liquid-service stainless steel filter
- recirculation flow such that 5 m/s (15 ft/s) maximum linear crossflow velocity can be achieved through the filter tube with water
- maximum trans-membrane pressure 80 psid with water.

A process flow diagram of the CUF is shown in Figure 2.2. The slurry feed is introduced into the CUF through the slurry reservoir. Three baffles are installed in the slurry reservoir to prevent vortex

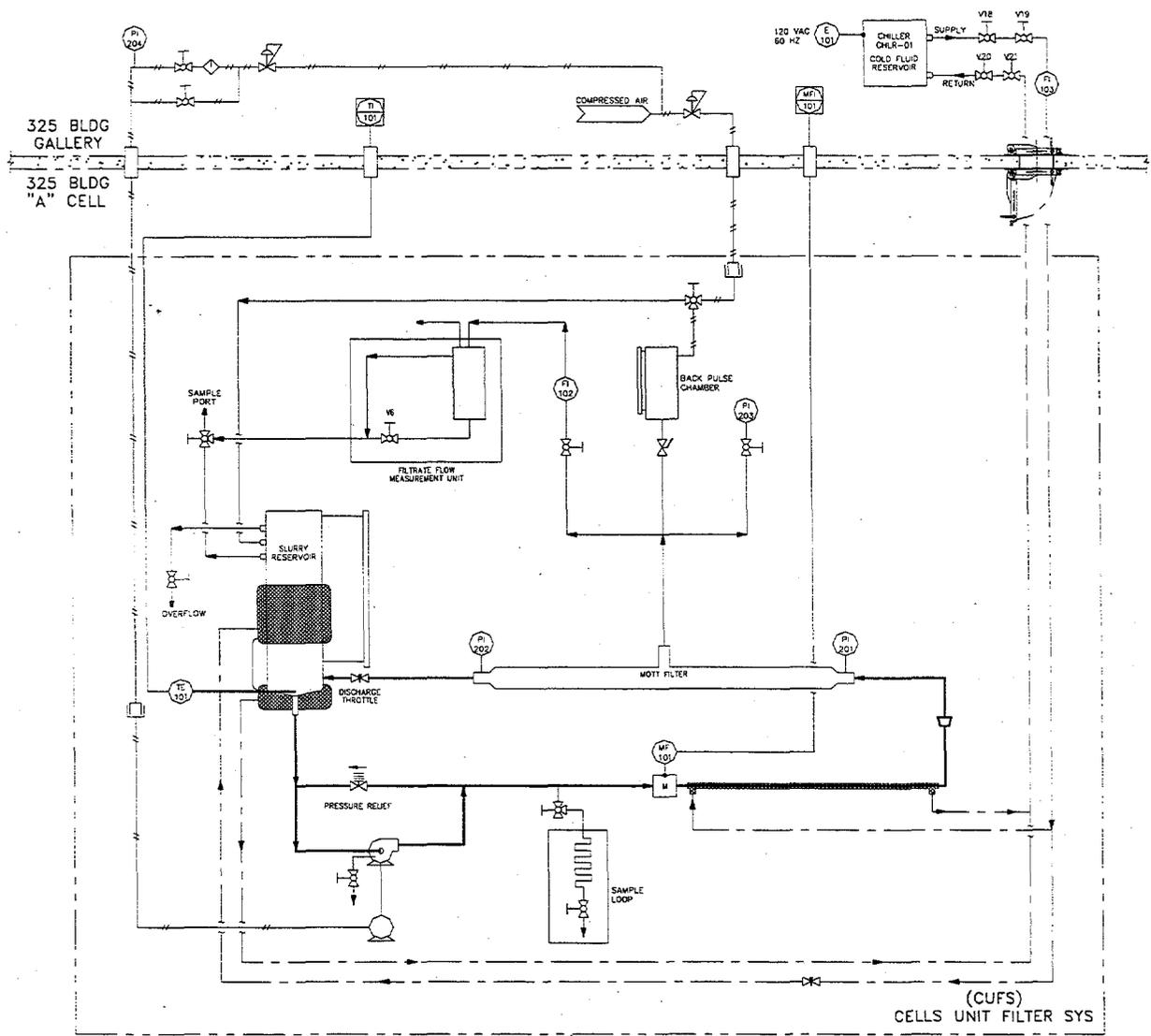


Figure 2.2. Crossflow Filtration Process Flow Diagram

formations. An Oberdorfer progressive cavity pump (powered by an air motor) pumps the slurry from the slurry reservoir through the magnetic flow meter and the filter element. The axial velocity and transmembrane pressure are controlled by the pump speed (which is controlled by the pressure of the air supplied to the air motor) and the throttle valve position. An air booster was added outside of the hot cell to increase the building pressure to the air motor. This was done in an attempt to achieve higher axial velocities during actual slurry testing. The impact of this pressure booster on the transmembrane pressure is described in Section 2.5.

Deionized (DI) water and dilute caustic (0.01 M NaOH) additions into the CUF were made through the chemical addition tank located outside of the hot cell. The chemical addition tank is hard piped into the cell where it is attached to a long piece of flexible tubing that can be gravity drained into the slurry reservoir. Concentrated caustic or acid solutions were added to the CUF using pre-filled polyethylene bottles transferred manually into the cell.

Filtrate that passes through the filter can be either sent to the backpulse chamber, reconstituted with the slurry in the slurry reservoir, or removed. The filtrate flow rate is measured by means of a graduated glass-flow monitor that is fill-and-drain operated. Higher filtrate flow rates can be monitored with an in-line rotameter. Filtrate samples are taken at the three-way valve upstream from the slurry reservoir. This is also the point at which filtrate is removed for the de-watering step. Filter backpulsing was conducted by partially filling the backpulse chamber with filtrate, pressurizing the backpulse chamber with air, and forcing the filtrate in the backpulse chamber back through the filter.

During the majority of the testing in the CUF, the slurry temperature was maintained at $25 \pm 5^\circ\text{C}$ by flowing cooling water in jackets around the slurry reservoir and on the tube between the magnetic flow meter and the filter. The slurry temperature was measured by a thermocouple installed in the slurry reservoir and controlled by a 1000-watt chiller. When filtering leachate and washes at elevated temperatures (85°C), the chiller was turned off. A heat tape surrounding the slurry reservoir and pump inlet tubing heated the slurry to the required temperature. The temperature was then maintained using a proportional-integral-derivative (PID) temperature controller.

The elevated-temperature caustic leaches/washes were performed in a 2-L stainless steel beaker. The slurry in the stainless steel container was continuously stirred with a mixing blade while being heated on a hotplate. A thermocouple, immersed in the slurry, measured temperature and fed the data into the temperature controller. This allowed for automatic temperature control for the 8-h wash cycles. To minimize evaporation loss, a stainless steel lid with a small hole for the mixer shaft was used.

The critical CUF measuring equipment included

- a magnetic flow meter to measure the slurry recirculation rate
- two flowmeters to measure the filtrate flowrate at high and low levels
- three pressure gauges to measure the filter module inlet, filter module outlet, and filtrate pressures
- two thermocouples to measure the slurry temperature in the CUF and in the leaching beaker.

All measuring equipment was calibrated, and the calibration information was recorded in the test instruction document.

2.4 CUF System Verification Testing

Testing to establish a background filtrate flux was conducted with de-mineralized, 0.1 μ m filtered water in the CUF at 20, 10, and 30 psid. The filtrate fluxes for these tests averaged 1.59, 0.95, and 2.38 gpm/ft², respectively. The water testing showed very little filtrate flux reduction over the 30-min testing times. This confirmed that the CUF was sufficiently clean to begin testing with the C-104.

2.5 Experimental Approach

A flowsheet of the testing is shown in Figure 2.3. The test instruction for this work is found in Appendix A, and a mass-balance spreadsheet is provided in Appendix B. The homogenized C-104 Comp C, RIN, and RIN2 were added to the CUF followed by 1458 g of inhibited water (0.01 M NaOH) to bring the insoluble solids concentration down to ~10 wt%. Approximately 1 L of the slurry was removed due to maximum CUF volume limitations. Approximately 1185 g of inhibited water was added to the remaining slurry, creating a solids concentration of 6.9 wt%. The removed slurry was later added back into the CUF, de-watered, and used for the second testing matrix with higher solids loading.

The first testing matrix to determine the optimum de-watering conditions for dilute slurry conditions was run at five combinations of TMP and crossflow velocity with two conditions being repeated to determine filter fouling over the course of testing (see Table 2.1). The conditions below were performed in the order described. The system was backpulsed two to four times between each condition, and only between conditions. It was not backpulsed during the course of any 1-hour condition. The cognizant engineer determined the required amount of backpulses needed based on the drop in flux during the previous condition and on the amount of recovery achieved with the first two backpulses. The filtrate flux results are found in Section 3.1.

An external air-booster was used for the test conditions requiring both a high TMP and a high flow rate. These conditions are noted in the tables with an asterisk. When the air-booster was in use, the air-pressure supplied to the air-motor driving the pump pulsed rhythmically over a 10- to 20-psig range. The pulse frequency was approximately 60 cycles/min. This caused fluctuations in the crossflow velocity and corresponding pulses in the TMP of 5 to 10 psig. During normal operations without the air-booster, the equipment vibrations created vibrations in the pressure gauges of between 0 and 5 psig.^(a)

(a) At the conclusion of the testing, the C-104 slurry was filtered at 50 psid and 7.2 ft/s both with and without the air booster over the course of 10 minutes. No change in filtrate flux was observed due to the pulsations of the air booster.

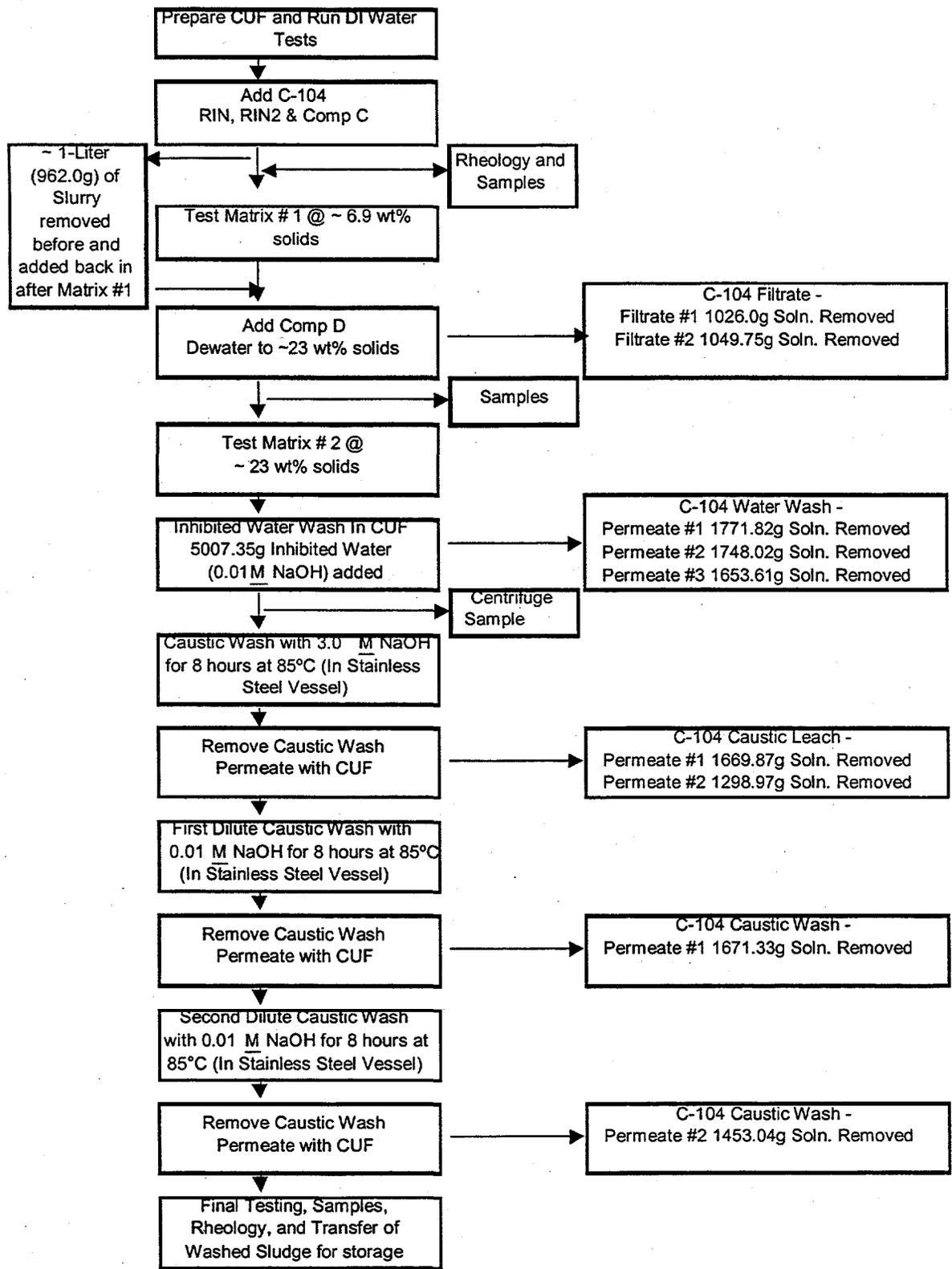


Figure 2.3. C-104 Crossflow Filtration Test Experimental Steps

Table 2.1. Test Conditions for Dilute Slurry^(a)

Condition #	Target Flowrate (ft/s)	Average Velocity (ft/s)	Target Pressure (psid)	Average Pressure (psid)
1	12.2	12.8	50	50.7
2	12.2	12.0	30	30.6
3 ^(b)	12.2	12.1	70	70.2
4	9.1	9.0	50 ^(c)	50.2
5 ^(b)	15.2	15.1	50 ^(c)	49.3
6	12.2	12.3	50	49.9
7	9.1	9.0	50 ^(c)	51.2

- (a) Matrix from pre-October 1999 BNFL experimental design change.
- (b) Air Booster used to attain required pressure and flow.
- (c) Determined to be the optimum pressure condition, based on raw data. Including filter fouling over the course of the study, the optimum pressure condition was later determined to be 30 psid.

After completion of the first test matrix, the slurry was de-watered. The liter of slurry that was removed was re-added to the CUF, and de-watering commenced a second time. Sample C-104 Comp D was added to the CUF, and the combined slurry was de-watered a third time until an estimated pre-wash target of 20 wt % solids was reached. The filtrate from these three de-watering processes was collected in 1-L bottles labeled "Filtrate #1" and "Filtrate #2," and totaled 2075.75 g. Representative samples of these filtrates and the final combined slurry were taken for analysis.

This concentrated slurry with a solids concentration calculated at 23.2 wt% was run through a second test matrix to determine optimum de-watering conditions at higher solids loading. Testing procedures for Matrix 2 were identical to the testing for Matrix 1. The actual and targeted conditions are shown in Table 2.2.

Table 2.2. Test Conditions for High-Solids-Loading Matrix^(a)

Condition #	Target Flowrate (ft/s)	Average Flowrate (ft/s)	Target Pressure (psid)	Average Pressure (psid)
1	12.2	11.24	50	53.0
2	12.2	11.53	30	33.67
3 ^(b)	12.2	11.77	70	71.33
4	9.1	9.09	50 ^(c)	52.43
5 ^(b)	15.2	14.35	50 ^(c)	52.86
6	12.2	11.68	50	50.83

- (a) Matrix from pre-October 1999 BNFL experimental design change.
- (b) Air Booster used to attain required pressure and flow.
- (c) Determined to be the optimum pressure condition, based on raw data. Including filter fouling over the course of the study, the optimum pressure condition was later determined to be 70 psid.

The original test plan called for the slurry to be washed three times after the testing at ~20 wt% solids was completed. Due to maximum volume limitations, it was decided to conduct the wash as a continuous process and collect the wash filtrate progressively in three equal volumes. The total wash volume was divided into seven equal parts of approximately 715 g and added separately to the CUF. After each addition, the system was de-watered by collecting the filtrate until a similar volume was removed. About half of each addition was added through the backpulse chamber to effect two backpulses between each de-watering stage. The remainder of the wash water was added directly into the mixing tank.

During these water washes, approximately 600 g of wash solution or permeate was lost. This resulted in a slurry of approximately 33 wt% solids that did not mix homogeneously in the slurry reservoir and did not produce homogeneous samples. To return the slurry to the correct volume and weight percent solids, 641.11 g of additional inhibited water was added, and new "reconstituted" slurry samples were taken.^(a)

After completion of the water-washing steps, the slurry was removed from the CUF and leached at 85°C with 3 M NaOH for 8 hours. The slurry was pumped into the leaching beaker and then 2899.26 g of 3 M NaOH was divided into two batches and used to rinse residual solids from the CUF before being pumped into the leaching beaker to mix with the slurry. This resulted in a slurry with ~8.6 wt% solids in the leaching beaker. The temperature of the slurry was raised to $85 \pm 5^\circ\text{C}$ and maintained at 85°C for 8 hours. The slurry was continuously stirred with a mixer blade throughout the caustic leaching. Approximately 15 minutes before the end of the leaching time, the heat tape on the CUF was activated to pre-warm the system so that slurry could be maintained at $85 \pm 5^\circ\text{C}$ for the de-watering process. After 8 hours, the slurry was transferred back into the CUF to de-water back to original solids concentration (~20 wt% solids).

The slurry was pumped around in the CUF and heated to bring its temperature back to $85 \pm 5^\circ\text{C}$ before the de-watering process was started. Filtrate flux, TMP, temperature, and flow-rate data were taken every 10 minutes during all de-watering steps when possible.

Following the caustic leaching steps, the solids were washed twice with inhibited water, 0.01 M NaOH. Each inhibited water wash was conducted in the same manner as the caustic leach. Dilute Caustic Wash #1 consisted of 1405.22 g of 0.01 M NaOH added to the slurry. This produced an ~11.8 wt% solids concentration in the leaching beaker. The solution was heated to ~85°C and held for a period of 8 hours, then de-watered to the pre-wash concentration. Dilute Caustic Wash #2 consisted of 1512.18 g of 0.01 M NaOH added to the slurry. The solution was heated to $85 \pm 5^\circ\text{C}$ and held for a period of 8 hours, then de-watered at $85 \pm 5^\circ\text{C}$ to the pre-wash concentration. The extent of de-watering was limited by the minimum volume of the CUF, approximately 800 mL. Duplicate sub-samples of each permeate were taken and analyzed for soluble components removed.

The slurry was drained and collected in a 2-L bottle. Approximately 1018 g of washed and leached C-104 sludge (wet) was collected from the CUF. The CUF was rinsed twice with 1000 mL of DI water, and the solids from this were also collected and saved.

^(a) Mass balance results on individual analytes indicate that the wash solution lost contained very little if any non-water components. Whether it was water not added or permeate lost is unclear.

The CUF was then rinsed multiple times with water to remove all of the remaining solids and to attempt to recover the initial clean water fluxes. To assist in cleaning the filter, the water was added to the backpulse chamber and forced backward through the filter. When the water being removed was relatively clean, an external cartridge filter with a 0.05- μm rating was attached to the system. Roughly 2/3 of the flow continued through the CUF, and the remaining 1/3 of the flow was circulated through the cartridge filter in an attempt to remove the remaining solids. This recirculation continued for several hours while intermittently backpulsing. At the conclusion of this cleaning step, the filtrate flux was measured with clean water. The filtrate flux was significantly below that measured before the C-104 test, and it was determined that acid cleaning would be required.

One liter of 1 M HNO_3 was backpulsed into the CUF and allowed to recirculate for several hours. The nitric acid was then drained from the CUF and found to be very dark and full of solids. A second batch of 1 M HNO_3 was added to the CUF through the backpulse chamber and allowed re-circulate through the system. This second batch of nitric acid was considerably cleaner. The system was then rinsed until a neutral pH was obtained, and the clean-water flux was measured again at 20, 10, and 30 psid. The resultant fluxes over 20 min were 1.38, 0.84 and 1.77 gpm/ft^2 , respectively. These values are approximately 12 to 25% lower than the initial fluxes. Further cleaning in the plant may be required to maintain a high clean water flux. In the CUF system, the acid can damage the pump stator, so less extensive cleaning is possible.

2.6 Sample Analyses

The sample names, descriptions and associated analyses are shown in Table 2.3. For both permeates and slurry samples, analyses included

- total organic carbon (TOC) and total inorganic carbon (TIC)
- ion chromatography (IC) (for soluble anions)
- inductively coupled plasma-atomic emission spectroscopy (ICP-AES) (for metals)
- gamma energy analysis (GEA) (^{137}Cs , ^{154}Eu , ^{155}Eu , ^{241}Am)
- strontium chemical separation followed by beta counting (^{90}Sr)
- inductively coupled plasma-mass spectrometry (ICP-MS) (for ^{99}Tc)
- alpha emission analysis (AEA) (for ^{241}Am , ^{243}Cm , ^{244}Cm)
- total alpha analysis
- laser fluorescence (for total U).

Table 2.3. Samples and Analyses Performed

Sampling Step	Sampling Number	Sample Type	Analysis
"Initial Slurry Sample" ^(a) (Before Matrix 1)	CUF-C104-001	Slurry	PSD
	CUF-C104-002		Physical Properties
	N/A		Rheology
"Initial Permeate" (First De-Watering After Matrix 1)	CUF-C104-019	Permeate	Chemical and Radiochemical
"Initial Permeate" (Second De-Watering After Matrix 1)	CUF-C104-005	Permeate	Chemical and Radiochemical
"Water Wash Permeate 1"	CUF-C104-016	Permeate	Chemical and Radiochemical
"Water Wash Permeate 2"	CUF-C104-017	Permeate	Chemical and Radiochemical
"Water Wash Permeate 3"	CUF-C104-018	Permeate	Chemical and Radiochemical
"Washed Slurry" (After Water Wash, high solids loading)	CUF-C104-006	Slurry	Physical Properties
	CUF-C104-007		PSD
	N/A		Rheology
"Intermediate Slurry" or "Reconstituted Washed Slurry" (After Water Wash, diluted slurry)	CUF-C104-009	Slurry	Physical Properties
	N/A		Rheology
	CUF-C104-010		Chemical and Radiochemical
"Caustic Leach Permeate"	CUF-C104-011	Permeate	Chemical and Radiochemical
"Caustic Wash Permeate 1"	CUF-C104-021	Permeate	Chemical and Radiochemical
"Caustic Wash Permeate 2"	CUF-C104-022	Permeate	Chemical and Radiochemical
"Final Slurry Sample"	CUF-C104-012	Slurry	PSD
	CUF-C104-013		Chemical and Radiochemical
	CUF-C104-014		Chemical and Radiochemical
	CUF-C104-Final Rheology		Rheology & Physical Properties
Final Decanted Supernatant	CUF-C104-014	Permeate	Acid Digest/ICP-AES (for Na)

(a) Names in quotes are the names of the samples used in this report.

The slurry samples were prepared by both acid digestion and KOH fusion to obtain complete dissolution as well as measure the K and Ni concentration. A precious metals group (PMG) fusion was also performed to obtain the Pt concentration in the slurry. In addition to the above analysis, the following additional analyses were performed only on the solids:

- ICP-MS (for ²³⁷Np, ¹²⁶Sn, ¹²⁹I, Pr, Rb, Ta, Pt)
- extraction and Beta Count (for ³H)
- combustion release and beta count (for ¹⁴C)
- cold vapor atomic adsorption spectroscopy (for Hg)

- cyanide and ammonia concentrations.

The physical analyses of the slurries included density, weight percent dissolved and undissolved solids, and volume percent settled and centrifuged solids. The results of these analyses, along with a further description of the experimental steps, are provided in Section 3.3. The rheological work measured shear stress as a function of shear rate. The results of this work, along with a further description of the experimental steps, are provided in Section 3.4. The particle size distribution (PSD) measurements were performed for selected samples using Microtrac X-100 and Microtrac UPA particle analyzers. The results of this work along with a further description of the experimental steps are provided in Section 3.5.

3.0 Results and Discussion

This results and discussion section is divided into five subsections: crossflow filtration results, water washing and caustic leaching results, physical property results, rheological results, and PSD results.

3.1 Crossflow Filtration Results

3.1.1 Low Solids Loading Matrix Results

The low solids loading matrix test consisted of seven conditions. All were performed at 6.9 wt% insoluble solids concentration, and each was 1 h in duration. The average filtrate fluxes from these conditions are shown in Table 3.1. A graph of the filtrate flux as a function of time for all seven conditions is shown in Figure 3.1. The high initial flux rates drop within a few minutes to a lower, more consistent flux rate that slowly decreases over time. For comparison of test conditions, the flux rate is averaged over the 1-h run time, except for the first 10 min of operation. All the flux data presented in this section have been corrected to 25°C using the following formula provided by BNFL to correct for viscosity changes:

$$Flux_{25C} = Flux_T e^{2500 \left(\frac{1}{273+T} - \frac{1}{298} \right)} \quad (3.1)$$

where $Flux_{25C}$ is the corrected filtrate flux, and T is the temperature (°C) at the flux measurement ($Flux_T$). All of the raw data for the filtrate flux measurements are included in Appendix D.

Table 3.1. Average Filtrate Flux for Low Solids Matrix (~6.9 wt%)

Condition #	Average Velocity (ft/s)	Average Pressure (psid)	Average Filtrate Flux (gpm/ft ²)
1	12.8	50.7	0.083
2	12.0	30.6	0.080
3 ^(a)	12.1	70.2	0.057
4	9.01	50.3	0.038
5 ^(a)	15.1	49.3	0.081
6	12.3	49.9	0.065
7	8.95	51.2	0.051

(a) Conditions #3 and #5 required the air booster to achieve the required pressure and flow.

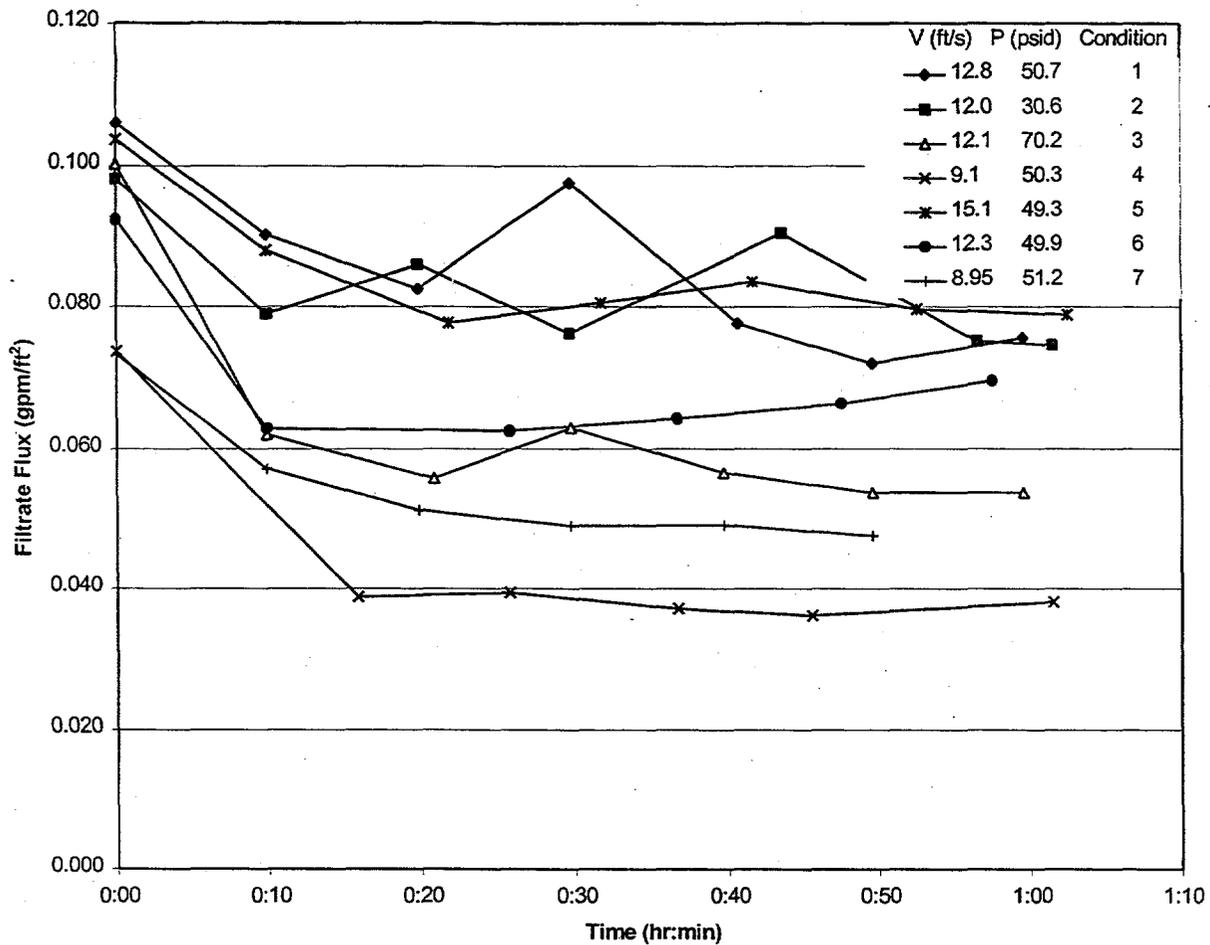


Figure 3.1. Filtrate Flux as a Function of Time for the Low Solids Matrix

The initial condition (50.7 psid, 12.8 ft/s) had the highest average filtrate flux. As with previous crossflow filtration studies (Brooks et al. 1999, Geeting & Reynolds 1997) on Hanford tank wastes, during each condition and between conditions, the filtrate flux steadily decreases over time. This is evidenced by the decrease in filtrate flux that was observed in Conditions #1 and #6. With nearly identical pressure and velocity, Condition #6 (49.9 psid, 12.3 ft/s) had a filtrate flux 21% lower than the initial condition. Condition #4 (50.3 psid, 9.01 ft/s) and Condition #7 (51.2 psid, 8.95 ft/s) are also nearly identical in pressure and velocity. In this case, there is actually an increase in filtrate flux between these two conditions. These results may indicate that Condition #2 (30.6 psid, 12.0 ft/s) and more likely Condition #3 (70.2 psid, 12.1 ft/s) produced most of the overall decrease in filtrate flux and thus the irreversible fouling of the filter.

The initial, final, and average filtrate flux results are shown in Figure 3.2. The first three conditions show the effect of trans-membrane pressure on the filtrate flux. While the initial filtrate flux is nearly identical for all three conditions (suggesting no significant fouling of the filter), the average and final filtrate flux of the 70-psid condition (#6) is much lower than 30 and 50 psid. It appears that higher pressures may compact the filter cake or foul the filter, reducing the benefits of increased pressure. It is also interesting to note that the lower axial velocity Conditions #4 and #7 both have a significantly lower initial as well as average filtrate flux. It appears that the lower axial velocity prevents the complete removal of surface fouling during backpulsing, reducing the initial filtrate flux.

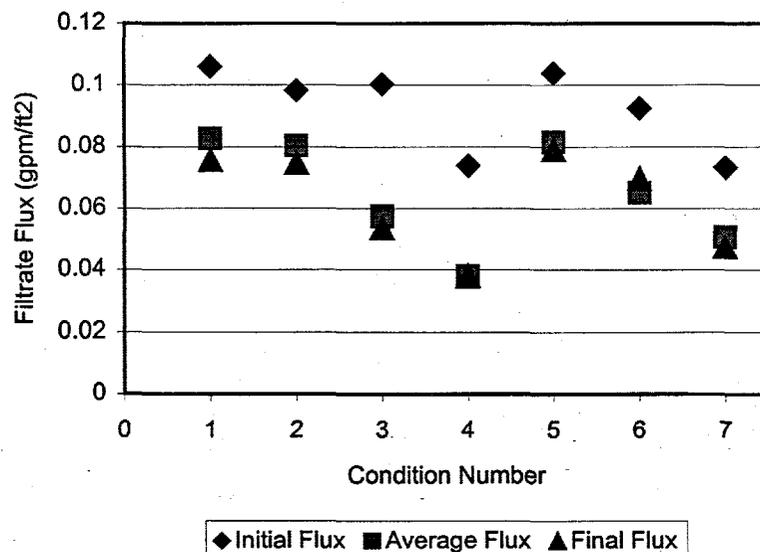


Figure 3.2. Initial, Average, and Final Filtrate Flux for Each Condition in the Low Solids Matrix

In spite of the overall decreasing trend in the filtrate flux, the influence of trans-membrane pressure and axial velocity can be seen. Between 50 and 30 psid (Condition #1 and #2), there is little decrease in filtrate flux, and there is a substantial decrease when the trans-membrane pressure is increased to 70 psid (Condition #3). The effect of pressure can best be seen as a function of permeability. In Figure 3.3, permeability is plotted as a function of pressure for the first three data points. These three points are nearly linear with a two-fold increase in pressure, resulting in a 3-fold decrease in permeability.

In contrast, higher axial velocities show a significant increase in filtrate flux for similar trans-membrane pressure. The higher axial velocities at constant trans-membrane pressure were measured for Conditions #4 through #7. The results of these tests are shown in Figure 3.4. There is a linear impact of initial and average filtrate flux with respect to axial velocity. Higher axial velocities produced higher filtrate fluxes. Overall, a 40% increase in axial velocity resulted in a 40% increase in filtrate flux. Higher axial velocities also improve the recovery of the backpulse following a 1-h test condition. These results

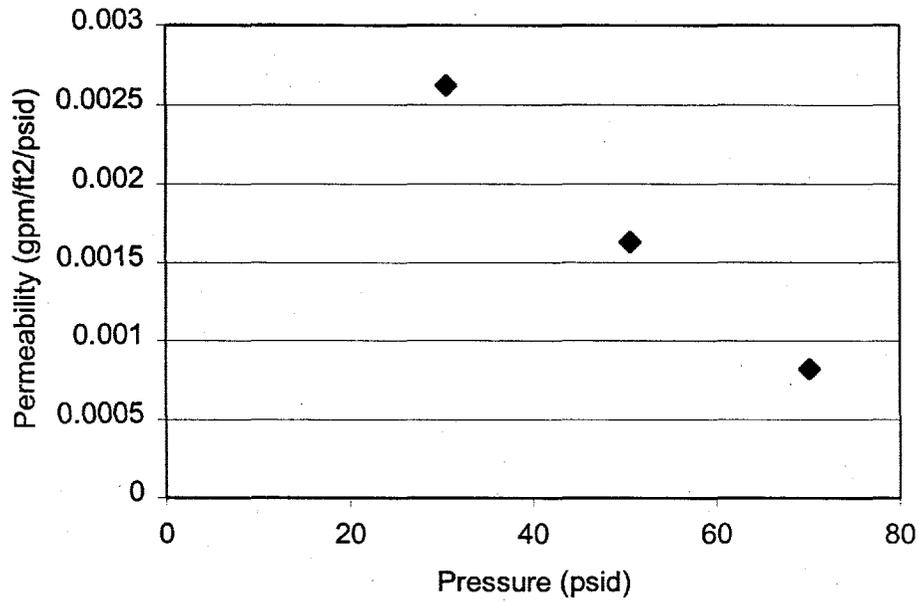


Figure 3.3. Average Permeability versus Pressure for the Low-Solids-Loading Matrix at Approximately 12 ft/s Axial Velocity

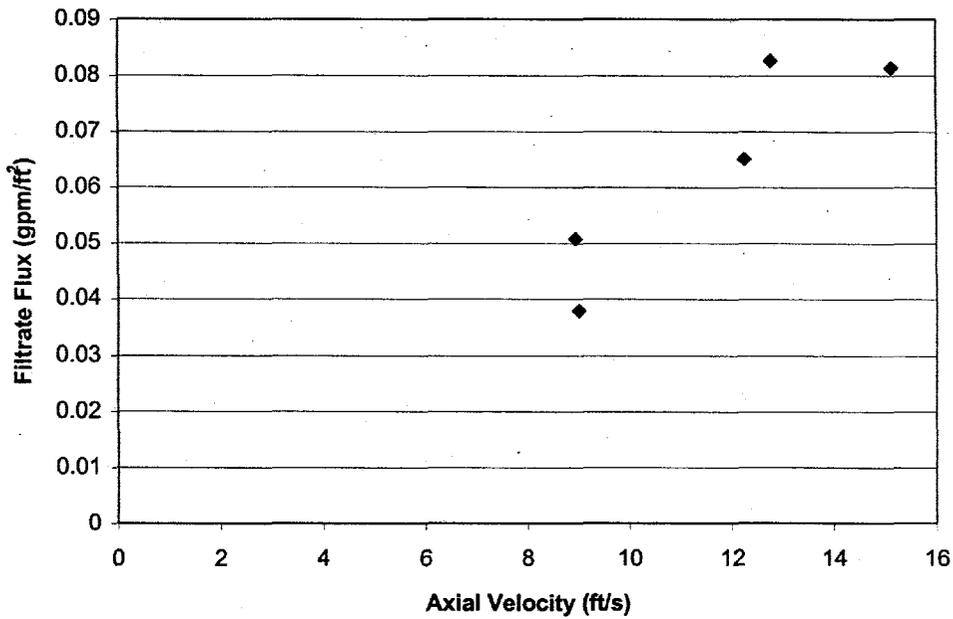


Figure 3.4. Average Filtrate Flux as a Function of Axial Velocity for the Low-Solids-Loading Matrix at Approximately 50 psid Transmembrane Pressure

would indicate that at 6.9 wt% solids, particle convection from the surface of the filter membrane dominates the filtration rate.

3.1.2 De-Watering from Low to High Solids Loading

The slurry was de-watered in two steps, once on the initial slurry containing C-104 RIN 1 & 2 and C-104 COMP C, and again on the slurry after it contained all C-104 sludge material to be used, including C-104 COMP D. The initial de-watering removed approximately 1500 mL of supernatant over the course of 28 min for an average flux of 0.072 gpm/ft². The filtrate flux for this dewatering step is within 10% of the flux for Condition #6 (49.9 psid, 12.3 ft/s) of the low-solids-loading matrix. The solids concentration varied from 6.9 wt% to approximately 17 wt% solids over this time. The second de-watering step removed an additional 500 mL of supernatant. This de-watering step required approximately 43 min. The filtrate flux for this step was measured at 0.0175 gpm/ft². During this de-watering, the solids concentration ranged from an estimated 16 to 20 wt% solids. The average filtrate flux during this step of dewatering was 35% higher than the initial condition for the high-solids-loading matrix.

3.1.3 High-Solids-Loading Matrix Results

The second filtration matrix was performed after the slurry had been de-watered to a calculated solids concentration of 23.2 wt% solids. This matrix consisted of six conditions. The first and last conditions were repeated to evaluate filter fouling during the course of the testing. The average filtrate fluxes for this matrix are shown in Table 3.2. The filtrate fluxes as a function of time are shown in Figure 3.5. As done previously, each condition was performed over the course of 1 h with 3 to 4 backpulses between each condition. During the de-watering step between these two test matrices, the outlet pressure gauge became plugged. Thus, the pressures provided in this discussion are only for the filter inlet and not the filter outlet pressure (typically 1 to 4 psid lower than the inlet). Once again, to achieve the correct pressure and velocity for Conditions #3 and #5, the air booster was used, resulting in slight fluctuations in pressure and velocity.

Table 3.2. Average Filtrate Flux for High-Solids Matrix (~23 wt%)

Condition #	Average Velocity (ft/s)	Average Pressure (psid)	Average Filtrate Flux (gpm/ft ²)
1	11.2	53.0	0.013
2	11.5	33.7	0.014
3 ^(a)	11.8	71.3	0.014
4	9.09	52.4	0.010
5 ^(a)	14.4	52.9	0.017
6	11.7	50.8	0.0095

(a) Conditions #3 and #5 required the air booster to achieve the required pressure and flow. This resulted in greater fluctuations in pressure and flow during the test than were seen during other conditions.

Unlike the low-solids matrix, the highest average flux did not occur at the first condition. Instead, it occurred at the condition of highest axial velocity. Furthermore, Condition #2 and #3 both had higher average filtrate fluxes. This may indicate incomplete backpulsing following the de-watering and before starting Condition #1. Once again, the average filtrate flux appeared to decrease over the entire matrix as evidenced in the decrease in filtrate flux that was observed in Conditions #1 and #6. With nearly identical pressure and velocity, Condition #6 had a filtrate flux 27% lower than Condition #1.

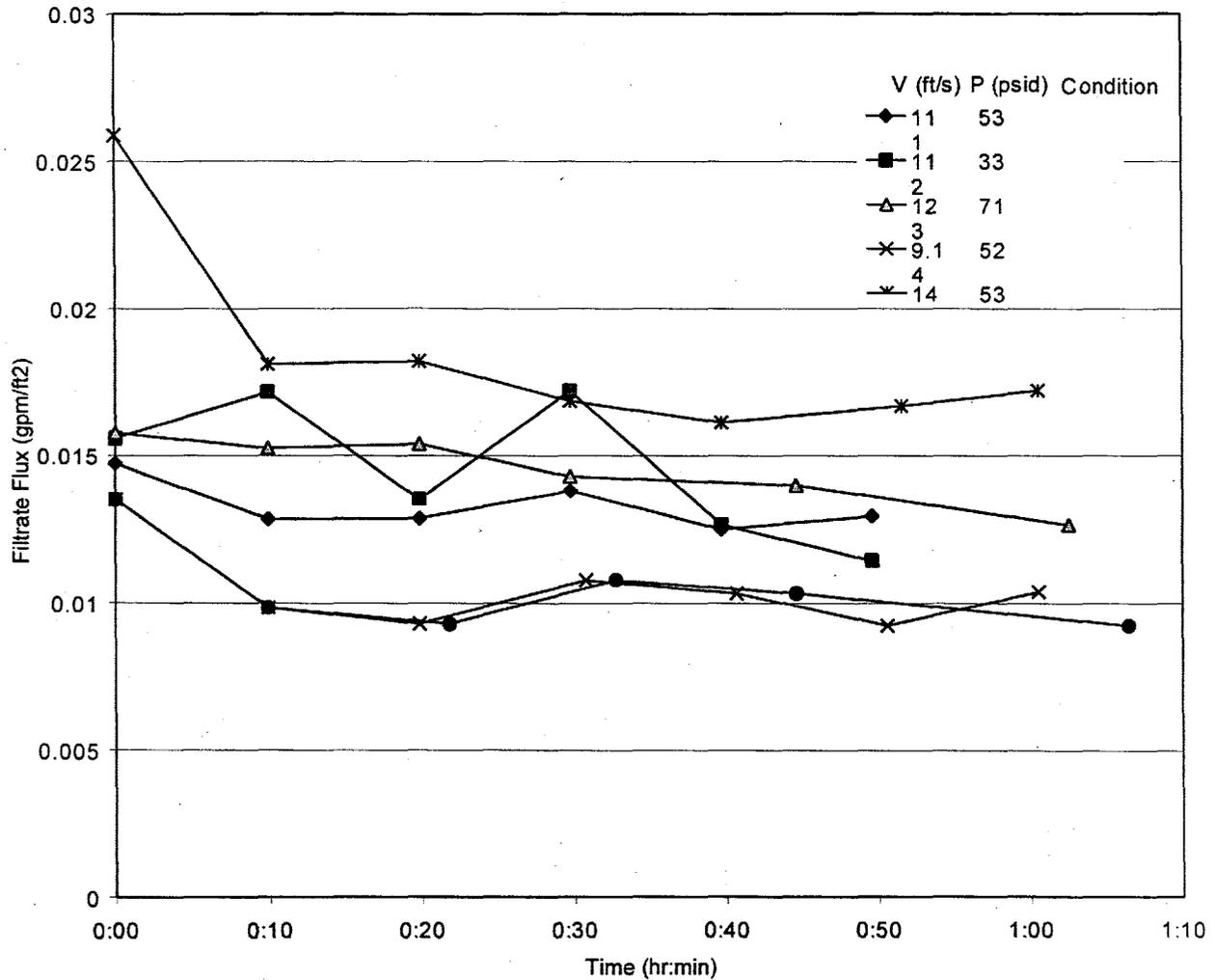


Figure 3.5. Filtrate Flux as a Function of Time for the High Solids Matrix

The initial, final, and average filtrate-flux results are shown in Figure 3.6. For the first four conditions, there is little difference between the initial, average, and final filtrate fluxes. In contrast, the low solids matrix exhibited a 50% decrease between the initial and average flux as compared to only 25%

for the high-solids matrix. The initial filtrate fluxes also appeared to be controlled significantly by the axial velocity. Apparently, the higher axial velocities prevent significant filter cake build-up throughout the test.

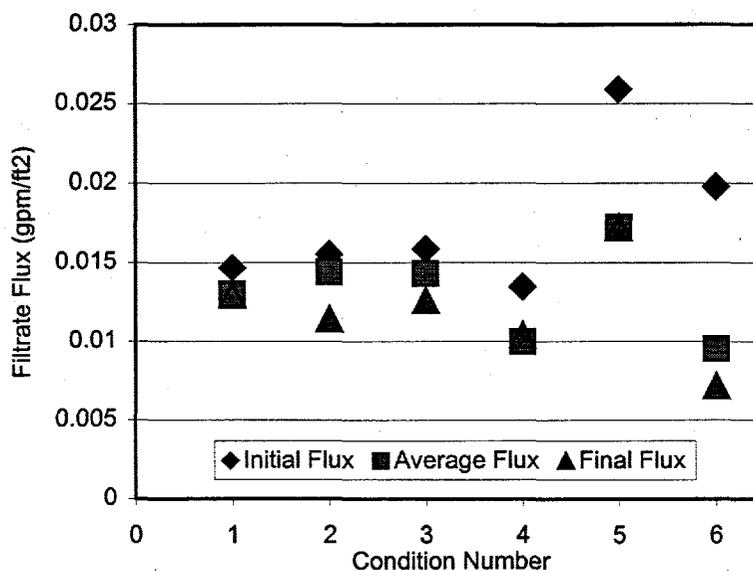


Figure 3.6. Initial, Average, and Final Filtrate Flux for Each Condition in the High-Solids Matrix

The filtrate fluxes for this test were significantly lower than those seen in the low-solids matrix. With the 3.3-fold increase in solids concentration, the filtration flux decreased 5 fold (see Figure 3.7).

Similar to the low solids matrix, the axial velocity has a significant effect on both the average and initial filtrate flux for the high solids loading matrix. The filtrate flux is presented as a function of axial velocity for the first five conditions (Figure 3.8). In spite of pressure variations, the data are nearly linear. Only the final data point falls outside of this trend. Transmembrane pressure, on the other hand, has little to no effect at all on filtrate flux at this solids loading. Within experimental error, fluxes for 30, 50, and 70 psid have nearly identical average filtrate fluxes.

3.1.4 Filtration Results During the Washing and Caustic Leaching Steps

Washing with dilute caustic was performed in seven steps. In each step, approximately 715 mL was added to the CUF, and then an equal quantity of filtrate was removed. During each of the steps, in theory, the solids concentration should increase from approximately 17% up to 23%. The filtrate flux curves for these seven steps are presented in Figure 3.9. As can be seen from the data, de-watering steps 1, 2, 3, and 4 are nearly identical in filtrate flux over time. De-watering Step 5 and especially 6 and 7 are significantly lower. As discussed in Section 2.5, at the end of the seven dewatering steps, the volume was ~600 mL lower than expected. This resulted in the final solids concentration being 33 wt% rather than

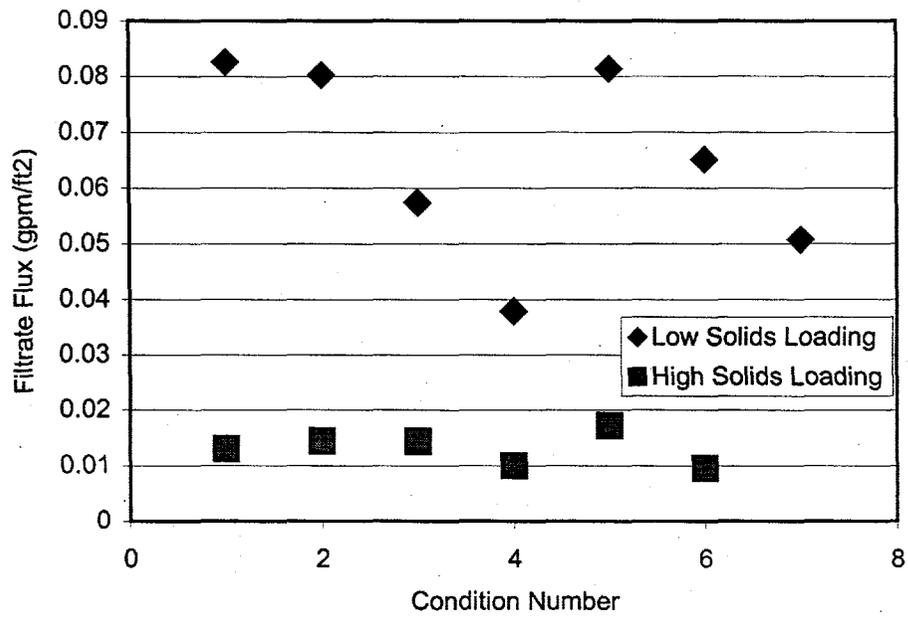


Figure 3.7. Filtrate Flux Comparison between High and Low Solids Loading Matrices

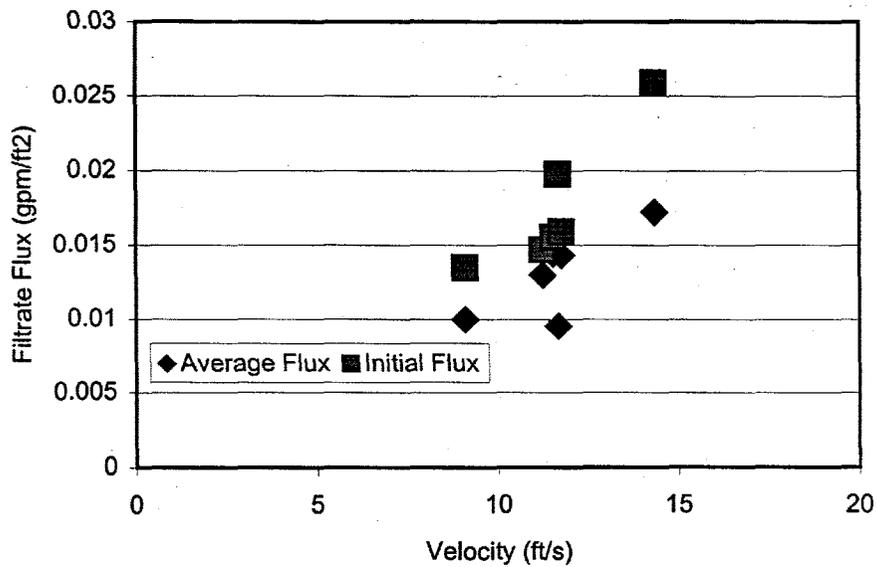


Figure 3.8. Average and Initial Filtrate Flux as a Function of Velocity for the High-Solids-Loading Matrix

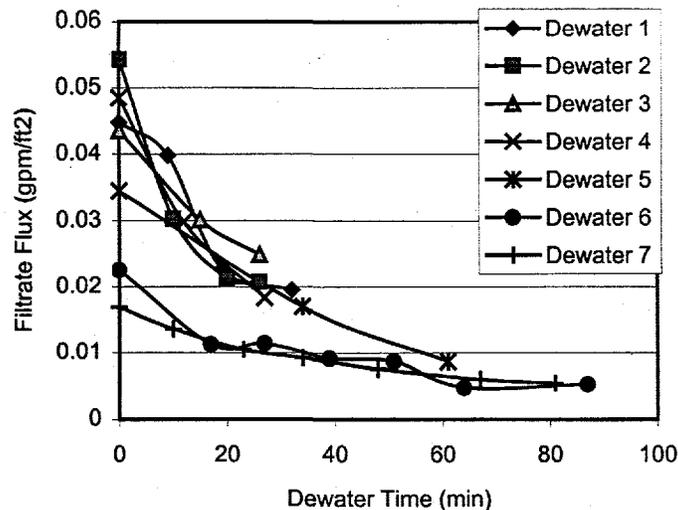


Figure 3.9. Filtrate Flux as a Function of Time for De-watering During Washing Steps

23 wt%. This higher solids concentration due to the material loss may be the cause of the lower filtrate flux during Steps 6 and 7.

Filtrate flux data was also taken during the caustic leach de-watering. During these tests, the filtration was performed at elevated temperatures. The filtrate flux rates at 85°C, not corrected for the elevated temperature, are shown in Figure 3.10. During this test, caustic leach slurry (4 L total) was added in three batches to the CUF for de-watering. Overall, the insoluble solids concentration was increased from ~8.6 to ~22.5 wt% solids. The filtrate flux was much higher than matrices and de-watering done previously. Using the correction in Equation 3.1, the filtrate flux is reduced by 50%. However, the filtrate flux values were still higher than previous flux data. Similarly high filtrate fluxes were also seen during the 85°C de-watering steps following the caustic wash steps. While higher temperatures may be beneficial for filtration, after several days, white flaky solids were found in caustic leach permeate from this de-watering step. These are probably the result of aluminum precipitating out of solution after the permeate was allowed to cool to ambient temperature (~35°C), probably in the form of $Al(OH)_3$. The caustic leach permeate and solids were archived for possible future analysis. No solids were seen in other samples including the two washes after the caustic leach.

3.1.5 Statistical Analysis

The goal of the statistical analysis is to determine the error associated with these tests and to develop a model that best predicts the average flux for C-104 over the range of conditions studied.

By comparing how well each of the filtrate-flux measurements are to be repeated, the pure error of the measurement technique can be estimated. For each test condition, seven measurements were taken at

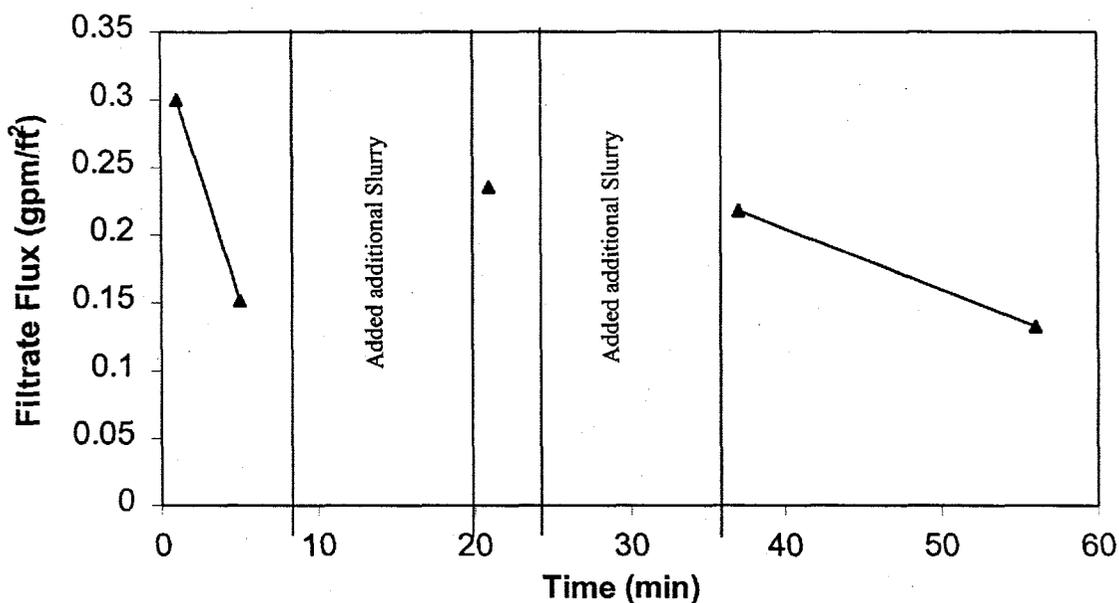


Figure 3.10. Filtrate Flux as a Function of Time for De-Watering During Caustic Leaching
 (Note: Due to CUF volume constraints, during filtration, additional slurry was added twice during dewatering.)

the same relative time over a period of 1 hour. If it is assumed that the change in flux (absolute) with respect to time after the first 10 minutes is considered to be constant, the error can be calculated. While it is true that the flux does continue to decrease even after the first 10 min, the values calculated in this way provide an upper bound on the possible error in the measurements. The idea behind calculating pure error is that repeated measurements should be identical. The differences found from repeated measurements provide a means of estimating the error associated with measurement. In this experiment, since replicates were not available, points that were "close" were treated as replicates, and an approximate pure error calculated. The results of the error calculation for each matrix individually and for the combined set are shown below.^(a)

- low solids matrix only (7 points): 0.0017 gpm/ft²
- high solids matrix only (6 points): 0.0004 gpm/ft²
- both matrices (13 points): 0.0009 gpm/ft²

(a) The calculation is done by subtracting the mean of the data points taken at the same location (replicates) from each raw measurement, squaring those differences, adding them up, and dividing that total by the number of degrees of freedom.

A statistical model can be used to understand the important factors, predict filtrate-flux performance, and eliminate effects particular to the CUF test and equipment that would not be seen in actual operation (i.e., run number). Four possible factors were evaluated: linear velocity in ft/s (Velocity), pressure in psid (Pressure), time or run order in hours (Run), solids concentration given as fraction of insoluble solids (Solids), or any combination of those variables. The following assumptions are used for fitting these models:

- The fixed components of the errors are negligible. That is, the errors have a zero mean.
- The errors are mutually uncorrelated, or their covariances are zero. This means that the value of one error does not depend on or help determine the value of any other error.
- Though generally unknown, the variances of the errors are equal.
- The errors are normally distributed.

A model was developed that incorporated both the low and high solids loading matrix and was found to take on the following form:

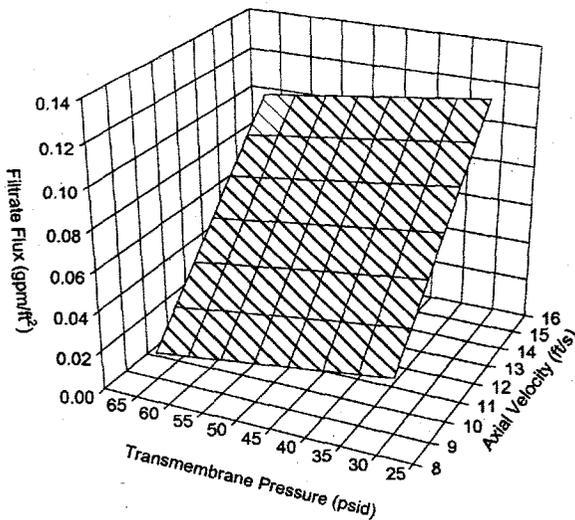
$$\text{Flux (gpm/ft}^2\text{)} = -0.03711 + 0.022206*\text{Run} - 1.116*\text{Solids} + 0.01298*\text{Velocity} - 0.00063*\text{Pressure} \dots \\ \dots - 0.00199*\text{Run}* \text{Velocity} + 0.05746*\text{Solids}* \text{Velocity} + 0.00313*\text{Solids}* \text{Pressure}.$$

The model was a good fit, yielding a high R^2 of 0.9959 and a low mean squared error (MSE) of 0.00292. For a model to be considered a good prediction model, it is desired, among other things, that the R^2 should be as close to 1 as possible, while at the same time, the MSE should be as low as possible.

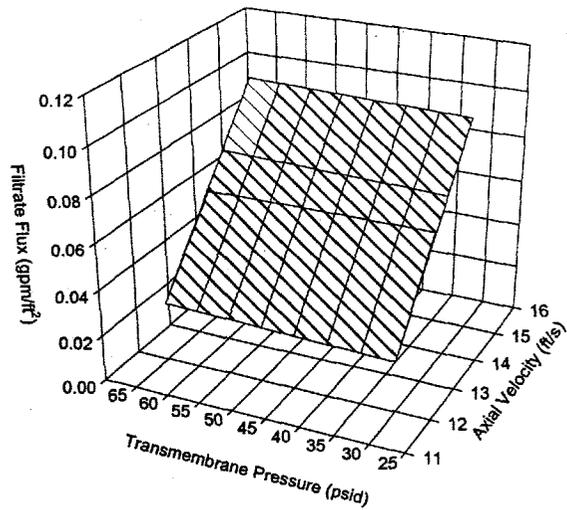
This model shows, as would be expected, that solids loadings and pressure have a negative relationship to the filtrate fluxes, and axial velocities possess a positive relationship with the filtrate fluxes at the solids loadings tested. Although it's not intuitive, the requirement of the second order terms in the model show that an interaction exists within the factors and their relationship with filtrate flux in order to give a better prediction of filtrate fluxes. Simply using a model without the interaction information would yield an inferior model in terms of having more unexplained error, as opposed to the chosen model, which has very little unexplained error.

This model was used to determine the optimum conditions without the effect of run order for both solids concentrations. In the case of 7 wt% solids, the optimum filtration condition is 30 psid and 15 ft/s. At 23 wt% solids, the optimum condition is 70 psid and 15 ft/s. In both cases, but especially at 23 wt% solids, the pressure effect is very small. This can be seen in Figure 3.11. Filtrate flux is a strong function of axial velocity and a weak function of pressure in both cases.

This model should allow an estimate of the filtrate flux for tests run between 1 and 13 hours, for C-104 solids concentrations between 7 and 23 wt%, and over the transmembrane pressures and axial velocities studied. It should not be used for conditions outside these parameters, especially since it is an empirical and not theoretical model.



(a)



(b)

Figure 3.11. Filtrate Flux model for constant run number at (a) 7 wt% and (b) 23 wt%. Note that the highest filtrate flux occurs at lowest pressure with 7 wt% solids as compared to the highest pressure at 23 wt% solids.

3.2 Sludge Washing and Caustic Leaching Results

The chemical and radiochemical analyses obtained from the slurry-washing and caustic-leaching test are presented in this section. Slurry samples were taken on the initial feed after all C-104 sludge was added to the CUF, following the three water washes, and at the conclusion of the tests. Liquid samples were taken on all permeates removed during the course of testing. The results of these analyses are shown in Table 3.3.

Table 3.3. Non-Radioactive Component Concentrations

Analyte	Initial Slurry Sample µg/g	Initial Permeate µg/mL	Water Wash Permeate No. 1 µg/mL	Water Wash Permeate No. 2 µg/mL	Water Wash Permeate No. 3 µg/mL	Intermediate Slurry Sample µg/g	Caustic Leach Permeate µg/mL	Caustic Leach Permeate No. 1 µg/mL	Caustic Leach Permeate No. 2 µg/mL	Final Slurry Sample µg/g
Ag	996	<0.08	<0.08	<0.08	<0.08	979	<0.4	<0.4	0.16	1895
Al	125,000	207	324	214	142	155,667	15,600	7770	2650	36700
As	<261	<0.4	<0.4	<0.4	<0.4	97	<2	<2	1.4	<173
B	250	3.4	3.9	3.5	2.9	476	7.1	3.3	2.8	52
Ba	238	0.086	1.2	0.23	0.15	218	<0.3	<0.3	<0.09	426
Be	31	<0.05	<0.05	<0.05	<0.05	40	1.1	0.29	<0.05	58
Bi	<105	0.61	2	1.2	1.2	40	<3	<3	1.9	71
Ca	4295	<0.5	1.5	<0.7	0.52	5033	<3	<3	<0.5	8547
Cd	747	0.45	0.38	0.1	<0.08	921	<0.4	<0.4	<0.08	1669
Ce	575	<0.5	<0.5	<0.5	<0.5	570	<3	<3	<1	1868
Co	62	0.22	0.38	<0.1	<0.1	11	<0.6	<0.6	0.2	58
Cr	1495	7.4	13	4.5	2.3	1717	59	58	28	1953
Cu	164	0.56	0.78	0.14	1.5	237	0.51	<0.4	0.23	465
Dy	31	<0.3	<0.3	<0.3	<0.3	24	<1	<1	<0.3	76
Eu	<105	<0.5	<0.5	<0.5	<0.5	<54	<3	<3	<0.5	32
Fe	38,450	<0.1	0.34	<0.1	9.41	48,567	3.4	1.8	0.81	89,029
Hg	21	na	na	na	na	22	na	na	na	32
K	<992	42	73	14	<10	<1087	51	<50	28	500
La	110	<0.1	<0.1	<0.1	<0.1	160	<0.6	<0.6	0.2	294
Li	404	3.4	2.6	1.1	0.34	532	30	13	5.1	478
Mg	430	<0.5	0.54	<0.5	0.5	759.7	<3	<3	<0.8	1066
Mn	8830	<0.03	<0.03	<0.03	0.062	10,933	<0.1	<0.1	0.039	19,671
Mo	36	1	1.4	0.44	0.23	27	1.7	0.95	0.56	31
Na	112,500	12,500	18,450	7015	3595	34,567	46,500	21,050	8005	58,529
Nd	230	<0.5	<0.5	<0.5	<0.5	300	<3	<3	<1	558

Table 3.3. Non-Radioactive Component Concentrations (Contd)

Analyte	Initial Slurry Sample	Initial Permeate	Water Wash Permeate No. 1	Water Wash Permeate No. 2	Water Wash Permeate No. 3	Intermediate Slurry Sample	Caustic Leach Permeate	Caustic Leach Permeate No. 1	Caustic Leach Permeate No. 2	Final Slurry Sample
	µg/g	µg/mL	µg/mL	µg/mL	µg/mL	µg/g	µg/mL	µg/mL	µg/mL	µg/g
Ni	2420	13	17	5.0	2.4	3127	0.78	0.8	0.34	5664
P	3930	101	137	30	13	3953	362	142	40	4290
Pb	1535	<0.3	0.44	<0.3	<0.3	1730	4.9	3	2.2	2949
Pr	55.8	na	na	na	na	102	na	na	na	100
Pt	<1	na	na	na	na	<1	na	na	na	<1
Rb	71	na	na	na	na	75	na	na	na	26
Rh	<314	<2	<2	<2	<1.5	<163	<8	<8	<2	852
Ru	<1150	0.45	0.72	<0.4	<0.4	<598	<2	<2	<0.6	390
Sb	<523	<0.3	<0.3	<0.3	<0.3	<272	<1	<1	<0.8	<345
Se	<261	<0.3	0.34	<0.3	<0.3	<136	3.3	2.8	1.6	79
Si	10,950	98	157	68	27	25,200	125	29	15	21,950
Sn	930	<5	8.4	<5	<5	1400	55	27	8.1	1700
Sr	86	<0.03	0.031	<0.03	<0.03	106	<0.1	<0.1	<0.03	189
Ta	2.5±0.8	na	na	na	na	9±2.5	na	na	na	1.1±0.3
Te	<1569	<3	<3	<3	<3	<815	<13	<13	<3	<1035
Th	55,100	<4	<4	<4	<4	68,900	<20	<20	<4	113,043
Ti	205	<0.03	0.054	<0.03	<0.03	250	<0.1	<0.1	0.057	301
Tl	<522	<1	<1	<1	<1	<272	<6	<6	<1	<345
U	42,700	17	17	12	13	55,900	<50	<50	22	99,914
V	27	0.085	0.11	<0.08	<0.08	34	0.76	0.58	0.37	65
W	<2092	<3	<3	<3	<3	<1087	<13	<13	<3	<1380
Y	29	<0.05	<0.05	<0.05	<0.05	37	<0.3	<0.3	<0.06	74
Zn	287	0.4	7.7	1.3	0.98	303	8.0	4.1	0.49	815
Zr	46,850	<0.1	<0.1	<0.1	0.66	60,967	0.72	<0.6	<0.1	112,250
Cl	875	270	220	160	70	185	<25	<25	<10	<11

3.14

Table 3.3. Non-Radioactive Component Concentrations (Contd)

Analyte	Initial Slurry Sample	Initial Permeate	Water Wash Permeate No. 1	Water Wash Permeate No. 2	Water Wash Permeate No. 3	Intermediate Slurry Sample	Caustic Leach Permeate	Caustic Leach Permeate No. 1	Caustic Leach Permeate No. 2	Final Slurry Sample
	µg/g	µg/mL	µg/mL	µg/mL	µg/mL	µg/g	µg/mL	µg/mL	µg/mL	µg/g
NO ₂ ⁻	18,690	3100	4100	1400	490	690	220	70	30	100
NO ₃ ⁻	9590	1500	2200	610	230	730	180	120	90	320
PO ₄ ³⁻	1490	190	<200	<50	<50	150	630	330	90	525
SO ₄ ²⁻	2290	320	<200	110	80	210	<50	<50	<20	65
C ₂ O ₄ ²⁻	7290	1100	260	240	120	240	<50	<50	<20	50
CN ⁻	18.5	-na-	-na-	-na-	-na-	18.0	-na-	-na-	-na-	14.2
NH ₃	2.64	-na-	-na-	-na-	-na-	<0.8	-na-	-na-	-na-	<0.8
	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g
TIC	15,800	6340	995	320	145	5760	280	320	190	18,450
TOC	5180	3785	980	250	122.5	17,850	<90	175	145	20,900

na: no analysis performed.

Note: <Y denotes a value less than the detection limit, Y, for that analyte.

Note: Analytical data for slurry samples are on a dry weight basis (dried at 105°C), liquids are on a wet basis.

Slurry samples were measured on a dry basis (water removed by drying at 105°C), and permeate samples were measured on a wet basis. These results indicate that the primary metals in the initial slurry were, from highest to lowest concentration, aluminum, sodium, thorium, zirconium, uranium, and iron. The dilute caustic washing removed a majority of the sodium species while the caustic leaching removed a majority of the aluminum species. Thus, the metals in the final slurry were, from highest to lowest concentration, thorium, zirconium, uranium, iron, sodium, and aluminum. During the course of washing and leaching, most of the soluble anions measured by IC were washed from the solids. TIC and TOC concentrations, however, remained high in the solid portion of the sample.

The radioactive component concentrations are shown in Table 3.4. Of the major radioactive isotopes, only ^{137}Cs was significantly removed during leaching and washing. As would be expected, the ^{90}Sr and transuranic (TRU) isotopes remained with the slurry.

Table 3.4. Radioactive Component Concentrations

	Initial Slurry Sample	Initial Permeate	Water Wash Permeate No. 1	Water Wash Permeate No. 2	Water Wash Permeate No. 3	Intermediate Slurry Sample	Caustic Leach Permeate	Caustic Leach Permeate No. 1	Caustic Leach Permeate No. 2	Final Slurry Sample
	μCi/g	μCi/mL	μCi/mL	μCi/mL	μCi/mL	μCi/g	μCi/mL	μCi/mL	μCi/mL	μCi/g
Gross α	9.8	3.1E-4	5.1E-4	2.9E-4	3.7E-4	13	2.3E-4	1.9E-4	1.3E-4	25
¹⁴ C	0.00373	na	na	na	na	0.00431	na	na	na	0.00343
⁹⁰ Sr	572	<0.02	<0.03	<0.03	<0.03	699	<0.03	<0.03	<0.025	1280
²⁴¹ Am	6.0	1.7E-6	5.5E-6	2.5E-6	6.5E-5	7.4	1.9E-6	2.0E-6	3.6E-7	13
²⁴³ Cm, ²⁴⁴ Cm	0.087	<3.E-7	<4.5E-7	<4.E-7	6.0E-7	0.11	<2.E-7	<2.E-7	<2.5E-7	0.17
²⁴⁰ Cm	0.011	<1.8E-7	<2.5E-7	<2.E-7	2.1E-7	0.01	<2.E-7	<2.E-7	<1.4E-7	0.017
²³⁵ U	0.22	na	na	na	na	0.23	na	na	na	0.42
²³⁹ Pu, ²⁴⁰ Pu	5.5	na	na	na	na	6.4	na	na	na	13
Sum of α's	12	na	na	na	na	14	na	na	na	26
⁶⁰ Co	0.25	4.3E-3	5.8E-3	1.6E-3	6.7E-4	0.22	7.6E-4	5.2E-4	2.7E-4	0.41
⁹⁴ Nb	0.11	na	na	na	na	0.11	na	na	na	0.24
¹³⁴ Cs	<0.025	na	na	na	na	0.16	na	na	na	<0.03
¹²⁷ Cs	75	3.5	4.8	1.6	0.69	54	2.1	1.6	0.93	53
¹²⁵ Sb	<0.3	<0.007	<0.009	<0.004	<0.002	0.26	<0.006	<0.005	<0.004	0.39
¹²⁶ Sb	<0.08	<0.003	<0.003	<0.002	<0.0003	<0.04	<0.0008	<0.002	<0.00055	<0.04
¹⁵² Eu	<0.2	<0.004	<0.006	<0.002	<0.0008	0.086	<0.004	<0.003	<0.003	<0.3
¹⁵⁴ Eu	1.8	<0.0004	<0.00035	<0.00014	<0.00015	2.1	<0.0004	<0.0003	<0.0003	3.9
¹⁵⁵ Eu	1.1	<0.005	<0.007	<0.002	<0.002	1.2	<0.005	<0.004	<0.004	2.3
²⁴¹ Am	5.3	na	na	na	na	6.4	na	na	na	13
ICP/MS	ng/g	ng/mL	ng/mL	ng/mL	ng/mL	ng/g	ng/mL	ng/mL	ng/mL	ng/g
⁹⁹ Tc	2.7	87	120	38	22	2.5	21	52	28	2.5
¹⁰¹ Ru	25	195	249	72	36	43	25	13	12	44
¹²⁶ Sn	25±1	na	na	na	na	3±1	na	na	na	3±1
¹²⁹ I	3.7±0.8	na	na	na	na	1.65±3	na	na	na	<0.4
¹²⁷ Np	8.9	na	na	na	na	15.5±1	na	na	na	14±3

3.17

Table 3.4. Radioactive Component Concentrations

	Initial Slurry Sample μCi/g	Initial Permeate μCi/mL	Water Wash Permeate No. 1 μCi/mL	Water Wash Permeate No. 2 μCi/mL	Water Wash Permeate No. 3 μCi/mL	Intermediate Slurry Sample μCi/g	Caustic Leach Permeate μCi/mL	Caustic Leach Permeate No. 1 μCi/mL	Caustic Leach Permeate No. 2 μCi/mL	Final Slurry Sample μCi/g
Tritium										
Weight % Solids	28%	na	na	na	na	22%	na	na	na	19%
Entire Slurry, μCi/g	4.4E-3	na	na	na	na	3.9E-3	na	na	na	3.2E-3
Dried Solids, μCi/g	0	na	na	na	na	0	na	na	na	0
Water, μCi/mL	6.1E-3	na	na	na	na	5.0E-3	na	na	na	3.9E-3

na: no analysis performed.

Note: <Y denotes a value less than the detection limit, Y, for that analyte.

Note: Analytical data for slurry samples are on a dry weight basis (dried at 105°C), liquids are on a wet basis.

The removal efficiencies both for the initial dilute washing and the caustic leaching of the non-radioactive and radioactive components are shown in Tables 3.5 and 3.6, respectively. The results indicate that 91% of the sodium was removed from the slurry during the water-washing steps. Nearly all of the soluble chloride, nitrate, nitrite, and oxalate were removed during the first water washes. Phosphate was the only exception, having only 14% removal during the water washes. Other non-radioactive components with significant removal efficiencies during the water wash were Ru with 69% removal, Bi with 61% removal, K with 46% removal, Mo with 31% removal, and B with 42% removal.

In terms of radioactive components, 50% of the ^{137}Cs , 44% of the ^{99}Tc , and 22% of the ^{60}Co were removed during the initial water-wash steps.

The caustic leach and subsequent caustic washing steps were performed at estimated 2.0, 0.9, and 0.3 M NaOH concentrations. The leaching efficiencies of these steps are also shown in Tables 3.5 and 3.6. While only 2.4% of the aluminum was removed during the dilute caustic washing, 91% was removed during caustic leaching and this almost exclusively in the first leaching step. It appears that subsequent leaches would not be required for aluminum oxide removal from the C-104 sludge.

The as-received C-104 waste did not fall within the DOE Contract feed limits due to its high thorium concentration. Thus the original HLW glass limits described in the DOE contract do not apply. Instead, BNFL Inc. is to formulate a glass composition that maximized waste oxide loading to the extent practical. Thus, no attempt will be made here to determine the reduction in HLW glass produced due to dilute caustic washing and caustic leaching.

Other non-radioactive constituents significantly removed during the caustic-leaching process were B, Be, Cr, K, Li, Mo, P, Se, Sn, and V. Greater than 50% of Al, B, Cr, K, Li, P, all of the IC measured anions, TOC, and TIC were removed during the combined washing and leaching steps. Of the radioactive components, only ^{137}Cs and tritium were removed in significant quantities during the caustic-leaching process.

Tables 3.7 and 3.8 show the masses calculated present at each process step. The final column shows the percentage recovery. The mass recovery is the comparison of the total mass of analyte recovered throughout the test (what is removed in each process step plus what remains in the sludge residue) to the mass of analyte present in the initial sludge. The mass recovery can be represented as

$$\text{Recovery} = \frac{\sum \text{Analyte}_{\text{leach/wash}} + \text{Analyte}_{\text{residue}}}{\text{Analyte}_{\text{initialsludge}}} * 100 \quad (3.2)$$

This value provides a means of evaluating the closure of the mass balance (i.e., how much of each component is not accounted for). In general, the recoveries are reasonably close to 100%. There are some cases where the recoveries are very much larger or much smaller than 100%. These values most likely indicate significant error in one or more of the analyses (e.g. Mo). It is not surprising that the mass recoveries of the IC anions are not close to 100%. IC measures only the soluble fraction of these anions, which may change during the course of washing and leaching. The major constituents indicate that

Table 3.5. Distribution of Non-Radioactive Analytes in the Wash Steps

Analyte %	Dilute Wash Efficiency %	Caustic Leach Efficiency %	Caustic Wash #1 Efficiency %	Caustic Wash #2 Efficiency %	Residue %
Ag	0.01	<0.3	<0.2	0.10	<99.5
Al	2.4	90.8	<0.003	<0.0006	6.8
As	0.8	na	na	99.2	0
B	42.0	41.5	<3.1	4.5	<11.9, >8.9
Ba	<0.3	<0.8	<0.5	<0.08	>98.3
Be	<1.7	28.1	<2.5	<0.4	<71.9, >67.4
Bi	60.9	na	na	39.1	0
Ca	0.09	<0.4	<0.2	<0.04	>99.2
Cd	0.4	<0.3	<0.2	<0.03	<99.6, >99,
Ce	<0.7	<1.9	<1.1	0.4	>95.9
Co	8.4	<13.6	<7.6	3.6	<88.0, >66.7
Cr	7.0	32.9	10.6	<0.02	49.5
Cu	7.0	0.9	<0.6	0.5	<91.5, >91.0
Dy	<8.9	<23.3	<13.0	<2.3	>52.5
Eu	nd	nd	nd	nd	nd
Fe	0.01	0.08	<0.006	<0.0010	99.9
Hg	nd	nd	nd	nd	nd
K	46.2	41.0	<15.5	12.9	0
La	<1.1	<3.0	<1.7	0.7	<99.3, >93.5
Li	6.2	54.0	<0.09	<0.02	39.9
Mg	0.011	<3.3	<1.9	0.2	>94.6
Mn	0.005	<0.009	<0.005	0.002	99.99
Mo	31.3	36.7	<6.4	1.5	<30.6, >24.2
Na	91.1	na	na	na	4.6
Nd	<2.4	<6.3	<3.5	1.5	<98.5, >86.3
Ni	5.9	0.1	<0.1	0.07	94.0
P	18.9	52.2	<0.1	<0.02	29.0
Pb	0.02	3.0	0.3	<0.2	96.5
Pt	nd	nd	nd	nd	nd
Ru	69.2	na	na	30.8	0
Se	3.4	75.5	17.4	3.7	0
Si	11.7	8.9	<0.07	0.1	79.3
Sn	2.6	39.4	<6.9	<1.2	<58.0, >49.9
Sr	<0.4	<0.9	<0.5	<0.09	98.2
Th	<0.10	<.03	<0.1	<0.02	>99.5
Ti	0.024	<0.6	<0.3	0.05	>99.0
U	0.6	<0.7	<0.4	0.3	<99.2, >98.1
V	2.1	18.4	3.4	1.4	74.7
Y	<1.8	<4.8	<2.7	0.6	<99.4, >
Zn	2.2	6.2	<0.4	<0.08	< 91.6, > 91.1
Zr	0.009	0.01	<0.004	<0.0008	99.98
Cl	100	0	0	0	0

Table 3.5. Distribution of Non-Radioactive Analytes in the Wash Steps

Analyte %	Dilute Wash Efficiency %	Caustic Leach Efficiency %	Caustic Wash #1 Efficiency %	Caustic Wash #2 Efficiency %	Residue %
NO ₂ ⁻	97.0	3.0	0	0	0
NO ₃ ⁻	92.2	6.0	1.0	9.0	0
PO ₄ ³⁻	13.9	83.2	2.4	0	0.5
SO ₄ ²⁻	99.0	0	0	0	1.0
CO ₃ ²⁻	99.8	0	0	0	0.2
TIC	73.9	5.7	2.5	0.5	17.5
TOC	67.7	0	3.4	1.0	27.9
CN ⁻	nd	nd	nd	nd	nd
NH ₃	nd	nd	nd	nd	nd

(a) Accounts for carry-over of interstitial liquid.
 (b) Percentages based on total material removed and what remained in the residue. Sodium percentages based on starting material.
 Note: na: not applicable-detection limit too large (or too much Na added in washes.)
 Note: nd: no detect-analysis not performed.
 Note: <Y denotes less than the detection limit (Y), >Z denotes a value greater than the value Z.
 Note: <P, >Q denotes a value determined to be greater than Q but less than P.

Table 3.6. Distribution of Radioactive Analytes in the Wash Steps

Analyte	Dilute Wash Efficiency %	Caustic Leach Efficiency %	Caustic Wash #1 Efficiency %	Caustic Wash #2 Efficiency %	Residue %
⁹⁰ Sr	0	0	0	0	100
²⁴¹ Am	0.008	0	0.0001	0	99.99
²⁴³ Cm, ²⁴⁴ Cm	0.007	0	0	0	99.99
²⁴² Cm	0.02	0	0	0	99.98
⁶⁰ Co	21.9	2.5	0.4	0.07	75.2
¹³⁷ Cs	50.3	20.8	3.7	1.2	24
¹²⁵ Sb	0	0	0	0	100
¹⁵⁴ Eu	0	0	0	0	100
¹⁵⁵ Eu	0	0	0	0	100
⁹⁹ Tc	44.6	6.1	9.9	0.9	38.5
¹⁰¹ Ru	10.6	0.7	0.03	0.1	88.5

Note: ** accounts for carry-over of interstitial liquid.

Table 3.7. Mass Recovery of the Radioactive Components

	Initial Sample	Initial Dewater	Water Wash Permeate #1	Water Wash Permeate #2	Water Wash Permeate #3	Caustic Leach Permeate	Caustic Wash Permeate 1	Caustic Wash Permeate 2	Final Sample	Recovery, %
	μCi	μCi	μCi	μCi	μCi	μCi	μCi	μCi	μCi	
Gross α	5772	0.60	0.88	0.50	0.60	0.62	0.30	0.19	5152	99
¹⁴ C	2.19	na	na	na	na	na	na	na	0.72	
⁹⁹ Sr	335,885	<39	<52	<52	<50	<82	<48	<36	268,848	89
²⁴¹ Am	3498	3.4E-3	9.4E-3	4.3E-3	1.1E-1	5.1E-3	3.2E-3	5.1E-4	2677	85
²⁴³ Cm, ²⁴⁴ Cm	51	<5.8E-4	<6.7E-4	<6.9E-4	1.0E-3	<5.5E-4	<3.2E-4	<3.6E-4	36	79
²⁴² Cm	6.2	<3.4E-4	<3.2E-4	3.5E-4	3.4E-4	5.5E-4	<3.2E-4	2.0E-4	3.6	66
²³⁵ U	130	na	na	na	na	na	na	na	89	76
²³⁹ Pu, ²⁴⁰ Pu	3249	na	na	na	na	na	na	na	2646	90
Sum of α's	6933	na	na	na	na	na	na	na	5449	87
⁶⁰ Co	145	8.3	9.9	2.8	1.1	2.1	0.83	0.38	84	77
⁹⁹ Nb	62	na	na	na	na	na	na	na	<25, >17	>35
¹³⁴ Cs	<14	na	na	na	na	na	na	na	<6.2	<58
¹³⁷ Cs	44,109	6796	9288	2725	1144	5835	2521	1334	11,013	80
¹²⁵ Sb	<166	<14	<16	<6.9	<3.3	<16	<8.8	<5.7	81	<90
¹²⁶ Sn	<44	<5.8	<5.2	<3.5	<0.50	<2.2	<3.2	<0.79	<11	<65
¹⁵² Eu	<111	<7.8	<10	<3.5	<1.3	<11	<4.8	<4.3	<48	<82
¹⁵⁴ Eu	1050	<0.68	<0.60	<0.24	<0.25	<1.1	<0.48	<0.43	811	86
¹⁵⁵ Eu	648	<9.7	<12	<3.5	<3.3	<14	<6.4	<5.7	470	80
²⁴¹ Am	3136	na	na	na	na	na	na	na	2644	93
ICP/ MS	μg	ng	ng	ng	ng	ng	ng	ng	μg	
⁹⁹ Tc	1.5E+3	1.7E+5	2.1E+5	6.5E+4	3.6E+4	5.5E+4	8.4E+4	4.3E+4	4.9E+2	83
¹⁰¹ Ru	1.5E+4	3.8E+5	4.3E+5	1.2E+5	5.9E+4	6.8E+4	2.1E+4	1.7E+4	9.2E+3	77
¹²⁶ Sn	1.9E+3	na	na	na	na	na	na	na	8.4E+2	>24
¹²⁹ I	2.8E+3	na	na	na	na	na	na	na	4.2E+1	<6.1
²³⁷ Np	5.2E+3	na	na	na	na	na	na	na	3.5E+3	>58

3.22

Table 3.7. Mass Recovery of the Radioactive Components

	Initial Sample	Initial Dewater	Water Wash Permeate #1	Water Wash Permeate #2	Water Wash Permeate #3	Caustic Leach Permeate	Caustic Wash Permeate 1	Caustic Wash Permeate 2	Final Sample	Recovery , %
	μCi	μCi	μCi	μCi	μCi	μCi	μCi	μCi	μCi	
Tritium										
Weight % Solids	28%	na	na	na	na	na	na	na	19%	na
Entire Slurry, μCi	9.4	na	na	na	na	na	na	na	3.6	38
Dried Solids, μCi	0	na	na	na	na	na	na	na	0	nd
Water, μCi	9.4	na	na	na	na	na	na	na	3.6	38
na: no analysis performed, na: not applicable, na: not determined due to inadequate information.										
Note: <Y denotes less than the detection limit, Y, >Z denotes a value greater than Z.										
Note: <P, >Q denotes a value determined to be greater than Q but less than P.										

3.23

Table 3.8. Mass Recovery of the Non-Radioactive Components

Total Material	Initial Sample	Initial Permeate	Water Wash Permeate #1	Water Wash Permeate #2	Water Wash Permeate #3	Caustic Leach Permeate	Caustic Wash Permeate 1	Caustic Wash Permeate 2	Final Sample	Recovery
Analyte	µg	µg	µg	µg	µg	µg	µg	µg	µg	%
Ag	5.8E+5	<146	<129	<130	<124	<1023	<600	222	4.0E+5	76
Al	7.4E+7	40,674	556,109	370,302	233,659	4.3E+7	1.2E+7	3.8E+6	7.7E+6	98
As	<1.5E+5	<777	<687	<694	<661	<5454	<3202	1052	<3.6E+4	nd
B	1.5E+5	6631	6654	6006	4736	19,361	5283	3975	1.1E+4	47
Ba	1.3E+5	<97	<86	<87	<83	<682	<400	<72	9.0E+4	76
Be	1.8E+4	<97	<86	<87	<83	3000	464	37	1.2E+4	95
Bi	<6.1E+4	<971	2035	581	711	<6817	<4002	1557	1.5E+4	nd
Ca	2.5E+6	<971	1374	<868	<827	<6817	<4002	<718	1.8E+6	80
Cd	4.4E+5	676	477	<130	<124	<1023	<600	<108	3.5E+5	90
Ce	3.4E+5	<971	<859	<868	<827	<6817	<4002	1005	3.9E+5	131
Co	3.7E+4	427	644	<217	<207	<1704	<1001	287	1.2E+4	41
Cr	8.9E+5	14,311	22,925	7871	3810	161,702	92,131	40,613	4.1E+5	92
Cu	9.7E+4	1078	1334	234	2480	1391	<600	323	9.8E+4	121
Dy	1.8E+4	<485	<429	<434	<413	<3409	<2001	<359	1.6E+4	97
Eu	<6.1E+4	<971	<859	<868	<827	<6817	<4002	<718	6.8E+3	nd
Fe	2.2E+8	<243	575	<217	670	9271	2802	1155	1.9E+7	94
Hg	1.2E+4	na	na	na	na	na	na	na	6.8E+3	63
K	8.3E+4	81,554	124,498	23,432	<16,531	139,069	<80,045	40,183	1.1E+5	nd
La	6.5E+4	<243	<215	<217	<207	<1704	<1001	251	6.2E+4	107
Li	2.4E+5	6531	4445	1872	509	81,706	20,113	7317	1.0E+5	99
Mg	2.5E+5	<971	26	<868	<827	<6817	<4002	323	2.2E+5	101
Mn	5.2E+6	<49	<43	<43	102	<341	<200	56	4.1E+6	89
Mo	3.5E+3	1942	2404	755	380	4636	1521	796	6.5E+3	1130
Na	9.0E+7	2.4E+7	3.2E+7	1.2E+7	5.9E+6	1.3E+8	3.4E+7	1.1E+7	1.2E+7	nd
Nd	1.3E+5	<971	<859	<868	<827	<6817	<4002	1005	1.2E+5	98

Table 3.8. Mass Recovery of the Non-Radioactive Components

Total Material	Initial Sample	Initial Permeate	Water Wash Permeate #1	Water Wash Permeate #2	Water Wash Permeate #3	Caustic Leach Permeate	Caustic Wash Permeate 1	Caustic Wash Permeate 2	Final Sample	Recovery
Analyte	µg	µg	µg	µg	µg	µg	µg	µg	µg	%
Ni	1.4E+6	25,146	29,364	8592	3959	2127	<1201	488	1.2E+6	100
P	2.3E+6	195,148	235,258	52,071	21,738	987,116	226,526	57,835	9.1E+5	126
Pb	8.4E+5	<583	<515	<521	<496	12,175	4507	2526	6.2E+5	85
Pr	3.3E+4	na	na	na	na	na	na	na	2.1E+4	74
Pt	<697	na	na	na	na	na	na	na	<211	nd
Rb	4.2E+4	na	na	na	na	na	na	na	5.6E+3	18
Rh	<1.8E+5	<2913	<2576	<2604	<2480	<20,451	<12,007	1363	<4.4E+4	nd
Ru	8.7E+2	874	1228	<651	<620	<5113	<3002	588	<1.6E+5	nd
Sb	<3.1E+5	<485	<429	<434	<413	<3409	<2001	933	<7.3E+4	nd
Se	<1.5E+5	<485	584	<434	<413	8999	4402	2224	<3.6E+4	nd
Si	6.6E+6	190,779	268,743	118,288	45,295	340,855	44,345	21,527	4.6E+6	96
Sn	5.5E+5	<9709	14,425	<8678	<8266	149,976	42,424	11,533	3.6E+5	115
Sr	5.0E+4	<49	<43	<43	<41	<341	<200	<36	4.0E+4	88
Ta	2.0E+3	na	na	na	na	na	na	na	3.2E+2	<46, >15
Te	<9.2E+5	<9.2E+5	<4293	<4339	<4133	<34,086	<20,011	<3588	<2.2E+5	nd
Th	3.2E+7	3.2E+7	<6869	<6943	<6612	<54,537	<32,018	<5740	2.4E+7	83
Ti	1.2E+5	1.2E+6	15	<43	<41	<341	<200	17	6.3E+4	61
Tl	<3.1E+5	<3.07E+5	<2147	<2170	<2066	<17,043	<10,006	933	<7.3E+4	nd
U	2.5E+7	2.51E+7	25,334	20,828	20,664	<136,342	<80,045	31,572	2.1E+7	95
V	1.6E+4	1.60E+4	189	<130	<124	2072	929	524	1.4E+4	122
W	<1.2E+6	<1.23E+6	<4203	<4339	<4133	<34,086	<20,011	<3588	<2.9E+5	nd
Y	1.7E+4	1.70E+4	<86	<87	<83	<682	<400	51	1.6E+4	102
Zn	1.6E+5	1.60E+5	4113	<174	<165	7335	<800	<144	1.7E+5	128
Zr	2.7E+7	2.75E+7	<215	<217	1091	1963	<1001	<179	2.4E+7	96
Cl ⁻	5.1E+5	5.2E+5	3.8E+5	2.8E+5	1.2E+5	<6.8E+	<4.0E+	<1.4E+	<1.3E+	161
NO ₂ ⁻	1.1E+7	6.0E+6	7.0E+6	2.4E+6	7.9E+5	6.0E+5	1.1E+5	4.3E+4	1.9E+4	105

Table 3.8. Mass Recovery of the Non-Radioactive Components

Total Material	Initial Sample	Initial Permeate	Water Wash Permeate #1	Water Wash Permeate #2	Water Wash Permeate #3	Caustic Leach Permeate	Caustic Wash Permeate 1	Caustic Wash Permeate 2	Final Sample	Recovery
Analyte	µg	µg	µg	µg	µg	µg	µg	µg	µg	%
NO ₃ ⁻	5.6E+6	2.9E+6	3.8E+6	1.1E+6	3.8E+5	4.9E+5	1.9E+5	1.3E+5	6.5E+4	113
PO ₄ ³⁻	8.7E+5	3.7E+5	<3.4E+5	<8.7E+4	<8.3E+4	1.7E+6	5.3E+5	1.3E+5	1.1E+5	287
SO ₄ ²⁻	1.3E+6	6.2E+5	<3.4E+5	1.9E+5	1.3E+5	<1.4E+	<8.0E+	<2.9E+	1.2E+4	31
C ₂ O ₄ ²⁻	4.3E+6	2.1E+6	4.5E+5	4.2E+5	2.0E+5	<1.4E+	<8.0E+	<2.9E+	8.4E+3	29
CN ⁻	1.09E+4	na	na	na	na	na	na	na	3.0E+3	31
NH ₃	1.55E+3	na	na	na	na	na	na	na	<1.7E+2	<11
TIC	9.3E+6	1.3E+7	1.8E+6	5.6E+5	2.4E+5	8.4E+5	5.3E+5	2.8E+5	3.9E+6	93
TOC	3.0E+6	7.5E+6	1.7E+6	4.4E+5	2.0E+5	<2.7E+5	2.9E+5	2.1E+5	4.4E+6	259

na: no analysis performed, na: not applicable, na: not determined due to inadequate information.
 Note: <Y denotes less than the detection limit, Y, >Z denotes a value greater than Z.
 Note: <P, >Q denotes a value determined to be greater than Q but less than P.

approximately 5 to 10% of the total slurry mass was unaccounted for during the process. This could be due to actual material loss, but is more likely due to analysis and measurement errors, which in some cases are within this range.

The insoluble radioactive-component concentrations provide a means of measuring the capability of the filter to separate the insoluble solids from the liquids. The isotope ^{241}Am is basically insoluble in caustic solutions and its concentration was measured for all permeates and slurries so it was used to measure filter removal efficiency. This can be done in terms of a decontamination factor (DF) for each step of the process using the following equation:

$$DF = \frac{C_{Am,solids} \text{ wt}\%_{solids}}{100 C_{Am,permeate}} \quad (3.3)$$

where $C_{Am,permeate}$ is the ^{241}Am concentration in a given permeate sample, $C_{Am,solids}$ is the ^{241}Am concentration in the dried slurry taken during that time, and wt% slurry is the weight percent solids in the dried slurry. For the initial de-watering, the decontamination factor for ^{241}Am was 243,000. The water-wash decontamination factor was >23,000, and the caustic-leach decontamination factors were >1,300,000. These high decontamination factors indicate good solid/liquid separations using the Mott 0.1- μm sintered metal filter.

Besides the sludge wash/caustic leach study described in this report, Lumetta et al. (2000) also performed sludge washing and caustic leaching on a subsample of the initial C-104 material. In that work, the dilute caustic washing and caustic leaching were performed on separate subsamples of material. The leaching efficiencies (defined as a percentage of the starting material) for these two tests are compared in Table 3.9. The relative percent difference is also provided for ease of comparison, a negative value indicating better efficiency in the CUF than in the beaker-scale tests.^(a) The major metals that were significantly removed, such as Al, Na, Cr, P and most anions, are very similar in washing and leaching efficiency, indicating good fit between the two sets of data. Minor constituents, and those that were only slightly leached out of solution, do not correlate as well. It should be noted that the CUF material was washed and leached, while the bench-scale material was either washed or leached but not both. This may also account for some of the differences.

The final caustic leached slurry is also compared in Table 3.9. No initial characterization of the sludge sample in the small-scale testing was performed. Thus, only the final caustic-leached material can be compared. The metals compositions between the two leached samples are very similar. This would indicate similar homogeneity during initial sampling, similar leaching characteristics, and good scale-up from small to pilot scale. The anions and radionuclides do not compare quite as well. The reason for this difference is not clear.

(a) The relative percent difference is defined here as the difference between the two values divided by their sum.

Table 3.9. Comparison in Washing/Leaching Efficiencies between the Small-Scale and CUF Test

Analyte	Dilute Caustic Wash Efficiency			Caustic Leach Efficiency			Caustic Leached Slurry Composition ^(a)		
	Small Scale %	CUF %	RPD %	Small Scale %	CUF %	RPD %	Small Scale (µg/g)	CUF (µg/g)	RPD %
Ag	1.33	0.01	99	1.45	0.54	91	1790	1895	-6
Al	1.67	2.41	-18	94.96	93.22	2	34250	36700	-7
Ca	4.71	0.09	96	4.17	0.77	1.37	8131	8547	-5
Cr	14.57	7.02	35	56.77	50.53	12	1895	1953	-3
Fe	0.01	0.01	16	0.05	0.09	-58	81350	89029	-9
Mn	0.003	0.005	-16	0.03	0.02	34	18775	19671	-5
Mo	15.40	31.26	-34	53.96	75.85	-34	51	31	49
Na	92.55	91.11	1	98.51	nm		34850	58529	-51
Ni	6.43	5.87	5	2.14	6.14	-97	5550	5664	-2
P	30.97	18.87	24	70.76	71.20	-1	4690	4290	9
Pb	0.24	0.02	87	10.37	3.50	99	3042.5	2949	3
Si	15.42	11.72	14	9.98	20.79	-70	22400	21950	2
Th	0.16	0.10	26	0.76	0.51	39	116500	113043	3
U	1.16	0.58	34	0.22	1.93	-159	100100	99914	0.2
Zr	0.004	0.01	-42	0.03	0.02	9	102500	112250	-9
TOC	60.66	67.72	-5	56.46	72.10	-24	16950	20900	-21
TIC	84.88	73.86	7	76.21	82.51	-8	6900	18450	-91
Cl	96.23	100.00	-2	96.69	100.00	-3	160	<11	
NO ₃	95.48	92.16	2	96.19	100.00	-4	1250	320	118
SO ₄	93.15	99.02	-3	>51	99.02		240	65	115
PO ₄	89.00	13.87	73	>67	99.49		240	525	-75
							µCi/g	µCi/g	
¹³⁷ Cs	64.03	50.28	12	44.63	75.95	-52	135.5	53	88
⁹⁰ Sr	0.01	<0.06		0.004	<0.15		2820	1280	75
⁹⁹ Tc ^(b)	43.36	44.59	-1	44.16	61.49	-33	0.05945	0.0025	184
²⁴¹ Am	1.44	0.01	99	0.52	0.01	194	26.3	13	68

(a) Concentration on a dry solids basis

(b) ⁹⁹Tc concentration in µg/g

3.3 Physical Property Results

The C-104 samples were analyzed for density of the bulk slurries, settled solids, settled supernatant, centrifuged solids, and centrifuged supernatant. The density results are listed in Table 3.10. The weight percent (wt%) and volume percent (vol%) settled solids, wt% and vol% centrifuged solids, and wt% total solids were measured for these samples as well. The wt% and vol% solids results are listed in Table 3.11.

A known mass of each slurry was placed in a volume-graduated centrifuge cone. If sufficient material was available, samples were prepared in duplicate. The samples were then allowed to settle for 3 days. The total mass (M_B) and volume (V_B) of the settled samples were recorded, and the density of the bulk slurries was calculated ($D_B = M_B/V_B$). In addition, the volume of the settled solids (V_{ss}) and volume of settled supernatant (V_{sl}) were recorded. The vol% settled solids was then calculated ($\text{Vol}\%_{ss} = V_{ss}/V_B \times 100\%$). A portion of the settled supernatant was then transferred to a graduated cylinder, and its mass (M_{slb}) and volume (V_{slb}) were recorded. Using this data, the density of the settled supernatant was calculated ($D_{sl} = M_{slb}/V_{slb}$).

Since all of the settled supernatant could not be removed from the centrifuge cone without disturbing the settled solids, the mass of the settled solids (M_{ss}) could not be measured directly. Therefore, the mass of the settled solids was calculated. This was done by first calculating the mass of the supernatant (on top of the settled solids) from the measured supernatant density and volume ($M_{sl} = \rho_{sl} \times V_{sl}$), then subtracting this mass from the mass of the bulk slurry to get the mass of the settled solids ($M_{ss} = M_B - M_{sl}$). The density of the settled solids was then calculated ($\rho_{ss} = M_{ss}/V_{ss}$) as well as the wt% settled solids ($\text{Wt}\%_{ss} = M_{ss}/M_B \times 100\%$).

The settled supernatant was then added back to the centrifuge cones and centrifuged at approximately 1000 times the force of gravity for 1 h. All of the centrifuged supernatant was then transferred to a graduated cylinder, and its mass (M_{cl}) and volume (V_{cl}) were recorded. Then the density was calculated ($\rho_{cl} = M_{cl}/V_{cl}$). The mass (M_{cs}) and volume (V_{cs}) of the centrifuged solids were then recorded, and the density was calculated ($\rho_{cs} = M_{cs}/V_{cs}$). In addition, the wt% centrifuged solids ($\text{Wt}\%_{cs} = M_{cs}/M_B \times 100\%$), and vol% centrifuged solids ($\text{Vol}\%_{cl} = V_{cl}/V_B \times 100\%$) were also calculated.

The centrifuged solids and supernatants were then each dried at 105°C for 24 h. The mass of the dried centrifuged supernatant (M_{dcl}) and dried centrifuged solids (M_{dcs}) were then measured. Assuming all mass lost during the drying process is water and not another volatile component, the weight percent total solids in the bulk slurry was calculated:

$$\text{Wt}\% \text{ total solids} = (M_{dcs} + M_{dcl}) / (M_{cs} + M_{cl}) \times 100\% \quad (3.3)$$

The results in Table 3.10 suggest that the density of the supernatant did not vary by more than 2% before and after the processing step with a value of ~1.01 g/mL. Since dilute caustic and water were the only solutions added to the system, this consistent density result is not surprising.

Table 3.10. Density Measurement for Samples of C-104 Slurry Feed Samples

Sample	Description	Density, g/mL				
		Slurry	Settled Solids	Settled Supernatant	Centrifuged Solids	Centrifuged Supernatant
C104-002	Diluted Slurry Feed	1.07	1.21	1.01	1.37	1.01
	Duplicate	1.04	1.18	1.02	1.49	1.02
	Average	1.06	1.20	1.02	1.43	1.02
	Relative Percent Difference	3%	2%	1%	8%	1%
C104-006 ^a	Washed Slurry	1.22	1.28	NA	1.43	0.98
C104-009 ^a	Reconstituted Washed Slurry	1.16	1.18	NA	1.34	1.00
C104-FINAL	Final Slurry	1.13	1.18	NA	1.38	1.00
	Duplicate	1.14	1.18	NA	1.36	1.01
	Average	1.14	1.18	NA	1.37	1.00
	Relative Percent Difference	1.0%	0%	NA	2%	1%

^a Insufficient sample for duplicate analysis.

NA = insufficient settled liquid for density measurement.

Table 3.11. Wt% and Vol% Solids Data for C-104 Slurry Feed Samples

Sample	Description	Wt% Settled	Wt% Centrifuged	Vol% Settled	Vol% Centrifuged	Wt% Total
C104-002	Diluted Slurry Feed	35.4	16.0	31.3	12.5	10.4
	Duplicate	30.5	13.4	26.9	9.4	9.0
	Average	33.0	14.7	29.1	11.0	9.7
	Relative Percent Difference	15%	18%	15%	28%	14%
C104-006 ^a	Washed Slurry	98.1	54.2	93.8	46.2	24.9
C104-009 ^a	Reconstituted Washed Slurry	96.8	58.3	95.0	50.6	22.1
C104-FINAL	Final Slurry	98.9	52.1	95.2	42.8	21.7
	Duplicate	98.4	49.7	95.2	41.7	20.8
	Average	98.6	50.9	95.2	42.2	21.2
	Relative Percent Difference	1%	5%	0%	3%	4%

^a Insufficient sample for duplicate analysis.

Note: Wt% in this table includes interstitial liquid.

The density of the bulk slurry follows a trend expected from the processing flow. The diluted feed had a bulk density of 1.06 g/mL. Following washing and concentration, this density increased to 1.22 g/mL and then dropped to 1.16 g/mL following the reconstitution step. The material was then washed with only a small change in the density (a drop from 1.16 to 1.14 g/mL). The density of the settled solids followed this same trend except there was no final drop in density following the final washing. The density of the centrifuged solids, probably a much better measure of the actual solids density, did not vary significantly during the testing, although some variability between samples is apparent. The initial centrifuged solids density for the duplicates were 1.37 and 1.49 g/mL. The values following the processing steps were between 1.43 and 1.36 g/mL; therefore, it is likely that the initial value of 1.49 g/mL is high. Ignoring this high initial value, the density of the centrifuged solids was between 1.43 and 1.34 g/mL with no apparent trend.

From Table 3.11, the settled solids content of the initial diluted feed was 29.1 vol%. After the first washing, the settled solids content increased to >93 vol%. Given the high level of solids, the settling solid wt% and vol% data for the remaining samples are all very similar and >94%. The wt% total solids (dissolved and undissolved determined by drying) is the best measure of the solids content in Table 3.11 and follows the trend expected from the process flow. The initial total solids were 9.7 wt%. After washing, this increased to 24.9 wt% and then dropped to 22.1 wt% following reconstitution. The solids content then decreased slightly following the final processing steps to 21.2 wt%. This is the same trend seen in the bulk slurry density discussed above, showing that the trend in bulk density is the result of the trend in solids content.

An additional calculation was performed to determine the wt% solids in the samples, excluding all interstitial liquid (wt% undissolved solids). This wt% undissolved can also be thought of as the solids left if all the supernatant could be removed from the bulk slurry. The following equation was used:

$$Wt\% \text{ undissolved solids} = \left(1 - \frac{1 - \frac{M_{dsc}}{M_{cs}}}{1 - \frac{M_{dcl}}{M_{cl}}} \right) \times \frac{M_{cs}}{M_B} \times 100\% \quad (3.4)$$

This calculation assumes 1) that the supernatant and the interstitial liquid have the same composition, and 2) that all mass loss during the drying of the centrifuged solids is water loss from interstitial liquid. The results of this calculation are listed in Table 3.12 along with the weight percent dried residue from the centrifuged solids (Solids Residue = $M_{cs}/M_{dcs} \times 100\%$), and dried centrifuged supernatant (Supernatant Residue = $M_{dsl}/M_{sl} \times 100\%$).

Table 3.12 shows that the wt% undissolved solids in the initial diluted feed was 6.9 wt%. Following initial washing, the undissolved solids content was increased to 24 wt% and decreased to approximately 21 wt% following water additions for reconstitution. The undissolved solids content decreased slightly to 20 wt% following the final treatment steps. This trend is what would be expected based on the process flow as well as the trend observed in bulk density and wt% total solids content discussed above.

Table 3.12. Results of Wt% Residual Solids and Undissolved Solids Calculation Following Drying at 105°C for 24 h

Sample	Description	Wt% Residual Centrifuged Solids	Wt% Residual Centrifuged Supernatant	Wt% Undissolved Solids	Average Wt% Undissolved Solids
C-104-002	Diluted Slurry Feed	49.1	3.1	7.6	6.9
	Duplicate	47.9	3.1	6.2	
C-104-006 ^a	Washed Slurry	45.2	10	24	NA
C-104-009 ^a	Reconstituted Washed Slurry	37.6	0.5	22	NA
C-104-FINAL	Final Slurry	40.5	1.4	21	20.5
	Duplicate	40.4	1.5	20	

^a Insufficient sample for duplicate analysis.

3.4 Rheological Property Results

The C-104 slurries were analyzed for shear stress as a function of shear rate from approximately 0.1 to 300 s⁻¹ according to procedure BNFL-TP-29953-010. Several of the final slurry samples were also analyzed from 0.1 to 1000 s⁻¹. The slurries were analyzed using a Haake M5 measuring head modified for hot cell operations. An MVI measuring geometry was used. Samples were analyzed in duplicate at 25°C. A 49.9 cP standard, Brookfield lot 102298, was used to check the calibration of the instrument before samples were analyzed.

Prior to shear stress as a function of shear rate analysis, the samples were stirred to combine the separated liquid and solid layers. Shear stress as a function of shear rate data were obtained by measuring the shear stress produced at a specific shear rate. The shear rate was gradually increased from approximately 0.1 to 300 s⁻¹ (0.1 to 1000 s⁻¹ in some cases) generating the increasing shear rate curve, and then back down to 0.1 s⁻¹ generating the decreasing curve. The shear rate analysis was conducted again with the same sample still in the instrument. A difference between the first and second run would indicate potentially unusual behavior in the samples including (but not limited to) settling of the solids within the instrument, the sample being effected by shearing in the instrument, or water loss through evaporation. The sample cup was then cleaned and a duplicate sample was analyzed using the same parameters.

The tabular results for viscosity and yield stress are listed in Table 3.13. The rheograms are presented in Figures 1 through 18 in Appendix F. The initial diluted slurry displayed a nearly linear relationship between shear stress and shear rate over the shear rate range examined with no detectable yield stress. This is referred to as Newtonian behavior. Since the viscosity is the ratio of the shear stress to the shear rate, the viscosity was nearly constant (between 2 and 4 cP) over the shear rate range examined.

Table 3.13. Viscosity and Yield Stress for C-104 Slurry Samples During Increasing Rate Analysis

Sample	Sample	Analysis	Yield Stress (Pa)	Viscosity (cP)			
				100 s ⁻¹ Increasing Curve	100 s ⁻¹ Decreasing Curve	300 s ⁻¹	1000 s ⁻¹
Diluted Slurry	1	1	ND	3	NA	2	NM
		2	ND	4	NA	2	NM
	2	1	ND	2	NA	2	NM
		2	ND	2	NA	2	NM
Washed Slurry	1	1	20-38 ^a	300	182	76	NM
		2	27	270	191	85	NM
	2	1	25-40 ^a	360	240	110	NM
		2	28	300	230	103	NM
Reconstituted Washed Slurry	1	1	3	38	21	13	NM
		2	2	29	21	11	NM
	2	1	2	28	21	11	NM
		2	3	41	25	14	NM
Final Slurry	1	1	3	42	27	18	NM
		2	3	37	34	16	NM
		3	3	35	31	14	7
	2	1	3	40	33	15	8
		2	3	37	31	16	8

ND = Yield stress not detected or below 1 Pa.

NA = Thixotropy not observed.

NM = Not Measured.

^a Thixotropy makes interpretation of yield point difficult.

The remaining slurry samples had a much higher solids content. As a result of this higher solids content, the rheological behavior is more complex. These materials exhibited a yield stress along with a non-linear relationship between shear stress and shear rate. The concentrated washed slurry and the reconstituted washed slurry exhibit behavior referred to as yield pseudoplastic. These samples also exhibit a higher viscosity on the increasing shear rate curve portion of the curve than on the decreasing shear rate portion of the curve. This shear history dependence is referred to as thixotropy. Thixotropy was greatest for the washed slurry as would be expected given this material has a higher solid content compared to the other C-104 slurry samples. High solids concentrations provide more opportunity for particle interactions--one of the primary causes of thixotropy.

For a yield pseudoplastic, the viscosity, or resistance to flow, decreases as the shear rate increases. This is an important parameter when considering transport, processing or pumping. Pseudoplastic behavior is common for emulsion, suspensions and dispersions. Under shear conditions, particles find an orientation more conducive to flow, reducing the particle contribution to resistance. This reduces the apparent viscosity. When the material is pseudoplastic without thixotropy there are no shear history effects, so when the shear rate drops the apparent viscosity climbs back up as the particle orientation is lost. However, with thixotropy a more complex system exists. Slurries that exhibit thixotropy usually

form a structured network of some kind within the slurry. This structure is easily broken down under the influence of shear but will begin rebuilding when the shear forces are decreased or removed. If a thixotropic liquid is measured at a constant shear rate, the apparent viscosity will drop asymptotically with time until it reaches the sol-state, or lowest viscosity achievable at that shear rate. The sol-state numbers can not be acquired from a standard rheogram. The combination of pseudoplastic and thixotropic behavior in the C-104 slurries is consistent with testing done on other tank waste slurries.

3.5 Particle Size Distribution Results

The particle size distributions (PSDs) of the initial slurry (sample CUF-C104-001), the water washed slurry (sample CUF-C104-007), and the final slurry (sample CUF-C104-012) are described below. The first sample was C-104 that was fed into the CUF and diluted to 6.9 wt% solids (initial slurry). The second sample was the slurry that was taken at the end of the washing steps (washed slurry). The third sample was the final slurry, which was caustic leached and was caustic washed twice (final slurry). The sample number associated with each cross flow filtration experimental step is presented in Table 2.3 and the experimental process is described in detail in Section 2.

A Microtrac X-100 Particle Analyzer and a Microtrac Ultrafine Particle Analyzer (UPA) were both used to measure the PSD of these samples. The operation of Microtrac X-100 and Microtrac UPA analyzers were checked against NIST traceable standards from Duke Scientific Corporation. The PSD results of NIST traceable standards are documented in Appendix H.

The Microtrac X-100 Particle Analyzer measures particle diameter by scattered light from a laser beam projected through a stream of the sample particles diluted in a suspending medium. The amount and direction of light scattered by the particles is measured by an optical detector array and then analyzed to determine the size distribution of the particles. This measurement is limited to particles with diameters between 0.12 and 700 μm . The Microtrac UPA measures particle diameter by Doppler-shifted scattered light. This method is limited to particles with diameters between 3 nm and 6.5 μm .

The PSDs of these three samples and their duplicates were measured on the Microtrac X-100 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, 2) circulation at 60 mL/s, 3) circulation at 60 mL/s with 40 W sonication for 90 seconds, and 4) circulation at 60 mL/s with 40 W sonication for 90 seconds for the second time. The PSDs of these three samples and their duplicates were then repeated using the Microtrac UPA under conditions of Brownian motion. For each sample replicate, the PSD was measured three times and averaged. The PSD of the averaged data on a volume-weighted basis and on a number-weighted basis is reported. The suspending medium for these analyses were surrogate supernatants based on the ICP-AES and IC data obtained for the applicable C-104 supernatant. The composition of these three supernatants is reported in Table 3.14.

Table 3.14. Surrogate Supernatant Composition

	Initial Slurry	Water-Washed Slurry	Final Slurry
Component	Concentration (M)	Concentration (M)	Concentration (M)
NaNO ₃	5.64 E-02	-	-
NaOH	6.87 E-01	1.12 E-01	3.02 E-01
Al(NO ₃) ₃ - 9 H ₂ O	8.61 E-03	5.30 E-03	-
Na ₂ C ₂ O ₄	3.64 E-02	1.36 E-03	-
Na ₂ SO ₄	1.03 E-02	8.33 E-04	-
Na ₂ HPO ₄ - 7 H ₂ O	7.90 E-03	-	9.47 E-04
NaCl	1.84 E-02	1.98 E-03	-
NaNO ₂	2.07 E-02	1.04 E-02	6.52 E-04
NaHCO ₂	1.19 E-01	7.44 E-03	1.21 E-02
Na ₂ CO ₃	1.85 E-01	1.12 E-02	1.58 E-02
Al(OH) ₃	-	-	9.81 E-02

In Appendix H, the PSD plots for the samples and their duplicates under all conditions measured are presented in volume-weighted distribution and number-weighted distribution form. 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. In general, the PSD plots show that under all conditions the samples were polydispersed, and as a result the mean size of the volume distribution is much larger than the mean size of the number.

In Figure 3.12, the averaged PSDs for the initial slurry, water-washed slurry, and final slurry in cumulative under-size-percentage form are presented for the Microtrac X-100 system. The volume-weighted PSDs for these samples are illustrated in Figure 3.13. The reproducibility of the two replicate PSD plots for each sample (initial slurry, water-washed slurry and final slurry) suggest that the slurry was thoroughly homogenized and each extracted sample was a representative specimen. The cumulative under-sized-percentage plots using the UPA system (see Figure 3.14) show the samples and their duplicates. There is more scatter between the sample and its duplicate. Because of settling and the smaller sample size, reproducibility is more difficult to achieve with this instrument.

The PSD analysis (Figures 3.12 and 3.14 combined) of all three C-104 slurry samples indicate that the large majority (> 99 %) of the volume and number of the particles have diameters greater than 0.2 and less than 50 μm. On a volume-weighted basis, approximately 80% of the particles in the initial slurry sample and 90% of the particles in the water washed slurry were greater than 1μm, whereas only 50% of the particles in the caustic leached slurry were greater than 1μm. In general the plots indicate a reduction in particle size from the initial slurry to the final slurry.

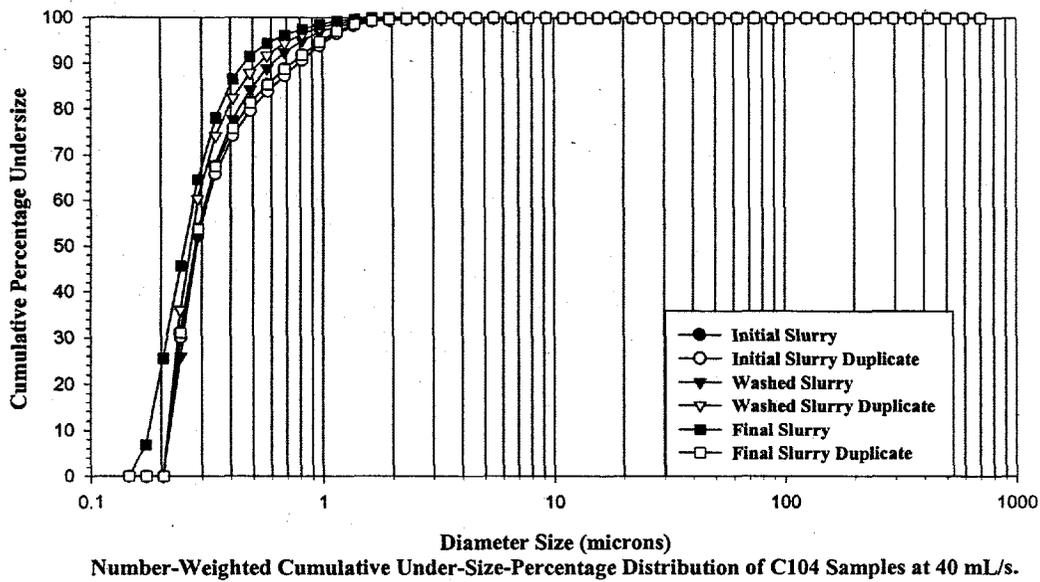
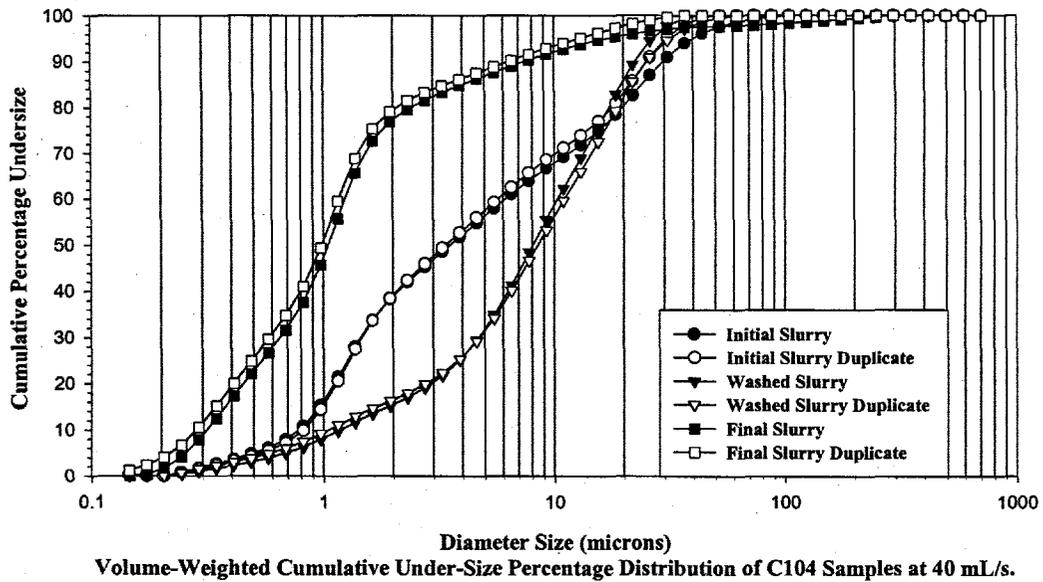


Figure 3.12. Cumulative Under-Size Percentage Distribution for C-104 slurries using the Microtrac X-100

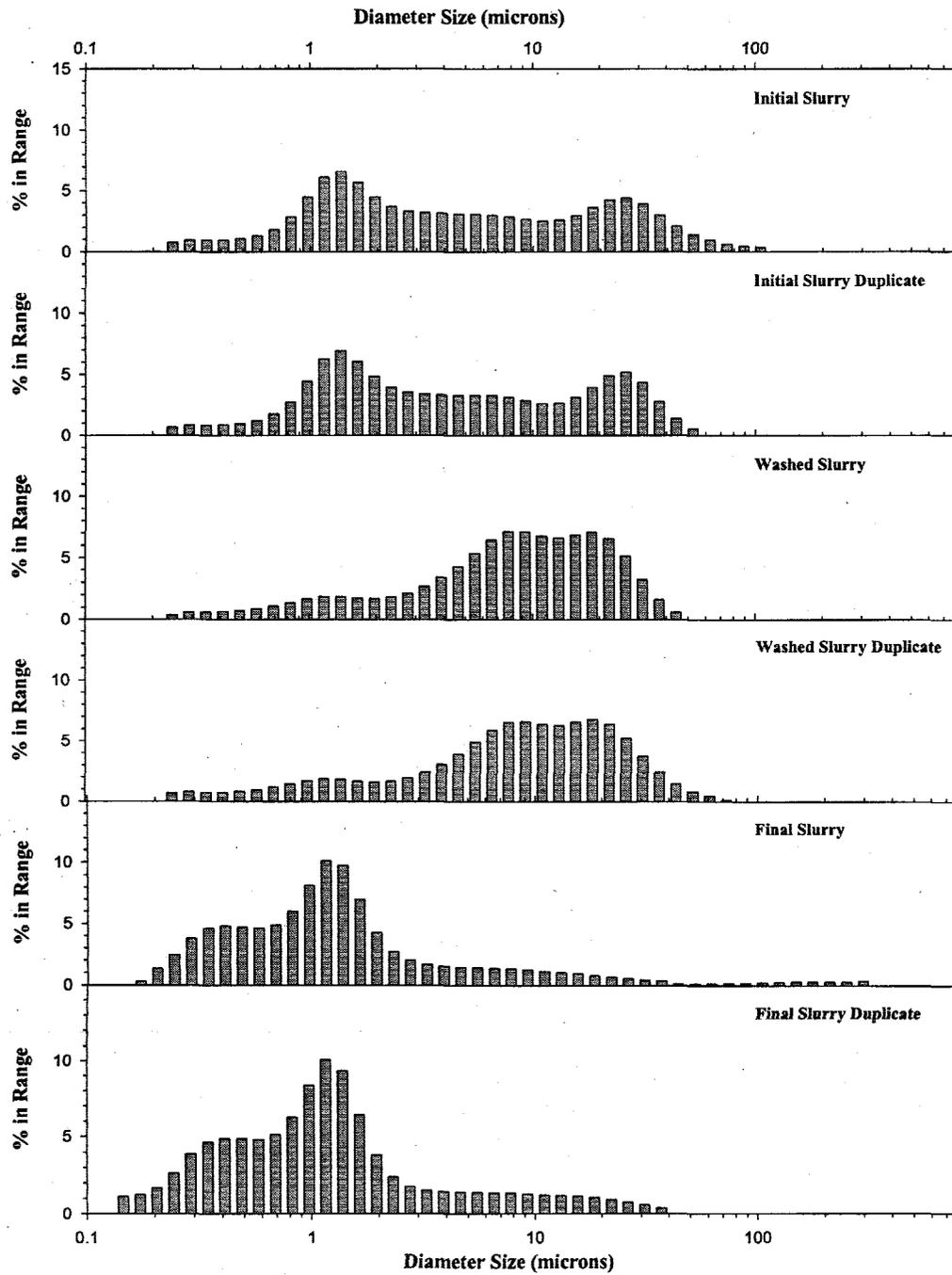


Figure 3.13. Volume-Weighted Distribution for C-104 Slurries Using the Microtrac X-100

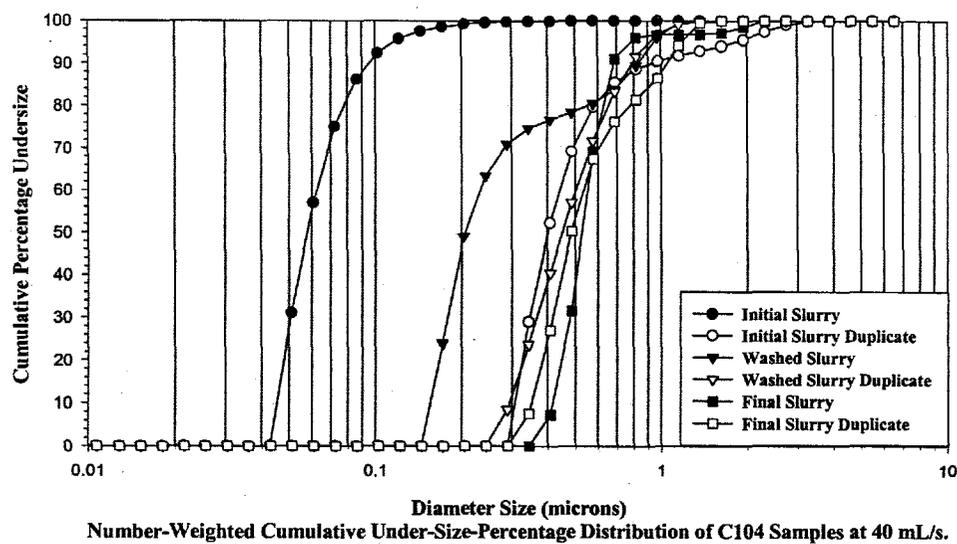
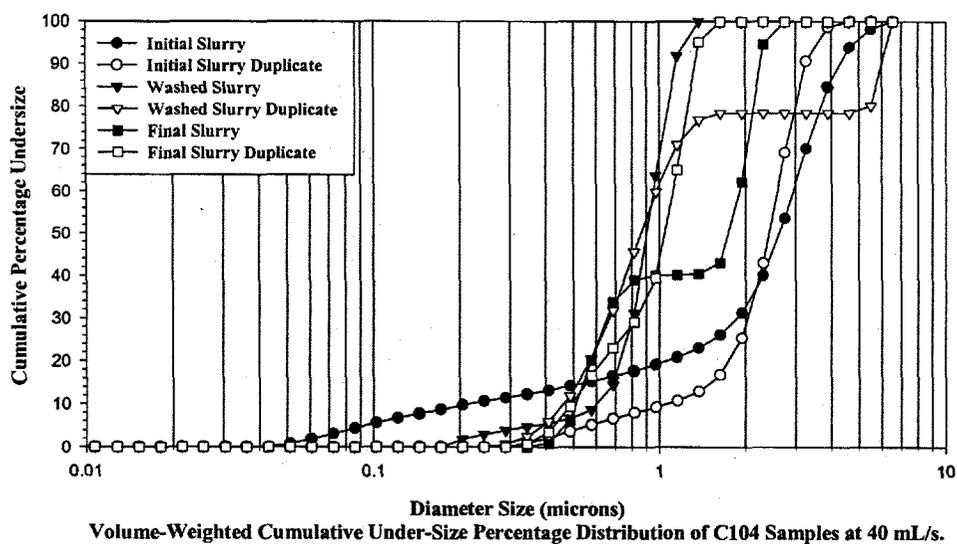


Figure 3.14. Cumulative Under-Size Percentage Distribution for C-104 Slurries Using the Microtrac UPA

The volume-weighted distribution plots (see Figure 3.13) of the initial slurry and the final slurry show a bimodal distribution formed from overlapping two Gaussian distribution peaks. While the water washed slurry distribution plots show a tetramodal distribution. The major particle-size peaks along with the relative volume or number percentage that each peak represents are summarized in Tables 3.15 and 3.16.

Table 3.15. Peak Mode Location for Volume-Weighted Particle Distribution of C-104 Samples

Sample	X-100 (40 mL/sec)		UPA (Brownian Motion)	
	Mode Diameter (μm)	Vol %	Mode Diameter (μm)	Vol %
Initial Slurry (CUF-C104-001)	22.6	33 %	2.8	89 %
	1.6	67 %	0.1	11 %
Water Washed Slurry (CUF-C104-007)	18.8	40 %	0.92	95 %
	6.0	45 %	0.22	5 %
	0.9	14 %		
	0.25	1 %		
Final Slurry (CUF-C104-012)	130	3 %	2.0	60 %
	1.2	75 %	0.58	40 %
	0.33	22 %		

Volume- and number-weighted histograms of the initial slurry, water-washed slurry and the final slurry are presented in Figures 3.15 and 3.16 for the Microtrac X-100 system and in Figure 3.17 for the UPA system, respectively. Once again, these figures indicate a reduction in particle size. The decrease in the particle size distribution from the initial slurry to the water-washed slurry after pumping in the CUF may indicate that the solids are eroded and smaller particles or agglomerates are probably formed due to vigorous mixing and shearing of particles in the CUF re-circulation line. The significant reduction in the PSD from the water-washed slurry to the final slurry is probably attributed to the dissolution of the particles or agglomerates at high caustic concentration during the caustic leaching step. In addition, the PSD analysis of the UPA system (Figures 3.17 and 3.18) reveal that the number of sub-micron particles ($>0.1 \mu\text{m}$) is reduced, and the broad distribution of particles denoted in the initial slurry is narrowed as the slurry was water washed and caustic leached.

Table 3.16. Peak Mode Location for Number-Weighted Particle Distribution of C-104 Samples

Sample	<u>UPA (Brownian Motion)</u>		<u>X-100 (40 mL/sec)</u>	
	Mode Diameter (μm)	Num %	Mode Diameter (μm)	Num %
Initial Slurry (CUF-C104-001)	0.3	100 %	0.06	100 %
Water Washed Slurry (CUF-C104-007)	0.2	100 %	0.8	23 %
			0.2	77 %
Final Slurry (CUF-C104-012)	0.25	100 %	1.9	3 %
			0.52	97 %

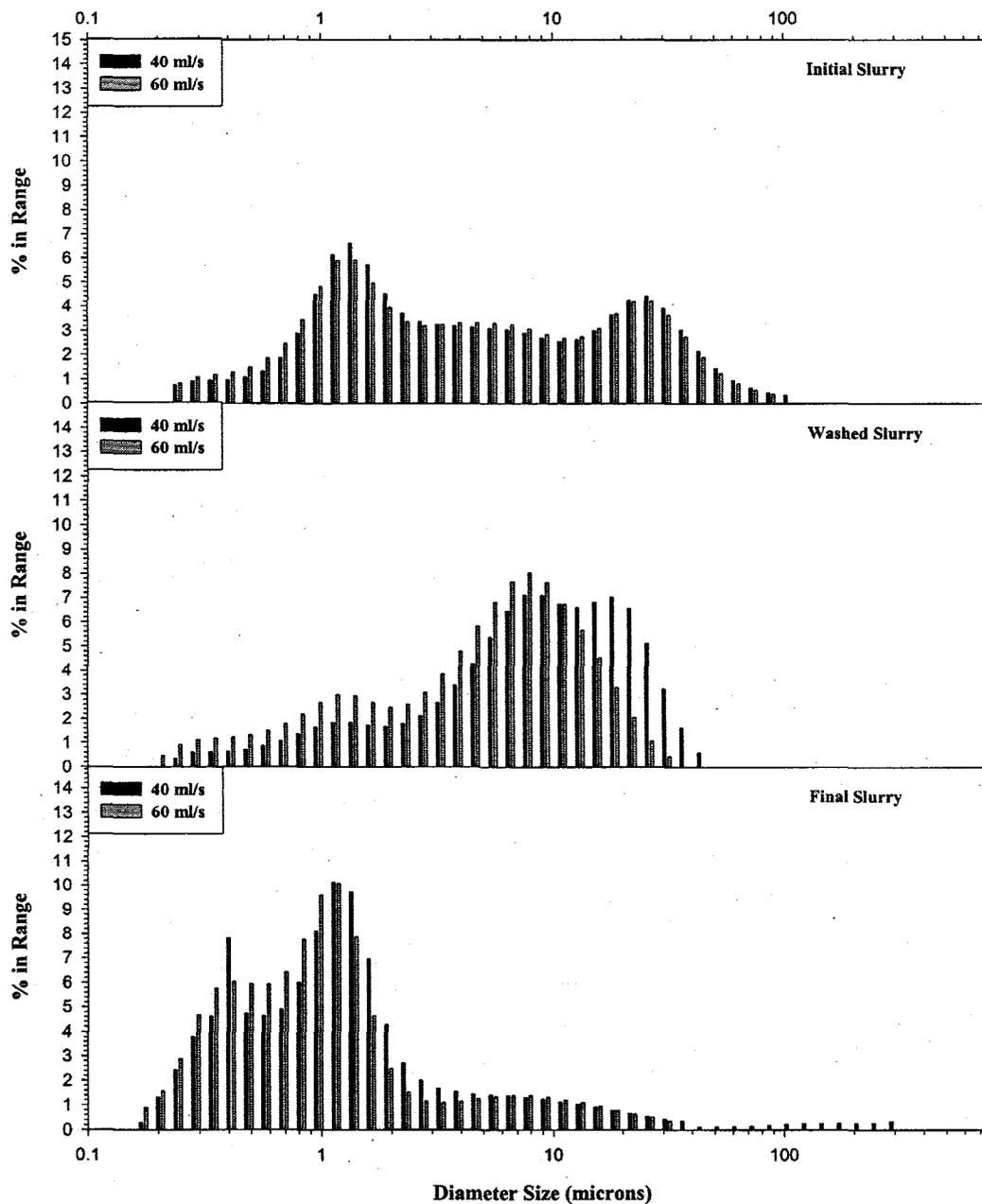


Figure 3.15. Histogram of the Volume-Weighted C-104 Slurries Using the Microtrac X-100 at 40 and 60 mL/s Circulation Flow Rate

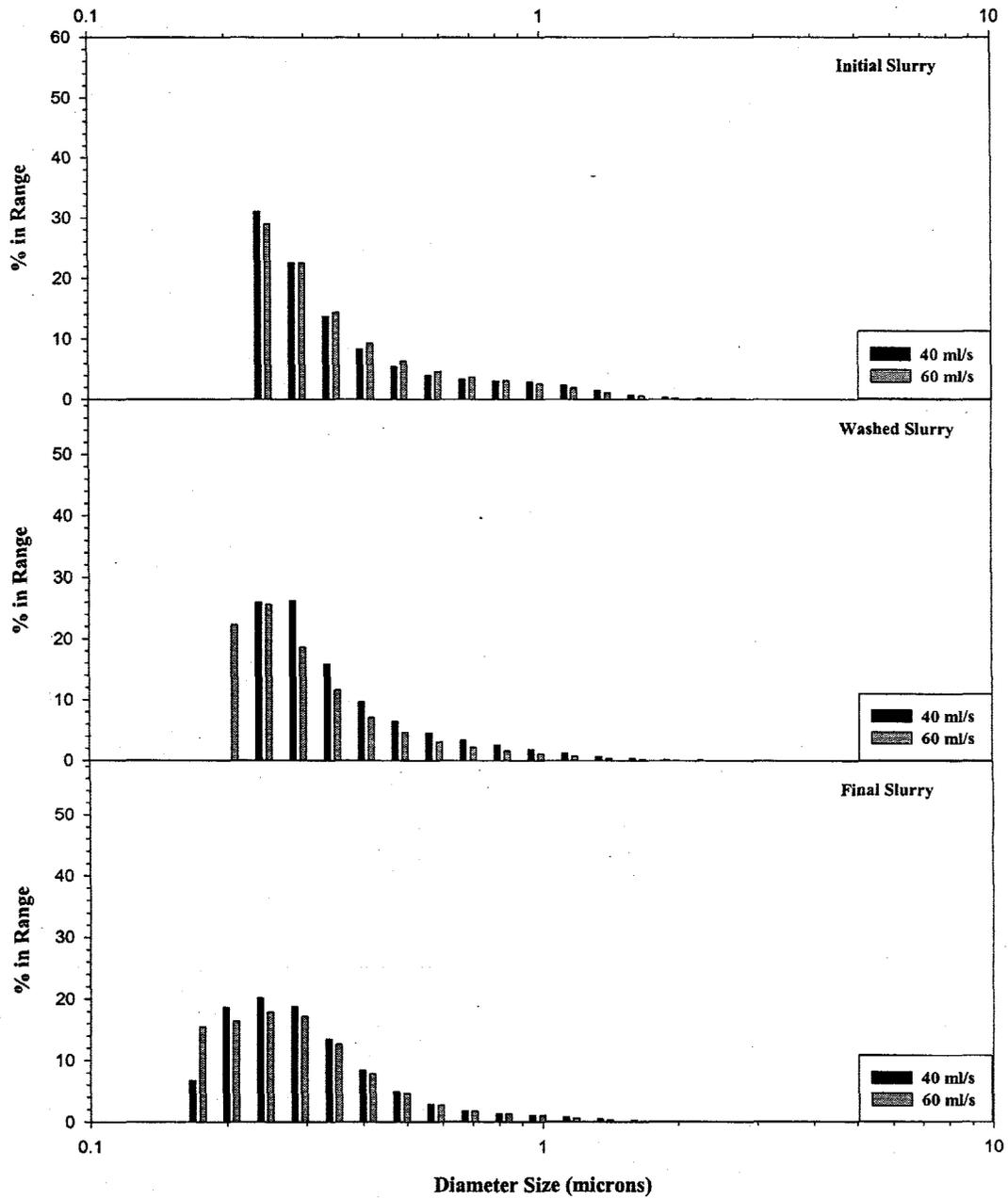


Figure 3.16. Histogram of the Number-Weighted C-104 Slurries Using the Microtrac X-100 at 40 and 60 mL/s Circulation Flow Rate

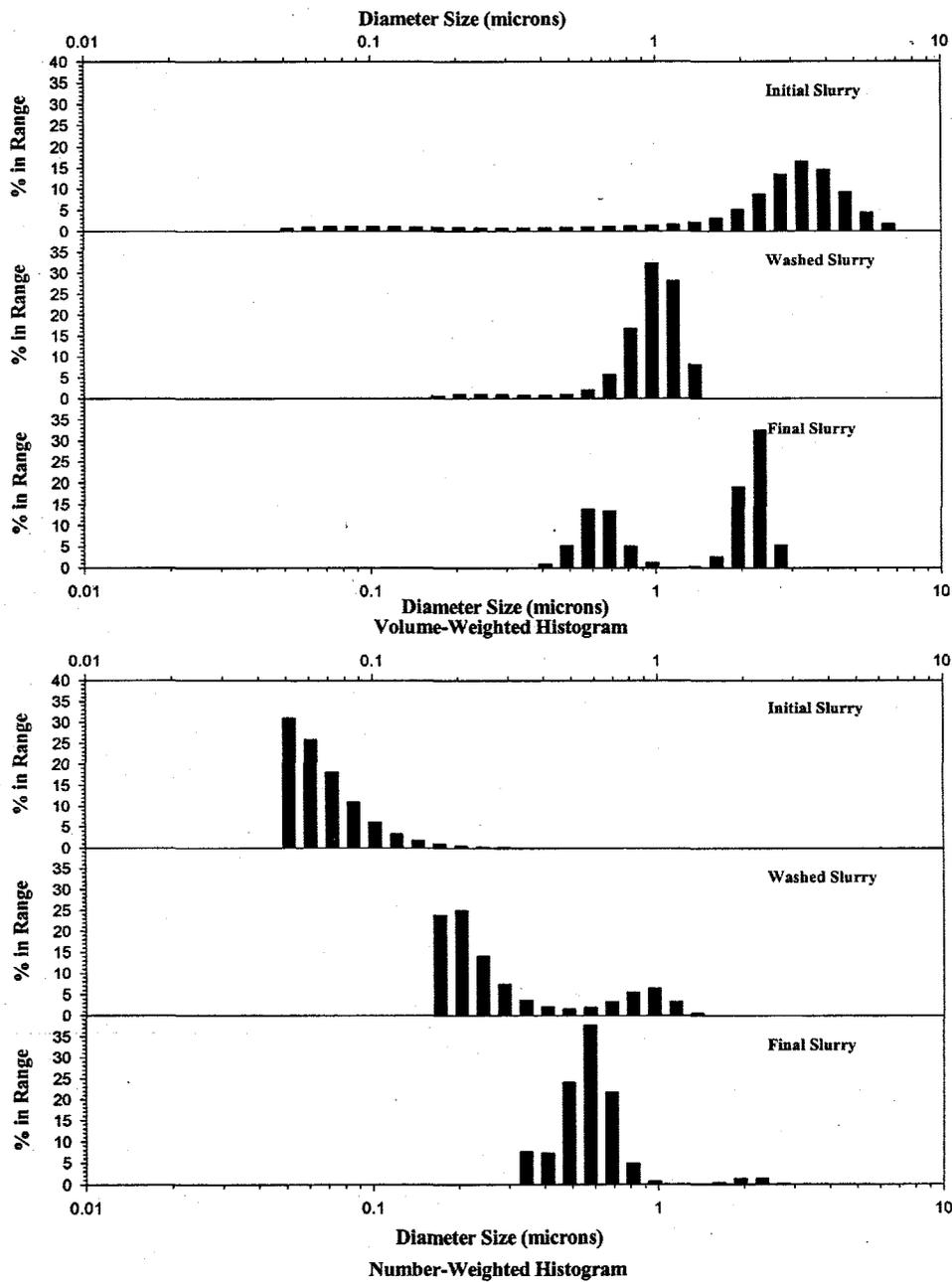


Figure 3.17. Histogram of the Volume- and Number-Weighted C-104 Slurries Using the Microtrac UPA

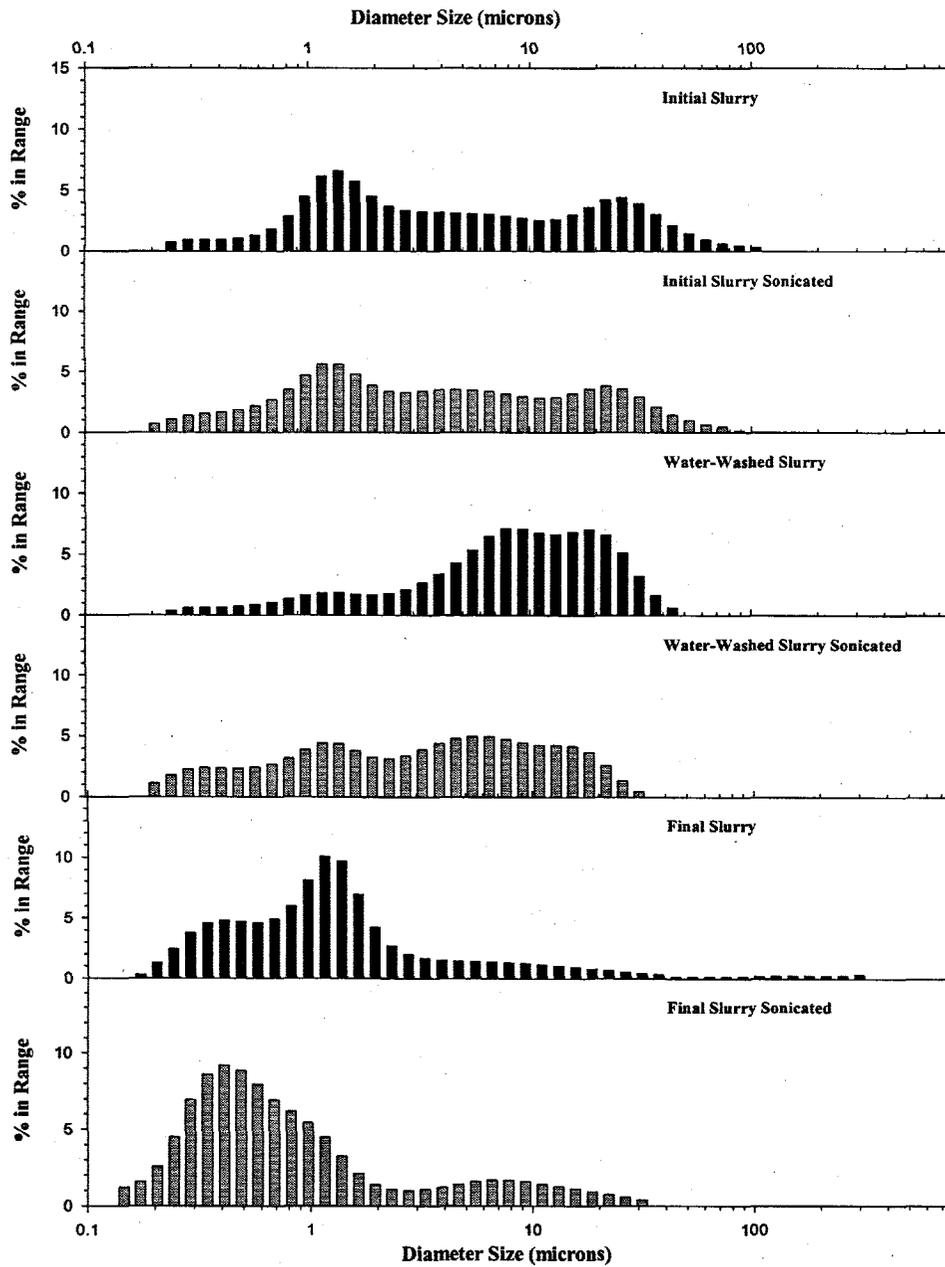


Figure 3.18. Histogram of the Volume-Weighted C-104 slurries Before and After Sonication Using the Microtrac X-100

Furthermore Figures 3.15 and 3.16 compare the volume- and number-weighted histograms of the three samples for the Microtrac X-100 at 40 and 60 mL/s circulation flow rate. These plots indicate that as the circulation flow rate increased from the 40 mL/s to 60 mL/s a large fraction of agglomerates in the water-washed slurry and the final slurry were broken down. It can be seen from Table 3.17 that the Reynolds numbers for the crossflow filtration and the Microtrac X-100 particle size analyzer are comparable. Thus, it is speculated that in qualitative terms the particles experience similar vigorous mixing and shearing in the particle analyzer and crossflow filtration unit. It should be noted that this comparison does not account for the kinetics of de-agglomeration as a function of circulation time.

Table 3.17. Calculated Reynolds Number for the Crossflow Filtration and Particle Analyzer

Crossflow Filtration System					
Solids Loading (wt %)	Velocity		Slurry Viscosity (mPa.s)	Slurry Density (kg/m ³)	Reynolds Number
	(ft/s)	(m/s)			
6.9	12.2	3.7	2	1070	18800
22			12	1160	3400
Microtrac X-100 Particle size analyzer					
Tubing Diameter (mm)	Flow Rate		Slurry Viscosity (mPa.s)	Slurry Density (kg/m ³)	Reynolds Number
	(ml/s)	(m/s)			
6.3	40	1.26	1.00	1	7800
6.3	60	1.90	1.00	1	12000

Following sonication (see Figure 3.18) a considerable fraction of agglomerates in the washed slurry and the final slurry were further broken down. In contrast, changing the flow rate did not influence the PSD of the initial slurry. The washed slurry and final slurry agglomerates are weaker than those of the initial slurry. This observation may suggest that during cross-flow filtration testing of washed and caustic leached slurries, there could be an initial decrease in filtration flux due to rapid agglomerate breakage. This hypothesis needs to be further validated with cross-flow filtration testing.

4.0 Conclusions

Based on the testing and analysis performed on C-104 sludge described in this document, the following conclusions have been obtained. They have been divided into categories for clarity.

C-104 Crossflow Filtration

- For a slurry at 6.9 wt% insoluble solids, the average filtrate fluxes over 50 min of operation ranged from 0.038 to 0.083 gpm/ft². Over the range of conditions studied, higher axial velocities appear to improve the filtrate flux while lower axial velocities and higher trans-membrane pressures appear to foul the filter and decrease overall filtrate flux. Based on modeling, the optimum condition for high solids loading over the range of conditions studied is 30 psi and 15 ft/s.
- For a slurry at 23 wt% insoluble solids, the average filtrate fluxes ranged from 0.0095 to 0.0172 gpm/ft² over 1 h. This filtrate flux is 4 to 5 times less than the first matrix, although it is still nearly an order of magnitude larger than the required plant flux for C-104 slurry of 0.00126 gpm/ft². Over the range of conditions studied, similar to the first matrix at lower solids loading, higher axial velocities improve filtrate flux. Trans-membrane pressures, in contrast, appear to have little effect. Based on modeling, the optimum condition for high solids loading over the range of conditions studied is 70 psi and 15 ft/s.
- In all cases, there is a reduction in filtrate flux over time, although it is not as significant as seen in the case of AW-101 or AN-107 supernatants (Brooks et al. 1999; Hallen et al. 2000).
- The filtrate decontamination factors for ²⁴¹Am (ratio of concentrations in the slurry to the concentration in the filtrate) are > 10⁴ to >10⁶ for the filtrates collected, indicating excellent solid-liquid separations.
- Filtrate fluxes at 85°C during the caustic leaching and washing were roughly 3 to 4 times higher than similar solids concentrations at ambient. Elevated temperature filtration however, allowed precipitation of solids after the filtrate cooled.
- A statistical model of filtrate flux as a function of solids concentration, trans-membrane pressure, axial velocity, and run order was developed for the conditions studied. This model could provide a means of comparing transmembrane pressure, axial velocity, and solids concentration without the induced effect of run order.
- Nitric acid (1 M) cleaning appears to be required to recover most of the clean water filtrate flux.

C-104 Wash and Caustic Leach Testing

- Dilute caustic washing removed 91% of the sodium and nearly 100% of soluble anions. Approximately 74 and 68% of the TIC and TOC were removed during washing. The only radioactive isotopes significantly removed by washing were ¹³⁷Cs and ⁹⁹Tc.
- The first caustic leach removed 91% of the aluminum. This indicates that a second and third caustic leach is not required. Caustic leaching also increased the removal of P and Cr as well as the water-soluble components such as K, Li, and ¹³⁷Cs.

- Mass recoveries were close to 100%, indicating good overall mass balance closure during testing.
- Th, Zr, U, and Fe were the analytes of highest concentration in the final composition of the final leached/washed sludge.
- The total mass of solids was reduced by approximately 50% during the course of water washing and caustic leaching.

C-104 Physical, Rheological, and Particle Size Properties

- The initial slurry sample at 6.9 wt% exhibited Newtonian behavior with a viscosity between 2-4 cP.
- The water-washed slurry that had a measured solids concentration of 24 wt% and a calculated solid concentration of 33 wt% was found to be yield pseudoplastic and thixotropic.^(a) With the initial increase in shear, the viscosity of the slurry at 100 s^{-1} was 300-360 cP. During the decrease in shear, viscosity of the slurry at 100 s^{-1} was 180-240 cP.
- The final slurry samples, washed and leached, at ~ 20 wt % solids exhibited a yield pseudoplastic behavior with a viscosity at 100 s^{-1} of 30 to 40 cP.
- The decrease in the particle size distribution from the initial slurry to the washed slurry after pumping in the CUF may indicate that the solids are eroded and smaller particles or agglomerates are formed due to vigorous mixing and shearing of particles in the CUF re-circulation line. The significant reduction in the PSD from the washed slurry to the final caustic leached slurry is primarily attributed to the dissolution of the particles or agglomerates at high caustic concentration during the caustic leaching step.
- As the PSD circulation flow rate increased a large fraction of agglomerates in the water-washed slurry and the final slurry were broken down. Following sonication, a considerable fraction of agglomerates in the water-washed and the final slurry were further broken down. In contrast, changing the flow rate did not influence the PSD of the initial slurry. The results indicate that the strength of the agglomerates in the initial slurry is stronger than the other steps.

(a) The difference in the calculated and measured solids concentration was caused by inhomogeneity in the CUF sampling at these high solids concentrations. For example, videos of the feed tank during this time showed it was not well mixed and there were large stagnant areas that appeared to be at high solids concentration.

5.0 References

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**Appendix A: CUF Filtration, Washing and
Leaching Test Instructions and Laboratory
Record Book**

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736-6011

PNNL Test Instruction

Document No.: BNFL-TP-29953-029

Rev. No.: 0

Title: HLW Filtration and Caustic Leaching of C-104

Work Location: RPL SFO HLRF

Page 1 of 19

Author: Kenneth G. Rappé

Effective Date: Upon Final Approval
Supercedes Date: New

Use Category Identification: Information

Identified Hazards:

- Radiological
- Hazardous Materials
- Physical Hazards
- Hazardous Environment
- Other:

Required Reviewers:

Technical Reviewer SFO Manager

Are One-Time Modifications Allowed to this Test Instruction? Yes No

NOTE: If Yes, then modifications are not anticipated to impact safety. For documentation requirements of a modification see SBMS or the controlling Project QA Plan as appropriate.

On-The Job Training Required? Yes or No

FOR REVISIONS:

Is retraining to this procedure required? Yes No

Does the OJT package associated with this procedure require revision to reflect procedure changes?
 Yes No N/A

Approval

Signature

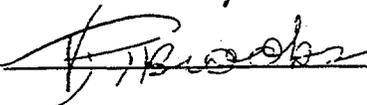
Date

Author



8/24/99

Technical Reviewer



8/24/99

1.0 Applicability

This test instruction is to be used to perform the testing of the Cell Unit Filter (CUF) in the HLRF A-cell with approximately 142 mL of C-104 rinse solution and 835 mL of C-104 slurry.

2.0 Supporting Documents

This test instruction is not a stand-alone document. It will be used in conjunction with PNNL Operating Procedure BNFL-TP-29953-020, which contains the necessary procedural information for the safe operation of the CUF. It is also linked to PNNL Test Plan No. BNFL-TP-29953-047, which contains an overall description of the project, ES&H compliance, emergency response, and the hazards assessment and mitigation.

3.0 Responsible Staff

The staff responsible for executing this test plan are:

- Task Manager – Kriston Brooks
- SFO Manager – Randy Thornhill
- Test Engineers – Kriston Brooks, Ken Rappé, Lynette Jagoda
- Hot Cell Technician – Don Rinehart, Ralph Lettau, Mac Zumhoff
- Radiological Control Technician

4.0 Materials, Equipment, Supplies and Reagents Needed

4.1 Materials Required

- Twenty-eight 20-mL glass scintillation vials for filtrate and slurry samples, pre-labeled as follows:
CUF-C104-001 through CUF-C104-028.
- Three 1-L polyethylene bottles. They should be labeled as follows: “C-104 Filtrate#1,” “C-104 Filtrate#2,” and “CUF C-104 First Wash.” The bottle labeled “C-104 Filtrate#1” should be marked with a line for 530-mL volume. The bottle labeled “CUF C-104 First Wash” should be marked with a line for 1-L volume.
- Three 2-L polyethylene bottles labeled “C-104 water wash permeate#1” through “C-104 water wash permeate#3”. “C-104 water wash permeate#1” should have marks indicating 715, 1430, and 1670-mL volume. “C-104 water wash permeate#2” should have marks indicating 475, 1190, and 1670-mL volume. “C-104 water wash permeate#3” should have marks indicating 235, 950, 1660-mL, 1700-mL, and each 50-mL increment from 1700-mL to 2000-mL volume.
- Two 2-L polyethylene bottles labeled “C-104 caustic leach permeate#1” and “C-104 caustic leach permeate#2”. “C-104 caustic leach permeate#1” should have marks indicating increments of 500-mL. “C-104 caustic leach permeate#2” should have marks in increments of 100-mL. These bottles will need to be marked and placed in the cell at a later time.
- Two 2-L polyethylene bottles labeled “C-104 caustic wash permeate#1” and “C-104 caustic wash permeate#2”. Both bottles should have marks indicating increments of 500-mL. These bottles will need to be marked and placed in the cell at a later time.
- Two 2-L polyethylene bottles labeled “C-104 final sample” and “C-104 decant.”

- Three graduated centrifuge tubes.
- Tubing for de-watering mode.
- ½" tubing to attach to V10 and remove the slurry for caustic leaching.
- Two 10 liter containers, one labeled for the alkaline rinses and the other labeled for the acidic rinses.
- Containers for draining from the bottom of the pump and from the sample valve.
- Transfer C-104 RIN, C-104 RIN2, C-104 COMP C, and C-104 COMP D from C-cell to A-cell.
- 4 liters of 0.2-micron filtered DI water for washing the CUF upon completion of C-104 processing.

4.2 Equipment

- 4000 gram balance
- pH paper
- Hand held camera. Used for making difficult readings and volume estimations.
- Stopwatch
- Calculator
- CUF Ultrafiltration system with 100 mL plug in place
- Caustic Leach equipment: graduated SS beaker with lid, stirrer, thermocouple, and temperature controller
- 1000 watt Chiller

4.3 Reagents Needed

- 951.8g of 0.01-M NaOH_{aq} labeled "5 wt % dilution."
- 5-L of 0.01-M NaOH_{aq} labeled "water wash solution" split up into seven separate one-liter containers containing ~715-mL of solution in each.
- 3-M NaOH_{aq} labeled "caustic leach solution." Amount to be determined.
- Two containers of 0.01-M NaOH_{aq} labeled "caustic wash solution#1" and "caustic wash solution#2." Amount to be determined.
- 1-L of filtered, distilled water labeled "CUF rinse."
- 1-L of 2-M HNO_{3aq} labeled "CUF wash acid."
- 1-L of filtered, distilled water labeled "shut-down water."

4.4 Other Supplies

1. Workplace Copy of Operating Procedure BNFL-TP-29953-020
2. Extra Copies of Data Sheets 1, 2, and 3
3. Laboratory Record Book
4. DAS disk for recording data

5.0 Test Instructions

The laboratory record book (LRB) shall be used to record other testing information as required by this procedure and all test conditions not stated by this procedure.

Cross-contamination between samples and contamination of samples from outside sources must be minimized at each step. Use new tools and bottles for each sample as much as possible. Those tools that are reused should be washed and rinsed prior to reuse.

Keep all test materials in sealed containers as much as possible to prevent them from drying.

5.1 Pre-start

5.1.1 Inventory materials, equipment, supplies, and reagents to ensure all required items are available. Assure that all materials have been modified for remote handling.

5.1.2 Do the following and initial and date when each item is completed.

Review PNNL Operating Procedure BNFL-TP-29953-020.

Review the work instructions in BNFL-TP-29953-029.

5.1.3 Conduct the "0.0 Pre-Start" operations in BNFL-TP-29953-020. Drain the system overflow container.

5.1.4 Conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020.

5.2 Start-Up

5.2.1 Obtain the following information:

M&TE List:

_____ Balance 1:

Calib ID 360-06-01-019 Calib Exp Date 8/18/2000

Location HLRF Counter (by computer)

_____ Balance 2:

Calib ID 362-06-01-054 Calib Exp Date 8/2000

Location A-cell North

_____ Data Acquisition System: Fluke Hydra

Calib ID 2275 Calib Exp Date 2/2000

Location A-gallery south

_____ Thermocouple 1:

325-423

Calib ID 3003 Calib Exp Date 8/2000

Location A-cell south

_____ Thermocouple 2: 325-426
 Calib ID 3006 Calib Exp Date 8/00
 Location Caustic leach pot

5.2.2 Weigh each jar with lid and C-104 rinse solution. Record the weights below.

	C-104 RIN		C-104 RIN2
Total	<u>521.070</u> g	Total	<u>389.871</u> g

5.2.3 Conduct the "1.0 Start-Up" operations in BNFL-TP-29953-020 using C-104 RIN and RIN2. Shake the jars thoroughly before adding them to the slurry reservoir. There may be some solids left the jars that cannot be transferred by shaking. If so, consult with the cognizant engineer on recovering these solids. Water addition using "5 wt% dilution" or scraping the sides with a spatula could be attempted. Record the method of recovery in the LRB.

5.2.4 Record the weights of the jar and lid in the spaces provided below. Calculate the amount of material remaining in the jar. If the differences below are > 2 g or significant solids are visible, further solids recovery is warranted. Record the method of recovery in the LRB.

	C-104 RIN		C-104 RIN2
249.5120	Jar+Lid <u>249.996</u> g		Jar+Lid <u>249.948</u> g
	Tare <u>356.558</u> g		Tare <u>351.2679</u> g
	Total <u>0.484</u> g Lost		Total <u>0.369</u> g Lost

5.2.5 Add the contents of the container labeled "5 wt % dilution" via the tank outside the cell to RIN and RIN2 and then to the slurry reservoir following "1.0 Start-up" operations in BNFL-TP-29953-020.

* See LRB - Added comp C 4500 more ml of 0.01M NaOH

5.2.6 Record the level in the slurry reservoir.

* - See LRB Height ~ ≤ 4" inches →

5.2.7 Obtain two slurry samples of at least 20 grams each following "7.0 Slurry Sampling" in BNFL-TP-29953-020 and using the pre-labeled sample vials. These will be used for physical property and particle size distribution analyses. If required, more than two sample vials may be used. Record the weights and sample numbers in Data Sheet 3.

5.2.8 Using the rheometer sample cup, obtain 40 mL of slurry material for rheological measurements following "7.0 Slurry Sampling" in BNFL-TP-29953-020. This may require as many as eight samplings to obtain this material.

5.2.9 Adjust the flow to < 2 gpm and the pressure to < 10 psig or turn off the pump while the rheology of the material is measured. Pour the 40-mL used for rheological measurements back into the CUF.

5.3 Operation

First Test Matrix

5.3.1 Conduct the "3.0 Operation during Ultrafilter Recycle Mode" operations in BNFL-TP-29953-020 using the conditions below. Filtrate flow rate should be monitored and data collected as specified in the operating procedure. After each condition, the test engineer should initial and date the table below.

Condition	Flow rate (gpm)	Transmembrane Pressure (psig)	Initial and date when complete
1	4.20	50	AKC 8/25/99
2	4.20	30	FIB 8/25/99
3	4.20	70	FIB 8/25/99
4	3.13	Optimum from 1 - 3	50 FIB 8/25/99
5	5.23	Optimum from 1 - 3	50 FIB 8/25/99
6	4.20	50	FIB 8/25/99
7	3.13	Optimum from 1 - 3	VGL 8/25/99

Do below first!

5.3.2 Weigh each jar with lid and C-104 slurry. Record the weights below.

C-104 COMP C * ^{See} _{LRB} C-104 COMP D

Total 878.52 g Total 880.52 g ~980.64

5.3.3 Shake the jars thoroughly then add them to the slurry reservoir. There may be some solids left the jars that cannot be transferred by shaking. If so, consult with the cognizant engineer on recovering these solids. DI water addition or scraping the sides with a spatula could be attempted. Record the method of recovery in the LRB.

Dewater slurry removing 1500 mL permeate. FIB 8/25/99
 Permeate should be placed in C-104 Filtrate #1 & #2

Tare wt. 104.60g Weight C-104 Filtrate #1 1130.60

Tare wt. 104.28g Weight C-104 Filtrate #2 1154.03

5.3.4 Record the weights of the jar and lid in the spaces provided below. Calculate the amount of material remaining in the jar. If the differences below are > 2 g or significant solids are visible, further solids recovery is warranted. Record the method of recovery in the LRB.

* See LRB
Added much
swirler

C-104 COMP C

Jar+Lid 347.198 g
Tare 356.6538 g

Total 1.736 g

C-104 COMP D

Jar+Lid 349.79 g
Tare 351.2579 g

Total 2.434 g

5.3.4 b Add 10 of RING RIN 2 & Comp C to slurry reservoir ^{2 RB 8/25}

5.3.5 Obtain two slurry samples of at least 20 grams each following "7.0 Slurry Sampling" in BNFL-TP-29953-020 and using the pre-labeled sample vials. These will be used for chemical and radiochemical analysis. If required, more than two sample vials may be used. Record the weights and sample numbers in Data Sheet 3.

5.3.6 Tare-weigh the 1-L bottle labeled "C-104 Filtrate#1."

Weight of empty "C-104 Filtrate#1" with lid 1154.03 g
^{2 RB 8/25}

5.3.7 Pre-weigh one of the labeled 20-mL glass scintillation vials and record its weights. Mid-way through the following step (5.3.8), obtain two filtrate samples of at least 20 grams following "8.0 Filtrate Sampling" in BNFL-TP-29953-020. If required, more than two sample vials may be used. Record the weights and sample numbers in Data Sheet 3. These will be used for chemical and radiochemical analyses.

5.3.8 Conduct the "4.0 Operation during Ultrafilter De-watering Mode" operations in BNFL-TP-29953-020 using the optimum conditions from step 5.3.1. Fill "C-104 Filtrate#1" to the 500-mL line.

5.3.9 Replace the lid on the bottle and weigh.

Weight of "C-104 Filtrate#1" with lid 1154.03 g
Total weight of material removed 1130.60 g
1049.75 g #2
1026.00 g #1 } 2075.75

5.3.10 Record the level in the slurry reservoir.

Height Not done inches - cannot read! ^{2 RB 8/25}

Second Test Matrix

5.3.11 Conduct the "3.0 Operation during Ultrafilter Recycle Mode" operations in BNFL-TP-29953-020 using the conditions below. Filtrate flow rate should be monitored and data collected as specified in the operating procedure. After each condition, the test engineer should initial and date the table below.

Condition	Flowrate (gpm)	Transmembrane Pressure (psig)	Initial and date when complete
1	4.20	50	<i>KLP</i> 8/25/99
2	4.20	30	<i>KLP</i> 8/25/99
3	4.20	70	<i>KLP</i> 8/25/99
4	3.13	Optimum from 1-3	<i>KLP</i> 8/25/99
5	5.23	Optimum from 1-3	<i>KLP</i> 8/25/99
6	4.20	50	<i>KLP</i> 8/25/99

Three Water Washes

5.3.12 Weigh all seven one-liter bottles labeled "water wash solution."

wt. taken outside of Hot Cell and then added through the chemical addition tanks.

		<i>10:41 PM</i>				
<i>Bottle #1</i>	<i>with solution</i>	<i>716.11 g</i>	<i>#2</i>	<i>715.86 g</i>	<i>#3</i>	<i>715.18 g</i>
<i>Bottle #5</i>	<i>with solution</i>	<i>714.97 g</i>	<i>#6</i>	<i>715.4 g</i>	<i>#7</i>	<i>714.2 g</i>

5.3.13 Obtain the tare weight of the three empty two-liter bottles labeled "C-104 water wash permeate#1" through "C-104 water wash permeate#3."

	permeate#1	permeate#2	permeate#3
Empty bottle	<i>192.85 g</i>	<i>192.17 g</i>	<i>26726 g</i>
	<i>±63.984</i>	<i>±63.92</i>	

5.3.14 Add one bottle of "water wash solution" to the slurry reservoir via the tank outside the cell. If the filtrate fluxes have dropped significantly during the run, the water wash solution can be added to the backpulse chamber. This is done by opening V7 towards the slurry tank, opening the vent valve and then slowly adding solution to the top of the backpulse chamber. Close the vent valve and backpulse using "6.0 Back pulsing" operations in BNFL-TP-29953-020.

5.3.15 Conduct the "4.0 Operation during Ultrafilter De-watering Mode" operations in BNFL-TP-29953-020 using the optimum conditions from step 5.3.14. Fill "C-104 water wash permeate#1." Add a bottle of "water wash solution" for each of the first two graduated marks reached. Switch to "C-104 water wash permeate#2" when the

third mark is reached. Repeat until all of the "water wash solution" is added and "C-104 water wash permeate#3" is filled to its 1660-mL mark.

5.3.16 Weigh the seven empty one-liter bottles labeled "water wash solution."

Bottle without solution	#1 ^{tare = 90.66} 90.70 g	#2 ^{tare = 90.66} 90.71 g	#3 90.72 g	#4 90.71 g
Bottle without solution	#5 90.74 g	#6 90.68 g	#7 90.67 g	

5.3.17 Obtain the tare weight of a graduated centrifuge tube.

Tare weight of centrifuge tube = 6.9925 g

5.3.18 Obtain 1 slurry sample of at least 10 grams following "7.0 Slurry Sampling" in BNFL-TP-29953-020 and using the graduated centrifuge tube. Determine the weight of the sample ($W_{t\text{sample}}$) and the total sample volume (V_{sample}). Centrifuge for 15 minutes full speed, remove, and determine volume of centrifuged solids (V_{solids}).

Performed after #7 addition but before #7 permeate removal

Weight (tube and sample) = 15.957 g
 $W_{t\text{sample}}$ =
 Weight (tube and sample) - weight of centrifuge tube = 8.964 g
 V_{sample} = 7.4 mL
 V_{solids} = 3.75 mL

Shake sample & return material to tank → *Not possible #18*

5.3.19 Calculate the volume of required leaching solution, 3-M NaOH_{aq} and place in "caustic leach solution." Place a mark indicating this volume on the bottle labeled "C-104 caustic leach permeate#1".

Volume of 3-M NaOH_{aq} to be used =
 3 * slurry volume * ($V_{\text{solids}} / V_{\text{sample}}$) = 3430 mL
 $V_{\text{solids}} = 3.75$ mL
 $V_{\text{sample}} = 7.4$ mL

Slurry volume ≈ 1700 mL (at start of sampling) slurry volume at centrifuge ≈ 2250 mL #18

5.3.20 Determine the wt % solids ($Wt\%_{\text{solids}}$) in the slurry. *#18* decide if further de-watering is required prior to caustic leaching. From the following calculation, if it is determined the wt % solids is < 17 %, then continue to the next step (5.3.21). Otherwise, skip to step 5.3.23.

In the following calculation 1.75 g/mL is the estimated density of the wet centrifuged solids and 0.5 g is the estimated mass of solid material per gram of wet centrifuged solids.

$$Wt\%_{\text{solids}} = 100\% * V_{\text{solids}} * \frac{1.365}{1.75} * 0.5 / W_{t\text{sample}}$$

$$100\% * 3.75 * 1.75 * 0.5 / 8.964$$

$$Wt\% = \frac{36.6\%}{28.54\%}$$

#18
 $Wt\% \approx (V_{\text{solids}} / V_{\text{sample}}) * (f_{\text{solid}} / 100)$
 $Wt\% \approx \rho_{\text{solid}} (V_{\text{sample}} - V_{t\text{sample}}) / W_{t\text{sample}} (1 - f_{\text{solid}})$
 $Wt\% = 29\%$ for $\rho_{\text{solid}} = 2.5$
 26% for $\rho_{\text{solid}} = 3$

5.3.21 Determine the amount of additional permeate to filter out of the slurry.
 In the following calculation 5 is a factor accounting for 20 wt % solids and 1.05 g/mL is the estimated density of removed permeate.

Volume of permeate removed=

$$(V_{\text{slurry}} / V_{\text{sample}}) * [(100 / \text{Wt}\%_{\text{solids}}) - 5] * 1.75 * 0.5 * V_{\text{solids}} / 1.05 = 623.5$$

$$= \underline{\underline{N/A}} \text{ mL}$$

623.5 - removed

5.3.22 Conduct the "4.0 Operation during Ultrafilter De-watering Mode" operations in BNFL-TP-29953-020 using the optimum conditions from step 5.3.14. Continue filling "C-104 water wash permeate#3" to the closest 50-mL mark associated with the volume calculated in the previous step.

5.3.23 Record the level in the slurry reservoir.

Height Not available inches $7\frac{1}{2}$ " from the top of tank to the slurry

5.3.24 Weight the three full two-liter bottles labeled "C-104 water wash permeate#1" through "C-104 water wash permeate#3."

	permeate#1	permeate#2	permeate#3
with permeate	<u>2028.65</u> g	<u>2004.11</u> g	<u>1920.87</u> g

5.3.25 Obtain three slurry samples of at least 20 grams each following "7.0 Slurry Sampling" in BNFL-TP-29953-020 and using the pre-labeled sample vials. If required, more than two sample vials may be used. Record the weights and sample numbers in Data Sheet 3.

5.3.26 Using the rheometer sample cup, obtain 40 mL of slurry material for rheological measurements following "7.0 Slurry Sampling" in BNFL-TP-29953-020. This may require as many as eight samplings to obtain this material.

5.3.27 Adjust the flow to < 2 gpm and the pressure to < 10 psig or turn off the pump while the rheology of the material is measured. Pour the 40-mL used for rheological measurements back into the CUF.

Added 649.11 g 0.01 M NaOH
 Repeated steps 5.3.25 - 5.3.27

✓ 5.3.34 Obtain the tare weight of the two empty 2-L bottles labeled "C-104 caustic leach permeate#1" and "C-104 caustic leach permeate#2."

	permeate#1	permeate#2
Empty bottle	<u>193.20</u> g	<u>192.57</u> g

✓ 5.3.35 Begin heating the CUF approximately 15 minutes before until the end of the caustic leach. When leaching is complete, stop the heating/mixing the caustic leach. Add approximately 2000-mL of the leaching slurry to the slurry reservoir.

197.38g 196.58g

5.3.36 Begin pumping the slurry through the CUF with V4 closed using the optimum conditions from step 5.3.14. Allow the temperature to rise to 85°C. Continue heating the caustic leach/washing container during this time. Record the time 80°C is reached.

Time 80°C is reached 04:46 AM/PM

5.3.37 Conduct the "4.0 Operation during Ultrafilter De-watering Mode" operations in BNFL-TP-29953-020 using the optimum conditions from step 5.3.14. Fill "C-104 caustic leach permeate#1." Add an additional 500-mL of slurry to the reservoir for each 500-mL of permeate removed. Repeat until "C-104 caustic leach permeate#1" is filled to its mark made in step ~~5.3.14~~ and all of the slurry has been added to the reservoir. Rinse the caustic leach vessel with an additional 100-mL of DI water or the quantity of water evaporated (whichever is greater) to remove any residual solids on the bottom or sides and add to the reservoir. Determine the weight of DI water added.

1000 mL

Container with DI Water = _____ g
 Container without DI Water = _____ g ~150 mL added to CUF

5.3.38 Obtain 1 slurry sample of at least 10 grams following "7.0 Slurry Sampling" in BNFL-TP-29953-020 and using the graduated centrifuge tube. Determine the weight of the sample (W_{sample}) and the total sample volume (V_{sample}). Centrifuge for 15 minutes at full speed, remove, and determine volume of centrifuged solids (V_{solids}).

Weight (tube and sample) = 15.486 g
 W_{sample} = 7.0506 g
 Weight (tube and sample) - weight of centrifuge tube = 8.435 g

V_{sample} = 6.9 mL
 V_{solids} = 1.4 mL

5.3.39 Calculate the volume of required washing solution (0.01-M NaOH_{aq}) for the next two washes and place in "caustic wash solution#1" and "caustic wash solution#2." Place marks indicating this volume on the bottles labeled "C-104 caustic wash permeate#1" and "C-104 caustic wash permeate#2".

$$\begin{aligned} \text{Volume of 0.01-M NaOH}_{aq} \text{ to be used} &= \\ 4 * \text{slurry volume} * (V_{\text{solids}} / V_{\text{sample}}) &= \end{aligned}$$

Level 5.75-6 mm
↓

$$4 * 1600 \text{ mL} * 0.207 = \underline{1292.8} \text{ mL}$$

5.3.40 Determine the wt % solids (Wt%_{solids}) in the slurry to decide if further de-watering is required prior to the next two caustic washes. From the following calculation, if it is determined the wt % solids is < 17 %, then continue to the next step (5.3.38). Otherwise, skip to step 5.3.40.

In the following calculation 1.75 g/mL is the estimated density of the wet centrifuged solids and 0.5 g is the estimated mass of solid material per gram of wet centrifuged solids.

$$\begin{aligned} \text{Wt}\%_{\text{solids}} &= 100\% * V_{\text{solids}} * \sim 1.75 * 0.5 / \text{Wt}_{\text{sample}} \\ &= 11.6 \text{ wt}\% \text{ or } 14.52 \text{ wt}\% \end{aligned}$$

5.3.41 Determine the amount of additional permeate to filter out of the slurry.

In the following calculation 5 is a factor accounting for 20 wt % solids and 1.10 g/mL is the estimated density of removed permeate.

$$\begin{aligned} \text{Volume of permeate} &= [(100 / \text{Wt}\%_{\text{solids}}) - 5] * 1.75 * 0.5 * V_{\text{solids}} / 1.10 \\ &= \underline{\hspace{2cm}} \text{ mL} \end{aligned}$$

5.3.42 Conduct the "4.0 Operation during Ultrafilter De-watering Mode" operations in BNFL-TP-29953-020 using the optimum conditions from step 5.3.14. Fill "C-104 caustic leach permeate#2" to the closest 50-mL associated with the volume calculated in the previous step.

No slurry was removed.

5.3.43 Record the level in the ~~slurry reservoir~~ caustic leach container

Height 5.75 cm inches

5.3.44 Weight the two full two-liter bottles labeled "C-104 caustic leach permeate#1" and "C-104 caustic leach permeate#2."

	permeate#1	permeate#2
With permeate	<u>1867.25 g</u>	<u>1495.55 g</u>

First Dilute Caustic Wash

5.3.45 Drain the system of slurry following "10.0 Draining the System" with the following changes: rather than placing a container underneath sample port V10 to collect slurry, tubing will be connected to the sample port and flow directed into the caustic leaching/washing container.

5.3.46 Add the above volume of 0.01-M NaOH_{aq} to the slurry reservoir from "caustic wash solution#1" and pump through the system for five minutes. Drain as described above (5.3.42) into the caustic-leaching vessel. Determine the weight of 0.01-M NaOH_{aq} added.

Container with 0.01-M NaOH_{aq} = _____ g
Container without 0.01-M NaOH_{aq} = _____ g

1405.22g
0.01 M NaOH

5.3.47 Begin heating the slurry and set the controller temperature to 85°C. Secure the top on the leaching/washing vessel along with the attached mixer. Initiate steady mixing. Wait until the slurry temperature reaches 85°C and note the time. Leaching will continue for 8 hours from this time.

Time 85°C is reached 08:15 AM/PM

Shutdown time (above time plus 8 hours) 04:31 AM/PM

5.3.48 Obtain the tare weight of the empty two-liter bottle labeled "C-104 caustic wash permeate#1."

permeate#1
Empty bottle 195.88g

5.3.49 Begin heating the CUF approximately 15 minutes before until the end of the caustic wash. When washing is complete, stop the heating/mixing the caustic wash. Add approximately 2000-mL of the washing slurry to the slurry reservoir.

5.3.50 Begin pumping the slurry through the CUF with V4 closed using the optimum conditions from step 5.3.14. Allow the temperature to rise to 85°C. Continue heating the caustic leach/washing container during this time. Record the time 80°C is reached.

Time 80°C is reached 17:40 AM/PM
83.8 C

50 psi
High 50 psi

5.3.51 Conduct the "4.0 Operation during Ultrafilter De-watering Mode" operations in BNFL-TP-29953-020 using the optimum conditions from step 5.3.14. Fill "C-104 caustic wash permeate#1." Add an additional 500-mL of slurry to the reservoir for each 500-mL of permeate removed. Repeat until "C-104 caustic wash permeate#1" is filled to its mark made in step 5.3.36, and all of the slurry has been added to the reservoir.

5.3.52 Record the level in the slurry reservoir.

Height _____ inches

~ 1/2 up on 5.1k glass -
after last add. #1

* See LRB

Second Dilute Caustic Wash

5.3.53 Drain the system of slurry following "10.0 Draining the System" with the following changes: rather than placing a container underneath sample port V10 to collect slurry, tubing will be connected to the sample port and flow directed into the caustic leaching/washing container.

5.3.54 Add the same volume of 0.01-M NaOH_{aq} to the slurry reservoir as in the previous wash from "caustic wash solution#2" and pump through the system for five minutes. Drain as described above (5.3.49) into the caustic-leaching vessel. Determine the weight of 0.01-M NaOH_{aq} added.

Container with 0.01-M NaOH_{aq} = _____ g

Container without 0.01-M NaOH_{aq} = _____ g

1405 ml 0.01 M NaOH
+ 107.188 H₂O

5.3.55 Begin heating the slurry and set the controller temperature to 85°C. Secure the top on the leaching/washing vessel along with the attached mixer. Initiate steady mixing. Wait until the slurry temperature reaches 85°C and note the time. Leaching will continue for 8 hours from this time.

Time 85°C is reached

08:10 AM (PM) (2010)

Shutdown time (above time plus 8 hours)

04:18 AM (PM)

5.3.56 Obtain the tare weight of the empty two-liter bottle labeled "C-104 caustic wash permeate#2."

permeate#2

Empty bottle

196.74 g

5.3.57 Begin heating the CUF approximately 15 minutes before until the end of the caustic wash. When washing is complete, stop the heating/mixing the caustic wash. Add approximately 2000-mL of the washing slurry to the slurry reservoir.

Level in 55 Beaker 7.25 mm

~ 2 l total volume

Added 157.21 g 0.01 M NaOH

5.3.58 Begin pumping the slurry through the CUF with V4 closed using the optimum conditions from step 5.3.14. Allow the temperature to rise to 85°C. Continue heating the caustic leach/washing container during this time. Record the time 80°C is reached.

Time 80°C is reached 05:15 AM/PM

5.3.59 Conduct the "4.0 Operation during Ultrafilter De-watering Mode" operations in BNFL-TP-29953-020 using the optimum conditions from step 5.3.14. Fill "C-104 caustic wash permeate#2." Add an additional 500-mL of slurry to the reservoir for each 500-mL of permeate removed. Repeat until "C-104 caustic wash permeate#2" is filled to its mark made in step 5.3.36, and all of the slurry has been added to the reservoir. Rinse the caustic wash vessel with an additional 100-mL of 0.01-M NaOH_{aq} to remove any residual solids on the bottom or sides and add to the reservoir. Determine the weight of 0.01-M NaOH_{aq} added. Cover the washing vessel.

Container with 0.01-M NaOH_{aq} = _____ g
 Container without 0.01-M NaOH_{aq} = _____ g

Added
 157.71 g 2.91 l
 + 363.02 g DI

5.3.60 Record the level in the slurry reservoir.

Height 7 3/8 inches from top after 157.71 g addition

before 363.02 g DI addition 1025 mL

5.3.61 Weight the two full two-liter bottles labeled "C-104 caustic wash permeate#1" and "C-104 caustic wash permeate#2."

	permeate#1	permeate#2
	1867.21	
With permeate	<u>173.17</u> g	<u>1649.78</u>
	19 9/2/99	10/2/99

Wts are pre-sampling.

Testing Finale Tare weigh centrifuge tube 7.354 g

5.3.62 Obtain 1 slurry sample of at least 10 grams following "7.0 Slurry Sampling" in BNFL-TP-29953-020 and using the graduated centrifuge tube. Determine the weight of the sample (W_{sample}) and the total sample volume (V_{sample}). Centrifuge for 5 minutes, remove, and determine volume of centrifuged solids (V_{solids}).

Weight (tube and sample) = 15.166 g
 W_{sample} = _____
 Weight (tube and sample) - weight of centrifuge tube = 7.812 g

V_{sample} = 6.7 mL
 V_{solids} = 1.4 mL

$\rho_{bulk} = 1.16 \text{ g/mL}$

5.3.63 Determine the amount of additional permeate to filter out of the slurry to reach 25 wt % solids. Mark "C-104 permeate#2" at this volume.

In the following calculation 4 is a factor accounting for 25 wt % solids, 1.75 g/mL is the estimated density of the wet centrifuged solids, 0.5 g is the estimated mass of solid material per gram of wet centrifuged solids, and 1.05 g/mL is the estimated density of removed permeate.

$$\text{Volume of permeate} = [Wt_{\text{sample}} - (4 * 1.75 * 0.5 * V_{\text{solids}})] / 1.05 \left(\frac{V_{\text{total}}}{V_{\text{sample}}} \right)$$

$$= \frac{0.439 V_{\text{total}}}{1} \text{ mL}$$

5.3.64 Tare-weigh the 1-L bottle labeled "C-104 Filtrate#2."

Weight of empty "C-104 Filtrate#2" with lid _____ g

5.3.65 Conduct the "4.0 Operation during Ultrafilter De-watering Mode" operations in BNFL-TP-29953-020 using the optimum conditions from step 5.3.14. Fill "C-104 Filtrate#2" to the mark made in step 5.3.59.

5.3.66 Record the level in the slurry reservoir.

Height _____ inches

5.3.67 Replace the lid on the bottle and weigh.

Weight of "C-104 Filtrate#2" with lid _____ g
 Total weight of material removed _____ g

5.3.68 Using the rheometer sample cup, obtain 40 mL of slurry material for rheological measurements following "7.0 Slurry Sampling" in BNFL-TP-29953-020. This may require as many as eight samplings to obtain this material.

Tare 133.84 g (C-104 Final Sample Rheology)

5.3.69 Adjust the flow to < 2 gpm and the pressure to < 10 psig or turn off the pump while the rheology of the material is measured. Pour the 40-mL used for rheological measurements back into the CUF.

Final Weight 219.268 g Bottle C104 Final Sample Rheology

5.3.70 Increase the flow and pressure to its optimal value and repeat step 5.3.68 and 5.3.69 for a second 40-mL slurry sample.

5.3.71 Obtain three slurry samples of at least 20 grams each following "7.0 Slurry Sampling" in BNFL-TP-29953-020 and using the pre-labeled sample vials. If required, more than two sample vials may be used. Record the weights and sample numbers in Data Sheet 3.

Draining and Rinsing the System

- ✓ 5.4.1 Tare-weigh the 2-L bottle labeled "C-104 final sample."

Weight of empty "C-104 final sample" with lid 196.94 g
 Full wt → 1173.47g

- ✓ 5.4.2 Drain the system of slurry following "10.0 Draining the System" into "C-104 final sample."

219 9/2/99
pre and sample

- ✓ 5.4.3 Add the 1-L of filtered, distilled water in "CUF rinse" to the reservoir. Open V4 and pump through the entire system for 5 minutes.

CUF Rinse #1
 T = 104.37 g
 Full = 1077.33g

219 9/2/99
pre and sample

- ✓ 5.4.4 Back-pulse the system once following "6.0 Back-Pulsing."

- ✓ 5.4.5 Shut off pump and drain the system following "10.0 Draining the System" into "C-104 final sample."

- Not performed yet
 5.4.6 Allow the slurry to settle in the 2-L bottle. Analysis of the permeate from the second caustic wash, "C-104 caustic wash permeate#2," will determine if further washing is required. If no further washing is required, decant off the liquid on top of the settled slurry into "C-104 decant" until 1 to 2 inches of liquid remain. Weigh the amount of liquid removed.

Weight of empty "C-104 decant" with lid _____ g
 Weight of full "C-104 decant" with lid _____ g

- 5.4.7 Conduct the "9.0 Rinsing the system" operation in BNFL-TP-29953-020. The first rinse should be done with 1-L of filtered, distilled water. This liquid should be collected and saved in the container labeled "CUF C-104 First Wash." The second rinse should be done with 2-L of filtered, distilled water, and the final rinse with 1-L of filtered, distilled water. The second and third rinses should be collected separately from the first in the alkaline rinse storage container. Filtrate flow rates should be checked with the third rinse to determine if an acidic rinse is required. The acidic solutions, if necessary, should be placed in a separate container.

- 5.4.8 Conduct the "3.0 Operation during Ultrafilter Recycle Mode" operations in BNFL-TP-29953-020 using the conditions below and the 1-L of filtered, distilled water in "shut-down water." Filtrate flow rate should be monitored and data collected in the operating procedure. Each test should be performed for only 20 minutes and the system should be back-pulsed. After each condition, the test engineer should initial and date the table below.

Condition	Flowrate (gpm)	Transmembrane Pressure (psig)	Initial and date when complete
1	4.20	20	
2	4.20	10	
3	4.20	30	

5.4.9 Conduct the "11.0 Shutting down" operation in BNFL-TP-29953-020.

5.4.10 Conduct the "12.0 Lay Up" operation in BNFL-TP-29953-020.

5.4.11 Remove and weigh all appropriate samples from all permeate bottles.

6.0 Sample Analysis

The point of contact for physical property sample analysis of the slurry samples is Paul Bredt. The point of contact for the sample analysis of the filtrate, wash, and filtered solids samples is Mike Urie and Rick Steele.

6.1 Slurry Sample Physical Analysis

The slurry samples taken following the de-watering step will remain in A-cell for physical testing. These samples will be used for obtaining the following information: bulk density, supernatant density, particle size distribution, volume percent settled and centrifuged solids, and suspended solids loading. Each of these analyses will be done in duplicate. The viscosity of these samples was performed previously during testing in Section 5.0.

6.2 Chemical and Radiochemical Analysis

The following samples should be transferred to the SAL hot cells for prep work and analysis.

- One sample from the first de-watering step, 5.3.4.
- One sample taken from the bottle labeled "C-104 water wash permeate#1"
- One sample taken from the bottle labeled "C-104 water wash permeate#2"
- One sample taken from the bottle labeled "C-104 water wash permeate#3"
- One sample taken from the bottle labeled "C-104 caustic leach permeate#1"
- One sample taken from the bottle labeled "C-104 caustic wash permeate#1"
- One sample taken from the bottle labeled "C-104 caustic wash permeate#2"
- One sample taken from the bottle labeled "C-104 decant"

The following will be performed on these samples: TOC/TIC and IC on the original sample and acid digestion followed by GEA, Sr-90 analysis, ICP-MS for Tc-99, total alpha and ICP-AES.

Make-up of 0.01 M NaOH

$$\frac{10.0 \text{ L of } 0.01 \text{ M NaOH}}{\text{L}} \left| \frac{40.0 \text{ g NaOH}}{\text{mole NaOH}} \right| \frac{1 \text{ g NaOH powder}}{0.97 \text{ g NaOH}} = \boxed{4.12 \text{ target}}$$

4.1822 g NaOH powder (actual) added to 10.0 L DI
 Aldrich - NaOH 20-40 mesh beads 97% pure
 Lot #00723 DF

8/24/99
 6:13 pm

Sample Vials	Balance out of cell Last Cal 8/18/99	Tare	Used For
CUF-C104 - 001		17.0000	Slurry
CUF-C104 - 002		17.0662	Slurry
CUF-C104 - 003		17.0248	
CUF-C104 - 004		16.9448	
CUF-C104 - 005		16.9195	
CUF-C104 - 006		16.9275	
CUF-C104 - 007		17.0225	
CUF-C104 - 008		16.9471	
CUF-C104 - 009		17.0745	
CUF-C104 - 010		17.0053	
CUF-C104 - 011		16.9338	
CUF-C104 - 012		17.0128	
CUF-C104 - 013		16.9769	
CUF-C104 - 014		17.0017	
CUF-C104 - 015		17.1721	
CUF-C104 - 016		17.0447	
CUF-C104 - 017		16.9738	
CUF-C104 - 018		17.0115	CUF-C104 - 023 =
CUF-C104 - 019		17.0399	
CUF-C104 - 020		17.1269	
CUF-C104 - 021		16.9552	
CUF-C104 - 022		16.9383	

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8/24/99

~7:00pm CUF has been drained and is ready to go.
Viscosity Standard run and system ~~is~~ being cleaned up.

~8:00 While adding RIN & RIN2 a mistake was made and part of SAMPLE C was added as well.

8:25 After consulting with Kriston Brooks the following plan forward was decided.

Goal - 5 wt% slurry

RIN + RIN2 + C + 951.8g 0.01M NaOH + ~500g 0.01M NaOH gives a slurry of ~10.4 wt% with a vol of ~2L in the tank.

- 1) Add the rest of C & an extra 500g 0.01M NaOH
- 2) allow to mix well in the CUF.
- 3) Pull off 1 L ~~soln~~ slurry.
- 4) Add 1 L 0.01M NaOH

Gives \Rightarrow \cong 2L of 5 wt% slurry.

8:33	Sample	Final wt	tare wt	loss	Slurry added
	RIN	249.996	249.512	0.484	271.074
	RIN2	249.948	249.5786	0.369	139.923
	CMP C	347.198	345.4623	1.736	531.322

All Sample removal was accomplished with rinsing of the jars with the planned liquid addition soln.

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0.01M NaOH Added - 951.8 g
+ 301.72
+ 204.78

to total 1458.3 g of 0.01M Na

Allowed to pump for ~45 min to mix - See video

Removed ~1 L of slurry into pre-tered bottle -

Final = 1066.33

tare = 104.31
962.02

107.68g empty
g of slurry re.

10:06 1185.16 g of 0.01M NaOH added to tank

Float removed - mixing well. (see video)

10:20 Rheology sample pulled - ~40.0 g ¹⁰⁰ ml
11:23 Rheology finished -

11:46 Samples CUF-C104-001; CUF-C104-002
For Physical Properties
Section 5.2.7 in test instruction.

11:57 starting test condition #1 4.2 gpm - 50 PSI

11:58 - glass valve on flowmeter stuck -

12:16 Start test again - Filter outlet pressure gauge not
See Data sheet working properly.

1:18 Finished test #1
Back pulsing 3 times

1:33 Started test #2
See data sheet.

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#18

- 2:50 Started condition #3
See data sheet
Turned on air booster - create fluctuations of ~5 psi about every 1/2 sec
- 3:50 Completed test condition. Optimum appears to be 30 psi. 30 psi is only slightly lower fluxes
- 4:08 Started test condition #4
Initial flow was very high but quickly dropped
- 5:35 Started test condition #5
Filled oiler for pump
Required air booster to reach flow rate
- 6:52 Started test condition #6
Turned off air booster
- 7:59 Started test condition #7
- 9:32 started dewatering.
- 9:50 Filtrate #1 full.
Filling filtrate #2.
- 10:10 Pouring 1-L of slurry back into CuF.
- 10:27 Pouring Comp D into CuF.
Dewatering into Comp-D to rinse thoroughly.
Requiring multiple rinses. (3 to be exact).
Acquiring 2 slurry samples. -003 & -004
- 11:20 Back-pulsing, 3-times.
- 11:28 Beginning de-water #2. Outlet P gauge clogged.
Stopped de-water to acquire filtrate sample -005.
Continuing to de-water
- 12:11 Done de-watering.
- 12:20 Beginning second test matrix @ 12:20 pm.

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8/25/99
5:43pm Start condition 4 @ 50 psig \approx 3.13 gpm

6:44 finish #4

7:19 start #5 @ 50 psig / \approx 5.0 gpm

using Air booster -

8:20 finish #5

* very hard to keep @ desired pr
I + keeps climbing. - Require
readjustment!!

8:44 start #6 @ 50 psig / 4.2 gpm
(No air booster)

Fluxes dropped significantly on this test
Tubing popped off of glass flow meter - lost \approx 5%
of filtrate.

8:55 finished #6 * Hard to keep @ desired pressure

10:09 Started 2nd Backpulse / Did 4 backpulses total
11:09 Each one got faster on chamber fill-ups

10:45 Back pulsed in 2 chambers full of 1st wash / 0.01M
addition through the top.

Added rest to tank - tried to wash down sides
no visible effect. - 716.11 g added

11:09 - Opening V4 & starting wash-filtration cycle -
See test sheet. -

11:43 - Reached first mark on permeate #1
Adding 2nd wash volume of 715.86 (2 though 7. - Backpulse volume)

11:56 Start 2nd wash -

8/26
12:22am - V4 closed

12:39am - V4 open start 3rd wash volume - (2 volumes though)

Had to change permeate bottles

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- 2:48 restant permeate flow V4 open
- 3:12 reached next volume mark - adding wash volume #4
- 3:16 adding 2nd top fill / back pulse volume of 4th wash volume.
- 3:24 Starting Wash #4
- 3:55 - Finish wash #4 -
Filling with #5 wash volume -
2 twice from top - (back pulsing with fresh soln.)
rest directly into tank.
- 3:14 Start #5
- 3:44 change out bottle
- 3:48 back on / take data - see sheet.
- 3:21 Making video clip of water addition in the tank #6. There appears to be a region of less mixing in the tank. No water was added to the backpulse chamber. All was added directly to the tank.
- 4:58 Closed V4 - completed 950 ml in ^{P13} second third bottle. Backpulsing w/ bottle #7 liquid into backpulse chamber. This done twice & then the remaining liquid was added to the tank.
- 5:06 Video taped the slurry after #7 addition
- 5:13 Taking sample for centrifuge. Sample material sticks to everything!
- 5:32 Began pulling off permeate.
- 5:41 Pulled sample because we want to have low enough solids concentration to get a representative sample
- 5:52 Started centrifuging samples
- 6:27 Turned off centrifuge

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7:46

7 1/2" Depth from surface to top of tank
Using the cold CUF, this appears to be
a volume of ~1000 ml. This is consistent
w/ hot CUF calibrations.

There is a region of sludge that does not
move!

Put 649.11 g 0.01 M NaOH in container.

9:00 am Pulled two slurry samples

KGR. Added 0.01 M NaOH to slurry reservoir.
Used some to rinse rheology equipment.
Lost ~ 10-15 mL.

11:30a mixing looks good. A bit frothy.
Rheology measurements taken.

1:45p Taking 3 slurry samples. CUF-C104-008, -009, &
Kriston prepared & dropped off 3-M caustic for
caustic wash.

2:30p Rinsing out / off rheology equipment.
Using part of the 3-M caustic wash.

Took off slurry sample tube, in small bucket soaking in 3
Setting up mixer & heating equipment.

4:44 - V4 closed

4:50 - Transfer to tank the slurry from CUF

5:01 - 1st 3.0 M NaOH rinse in and mixing
- Brushed tank to clean solids off sides.

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Transferring
to wash
vessel

[Signature]

8/26/99
8/26/99

5:26 - Back pulsing in 3 times - 3M NaOH for final rinse into wash system.

5:41 - last rinse in and running.

5:46 - Draining into wash tank -

System relatively clean and well drained - except for actual pump housing.

Video taken @ various points during wash & drain process.

10:04 - Stirring (sys ~~AD~~)
Stir control @ 20

10:06 heat on T=21C Set pt @ 50C

10:18 $\Phi = 38$ changed set pt to 85C

10:26 T=54°C

⁴³
~~139~~ T=71°C

⁵²
~~48~~ T=81°C (hot plate is now cycling temp)

15 T=85°C

38 T=86°C

20 T=85°C

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8/26/99 8:40 T=86°C

9:22 T=85°C

10:06 T=86°C

11:01 T=86°C

8/27/99 12:00 am T=85°C

~~8/27/99~~ 1:15 am T=87°C

1:29 am T=83°C

The caustic leach vessel was calibrated for volume previously.

1 l = 3.5 cm on ruler

2 l = 7.3 cm on ruler

3 l = 11.1 cm on ruler

Calculating a least-squares fit:

$$\text{Volume (mL)} = 266.651 \text{ Height (cm)} + 62.337$$

$$R^2 = 0.99994$$

1:45 am - TC line from cell wall to controller was a J-type
87°C new temp - jumped up from 85°C w/ the
TC cable - apparently 2°C diff due to cable

2:10 am T=84°C

2:25 am T=86°C

2:55 am T=86°C

2:59 am Turned on ~~the~~^{the} heat tape on CUF

3:18 am T=86°C

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4:26 Poured ~2L into tank
spilled less than 20 ml of material during the pour

4:32 Turned on pump $T = 60^{\circ}\text{C}$

4:39 $T = 72^{\circ}\text{C}$

4:45 $T = 79.2^{\circ}\text{C}$

4:48 Started dewatering

4:51 $T = 81.9^{\circ}\text{C}$

Filled to 1000 ml instead of 500 ml since it is difficult to move the beaker.

5:01 $T = 73.3^{\circ}\text{C}$

5:07 $T = 80.7^{\circ}\text{C}$ Switched to second caustic leach bottle

5:20 Took filtrate sample $T = 82.7^{\circ}\text{C}$

5:24 Opened V4 & continued taking samples

5:35 Added 100 g water to the tank outside the cell. There was already ~50 ml in the tank.

5:45 Dewatered completely to bring it back to its original concentration before caustic leaching.
Pulled out approximately 2700 ml of permeate

6:25 Changed fitting on V10 & preparing to take centrifuge sample.

6:34 Pulled centrifuge sample - filled w/ 10 ml easily, unlike yesterday's which required 2 (one time) & viscosity ~ 20 open/close cycles

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CUF E-104

8/27/99 7:10

Emptied CUF of sludge. Measured height = 5.75 cm
~ 1600 ml

Added water to CUF and backpulsed 3 times.
Drained system into pot.

8:15 Started cooking T = 85°C KG Rappé 371

8:30 T = 85°C

9:00 T = 84°C

9:30 T = 85°C

10:25 T = 84°C

11:00 T = 85°C

11:30 T = 86°C

12:05 T = 85°C

12:35 T = 85°C

1:40 T = 85°C

2:48 T = 84°C

4:21 T = 84°C

4:31 T = 87 - Finished with heat time -

4:30 Heat tape on CUF turned on.

4:59 Slurry tank on CUF filled to 3/4 on sight glass with slurry.

R = 2.5 cm left intact (wash pot)

5:10 Running with V4 closed @ 50 psig / ~ 4.2 gpm

T₀ = tank

T₂ = Slurry left in wash vessel

T₁ = 52.4

Varac set @ ~ 65%

T₂ = 87 -

5:42 - T₁ = 84.5 - Opening V4

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Glass valve on flowmeter fused And un operable -
V4 closed

5:59 - V4 open -

6:08 - The flow rate keeps falling - opening V1
allows good mixing to start again and the flow rate
rises - then re-adjusting back to higher pressure

6:09 1st line - ~500 ml of permeate taken off
- Added rest of slurry from wash vessel.

6:14 - 40 psig - 4.8 gpm
 $T_1 = 89.7$

6:18 2nd line ~1000 ml of permeate removed

$P = 40 \text{ psig}$ $F = 4.1 \text{ gpm}$

$T_1 = 86.5$

6:24 - Took a flux rate with the perme scope UV7
10 ml / 14.59 sec

$T_1 = 89.1$ - dropping Uvac power

6:26 3rd line - reached - slurry getting very low in
tank.

Flow rate dropping - $< 2 \text{ gpm}$

dropped pressure to 30 psig / 4.2 gpm

28 psig in / 22 psig out.

6:29 $T_1 = 89$ dropping Uvac power again

6:33 $P = 29.0 \text{ psig}$ $F = 3.3 \text{ gpm}$ $V_i \text{ dlo}$

6:36 $T_1 = 83.2$

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2.

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CUF-C104

(18:37) Flow rate dropped to zero - / Sound like pump is
6:37 Pressure dropped to zero - or was empty

opened & then watched while bumped the pump
* Pump is still working - but not enough to pump.

~1600 ml of permeate removed.
(1750 ml based on volume graduations in container next to it)

~6:50 Added about 100 ml of the wash soln. - Slurry became marginally pumpable, - Started setting up to drain.

7:00 Draining back into wash container. - Having to do small drain/wash addition cycles to try and get all the solids out of the system.

7:30 added 107.18 g DI H₂O to replace evaporative loss to Chem addition tank.

7:35 (See Video) - added enough wash soln. to the tank to run - running for 5 minutes.

7:45 Draining rinse into wash container

8:00 finished drain system (wash vessel) on and stirring T ≈ 65C

8:00 Reached 85 start cook time - will finish @ 4:10

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5:56 - 84 C

9:32 T = 85°C

10:11 T = 86°C

10:43 T = 84°C

11:15 T = 86°C

11:41 T = 85°C

8-28-99

007 T = 84°C

031 T = 85°C

100 T = 83°C video

131 T = 84°C

202 T = 85°C video

233 T = 85°C

302 T = 85°C video

335 T = 85°C

405 T = 85°C video

410 Turned on heat tape to CUF.

418 Turned off heater to wash solution.

04:52 Added CUF liquid to CUF (slurry from second dilute caustic wash)

Added 197.21 g 0.01 M to beaker to rinse

Added 357 g to beaker & let sit

Rinsed off w/ some additional DI water

5:22 Started dewatering

5:31 Took movie at 1025 mL removal

Tank level measured at 7 3/8" from top

5:43am 363.02g DI water added to slurry reservoir

5:50 Dewatered to 1405 mL in permeate #2
~~118.8~~

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~~5:46:38~~ ~~V4 opened~~ ~~D19~~

5:55 Mixture was not homogeneous. Side of tank not mixing at all.

Added 118.8 g DI water

Rerecorded. Slurry is mixing considerably better.

There may still be a region of higher solids conc on side nearest CUF inlet.

6:30 Pulled centrifuge sample

6:50 Started Centrifuge

I want to assure that this information was included - full containers

C-104 Filtrate #1 1130.60 g

C-104 Filtrate # 1154.30 g

Caustic Leach Permeate #1 1867.25 g

Caustic Leach Permeate #2 1495.55 g

7:33 Taking 100 ml for Paul's rheology measurements at 2.4 gpm & 50 psi

8:31 Took samples $T = 78.7^\circ\text{C}$ - 48 psi & 2.8 gpm - high improved sample amount retrieved for each V2 port opening

8:34 Turned off centrifuge

9:00 Ran flux measurements w/ & w/o air booster

9:20 Drained CUF

9:33 First Rinse of CUF added 1000 mL (grad cy DI water. 2 Backpulses worth ^{of DI water} were sent through the bac chamber

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J. B. [Signature]

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10:00 Pumped it around & drained into "CUF Rinse #1."
Pumped for
Rinsed out SS beater & put ~300 ml into
"CUF Rinse #2"

Added 2 backpulses worth of water into
the backpulse chamber.

Used the scrub brush to remove solids from
sides of container.

Ran CUF w/ ~700 ml water for 10-15 minutes;
Drained liquid into "CUF Rinse #2"

10:53 Added 2 l DI water to CUF

Ran thru V4 & w/ V7 open to permeate line

11:12 ~~Unplugged~~ Unplugged V9 - 20 psi in to unplug
Shut down system

3/30/99 9:30 Start system up - very dirty visually.

~10:30 Backpulsed 3 times

~11:00 Manipulator broken - work in A cell is now
limited ~~and~~ kg

12:00 pm - Sending in bottle to collect rinse #3

Labeled CUF Rinse #3 Tare 192.71 g

1:30 emptied CUF into CUF Rinse #3

1:40 Backpulsed 4 chambers of clean H₂O from top
of system into CUF. VERY VERY slow - filters
plugged!!

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Date

8/28/99

8/29/99

CUF-C-104

1:50 Running - still dirty -
2:18 Draining Rins #11 into the 1 Rins (#3 bottle,
2:28 Refilled with ~ 1 L H₂O - Ran for 5 min.
Still VERY Dirty - Brownish Yellow color.
Shutting down so manipulator can be repaired

9/2/99 Took samples of the wash solns. (see Sample

Sent to SAZ

CUF-C-104-~~001~~-004

CUF-C-104-~~012~~-005

CUF-C-104-~~010~~-000

-011

-013

-014

-015

-016

-017

-018

-019

-021

-022

~20mR

Sent to 312 (Hot Colla

CUF-C-104-001

-012

-007

} ~50m

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Date

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Disclosed To and Understood By

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AT	Name	Tare	Final
A1	Filtrate #1	16.9310	38.73
A2	Filtrate #2	16.9478	39.00
A3	C-104 Caustic Leach	16.9283	39.73
A4	C-104 Caustic Wash #1	16.8760	38.37
A5	C-104 Caustic wash #2	16.9706	38.29
A6	Wash Filtrate #1	16.9123	37.50
A7	Wash Filtrate #2	16.9256	37.77
A8	Wash Filtrate #3	17.1146	38.15
A9		16.9143	

New bottles (plastic coated glass)

	Tare	Full
C-104 Washed Sludge Very brown black	506.03	1468.18
C-104 Washed Sludge #2 Rinse of main bottle + Solids from Rinse #1 - 1099.4	506.76	

	Tare	Sec	tot
E-104 Wash (H ₂ O) Filtrate Composite	508.03g		
#1 * Dark color (greenish) / no solids! after ~200ml #1	726.36g		218
#2 lighter green (no solids) After ~200ml of #2	930.44g		204
#3 Very light green After ~200ml of #3	1128.36g		195
↳ Some white fluffy flocs in the bottom (very little)			

C-104 Caustic Leach	Tare	with sample
	509.19	1024.13g

* Visual observations -
When the large bottle, filled straight from the CVF, was first observed the solution looked clear (greenish). After swirling, large sheet-like flakes (white) came off sides and bottoms.

Project No. _____ Date of Work 10/21-10/22/99

Entered By [Signature] Date 10/22/99

Disclosed To and Understood By _____

Signed 1. _____ Date _____

2. _____ Date _____

C-104 Caustic Wash Composite

Caustic Wash #1	Tare	Sam
No-solids	506.67	to
Caustic Wash #2	After addition [↑] bottle #1 = 820.86g	31%
	After addition from bottle #2 = 1134.98	31%
		<u>62%</u>

C-104 Filtrate #1

Lighter color - No Solids	<u>Tare</u>	<u>Final</u>
	360.35	809.90

C-104 Filtrate #2

Darker color - No Solids	<u>Tare</u>	<u>Final</u>
	360.72	819.14

Project No. _____ Date of Work 10/22/99

Entered By Alyssa K. Jacob Date 10/22/99

Disclosed To and Understood By _____

Signed 1. _____

Date _____

Appendix B: Testing Mass Balance

Appendix B: Testing Mass Balance

Date/Time	Description	Volume (mL)	Mass (g)	Density (g/mL)	Caustic Molarity (M)	Undissolved Solids (g)	Weight Percent Undissolved Solids
	Rin 1	265	272.4	1.03	0	55.2	20.3%
	Rin 2	140	140.9	1.01	0	13.1	9.3%
	Comp C	410	605.7	1.48	0	171.1	28.2%
	Initial Pre-test Sample #1	756	944.9	1.25	0	239.3	25.3%
	Amount lost	2	2.6	1.25	0	0.7	25.3%
	Effective Pre-test Sample #1	754	942.3	1.25	0	238.7	25.3%
	Inhibited Water Added	1458	1458.3	1.00	0.010	0	0%
	Slurry removed for later	886	962.02	1.09	0.007	95.6	9.9%
	Inhibited Water Added	1185	1185.2	1.00	0.010	0	0%
	Initial Test Sample #1	2511	2623.8	1.055	0.008	181.0	6.9%
	Slurry Sample -001	19	20.5	1.055	0.008	1.4	6.9%
	Slurry Sample -002	19	20.4	1.055	0.008	1.4	6.9%
24-Aug-99	First Test Matrix & Dewatering						
	Dewatered filtrate (-019)	1942	1975.8	1.02	0.008	0	0%
	Slurry Re-addition	886	962.02	1.09	0.007	95.6	9.9%
	Amount lost	3	3.0	1.09	0.007	0.3	9.9%
	Comp D addition	425	608.50	1.43	0	170.9	28.1%
	Amount lost	2	2.4	1.43	0	0.7	28.1%
	Slurry Sample -003	18	20.80	1.15	0.006	4.8	23.2%
	Slurry Sample -004	18	20.73	1.15	0.006	4.8	23.2%
	Test Sample #2	1801	2131	1.15	0.006	493.8	23.2%
	Dewatered filtrate	94	100.0	1.06	0.007	0	0%
	Filtrate Sample -005	20	21.6	1.0602	0.008	0	0%
25-Aug-99	Second Test Matrix						
25-Aug-99	Water Washes						
	Test Sample #2	1765	2089.1	1.18	0.006	484.2	23.2%
	Water Washes Added	#1	716	716.07	1.00	0	0
		#2	716	715.81	1.00	0	0
		#3	715	715.15	1.00	0	0
		#4	716	715.62	1.00	0	0
		#5	715	714.93	1.00	0	0
		#6	715	715.36	1.00	0	0
		#7	714	714.16	1.00	0	0
	Removed Wash Permeate	#1	1717	1771.82	1.0318	0	0%
		#2	1736	1748.02	1.01	0	0%
		#3	1653	1653.61	1.00	0	0%
	Lost Permeate		606	613.84	1.01	0	0%
	Removed Centrifuge Sample		7.4	8.96	1.21	2.03	22.6%
	Test Sample #2	1052.9	1300.0	1.22	0.009	432.2	33.2%
	Slurry Sample -006	10	11.95	1.22	0.009	4.0	33.2%
	Slurry Sample -007	9	10.52	1.22	0.009	3.5	33.2%
	Inhibited Water Added	649	649.1	1.00	0.010	0	0%
	Inhibited Water Lost	15	15.0	1.00	0.010	0	0%
	Test Sample #2	1648.0	1911.6	1.16	0.009	424.3	22.2%
	Slurry Sample -008	13.3	15.4	1.16	0.009	3.4	22.2%
	Slurry Sample -009	13.0	15.1	1.16	0.009	3.3	22.2%

Date/Time	Description	Volume (mL)	Mass (g)	Density (g/mL)	Caustic Molarity (M)	Undissolved Solids (g)	Weight Percent Undissolved Solids
	Slurry Sample -010	15.5	18.0	1.16	0.009	4.0	22.2%
26-Aug-99	Caustic Leach						
	3 M NaOH Added	2570	2899.3	1.13	3.0	0	0%
	Cooked for 8 hours @ 85°C						
	Evaporation	169	169	1.00	0	0	0%
	Test Sample #2	3955.4	4593.4	1.16	1.928	380.7	8.2878%
	Slurry lost in transfer	17	20	1.16	1.928	1.2	6.1599%
	Water Added	100	100	1.00	0.000	0	0%
	Removed Leach Permeate						
	#1	1525	1669.87	1.09	1.928	0	0%
	#2	1186	1298.97	1.09	1.928	0	0%
	Permeate Sample -011	15	16.5	1.09	1.93	0	0%
	Test Sample #2	1311.4	1688.0	1.29	1.782	379.5	22.4792%
	Removed Centrifuge Sample	6.9	8.44	1.22	1.782	1.5	17.2134%
	Water Added	107	107.18	1.00	0.000	0	0%
27-Aug-99	Caustic Wash #1						
	0.01 M NaOH Added	1405	1405.2	1.00	0.01	0	0%
	Cooked for 8 hours @ 85°C						
	Evaporation	100	100.0	1.00	0	0	0%
	Test Sample #2	2716.9	3092.0	1.14	0.861	347.8	11.2%
	Removed Wash Permeate	#1	1601	1671.33	1.044	0	0%
28-Aug-99	Caustic Wash #2						
	Water Added	107	107.19	1.00	0.000	0	0%
	0.01 M NaOH Added	1405	1405.0	1.00	0.01	0	0%
	Cooked for 8 hours @ 85°C						
	Evaporation	100	100.0	1.00	0	0	0%
	Test Sample #2	2528.2	2832.9	1.12	0.386	340.8	12.0%
	Removed Wash Permeate	#2	1435	1453.04	1.0125	0	0%
	Inhibited Water Added	157	157	1.00	0.01	0	0%
	Water Added	363	363	1.00	0	0	0%
	Test Sample #2	1629.6	1900.1	1.17	0.262	340.8	17.9%
	Actual Test Sample	954.8	1113.2	1.17	0.262	211.0	19.0%
	Removed Centrifuge Sample	6.7	7.81	1.17	0.262	1.5	19.0%
	Slurry Sample -012	9	10.72	1.17	0.262	2.0	19.0%
	Slurry Sample -013	14	16.53	1.17	0.262	3.1	19.0%
	Slurry Sample -014	14	16.22	1.17	0.262	3.1	19.0%
	Final Test Sample	910.8	1062.0	1.17	0.262	331.1	31.2%
	Theoretical Test Smpl #2 with #7 still in	2479	2803.3	1.13	0.009	484.2	17.3%
	Liquid that would have to be removed to reach 36.6wt%	1302	1328.0	1.02	0.009	0	0%
	Theoretical Test Sample #2	1177	1475.3	1.25	0.009	398.2	27.0%

Appendix C: Analytical Results

Appendix C: Analytical Results

Appendix C: Analytical Results

Slurry Samples Analytical Evaluation

The ICP and GEA data from the acid digestions and fusion for samples CUF-C104-010, -013, and -014 were evaluated. The samples and duplicates for samples -010 and -013 were found to be very inconsistent. The original material from these samples was acid digested a second time and re-run for both ICP and GEA. The results of these re-runs indicate that:

- The original acid digestion and fusion data for CUF-C104-010 and CUF-C104-013 "samples" (but not duplicates) are mixed up. That is, the CUF-C104-010 Sample results should be reported as CUF-C-104-013 Sample results and vice versa.
- The variability in the samples and duplicates are higher than normal, suggesting significant heterogeneity of the subsamples processed (by fusion and acid digestion) for analysis.
- Some of the variability noted is due to the differences between the digestion techniques and the after processing additions. The original acid digestions and fusions were process per procedure and had some small quantity of residue evident in the preparation media. The rerun acid digestions added a little hydrofluoric acid to assist in maintaining solubility of some of the analytes.
- A look at the overall analysis indicates that the KOH fusion for CUF-C104-013 D was not representative of the homogeneous sample and was thus not included in the mean.

The attached file summarizes selected analytes based on "as reported" and again after switching the sample results for the -010 and -013 "samples" and removing the KOH fusion for CUF-C104-013 D. Although the RSDs are a little higher than normal after "switching", the data appears quite consistent with the rerun acid digestions.

	010								013								Excluding	
	Acid	Acid	KOH	Acid	Acid	KOH	Average	RSD	Acid	Acid	KOH	Acid	Acid	KOH	Average	RSD	Average	RSD
	99-2424R	99-2524	99-2524	99-2424RD	99-2524D	99-2524D			99-2425R	99-2525	99-2525	99-2425RD	99-2525D	99-2525D				
Al	144000	34000	34800	165000	158000	151000	114467	54%	43700	100000	117000	41800	34800	74300	68600	55%	67460	56%
Ca	4000	8310	8650	4970	4850	5230	6152	30%	8680	3110	5900	8970	8640	6900	7033	21%	7060	36%
Cr	1620	1800	2020	1770	1720	1760	1782	8%	2100	1090	1840	2110	1860	1800	1800	8%	1800	23%
Fe	46000	87500	84300	50500	49200	48000	60917	29%	91300	31000	59500	97100	89900	65800	72433	25%	73760	38%
Mn	10200	19200	19300	11300	11300	10900	13700	30%	20000	7150	13500	20700	20000	15000	16058	22%	16270	36%
Na	33400	57300	54400	35200	33400	35100	41467	24%	69400	20200	42800	63300	59100	46300	50183	20%	50960	39%
Ni	2990	5450		3230	3160		3708	1%	5740	2010		5950	5700		4850	4%	4850	39%
Pb	1600	2760	3040	1790	1660	1800	2108	31%	3070	1050	2070	3180	2880	2360	2435	21%	2450	37%
Th	5900	117000	104000	72700	68900	64900	72233	25%	136000	43200	62400	134000	119000	77500	93683	36%	96920	42%
U	43000	98200	86500	53400	55900	49600	64267	26%	110000	34800	61300	114000	10400	68200	66450	64%	66100	69%
Zr	60600	41700	109000	63100	38600	59200	62033	48%	113000	26700	66700	123000	39900	82500	75300	46%	73860	58%
Co-60	0.218		0.436	0.229		0.259	0.286	39%	0.398		0.297	0.366		0.352	0.353	10%		
Cs-137	60.6		56.8	47.3		55.9	55.2	10%	51.4		57.6	47.5		55.9	53.1	10%		
Eu-154	1.97		4.13	2.15		2.27	2.63	42%	3.83		2.79	3.61		3.29	3.38	12%		
Eu-155	1.11		2.45	1.23		1.36	1.54	44%	2.12		1.76	2.05		1.94	1.97	7%		
Am-241	6.08		1.25	6.67		6.53	5.13	60%	12.9		8.43	12.0		10.2	10.9	16%		

blue highlight = 010 and 013 results swithed (i.e., assumed samples swithed)

	Switch								Switch								Excluding	
	Acid	Acid	KOH	Acid	Acid	KOH	Average	RSD	Acid	Acid	KOH	Acid	Acid	KOH	Average	RSD	Average	RSD
	99-2424R	99-2524	99-2524	99-2424RD	99-2524D	99-2524D			99-2425R	99-2524	99-2524	99-2425RD	99-2525D	99-2525D				
Al	144000	100000	117000	165000	158000	151000	139167	15%	43700	34000	34800	41800	34800	74300	43900	43%	37820	12%
Ca	4000	3110	5900	4970	4850	5230	4827	10%	8680	8310	8650	8970	8640	6900	8358	11%	8650	3%
Cr	1620	1090	1840	1770	1720	1760	1633	3%	2100	1800	2020	2110	1860	1800	1948	7%	1978	7%
Fe	46000	31000	59500	50500	49200	48000	47367	11%	91300	87500	84300	97100	89900	65800	85983	16%	90020	5%
Mn	10200	7150	13500	11300	11300	10900	10725	11%	20000	19200	19300	20700	20000	15000	19033	13%	19840	3%
Na	33400	20200	42800	35200	33400	35100	33350	13%	69400	57300	54400	63300	59100	46300	58300	13%	60700	10%
Ni	2990	2010		3230	3160		2848	2%	5740	5450		5950	5700		5710	3%	5710	4%
Pb	1600	1050	2070	1790	1660	1800	1662	10%	3070	2760	3040	3180	2880	2360	2882	12%	2986	6%
Th	5900	43200	62400	72700	68900	64900	53000	9%	136000	117000	104000	134000	119000	77500	112917	21%	120000	9%
U	43000	34800	61300	53400	55900	49600	49500	10%	110000	98200	86500	114000	104000	68200	96817	21%	102540	11%
Zr	60600	26700	66700	63100	38600	59200	52483	24%	113000	41700	109000	123000	39900	82500	84850	43%	85320	48%
Co-60	0.218		0.297	0.229		0.259	0.251	14%	0.398		0.436	0.366		0.352	0.388	12%		
Cs-137	60.6		57.6	47.3		55.9	55.4	10%	51.4		56.8	47.5		55.9	52.9	10%		
Eu-154	1.97		2.79	2.15		2.27	2.30	15%	3.83		4.13	3.61		3.29	3.72	11%		
Eu-155	1.11		1.76	1.23		1.36	1.37	20%	2.12		2.45	2.05		1.94	2.14	13%		
Am-241	6.08		8.43	6.67		6.53	6.93	15%	12.9		12.5	12		10.2	11.90	10%		

	014		Average
	Acid 99-2526	KOH 99-2526	
Al	33700	34100	33900
Ca	8310	8270	8290
Cr	1830	1950	1890
Fe	87800	85300	86550
Mn	19300	19200	19250
Na	54300	51900	53100
Ni	5480		
Pb	2740	2970	2855
Th	114000	77300	95650
U	100000	86700	93350
Zr	51200	104000	77600
Co-60		0.428	
Cs-137		57.8	
Eu-154		4.1	
Eu-155		2.52	
Am-241		12.7	

013 and 014	
Average	RSD
59925	55%
7348	27%
1823	18%
75963	29%
16856	28%
50913	30%
4976	33%
2540	28%
94175	35%
73175	50%
75875	47%
0.368	13%
54.0	8%
3.52	14%
2.08	14%
11.2	17%

	014		Average
	Acid 99-2526	KOH 99-2526	
Al	33700	34100	33900
Ca	8310	8270	8290
Cr	1830	1950	1890
Fe	87800	85300	86550
Mn	19300	19200	19250
Na	54300	51900	53100
Ni	5480		
Pb	2740	2970	2855
Th	114000	77300	95650
U	100000	86700	93350
Zr	51200	104000	77600
Co-60		0.428	
Cs-137		57.8	
Eu-154		4.1	
Eu-155		2.52	
Am-241		12.7	

013 and 014	
Average	RSD
(Excluding 013 KOH)	
36700	38%
8547	7%
1953	7%
89029	10%
19671	9%
58529	12%
5664	4%
2949	9%
113043	19%
99914	15%
83114	41%
0.396	9%
53.9	8%
3.79	9%
2.22	12%
12.1	9%

**Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report**

Project: 29953
Client: K. Brooks

ACL Number(s): 99-2523 through 99-2535

Client ID: "CUF-C104-005" through "CUF-C104-015"

ASR Number: 5500

Total Samples: 13

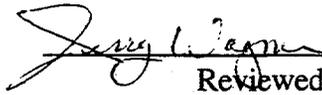
Procedure: PNL-ALO-211, "Determination of Elements by Inductively Coupled
Argon Plasma Atomic Emission Spectrometry" (ICP-AES).

Analyst: JJ Wagner

Analysis Date (Filename): 9-15-99 (A0543, ALO-128),
9-24-99 (A0544, ALO-115 K/Ni fusion),
9-28-99 (A0545, ALO-129 [K & Ni only])

See Chemical Measurement Center 98620: ICP-325-405-1 File for Calibration and
Maintenance Records.

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

 10-14-99
Reviewed by

 10-18-99
Concur

10/14/99

**Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report**

Nine radioactive liquid samples, CUF-C104-005 through CUF-C104-015 (ACL# 99-2527 through 99-2535), were analyzed by ICPAES after preparation by the Sample Receiving and Preparation Laboratory (SRPL). Samples were prepared by SRPL using PNL-ALO-128 acid digestion procedure and plastic vials. Approximately 4 ml of sample (weighed) was processed and diluted to a final volume of 20ml. The final volume was weighed and the density estimated. Density of each original supernatant sample was also estimated by weighing a ten ml aliquot. Upon addition of nitric acid some samples formed a gel (CUF-C104-016, the duplicate and CUF-C104-021, and duplicate). Additional acid solubilized the gel. After digestion all samples were brought to 20ml volume, remained clear, and did not require filtering. Some material was lost during processing of CUF-C104-011 duplicate and was therefore not analyzed.

Four radioactive solid samples were prepared by Shielded Analytic Lab (SAL), CUF-C104-004 through CUF-C104-014 (ACL# 99-2523 through 99-2526), three in duplicate (ACL# 99-2523 through 99-2525), and analyzed by ICPAES. Approximately 0.2g aliquots were used to prepare samples using fusion procedure PNL-ALO-115 (KOH/Ni). The fusion samples were diluted to a final volume of 100 ml and diluted 2.02-fold further using 2% v/v HCl because of ALARA radiation dose concerns. Additional dilution of 5-fold was sometimes necessary during ICPAES analysis because of high sodium, iron, thorium, uranium and/or zirconium concentration. Fusion prepared samples required additional HCl to dissolve brown precipitates or gels that formed after samples were fused. All solutions remained soluble after final dilution.

The four solid samples identified above were also processed using PNL-ALO-129 acid digestion procedure for solids by SAL. These samples were analyzed by ICPAES only for Ni and K. Sample size processed varied from about 0.19g to 0.31g. Samples were processed using plastic vials as requested by client. After digestion each sample was diluted to a final volume of approximately 20ml. A 1ml aliquot taken from each processed sample was weighed and the density estimated by dividing the aliquot weight by the weight of water using the same pipette. The final volume of each processed sample was determined using the final weight of processed sample divided by the estimated density.

Measurement results reported have been corrected for preparation and analytical dilution. All results reported are in $\mu\text{g/g}$ for the solids and $\mu\text{g/ml}$ for liquid samples. Volumes and weights have been recorded on bench sheets and included with this report. Please note, detection limit for fusion prepared samples have been modified to reflect uncertainties for some analytes due to unusually high concentration of thorium, uranium and zirconium in the solid samples. Detection limits were modified as judged by wavelength scans of fusion prepared samples.

10/14/99

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ... ICPAES Data Report

Specific analytes of interest requested for solid samples (ALO# 99-2523 through 99-2526) prepared using PNL-ALO-115 fusion procedure are identified in table 1.2, page 12, "Analytical Requirements for Filtrate, Washed Solids, and Wash Solutions" (Washed Solids). Analytes required included: Ag, Al, As, B, Ba, Be, Ca, Cd, Ce, Co, Cr, Cu, Fe, K, La, Li, Mg, Mn, Mo, Na, Nd, Ni, Pb, Pd, Sb, Se, Si, Sr, Te, Th, Ti, Tl, V, U, W, Y, Zn, and Zr.

An additional sample preparation for solid samples (ALO# 99-2523 through 99-2526) was performed using PNL-ALO-129 acid digestion of solids. These samples were analyzed for **K** and **Ni** only. Potassium was not detected in any of the samples.

Specific analytes of interest requested for liquid samples (ALO# 99-2527 through 99-2534) prepared using PNL-ALO-128 are identified in table 1.2, page 12, "Analytical Requirements for Filtrate, Washed Solids, and Wash Solutions" (Filtrate, Wash Solutions). Analytes required included: Ag, Al, Ba, Ca, Cd, Co, Cr, Cu, Fe, K, La, Mg, Mn, Mo, Na, Ni, Pb, Si, Ti, U, and Zn.

Sample CUF-C104-015 (ALO# 99-2535) was prepared using PNL-ALO-128 but analyzed only for sodium.

The fusion prepared solid samples contained high concentrations (1 to 15 Wt%) of aluminum, iron, sodium, silicon, thorium, uranium and zirconium. The acid digested solid samples also contained high concentrations of the same elements similar to the fused samples but were generally lower for zirconium, silicon and other refractory elements. Analyze concentration in the original sample aliquot of CUF-C104-010 was considerably different than the duplicate aliquot. Aluminum and several other analytes varied greatly (125 to 50%RPD) between sample and duplicate. The same acid digestion prepared solid sample also had similar high %RPD's.

The liquid samples contained high concentrations of sodium. A few samples had high concentrations of aluminum. All other analytes measured were much lower in concentration. Thorium was not detected in any of the liquid samples.

Quality control check-standard results met tolerance requirements for analytes of interest except as noted below. Following is a list of quality control measurement results relative to ICPAES analysis tolerance requirements under MCS-033.

10/14/99

**Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report**

Five fold serial dilution:

- (Solid samples/fusion) Results were within tolerance limit of $\leq 10\%$ after correcting for dilution except as follows. Thorium was somewhat high (approximately 17%) in only one of seven KOH/Ni fusion prepared samples, 99-2526-Ni. The discrepancy may be caused by non-linearity. The measured concentration was several times higher than the high calibration concentration of 10 $\mu\text{g/ml}$. The concentration in the undiluted sample measured about 75 $\mu\text{g/ml}$.
- (Solid samples/acid dig.) Analytes of interest included K & Ni only and were within tolerance limit of $\leq 10\%$ after correcting for dilution.
- (Aqueous samples) All results for analytes of interest were within tolerance limit of $\leq 10\%$ after correcting for dilution except barium, silicon, chromium, and nickel. Barium concentration varied up to 73% for sample 99-2527 and 99-2527-D (duplicate). This may be affected by the presence of sulfate in the sample. When diluted with additional acid, barium becomes more soluble. Thus the 5-fold dilution measured higher in concentration (dilution-corrected) than the undiluted sample for both original and duplicate of the same sample. Silicon concentration varied by more than 100% in nearly all samples. The reason for the large variability is not clear. Perhaps silicic acid had begun to precipitate. Depending upon where in the sample vial the dilution aliquot was withdrawn may have affected the amount of silicic acid measured in the diluted sample. The undiluted sample typically measured higher in concentration than the 5-fold diluted sample (dilution-corrected). Chromium and nickel concentration varied up to 23% difference only in sample 99-2534 but was within tolerance limit when measured in the duplicate sample.

Duplicate RPD (Relative Percent Difference):

- (Solid samples/fusion) All analytes of interest were recovered within tolerance limit of $\leq 20\%$ relative percent difference (RPD) except aluminum, thorium and zirconium in sample 99-2525-Ni and its duplicate, all analytes in sample 99-2524-Ni and its duplicate, and barium in sample 99-2523-Ni and its duplicate. Sample 99-2524-Ni and its duplicate do not appear to be related or is non-homogenous. Aluminum, thorium and zirconium appear to be non-homogenous in sample 99-2525-Ni also. Barium in sample 99-2523-Ni may be caused by the presence of sulfate in the sample.

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Duplicate RPD (Relative Percent Difference):

(Solid samples/acid dig.) All analytes of interest were recovered within tolerance limit of $\leq 20\%$ relative percent difference (RPD) except nickel. A large difference in RPD for nickel was found between sample duplicates 99-2524 and 99-2525. RPD for nickel in sample 99-2524 was about 53%, and about 96% RPD in sample 99-2525.

(Aqueous samples) All analytes of interest were recovered within tolerance limit of $\leq 20\%$ relative percent difference (RPD) except for barium, silicon and zinc. Barium varied by about 60%RPD in sample 99-2527 and 99-2527-D only. This may be related to the presence of sulfate in the sample as indicated by the poor recovery of barium when diluted as mentioned above (serial dilution). Silicon varied up to about 32%RPD in three samples (99-2527, 99-2531, and 99-2534). The variability between duplicates may be related to formation of silicic acid precipitate as mentioned above. Zinc varied up to 57%RPD in three samples (99-2527, 99-2529, and 99-2530). The reason for the variability is not apparent. It may be related to sample homogeneity. In general most of the analytes measured were found to be less than 10%RPD.

Post-Spiked Samples (Group A):

(Solid samples/fusion) All analytes of interest were recovered within tolerance of 75% to 125%.

(Solid samples/acid dig.) All analytes of interest were recovered within tolerance of 75% to 125%.

(Aqueous samples) All analytes of interest were recovered within tolerance of 75% to 125%.

Post-Spiked Samples (Group B):

(Solid samples/fusion) All analytes of interest were recovered within tolerance of 75% to 125%.

(Solid samples/acid dig.) All analytes of interest were recovered within tolerance of 75% to 125%.

(Aqueous samples) All analytes of interest were recovered within tolerance of 75% to 125%.

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Blank Spike:

(Solid samples/fusion) A blank spike is not require for fusion prepared samples.

(Solid samples/acid dig.) None prepared.

(Aqueous samples) All analytes of interest in the blank spike were recovered within tolerance limit of 80% to 120% except Ag (53/54%) in sample 99-2527-BS and 99-2527-BS-Duplicate and Zn (61%) in sample 99-2527-BS only. Chloride from the sample or from the hydrochloric acid used to prepare the sample using PNL-ALO-128 digestion procedure may have precipitated the silver resulting in low recovery. The apparent low recovery for Zn appears to be caused by zinc contamination in the associated process blank. The duplicate spike blank and duplicate process blank indicated a zinc recovery of 91%, well within tolerance limits.

Matrix Spiked Sample:

(Solid samples/fusion) A matrix spike is not require for fusion prepared samples.

(Solid samples/acid dig.) None prepared.

(Aqueous samples) A matrix spike was not prepared due to limited sample availability.

Quality Control Check Standards (solid samples/fusion):

Concentration of all analytes of interest in the KOH/Ni fusion prepared analytical run was within tolerance limit of $\pm 10\%$ accuracy in standards: QC_MCVA, QC_MCVB, and QC_SSTMVCV except as follows. Beryllium was low (-12%) in QC_MCVA one out of three times and silicon was slightly high (11%) in QC_SSTMVCV one out of two times.

Calibration Blank (ICP98.0) concentration was acceptable, less than two times IDL.

High Calibration Standard Check (solid samples/fusion):

Verification of the high-end calibration concentration for all analytes of interest is within tolerance of $\pm 5\%$ accuracy.

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Quality Control Check Standards (solid samples/acid dig.):

Concentration of all analytes of interest was within tolerance limit of $\pm 10\%$ accuracy in standards: QC_MCVA, QC_MCVB, and QC_SSTMCV. Calibration Blank (ICP98.0) concentration was acceptable, less than two times IDL.

High Calibration Standard Check (solid samples/acid dig.):

Verification of the high-end calibration concentration for all analytes of interest is within tolerance of $\pm 5\%$ accuracy.

Quality Control Check Standards (aqueous samples):

Concentration of all analytes of interest in the acid digested aqueous prepared analytical run was within tolerance limit of $\pm 10\%$ accuracy in the standards: QC_MCVA, QC_MCVB, and QC_SSTMCV except as follows. Sodium was high, up to 17% in three of five QC_MCVA check standard measurements. The high sodium value may be due to memory effect or carry-over from one measurement to the next. It may have been caused by not allowing sufficient time to clean out the sample delivery system between measurements of a previous sample containing very high concentration of sodium.

Concentration of silicon was greater than $2 * IDL$ (up to 10 times IDL) in the calibration blank ICP98.0 measurement checks during the run. As mentioned earlier, precipitation of silicic acid in the samples may have been the cause, requiring longer than normal clean-out time between sample measurements.

High Calibration Standard Check (aqueous samples):

Verification of the high-end calibration concentration for all analytes of interest was within tolerance of $\pm 5\%$ accuracy.

Process Blank:

(Solid samples/fusion)

All analytes of interest were within tolerance limit of $\leq EQL$ or $< 5\%$ of sample concentration in PNL-ALO-115 KOH/Ni fusion prepared samples. Sodium is known to be present in the reagents used to prepare the samples but was less than 5% of the lowest concentration of sodium found in any sample measured.

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(Solid samples/acid dig.) All analytes of interest were within tolerance limit of \leq EQL or $< 5\%$ of sample concentration.

(Aqueous samples) All analytes of interest were within tolerance limit of \leq EQL or $< 5\%$ of sample concentration except Ba ($\gg 100\%$ of the sample conc.) and Zn (greater than 17% to 91% of sample conc.) Both blanks appear to be contaminated with barium and zinc. Barium concentration in the blanks is much higher than that found in the samples. Zinc in one blank was five times higher than zinc measured in the duplicate blank. The source of contamination is not known. Since both barium and zinc are very high in the process blanks, sample measurements for these two analytes should be considered suspect.

Laboratory Control Standard (LCS):

(Solid samples/fusion) All analytes of interest except strontium, at a concentration equal to or greater than EQL were recovered within tolerance limit of 75% to 125% in fusion prepared LCS standards. SRM-2710 Montana Soil was used for the LCS in both PNL-ALO-114 and PNL-ALO-115 fusion preparations. Strontium recovery was high (127% recovery).

(Solid samples/acid dig.) No LCS was prepared for PNL-ALO-129 acid digested samples.

(Aqueous samples) No LCS was prepared for PNL-ALO-128 acid digested samples.

Analytes other than those requested by the client are for information only. Please note bracketed values listed in the data report are within ten times instrument detection limit and have a potential uncertainty much greater than 15%.

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Comments:

- 1) "Final Results" have been corrected for all laboratory dilution performed on the sample during processing and analysis unless specifically noted.
- 2) Detection limits (DL) shown are for acidified water. 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_3 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 $\mu\text{g/mL}$ (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.

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Det. Limit (ug/mL)	Analyte	Multiplier= ALO#= Client ID= Run Date= (ug/mL)	5.0 99-2527-B Process Blk 9/15/99 (ug/mL)	5.0 99-2527-B DUP Process Blk duplicate 9/15/99 (ug/mL)	5.0 99-2527 @1 CUF-C104-005 9/15/99 (ug/mL)	5.0 99-2527-D @1 CUF-C104-005 9/15/99 (ug/mL)	25.0 99-2528 @5 CUF-C104-011 9/15/99 (ug/mL)
0.015	Ag		-	-	[0.39]	[0.30]	-
0.060	Al		[0.31]	-	231	234	15,600
0.080	As		-	-	[0.46]	-	-
0.050	B		-	-	4.99	5.17	[7.1]
0.010	Ba		13.6	7.21	1.98	1.06	-
0.010	Be		-	-	-	-	[1.1]
0.100	Bi		[0.97]	[0.66]	[3.3]	[2.4]	-
0.100	Ca		[1.3]	-	[2.5]	[2.0]	-
0.015	Cd		[0.11]	[0.094]	2.11	2.02	-
0.100	Ce		-	-	-	-	-
0.025	Co		-	-	[0.65]	[0.66]	-
0.020	Cr		-	-	18.4	18.7	59.3
0.015	Cu		-	-	2.16	2.14	[0.51]
0.050	Dy		-	-	-	-	-
0.100	Eu		-	-	-	-	-
0.025	Fe		-	-	1.45	[1.2]	[3.4]
2.000	K		-	-	169	165	[51]
0.025	La		-	-	-	-	-
0.005	Li		[0.035]	[0.038]	8.43	8.54	30.0
0.100	Mg		[0.55]	-	-	-	-
0.005	Mn		-	-	-	-	-
0.030	Mo		-	-	2.56	2.63	[1.7]
0.300	Na		7.82	[3.7]	31,600	31,900	46,500
0.030	Nd		-	-	-	-	-
0.030	Ni		-	-	35.4	36.0	[0.78]
0.100	P		-	-	344	349	362
0.060	Pb		[0.47]	[0.40]	[1.8]	[1.0]	[4.9]
0.300	Pd		-	-	-	-	-
0.300	Rh		-	-	-	-	-
0.075	Ru		-	-	[1.5]	[1.5]	-
0.050	Sb		-	-	-	-	-
0.050	Se		-	-	[0.50]	[0.41]	[3.3]
0.100	Si		-	-	163	185	125
1.000	Sn		-	-	[11]	[11]	[55]
0.005	Sr		[0.13]	[0.066]	[0.038]	[0.030]	-
0.500	Te		-	-	-	-	-
0.800	Th		-	-	-	-	-
0.005	Ti		[0.090]	-	-	-	-
0.250	Tl		-	-	-	-	-
2.000	U		-	-	[21]	[21]	-
0.015	V		-	-	[0.17]	[0.17]	[0.76]
0.500	W		-	-	-	-	-
0.010	Y		-	-	-	-	-
0.020	Zn		8.94	1.62	1.20	1.51	7.97
0.025	Zr		-	-	-	-	[0.72]

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).

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Multiplie#	5.0	5.0	5.0	5.0	5.0
ALO#	99-2529 @1	99-2529-D @1	99-2530 @1	99-2530-D @1	99-2531 @1
Client ID	CUF-C104-016	CUF-C104-016	CUF-C104-017	CUF-C104-017	CUF-C104-018
Run Date	9/15/99	9/15/99	9/15/99	9/15/99	9/15/99
Det. Limit (ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.015	Ag	-	-	-	-
0.060	Al	321	327	212	215
0.080	As	-	-	-	-
0.050	B	3.69	4.06	3.32	3.60
0.010	Ba	1.13	1.17	[0.19]	[0.26]
0.010	Be	-	-	-	-
0.100	Bi	[2.3]	[1.7]	[1.3]	[1.0]
0.100	Ca	[1.5]	[1.4]	-	[0.99]
0.015	Cd	[0.38]	[0.38]	[0.10]	[0.10]
0.100	Ce	-	-	-	-
0.025	Co	[0.37]	[0.38]	-	-
0.020	Cr	13.3	13.4	4.50	4.57
0.015	Cu	0.773	0.781	[0.14]	[0.13]
0.050	Dy	-	-	-	-
0.100	Eu	-	-	-	-
0.025	Fe	[0.36]	[0.31]	-	-
2.000	K	[73]	[72]	[13]	[14]
0.025	La	-	-	-	-
0.005	Li	2.51	2.74	1.09	1.14
0.100	Mg	[0.58]	-	-	-
0.005	Mn	-	-	-	-
0.030	Mo	[1.4]	[1.4]	[0.43]	[0.44]
0.100	Na	18,700	18,200	7,030	7,000
0.100	Nd	-	-	-	-
0.030	Ni	16.9	17.3	4.89	5.01
0.100	P	136	138	29.7	30.3
0.060	Pb	[0.50]	[0.37]	-	-
0.300	Pd	-	-	-	-
0.300	Rh	-	-	-	-
0.075	Ru	[0.72]	[0.71]	-	-
0.050	Sb	-	-	-	-
0.050	Se	[0.35]	[0.33]	-	-
0.100	Si	136	177	62.2	74.1
1.000	Sn	[8.4]	[8.4]	-	-
0.005	Sr	[0.027]	[0.034]	-	-
0.500	Te	-	-	-	-
0.800	Th	-	-	-	-
0.005	Ti	[0.071]	[0.037]	-	-
0.250	Tl	-	-	-	-
2.000	U	[17]	[16]	[12]	[12]
0.015	V	[0.11]	[0.11]	-	-
0.500	W	-	-	-	-
0.010	Y	-	-	-	-
0.020	Zn	9.88	5.47	1.12	1.39
0.025	Zr	-	-	-	[0.66]

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)	5.0 99-2531-D @1 CUF-C104-018 9/15/99 (ug/mL)	25.0 99-2532 @5 CUF-C104-021 9/15/99 (ug/mL)	25.0 99-2532-D @5 CUF-C104-021 9/15/99 (ug/mL)	5.0 99-2533 @1 CUF-C104-022 9/15/99 (ug/mL)	5.0 99-2533-D @1 CUF-C104-022 9/15/99 (ug/mL)
0.015	Ag	-	-	-	[0.080]	[0.23]
0.060	Al	140	7,590	7,950	2,700	2,600
0.080	As	-	-	-	[0.42]	[2.3]
0.050	B	2.77	[3.3]	[3.3]	2.76	2.78
0.010	Ba	[0.069]	-	-	-	[0.12]
0.010	Be	-	[0.28]	[0.30]	-	[0.051]
0.100	Bi	[1.6]	-	-	[2.1]	[1.7]
0.100	Ca	-	-	-	-	-
0.015	Cd	-	-	-	-	-
0.100	Ce	-	-	-	-	[1.4]
0.025	Co	-	-	-	[0.13]	[0.27]
0.020	Cr	2.29	56.0	59.1	27.9	28.7
0.015	Cu	-	-	-	[0.15]	[0.30]
0.050	Dy	-	-	-	-	-
0.100	Eu	-	-	-	-	-
0.025	Fe	[0.40]	[1.7]	[1.8]	[0.75]	[0.86]
2.000	K	-	-	-	[12]	[44]
0.025	La	-	-	-	-	[0.35]
0.005	Li	0.339	12.3	12.9	5.32	4.95
0.100	Mg	-	-	-	-	[1.0]
0.005	Mn	[0.063]	-	-	[0.027]	[0.051]
0.030	Mo	[0.23]	[0.91]	[0.99]	[0.47]	[0.64]
0.100	Na	3,550	20,600	21,500	8,050	7,960
0.100	Nd	-	-	-	-	[1.4]
0.030	Ni	2.39	-	-	[0.28]	[0.40]
0.100	P	13.1	138	145	38.6	42.0
0.060	Pb	-	[2.9]	[3.6]	[1.2]	3.19
0.300	Pd	-	-	-	-	[3.3]
0.300	Rh	-	-	-	-	[1.9]
0.075	Ru	-	-	-	-	[0.82]
0.050	Sb	-	-	-	-	[1.3]
0.050	Se	-	[2.6]	[2.9]	[1.0]	[2.1]
0.100	Si	23.1	28.0	27.4	14.4	15.6
1.000	Sn	-	[25]	[28]	[6.1]	[10]
0.005	Sr	-	-	-	-	-
0.500	Te	-	-	-	-	-
0.800	Th	-	-	-	-	-
0.005	Ti	-	-	-	[0.026]	[0.088]
0.250	Tl	-	-	-	-	[1.3]
2.000	U	[13]	-	-	[14]	[30]
0.015	V	-	[0.55]	[0.61]	[0.31]	[0.42]
0.500	W	-	-	-	-	-
0.010	Y	-	-	-	-	[0.071]
0.020	Zn	[0.86]	[4.0]	[4.1]	[0.46]	[0.52]
0.025	Zr	[0.66]	-	-	-	-

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)	5.0 99-2534 @1 CUF-C104-019 9/15/99 (ug/mL)	5.0 99-2534-D @1 CUF-C104-019 9/15/99 (ug/mL)	5.0 99-2535 @1 CUF-C104-015 9/15/99 (ug/mL)		
0.015	Ag	-	-	-	-	-
0.060	Al	207	206	2,160	-	-
0.080	As	-	-	-	-	-
0.050	B	3.21	3.62	8.39	-	-
0.010	Ba	[0.12]	[0.051]	-	-	-
0.010	Be	-	-	-	-	-
0.100	Bi	[0.68]	[0.54]	[0.76]	-	-
0.100	Ca	-	-	-	-	-
0.015	Cd	[0.45]	[0.45]	-	-	-
0.100	Ce	-	-	-	-	-
0.025	Co	[0.22]	[0.22]	-	-	-
0.020	Cr	7.35	7.39	22.7	-	-
0.015	Cu	[0.55]	[0.56]	-	-	-
0.050	Dy	-	-	-	-	-
0.100	Eu	-	-	-	-	-
0.025	Fe	-	-	[0.72]	-	-
2.000	K	[41]	[43]	-	-	-
0.025	La	-	-	-	-	-
0.005	Li	3.40	3.40	3.86	-	-
0.100	Mg	-	-	-	-	-
0.005	Mn	-	-	[0.14]	-	-
0.030	Mo	[1.0]	[1.0]	[0.31]	-	-
0.100	Na	11,100	13,900	6,980	-	-
0.100	Nd	-	-	-	-	-
0.030	Ni	13.0	12.9	[0.18]	-	-
0.100	P	101	100	63.1	-	-
0.060	Pb	-	-	-	-	-
0.300	Pd	-	-	-	-	-
0.300	Rh	-	-	-	-	-
0.075	Ru	[0.44]	[0.46]	-	-	-
0.050	Sb	-	-	-	-	-
0.050	Se	-	-	[0.41]	-	-
0.100	Si	87.5	109	23.8	-	-
1.000	Sn	-	-	-	-	-
0.005	Sr	-	-	-	-	-
0.500	Te	-	-	-	-	-
0.800	Th	-	-	-	-	-
0.005	Ti	-	-	-	-	-
0.250	Tl	-	-	-	-	-
2.000	U	[16]	[17]	-	-	-
0.015	V	[0.083]	[0.087]	[0.11]	-	-
0.500	W	-	-	-	-	-
0.010	Y	-	-	-	-	-
0.020	Zn	[0.48]	[0.31]	[0.41]	-	-
0.025	Zr	-	-	[0.39]	-	-

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
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 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).

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Det. Limit (ug/mL)	Multiplier=	1072.4	1131.0	960.5	1141.9	1132.9
Run Date=	ALO#=	99-2523-PB-NI @1	99-2523-NI @1	99-2523-D-NI @1	99-2524-NI @1	99-2524-D-NI @1
(Analyte)	Client ID=	Process Blank	CUF-C104-004	CUF-C104-004	CUF-C104-010	CUF-C104-010
(ug/mL)	Run Date=	9/24/99	9/24/99	9/24/99	9/24/99	9/24/99
	(Analyte)	ug/g	ug/g	ug/g	ug/g	ug/g
0.025	Ag	-	1,020	971	1,720	977
0.060	Al	[98]	123,000	121,000	34,800	151,000
0.100	As	-	-	-	-	-
0.050	B	-	[250]	[250]	[65]	[80]
0.010	Ba	-	302	174	370	206
0.010	Be	-	[31]	[31]	[55]	[39]
0.175	Bi	[390]	-	-	-	-
0.250	Ca	[540]	4,280	4,310	8,650	5,230
0.015	Cd	-	740	738	1,600	902
1.150	Ce	-	-	-	-	-
0.050	Co	-	[64]	[59]	[82]	-
0.020	Cr	-	1,520	1,470	2,020	1,760
0.425	Cu	-	-	-	-	-
0.085	Dy	-	-	-	-	-
0.100	Eu	-	-	-	-	-
0.025	Fe	[120]	38,300	38,500	84,300	48,000
0.275	La	-	-	-	-	-
0.030	Li	-	354	355	400	447
1.500	Mg	-	-	-	-	-
0.050	Mn	-	8,800	8,860	19,300	10,900
0.060	Mo	-	-	-	-	-
0.150	Na	2,170	114,000	111,000	54,400	35,100
0.575	Nd	-	-	-	-	-
0.100	P	[310]	3,960	3,900	4,790	4,290
0.100	Pb	[110]	1,560	1,510	3,040	1,800
0.750	Pd	-	-	-	-	-
0.300	Rh	-	-	-	-	-
1.100	Ru	-	-	-	-	-
0.500	Sb	-	-	-	-	-
0.250	Se	-	-	-	-	-
0.500	Si	-	11,000	10,900	22,300	12,900
2.400	Sn	-	-	-	-	-
0.015	Sr	[29]	[87]	[85]	176	[100]
1.500	Te	-	-	-	-	-
1.000	Th	-	53,600	53,800	104,000	64,900
0.025	Ti	-	[200]	[210]	421	[250]
0.500	Tl	-	-	-	-	-
2.000	U	-	40,400	40,200	86,500	49,600
0.075	V	-	-	-	-	-
2.000	W	-	-	-	-	-
0.075	Y	-	-	-	-	-
0.050	Zn	-	[240]	[230]	724	[270]
0.050	Zr	-	46,500	47,200	109,000	59,200

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).

mult by
1.25
to get
13

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

Multipler=	1041.2	1108.7	1019.2		
ALO#=	99-2525-Ni @1	99-2525-D-Ni @1	99-2526-Ni @1		
Client ID=	CUF-C104-013	CUF-C104-013	CUF-C104-014		
Run Date=	9/24/99	9/24/99	9/24/99		
Det. Limit (ug/mL)	(Analyte)	ug/g	ug/g	ug/g	
0.025	Ag	1,190	1,360	1,740	--
0.060	Al	117,000	74,300	34,100	--
0.100	As	--	--	--	--
0.050	B	[64]	[63]	[80]	--
0.010	Ba	270	304	395	--
0.010	Be	[43]	[46]	[54]	--
0.175	Bi	--	--	--	--
0.250	Ca	5,900	6,900	8,270	--
0.015	Cd	1,110	1,230	1,590	--
1.150	Ce	--	--	--	--
0.050	Co	[56]	[66]	[70]	--
0.020	Cr	1,840	1,800	1,950	--
0.425	Cu	--	--	--	--
0.085	Dy	--	--	--	--
0.100	Eu	--	--	--	--
0.025	Fe	59,500	65,800	85,300	--
0.275	La	--	--	--	--
0.030	Li	447	402	411	--
1.500	Mg	--	--	--	--
0.050	Mn	13,500	15,000	19,200	--
0.060	Mo	--	--	--	--
0.150	Na	42,800	46,300	51,900	--
0.575	Nd	--	--	--	--
0.100	P	1,910	3,520	2,680	--
0.100	Pb	2,070	2,360	2,970	--
0.750	Pd	--	--	--	--
0.300	Rh	--	--	--	--
1.100	Ru	--	--	--	--
0.500	Sb	--	--	--	--
0.250	Se	--	--	--	--
0.500	Si	15,900	17,700	21,600	--
2.400	Sn	--	--	--	--
0.015	Sr	[120]	[140]	177	--
1.500	Te	--	--	--	--
1.000	Th	62,400	77,500	77,300	--
0.025	Ti	292	329	376	--
0.500	Tl	--	--	--	--
2.000	U	61,300	68,200	86,700	--
0.075	V	--	--	--	--
2.000	W	--	--	--	--
0.075	Y	--	--	--	--
0.050	Zn	[390]	[490]	644	--
0.050	Zr	66,700	82,500	104,000	--

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).

4.45

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

Det. Limit (ug/mL)	Run Date= (Analyte)	Multiplier= ALO#= Client ID= 9/28/99 ug/g	81.3 99-2523-PB @1 Process Blank 9/28/99 ug/g	486.6 99-2523 @5 CUF-C104-004 9/28/99 ug/g	504.9 99-2523-D @5 CUF-C104-004 9/28/99 ug/g	361.1 99-2524 @5 CUF-C104-010 9/28/99 ug/g	543.3 99-2524-D @5 CUF-C104-010 9/28/99 ug/g
0.025	Ag	--	--	172	[15]	207	295
0.060	Al	[23]	[23]	127,000	123,000	34,000	158,000
0.250	As	--	--	--	--	--	--
0.050	B	--	--	[160]	[190]	[25]	[33]
0.010	Ba	12.3	12.3	177	144	404	217
0.010	Be	--	--	[31]	[30]	51.7	[40]
0.100	Bi	[19]	[19]	--	--	--	--
0.250	Ca	--	--	3,800	3,700	8,310	4,850
0.015	Cd	--	--	756	734	1,620	944
0.200	Ce	--	--	[600]	[550]	757	[570]
0.050	Co	--	--	--	--	[44]	--
0.020	Cr	98.3	98.3	1,330	1,280	1,800	1,720
0.025	Cu	--	--	174	154	391	224
0.050	Dy	--	--	[30]	[32]	[64]	[38]
0.100	Eu	--	--	--	--	--	--
0.025	Fe	473	473	38,400	29,000	87,500	49,200
2.000	K	--	--	--	--	--	--
0.050	La	--	--	[120]	[100]	267	[160]
0.030	Li	--	--	411	396	473	539
0.100	Mg	--	--	[440]	[420]	896	564
0.050	Mn	[11]	[11]	8,800	8,210	19,200	11,300
0.050	Mo	[9.3]	[9.3]	[24]	--	[20]	--
0.150	Na	[43]	[43]	106,000	108,000	57,300	33,400
	Nd	--	--	[240]	[220]	539	[300]
	Ni	87.7	87.7	2,520	2,320	5,450	3,160
0.100	P	--	--	[400]	[350]	[210]	[86]
0.100	Pb	--	--	1,290	1,070	2,760	1,660
0.750	Pd	--	--	--	--	--	--
0.300	Rh	--	--	--	--	--	--
1.100	Ru	--	--	--	--	--	--
0.500	Sb	--	--	--	--	--	--
0.250	Se	--	--	--	--	--	--
0.500	Si	--	--	[1,800]	3,020	4,930	2,850
1.500	Sn	--	--	[1,100]	[760]	[1,700]	[1,400]
0.015	Sr	--	--	79.1	[72]	183	102
1.500	Te	--	--	--	--	--	--
1.000	Th	--	--	56,100	54,100	117,000	68,900
0.025	Ti	--	--	[120]	[110]	229	149
0.500	Tl	--	--	--	--	--	--
2.000	U	--	--	43,500	41,900	98,200	55,900
0.050	V	--	--	[27]	[27]	[36]	[34]
2.000	W	--	--	--	--	--	--
0.050	Y	--	--	[30]	[28]	[64]	[37]
0.050	Zn	[13]	[13]	343	[230]	1,250	288
0.050	Zr	--	--	39,100	36,500	41,700	38,600

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).

Analytes requested by client:
Potassium and Nickel only

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

Det. Limit (ug/mL)	Multiplier= ALO#= Client ID= Run Date= (Analyte)	332.5 99-2525 @5 CUF-C104-013 9/28/99 ug/g	364.3 99-2525-D @5 CUF-C104-013 9/28/99 ug/g	345.8 99-2526 @5 CUF-C104-014 9/28/99 ug/g		
0.025	Ag	[62]	141	162	-	-
0.060	Al	100,000	34,800	33,700	-	-
0.250	As	-	-	-	-	-
0.050	B	-	[44]	[29]	-	-
0.010	Ba	195	430	478	-	-
0.010	Be	[26]	53.7	53.0	-	-
0.100	Bi	-	-	-	-	-
0.250	Ca	3,110	8,640	8,310	-	-
0.015	Cd	596	1,690	1,630	-	-
0.200	Ce	[390]	[680]	976	-	-
0.050	Co	[19]	[46]	[43]	-	-
0.020	Cr	1,090	1,860	1,830	-	-
0.025	Cu	142	412	405	-	-
0.050	Dy	[26]	[69]	[67]	-	-
0.100	Eu	-	-	-	-	-
0.025	Fe	31,000	89,900	87,800	-	-
2.000	K	-	-	-	-	-
0.050	La	[100]	286	281	-	-
0.030	Li	336	495	478	-	-
0.100	Mg	393	923	881	-	-
0.050	Mn	7,150	20,000	19,300	-	-
0.050	Mo	-	[19]	[19]	-	-
0.150	Na	20,200	59,100	54,300	-	-
0.100	Nd	[200]	561	541	-	-
0.030	Ni	2,010	5,700	5,480	-	-
0.100	P	[66]	[330]	[250]	-	-
0.100	Pb	1,050	2,880	2,740	-	-
0.750	Pd	-	-	-	-	-
0.300	Rh	-	-	-	-	-
1.100	Ru	-	-	-	-	-
0.500	Sb	-	-	-	-	-
0.250	Se	-	-	-	-	-
0.500	Si	[940]	4,080	3,880	-	-
1.500	Sn	[900]	[1,800]	[1,800]	-	-
0.015	Sr	65.4	192	188	-	-
1.500	Te	-	-	-	-	-
1.000	Th	43,200	119,000	114,000	-	-
0.025	Tl	95.5	222	240	-	-
0.500	Tl	-	-	-	-	-
2.000	U	34,800	104,000	100,000	-	-
0.050	V	[23]	[38]	[39]	-	-
2.000	W	-	-	-	-	-
0.050	Y	[24]	[66]	[66]	-	-
0.050	Zn	628	726	974	-	-
0.050	Zr	26,700	39,900	51,200	-	-

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).

Analytes requested by client:
Potassium and Nickel only

**Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report**

Project: 29953
Client: R.T. Hallen & K.P. Brooks

ACL Number(s): 99-2524-R through 99-2525-DUP-R
[re-runs]

Client ID: "CUF-C104-010(Orange)" through "CUF-C104-013(Black)"

ASR Number: 5500.01

Total Samples: 4

Procedure: PNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry" (ICP-AES).

Analyst: D.R. Sanders

Analysis Date (Filename): 11-04-99 (A0550)

See Chemical Measurement Center 98620: ICP-325-405-1 File for Calibration and Maintenance Records.

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

Jerry Wagner 1-18-00
Reviewed by

M. W. Kin 1-19-00
Concur

1/18/00

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ... ICPAES Data Report

Four radioactive solid samples CUF-C104-010(Orange) through CUF-C104-013(Black) (ACL# 99-2524-R through 99-2525-DUP-R) were prepared by SAL using ALO-129 acid digestion of solids procedure. Prepared samples were analyzed by ICPAES. Approximately 0.02g to 0.32g aliquots were processed and diluted to a final volume of about 20 ml (weighed). Some dark-black residue remained insoluble from all the samples after processing. The final volume of each processed sample was determined using the final weight of processed sample divided by an estimated density. Analytical dilution of 5-fold to 50-fold was required because of high concentration of aluminum, iron, manganese, sodium, thorium, uranium, and zirconium. Sample CUF-C104-010(Orange) was prepared with only about one-tenth the amount of solids compared with the other three samples. As a result, only a 5-fold analytical dilution was required.

The concentration of palladium reported previously appears to be an artifact caused by spectral interference particularly from high concentrations of thorium and uranium. Because of this, the actual concentration of palladium is too low to determine by ICPAES without chemical separation of the interfering analytes. Sodium in sample CUF-C104-010(Orange) appeared to be about ten times higher than the other three samples. The reason for the discrepancy is not known.

Measurement results reported have been corrected for preparation and analytical dilution. All results reported are in $\mu\text{g/g}$ for the solids samples. Volumes and weights have been recorded on bench sheets and included with this report.

Quality control check-standard results met tolerance requirements for analytes of interest except as noted below. Following is a list of quality control measurement results relative to ICPAES analysis tolerance requirements under MCS-033.

Five fold serial dilution:

(Solid samples/acid dig.) Analytes of interest were within tolerance limit of $\leq 10\%$ after correcting for dilution except silver, magnesium and uranium. Because of the very high concentration of thorium, interference correction to silver, magnesium and uranium were incorrect leading to inaccurate concentration values in the 5-fold diluted sample. At 25-fold and 50-fold dilution all analytes of interest were within $\leq 10\%$ after correcting for dilution.

1/18/00

**Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report**

Duplicate RPD (Relative Percent Difference):

(Solid samples/acid dig.) All analytes of interest were recovered within tolerance limit of $\leq 20\%$ relative percent difference (RPD) except silicon in ACL# 99-2524-R and its duplicate analyzed at 5-fold dilution. As noted earlier the reason for the large (150% RPD) for silicon is not known.

Post-Spiked Samples (Group A):

(Solid samples/acid dig.) All analytes of interest were recovered within tolerance of 75% to 125%.

Post-Spiked Samples (Group B):

(Solid samples/acid dig.) All analytes of interest were recovered within tolerance of 75% to 125%.

Blank Spike:

(Solid samples/acid dig.) None prepared.

Matrix Spiked Sample:

(Solid samples/acid dig.) None prepared.

Quality Control Check Standards (solid samples/acid dig.):

Concentration of all analytes of interest was within tolerance limit of $\pm 10\%$ accuracy in standards: QC_MCVA.

Tin and thorium were low (-15% and -61% respectively) in QC_MCVB. Single element standards of 2 ppm tin and 10 ppm thorium were measured separately and were within the tolerance limits.

Several analytes: iron, potassium, manganese, sodium, nickel, lead, silicon and zirconium were a little high (11% to 15%) and out of tolerance limits in check-standard QC_SSTMCV.

Calibration Blank (ICP98.0) concentration was acceptable, less than two times IDL.

High Calibration Standard Check (solid samples/acid dig.):

Verification of the high-end calibration concentration for all analytes of interest is within tolerance of $\pm 5\%$ accuracy except potassium, which measured 22% high at the end of the run. Potassium was not detected in any of the samples.

1/18/00

**Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report**

Process Blank:

(Solid samples/acid dig.) All analytes of interest were within tolerance limit of \leq EQL or $< 5\%$ of sample concentration except silicon. Silicon concentration in the blank was equivalent to about 1300 $\mu\text{g/g}$. Silicon at about 25,000 $\mu\text{g/g}$ was only found in sample CUF-C104-010 (Orange) (ACL# 99-2524-R).

Laboratory Control Standard (LCS):

(Solid samples/acid dig.) No LCS was prepared for PNL-ALO-129 acid digested samples.

Analytes other than those requested by the client are for information only. Please note bracketed values listed in the data report are within ten times instrument detection limit and have a potential uncertainty much greater than 15%.

Comments:

- 1) "Final Results" have been corrected for all laboratory dilution performed on the sample during processing and analysis unless specifically noted.
- 2) Detection limits (DL) shown are for acidified water. 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_3 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 $\mu\text{g/mL}$ (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.

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

Multiplier=	4396.1	1842.6	1545.5	1589.3	
ALO#=	99-2524-R @5	99-2524-DUP-R @25	99-2525-R @25	99-2525-DUP-R @25	
Client ID=	CUF-C104-010 (Orange)	CUF-C104-010 (Black)	CUF-C104-013 (Orange)	CUF-C104-013 (Black)	
Run Date=	11/4/99	11/4/99	11/4/99	11/4/99	
Det. Limit (ug/mL)	(Analyte)	ug/g	ug/g	ug/g	
0.025	Ag	[900]	1,060	1,850	1,650
0.060	Al	144,000	165,000	40,100	39,400
0.250	As	--	--	--	--
0.050	B	[1,300]	--	[77]	[80]
0.010	Ba	[210]	226	412	472
0.010	Be	--	[49]	[69]	[66]
0.100	Bi	--	--	--	--
0.250	Ca	[4,900]	5,210	8,830	9,410
0.015	Cd	854	978	1,730	1,830
0.200	Ce	--	--	[420]	--
0.050	Co	--	--	--	--
0.020	Cr	1,620	1,830	2,130	2,190
0.025	Cu	[210]	[270]	485	523
0.050	Dy	--	--	--	--
0.100	Eu	--	--	--	--
0.025	Fe	46,000	52,500	91,300	97,100
2.000	K	--	--	--	--
0.050	La	--	[140]	[250]	[290]
0.030	Li	[470]	587	513	536
0.100	Mg	[910]	[990]	1,570	1,710
0.050	Mn	10,200	11,600	20,300	21,600
0.050	Mo	--	--	--	--
0.150	Na	33,400	36,100	68,700	63,800
0.100	Nd	--	[290]	[560]	[600]
0.030	Ni	2,990	3,350	5,860	6,230
0.100	P	5,080	2,510	[1,000]	[1,200]
0.100	Pb	[1,600]	1,870	3,170	3,350
0.750	Pd	--	--	[3,700]	--
0.300	Rh	--	--	--	--
1.100	Ru	--	--	--	--
0.500	Sb	--	--	--	--
0.250	Se	--	--	--	--
0.500	Si	25,200	[2,500]	[1,700]	[1,700]
1.500	Sn	--	--	--	--
0.015	Sr	[110]	[110]	[190]	[210]
1.500	Te	--	--	--	--
1.000	Th	[5,900]	73,600	126,000	134,000
0.025	Ti	[160]	[170]	[300]	[320]
0.500	Tl	--	--	--	--
2.000	U	[42,000]	52,600	93,500	100,000
0.050	V	--	--	[80]	--
2.000	W	--	--	--	--
0.050	Y	--	--	--	--
0.050	Zn	[340]	[300]	[650]	[760]
0.050	Zr	60,600	64,600	113,000	123,000

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 1 of 1

Det. Limit (ug/mL)	Multiplier= ALO#= Client ID= Run Date= (Analyte)	50.5 00-0083-PB Process Blank 11/4/99 ug/g	6177.8 00-0083 @125 WASHED SOLIDS 11/4/99 ug/g	1256.0 00-0083-DUP @25 WASHED SOLIDS 11/4/99 ug/g		
0.025	Ag	--	--	[97]	--	--
0.060	Al	[5.9]	8,500	8,400	--	--
0.250	As	--	--	--	--	--
0.050	B	--	--	--	--	--
0.010	Ba	[0.91]	[400]	387	--	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	[5,900]	5,750	--	--
0.015	Cd	--	--	[31]	--	--
0.200	Ce	--	[1,300]	[1,300]	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	--	3,580	3,510	--	--
0.025	Cu	--	--	--	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	[4.4]	53,900	50,700	--	--
2.000	K	--	--	--	--	--
0.050	La	--	[810]	785	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	139,000	126,000	--	--
0.050	Mo	--	--	--	--	--
0.150	Na	--	71,300	91,200	--	--
0.100	Nd	--	[2,500]	2,380	--	--
0.030	Ni	[8.6]	--	[130]	--	--
0.100	P	--	[700]	[710]	--	--
0.100	Pb	--	[5,300]	5,360	--	--
0.750	Pd	--	--	[1,000]	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	948	[3,800]	[4,000]	--	--
1.500	Sn	--	--	--	--	--
0.015	Sr	--	289,000	280,000	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	[33]	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	[320]	[320]	--	--
0.050	Zn	--	[330]	[310]	--	--
0.050	Zr	--	[2,300]	2,180	--	--

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).

WT% SOLIDS DATA SHEET (325 SHIELDED ANALYTICAL LABORATORY)

CLIENT: Kriston Brooks WORK PACKAGE: W48489 ASR/ARF/LOI/TI: ASR 5500

QA PLAN: MCS-033 IMPACT LEVEL: _____ PROCEDURE NUMBER: PNL-ALO-504

CUF-C104 SLURRIES SAMPLE IDENTIFICATION

ACL NUMBER	CLIENT IDENTIFICATION	TARE WEIGHT (G)	(A) SAMPLE WET WEIGHT PLUS TARE	(B) SAMPLE DRY WEIGHT PLUS TARE	WEIGHT % SOLIDS
99-2523	CUF-C104-004 (Orange)	16.9448	24.0008	7.0560 19.0043	(27.54) 29.19*
99-2523	CUF-C104-004 (Black)	16.7914	25.1998	8.4094 (18.8983) (18.9935)	Broken
99-2524	CUF-C104-010 (Orange)	17.0053	23.8762 (6.8709)	18.5316 (8.5208)	22.21
99-2524	CUF-C104-010 (Black)	16.8124	22.4954 5.6830	18.0759 (19.0759)	22.23
99-2525	CUF-C104-013 (Orange)	16.9769	22.3133 5.3364	17.9926 (17.9953)	19.03
99-2525	CUF-C104-013 (Black)	16.6846	22.9063 6.2217	17.4589 (17.8590)	18.87
99-2528	CUF-C104-014 (Orange)	17.0017	23.0598 6.0581		Broken
99-2528	CUF-C104-014 (Black)	16.7988	22.4140 5.6152	17.8628 (17.8645)	18.95

WT% SOLIDS = $\frac{B - TARE}{A - TARE} \times 100$

DATE/TIME IN: 9-13-99, 2:36 OVEN TEMPERATURE: 105

DATE/TIME OUT: 9-14-99 11:00 OVEN TEMPERATURE: 106

* 27.5% DRY SOLIDS is a better number for sample 99-2523 (orange)

BALANCE: CELL 2 (360-06-01-016) X

BALANCE: CELL 5 (360-06-01-039) _____

THERMOCOUPLE: 2116

Analyst: [Signature] Date: 9-13-99

Reviewer: [Signature] Date: 9/17/99

Battelle Pacific Northwest Laboratory
 Radiochemical Processing Group-325 Building
 Chemical Measurements Center

99-2523
 11/12/1999

Client : KP Brooks

Cognizant Scientist: L R Greenwood

Date : 11/15/99

Concur : T Trang-le

Date : 11/15/99

Procedures: PNL-ALO-418,474

Measured Tritium Activities

ALO ID		uCi/g Slurry	uCi/g Dried	uCi/ml Water
Client ID	Wt.% Solids	Error %	Error %	Error %
99-2523	27.54%	5.35E-3	0.00E+0	7.38E-3
CUF-C104-004		6%		6%
99-2523 MS*	27.54%	3.49E-3	0.00E+0	4.82E-3
CUF-C104-004		6%		6%
RPD		42%		
99-2524	22.22%	3.87E-3	0.00E+0	4.98E-3
CUF-C104-010		6%		6%
99-2525	18.95%	3.29E-3	0.00E+0	4.06E-3
CUF-C104-013		6%		6%
99-2526	18.95%	3.10E-3	0.00E+0	3.82E-3
CUF-C104-014		6%		6%
Blank		< 3.E-4		
Blank Spike		108%		

*Note: This sample was not spiked; it is a duplicate of CUF-C104-004.

The measured tritium is assumed to be in the water fraction. Drying would remove all of the tritium from the samples. Hence, results are reported per weight of the as received slurry and per volume of water in the slurry. Results per dried material are assumed to be zero.



Battelle

Pacific Northwest Laboratories

Project Number

Internal Distribution

Date October 1, 1999

To K.P. Books

From Tom Farmer

Subject ICP/MS Analysis of Submitted Samples
(ALO#99-2523 through 99-2526)

329/4 File
LSO Project File
Mike Urie

Pursuant to your request, the 9 Ni/KOH fusion samples that you submitted for analysis were analyzed by ICPMS for selected elements. The results of this analysis are reported on the attached page.

Johnson-Matthey standards for Rb, Ta, Pr, and Sn, an Amersham ^{99}Tc standard and Isotope Products ^{129}I and ^{237}Np standards were used to generate the calibration curves. Independent standards were used as the continuing calibration verification (CCV) standards. Unless otherwise specified, the overall uncertainty of the values is conservatively estimated at $\pm 10\%$, and is based on the precision between consecutive analytical runs as well as the accuracy of the CCV standard results.

Values for tin were obtained using the response of a natural tin standard. Because ^{126}Sn standards were not used and the concentrations were determined indirectly, these results should be considered semiquantitative.

The ^{99}Tc values reported assume that the Ru present is exclusively fission-product Ru, and therefore does not have an isotope at m/z 99; i.e., everything observed at m/z 99 is due to ^{99}Tc . The fingerprint we're seeing for Ru is obviously not natural, and is consistent with that observed in previous tank waste analyses. Approximate ^{101}Ru concentrations are provided for your information.

If you have any questions regarding this analysis, feel free to call me at 372-0700 or James Bramson at 372-0624

K.P. Brooks Analysis

October 1, 1999

Results are reported in µg analyte/g (ppm) of solid sample.
The uncertainty of the results is estimated at ±10%.

J.P. Brooks
10/5/99

Sample Number	Client Number	ICP/MS Number	Rb µg/g	⁸⁹ Tc µg/g	TRU-101 µg/g	¹²⁶ Sn µg/g	¹²⁹ I µg/g	Pr µg/g	¹³⁵ Ta µg/g	²³⁷ Np µg/g
1%HN03		9927a1 9927a26	<0.5 <0.5	<0.5 <0.5		<0.4 <0.4	<0.4 <0.4	<0.5 <0.5	<0.2 <0.2	<0.2 <0.2
99-2523-PB-Ni	Process Blank	9927a31	198	<0.5		<0.4	1.0±0.5	<0.5	1.5±0.2	<0.2
99-2523-Ni	CUF-C104-004	9927a14	296	2.9±0.3	26	2±1	5.1±0.7	56.0	5.0±0.9	8.87
99-2523-Dup-Ni	CUF-C104-004	9927a15	242	2.58	24	3±1	4.3±0.9	55.6	3.1±0.7	9.0±1.3
99-2524-Ni	CUF-C104-010	9927a17	295	2.8±0.4	53	4±1	2.3±0.4	128	10±2	19.1
99-2524-Dup-Ni	CUF-C104-010	9927a18	250	2.22	32	2±1	3.0±0.3	75.0	11±3	12±2
99-2525-Ni	CUF-C104-013	9927a19	215	2.50	36	3±1	<0.4	85.6	2.11	12±2
99-2525-Dup-Ni	CUF-C104-013	9927a20	230	2.8±0.3	41	3±1	1.0±0.3	94.3	1.6±0.2	14±3
99-2525-Ni + spike	CUF-C104-013	9927a32	300	8.54			16±3	151	12.0	20.7
Spike Recovery			108%	98%			100%	102%	105%	113%
99-2526-Ni	CUF-C104-014	9927a21	228	2.15	54	3±1	<0.4	120	4.1±0.5	16.5
SRM 2710-Ni	LCS/99-2523/Ni	9927a22	326	<0.5	<0.5	<0.4	<0.4	6.65	2.79	<0.2
2ppb ⁹⁹ Tc/50ppb ¹²⁹ I		9927a8		1.93			54.8			
2ppb ⁹⁹ Tc/10ppb ¹²⁹ I		9927a8		2.00			10.2			
1643d		9927a9	11.8							
True Value			13							
2ppb Rb, Pr		9927a10	1.90					1.99		
100ppb Rb, Pr		9927a28	99.0					99.7		
20ppb Ta/ 2ppb ²³⁷ Np		9927a4					18.8		1.98	
10ppb Ta/ 2ppb ²³⁷ Np		9927a18					9.02		2.15	

*Results are from procedure 9928a.

†Based on response from indium. For information only.

•Obtained using response of natural tin. Should be considered semiquantitative.

DATA REVIEW

Reviewed by:

J. J. Jarament

Date: 5 Oct 99 Pages: 1 of 1

Battelle Pacific Northwest Laboratory
 Radiochemical Processing Group-325 Building
 Chemical Measurements Center

99-2523
 10/5/1999

Client : KP Brooks

Cognizant Scientist: JR Greenwood

Date: 10/5/99

Concur: T Trang-le

Date: 10/6/99

Procedures: PNL-ALO-450

Gamma Energy Analysis
 Measured Activities (uCi/g)

ALO ID Client ID	Co-60 Error %	Nb-94 Error %	Cs-134 Error %	Cs-137 Error %	Sb-125 Error %	Sn-126 Error %	Eu-152 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
99-2523PB Process Blank	<3.E-3	<3.E-3	9.95E-3 9%	6.90E-1 2%	<1.E-2	<5.E-3	<7.E-3	<8.E-3	<9.E-3	<2.E-2
99-2523 CUF-C104-004	2.54E-1 4%	1.04E-1 8%	<3.E-2	7.61E+1 2%	<3.E-1	<8.E-2	<2.E-1	1.76E+0 2%	1.11E+0 7%	5.27E+0 10%
99-2523 DUP CUF-C104-004 RPD	2.41E-1 3%	1.09E-1 7%	<2.E-2	7.43E+1 2%	<3.E-1	<8.E-2	<2.E-1	1.82E+0 2%	1.10E+0 6%	5.42E+0 10%
	5%	5%		2%				3%	1%	3%
99-2524 CUF-C104-010	4.36E-1 3%	2.37E-1 8%	<3.E-2	5.68E+1 2%	4.29E-1 15%	<8.E-2	<3.E-1	4.13E+0 2%	2.45E+0 5%	1.25E+1 10%
99-2524 DUP CUF-C104-010 RPD	2.59E-1 3%	1.57E-1 6%	<3.E-2	5.59E+1 2%	2.17E-1 32%	<7.E-2	<2.E-1	2.27E+0 2%	1.36E+0 6%	6.53E+0 10%
	51%	41%		2%	66%			58%	57%	63%
99-2525 CUF-C104-013	2.97E-1 3%	1.70E-1 8%	<3.E-2	5.76E+1 2%	3.52E-1 22%	<8.E-2	<2.E-1	2.79E+0 2%	1.76E+0 5%	8.43E+0 10%
99-2525 DUP CUF-C104-013 RPD	3.52E-1 3%	2.44E-1 5%	<3.E-2	5.59E+1 2%	4.37E-1 15%	<8.E-2	<3.E-1	3.29E+0 2%	1.94E+0 5%	1.02E+1 10%
	17%	36%		3%	22%			16%	10%	19%
99-2526 CUF-C104-014	4.28E-1 3%	2.45E-1 7%	<3.E-2	5.78E+1 2%	3.80E-1 14%	<7.E-2	<3.E-1	4.10E+0 2%	2.52E+0 5%	1.27E+1 10%

Battelle Pacific Northwest Laboratory
 Radiochemical Processing Group-325 Building
 Chemical Measurements Center

99-2524R
 11/11/1999

Client : KP Brooks

Cognizant Scientist: J.R. Greenwood

Date: 11/11/99

Concur: Richard T. B.

Date: 11/11/99

Procedures: PNL-ALO-450

Gamma Energy Analysis
 Measured Activities (uCi/g)

ALO ID Client ID	Co-60 Error %	Nb-94 Error %	Cs-134 Error %	Cs-137 Error %	Sb-125 Error %	Sn-126 Error %	Eu-152 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
00-0083PB Process Blank	<5.E-4	<4.E-4	5.34E-4 27%	2.96E-2 3%	<2.E-3	<4.E-4	<2.E-3	<2.E-3	<2.E-3	<2.E-3
99-2524-Rerun CUF-C104-010	2.18E-1 4%	1.38E-1 8%	1.64E-1 8%	6.06E+1 2%	<3.E-1	<6.E-2	<3.E-1	1.97E+0 2%	1.11E+0 7%	6.08E+0 10%
99-2524 DUP-Rerun CUF-C104-010	2.29E-1 2%	8.65E-2 4%	<1.E-2	4.73E+1 2%	2.64E-1 8%	<2.E-2	8.63E-2 8%	2.15E+0 2%	1.23E+0 4%	6.67E+0 9%
RPD	5%	46%		25%				9%	10%	9%
99-2525-Rerun CUF-C104-013	3.98E-1 3%	<6.E-2	<3.E-2	5.14E+1 2%	4.33E-1 14%	<5.E-2	<3.E-1	3.83E+0 2%	2.12E+0 5%	1.29E+1 7%
99-2525 DUP-Rerun CUF-C104-013	3.66E-1 3%	<5.E-2	<3.E-2	4.75E+1 2%	3.34E-1 17%	<4.E-2	<8.E-2	3.61E+0 2%	2.05E+0 5%	1.20E+1 7%
RPD	9%			8%	26%			6%	3%	7%

Battelle Pacific Northwest Laboratory
 Radiochemical Processing Group-325 Building
 Chemical Measurements Center

99-2523
 10/21/1999

Client : KP Brooks

Cognizant Scientist: L.R. Greenwood

Date : 10-21-99

Concur: C. J. Soderstrom

Date : 10-21-99

Procedures: PNL-ALO-420/421, 476, 417

Measured Activities (uCi/g)

ALO ID Client ID	Gross Alpha Error %	Sr-90 Error %	Am-241 Error %	Cm-243/244 Error %	Cm-242 Error %	U-233 Error %	Pu-239/240 Error %	Sum of Alphas*
99-2523PB Process Blank	2.60E-2 6%	5.86E-1 3%	7.95E-3 5%	1.19E-3 9%	<2.E-5	<7.E-4	9.02E-3 11%	1.82E-2
99-2523 CUF-C104-004	9.58E+0 2%	5.72E+2 3%	5.77E+0 5%	6.61E-2 20%	1.54E-2 41%	2.50E-1 5%	5.44E+0 2%	1.15E+1
99-2523 DUP CUF-C104-004 RPD	8.61E+0 2% 11%	5.72E+2 3% 0%	6.10E+0 4% 6%	8.69E-2 11% 27%	5.32E-3 45% 97%	2.12E-1 5% 16%	5.51E+0 2% 1%	1.19E+1
99-2523 Rep CUF-C104-004	1.14E+1 2%	5.75E+2 3%	6.04E+0 4%	1.11E-1 14%	1.10E-2 48%	2.05E-1 5%	5.69E+0 2%	1.21E+1
99-2524 CUF-C104-010	2.57E+1 2%	1.22E+3 3%	1.21E+1 4%	1.48E-1 15%	1.76E-2 48%	3.23E-1 4%	1.25E+1 2%	2.51E+1
99-2524 DUP CUF-C104-010 RPD	1.29E+1 2% 66%	7.00E+2 3% 54%	7.41E+0 4% 48%	1.12E-1 11% 28%	1.14E-2 34% 43%	2.31E-1 5% 33%	6.36E+0 2% 65%	1.41E+1
99-2525 CUF-C104-013	1.81E+1 2%	8.75E+2 3%	9.06E+0 4%	1.30E-1 15%	<2.E-2	3.63E-1 4%	8.65E+0 2%	1.82E+1
99-2525 DUP CUF-C104-013 RPD	1.94E+1 2% 7%	9.93E+2 3% 13%	1.08E+1 4% 18%	9.63E-2 18% 30%	2.08E-2 50%	4.14E-1 4% 13%	9.58E+0 2% 10%	2.09E+1

Measured Activities (uCi/g)

ALO ID Client ID	Gross Alpha Error %	Sr-90 Error %	Am-241 Error %	Cm-243/244 Error %	Cm-242 Error %	U-233 Error %	Pu-239/240 Error %	Sum of Alphas*
99-2526 CUF-C104-014	2.32E+1 2%	1.33E+3 3%	1.33E+1 4%	1.93E-1 13%	1.66E-2 45%	5.22E-1 3%	1.26E+1 2%	2.66E+1
Blank	<2.E-3	<2.E-2	<1.E-4	<1.E-4	<4.E-5	<2.E-4	<4.E-4	
Blank spike	102%	93%	99%				105%	
Sample spike	109%	108%	96%				91%	

*Note: The sum of the individual alpha emitters is a better estimate of the total alpha activity due to absorption effects.

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Chemical Measurements Center

99-2523
1/24/2000

Client : KP Brooks

Cognizant Scientist: C Soderqvist
Concur: L R Greenwood

Date : 2-14-00
Date : 1/24/00

Measured Concentration (ug/g)

<u>ALO ID</u> <u>Client ID</u>	Ammonia	+/- 1 σ
99-2523PB Process Blank	< 1.0	
99-2523 CUF-C104-004	2.49	6%
99-2523 DUP CUF-C104-004	2.78	13%
99-2524 CUF-C104-010	< 0.8	
99-2525 CUF-C104-013	< 0.8	
99-2526 CUF-C104-014	< 0.8	



Battelle

Pacific Northwest Laboratories

Project Number

Internal Distribution

Date October 6, 1999

To K.P.Books

From Tom Farmer

Subject ICP/MS Analysis of Submitted Samples
(ALO#99-2523 through 99-2526)

329/4 File
LSO Project File
Mike Urie

Pursuant to your request, the 9 PMG fusion prep samples that you submitted for analysis were analyzed by ICPMS for platinum. The results of this analysis are reported on the attached page.

A Johnson-Matthey platinum standard was used to generate the calibration curve. An Aldrich platinum wire standard (99.99% LOT#06927CN) was used as the continuing calibration verification (CCV) standard.

The response for platinum in the samples was determined to be caused by oxide production from hafnium present in the samples. Since the highest concentration seen at platinum (mass 195) was $0.86 \pm 0.17 \mu\text{g/g}$, the results for the samples is reported as $<1 \mu\text{g/g}$ of original sample.

If you have any questions regarding this analysis, feel free to call me at 372-0700 or James Bramson at 372-0624

K.P. Brooks Platinum Analysis

October 7, 1999

Results are reported in μg analyte/ g of original material.
The uncertainty of the results is estimated at $\pm 10\%$.

Sample Number	Client Number	ICP/MS Number	Pt $\mu\text{g/g}$
1%HNO3		9a05a1	0.17 \pm 0.06
1%HNO3		9a05a7	<0.2
1%HNO3		9a05a18	<0.2
99-2523-PB-Zr	Process Blank	9a05a8	<1
99-2523-Zr	CUF-C104-004	9a05a10	<1
99-2523-Dup-Zr	CUF-C104-004	9a05a11	<1
99-2524-Zr	CUF-C104-010	9a05a12	<1
99-2524-Dup-Zr	CUF-C104-010	9a05a13	<1
99-2525-Zr	CUF-C104-013	9a05a14	<1
99-2525-Dup-Zr	CUF-C104-013	9a05a15	<1
99-2525-Zr + spike	CUF-C104-013	9a05a17	5.1
Spike Recovery			95%
99-2526-Zr	CUF-C104-014	9a05a16	<1
SRM 2710-Zr	LCS/99-2523/Zr	9a05a9	<1
CCV results are reported in ng/ml (ppb)			
0.491ppb Pt CCV		9a05a19	0.489
0.982ppb Pt CCV		9a05a4	0.992

DATA REVIEW

Reviewed by: *Quik Henry J. Smith*

Date: 7 Oct 99 Pages: 1 of 1

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

WO/Project: W48489/29953
Client: K. Brooks

ACL Numbers: 99-02523 through 99-02534
ASR Number 5500

Procedure: PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography"
Analyst: MJ Steele Analysis Date: September 24-27, 1999

M&TE: IC system (WD25214); Mettler AT400 Balance (360-06-01-031) See Chemical Measurement Center 98620 RIDS for IC File for Calibration, Standards Preparations, and Maintenance Records.

Analyst: MJ Steele

Approval: Michael W. Urie

Date 10-23-99

Notes:

- 1) "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- 2) The low calibration standards are defined as the estimated quantitation limit (EQL) for the reported results and assume non-complex aqueous matrices. Actual detection limits or quantitation limits for specific sample matrices may be determined, if requested.
- 3) Routine precision and bias is typically $\pm 15\%$ or better for non-complex aqueous samples that are free of interference and have similar concentrations as the measured anions.

Final Results:

The samples were analyzed by ion chromatography (IC) for inorganic anions as specified in ASR 5500. The liquid samples were diluted at the IC workstation up to 10,000-fold to ensure that all anions were within the calibration range, and the solids samples were diluted an additional 100-fold following leaching per procedure ALO-103. The anion results are presented in the table below. The solids samples are reported on a per-dry-mass basis.

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

Solids		Concentration: per g dry weight								
		Solids Prep Fctr	Wt% Solids	F ug/g	Cl ug/g	NO ₂ ug/g	NO ₃ ug/g	PO ₄ ug/g	SO ₄ ug/g	C ₂ O ₄ ug/g
Lab ID	Sample ID									
99-2523 PB	Process Blank	4.21	22.33	< 5	< 5	< 10	< 10	< 10	< 10	< 10
99-2523	CUF-C104-004	4.27	29.19	45,000*	830	17,800	9,200	1,400	2,200	6,900
99-2523 Dup	CUF-C104-004 Dup	4.27	29.19	46,000*	930	19,600	10,000	1,600	2,400	7,700
	RPD (%)			2%	11%	10%	8%	13%	9%	11%
	CUF-C104-004 MS Recovery			127%	95%	109%	108%	108%	109%	112%
	Working Spike Recovery			112%	94%	107%	108%	110%	110%	109%
99-2524	CUF-C104-010	4.38	22.22	8,800*	190	700	740	160	220	250
99-2525	CUF-C104-013	4.08	18.95	190*	< 11	120	320	550	70	60
99-2526	CUF-C104-014	4.11	18.95	110*	< 11	80	320	500	60	40

Liquids		F	Cl	NO ₂	NO ₃	PO ₄	SO ₄	C ₂ O ₄
Lab ID	Sample ID	ug/ml	ug/ml	ug/ml	ug/ml	ug/ml	ug/ml	ug/ml
99-2527	CUF-C104-005	12,000*	650	9,500	5,100	750	990	3,200
99-2534	CUF-C104-019	6,100*	270	3,100	1,500	190	320	1,100
99-2529	CUF-C104-016	1,200*	220	4,100	2,200	< 200	< 200	260
99-2530	CUF-C104-017	3,500*	160	1,400	610	< 50	110	240
99-2531	CUF-C104-018	2,100*	70	480	230	< 50	80	120
99-2528	CUF-C104-011	40*	< 25	220	180	630	< 50	< 50
99-2532	CUF-C014-021	50*	< 25	70	120	330	< 50	< 50
99-2533	CUF-C104-022	320*	< 10	30	90	90	< 20	< 20

RPD = Relative Percent Difference (between sample and duplicate/replicate)

* = Quantified by IC system as fluoride; however, slight retention time peak shift and peak shape suggest significant organic anion interference. High probability that little or no fluoride is actually present in the samples.

Q.C. Comments:

Besides the duplicate, matrix spike, and working spike QC, the following are results of analytical quality control checks performed during IC analyses. In general, quality control checks met the requirements of the governing QA Plan.

System Blank/Processing Blanks: Twelve system blanks were processed during the analysis of the sample. With the exception of only two nitrate values, no anions were detected above reportable concentrations in the system blanks or in the processing/dilution blank.

Quality Control Calibration Verification Check Standards: Fourteen mid-range verification standards were analyzed throughout the analysis runs. Except for a two fluoride values, the reported results for all analytes of interest were recovered within the acceptance criteria of $\pm 10\%$ for the verification standard. For two fluoride failures, no recoveries exceeded $\pm 15\%$ of the standard values.

Date November 3, 1999

File/LB

To K. Brooks

 From M. Urie *M. Urie*

 Subject Carbon analysis of CUF-C104 Solids
Samples

The analysis of the subject samples submitted under ASR 5500 was performed by the hot persulfate wet oxidation method, PNL-ALO-381, rev. 1. The hot persulfate method uses acid decomposition for TIC and acidic potassium persulfate oxidation at 92-95 °C for TOC, all on the same sample, with TC being the sum of the TIC and TOC.

The samples were analyzed on October 28, 1999 and Table 1 below shows the results, rounded to two to three significant figures. The raw data bench sheets and calculation work sheets showing all calculations are attached. All sample results are corrected for average percent recovery of system calibration standards and are also corrected for contribution from the blank.

All samples were analyzed directly (i.e., no preparative or analytical dilution), and are reported in microgram of carbon per gram of dried solids (as per your request). The analysis of the liquid samples submitted under ASR 5500 have not been completed, and will be reported under a separate report.

Table 1: TIC/TOC/TC Results

		Results on a per gram dry weight basis *						
ALO Number	Sample ID	Wt% Solids	TIC $\mu\text{gC/g}$	TIC RPD (%)	TOC $\mu\text{gC/g}$	TOC RPD (%)	TC $\mu\text{gC/g}$	TC RPD (%)
99-2523	CUF-C104-004	27.54	16,200		9,360		25,600	
99-2523 Dup	CUF-C104-004 Dup	27.54	15,400	5	10,000	7	24,500	0
99-2524	CUF-C104-010	22.22	5,730		16,500		22,200	
99-2524 Dup	CUF-C104-010 Dup	22.22	5,790	1	19,200	15	25,000	12
99-2525	CUF-C104-013	18.95	20,400		29,900		50,200	
99-2526	CUF-C104-014	18.95	16,500	21 **	11,900	86 **	28,500	55 **
99-2526 MS	CUF-C104-014 MS		104%		139%		115%	
	1000 μg TOC Std Rec.				105%			

RPD = Relative Percent Difference

 * $\text{ug per dry gram} = \text{ug C per gram sample} / (\text{weight \% solids} / 100)$

** RPD calculated between CUF-C104-013 and CUF-C104-014 (field duplicates)

QC Narrative

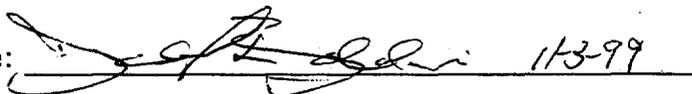
The TIC standard is calcium carbonate and TOC standard is α -Glucose (the certificates of purity are attached). The standard materials were used in solid form for system calibration standards as well as matrix spikes. TIC and TOC percent recovery are determined using the appropriate standard (i.e., calcium carbonate for TIC or glucose for TOC) in either solid or liquid form.

The QC for the methods involves calibration blanks, system calibration standards, sample duplicates, and one matrix spike per matrix type. The QC system calibration standards were all within acceptance criteria, with the average recovery being 98.1% for TIC and 95.6% for TOC. The calibration blanks were acceptable, averaging 19 μ gC for TIC and 37 μ gC for TOC.

The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. The matrix spike recoveries from sample CUF-C104-14 were 104% for TIC and 139% for TOC. The TOC matrix spike is outside the acceptance criteria of 75% to 125%. The sample and matrix spike sample were weighed in the hot cells and transferred to the laboratory for addition of the matrix spikes. The TIC matrix spike was added as a solid and the TOC matrix spike was added as a liquid. Analysis of the TOC liquid spike solution provided a recovery of 105%, confirming the matrix spike standard concentrations. The reason for the apparent high TOC recovery is unknown.

The precision (as Relative Percent Difference [RPD]) could not be determined since the carbon concentration in the sample is less than 5 times the method detection limit. For those samples for which samples and duplicates were analyzed (i.e., CUF-C104-004 and CUF-C104-010), the RPDs are within the acceptance criteria of 20%. The RPDs are also calculated for the field duplicates, CUF-C104-014 and CUF-C104-013. Based on these RPDs there is some concern about representative sampling, since the RPDs for these two samples is very large (especially for TOC). This variability may contribute to the poor TOC spike recovery obtained on CUF-C104-014.

Review/Approve:

 11/3-99

Archive Information:

Files: ASR 5550 Solids Brooks.doc

ASR 5500 Brooks - Solids/Liquids.xls

Battelle PNNL/RPG/Inorganic Analysis ---Hg Report

page 1 of 2

WO/Project: W48489/29953
Client: K. P. Brooks

ACL Numbers: 99-02523 through 99-02526
ASR Number 5500

Procedure: PNNL-ALO-131, "Mercury Digestion"
PNNL-ALO-201, "Mercury Analysis"

Analyst: J. J. Wagner

Digestion Date: October 21, 1999 **Analysis Date:** October 27, 1999

M&TE: Hg system (WD14126); Mettler AT400 Balance (360-06-01-029) See Chemical Measurement Center 98620 RIDS for Hg File for Calibration, Standards Preparations, and Maintenance Records.

Analyst: Jerry Wagner

Approval: MW

Date 11-5-99

Final Results:

The samples were analyzed by cold vapor atomic absorption spectrophotometry for inorganic mercury as specified in ASR 5500. The solids samples were diluted an additional 100-fold following sample digestion per procedure ALO-131. The mercury concentration results are presented in the table below.

Battelle PNNL/RPG/Inorganic Analysis ---Hg Report

page 2 of 2

Lab ID	Solid Sample ID	Solids Grams	Solids Dig Fctr	Solids Anal Fctr	Hg ug/g
99-2523 PB	Solids Process Blank	0.2743	91.1	1	<0.018
99-2523	CUF-C104-004	0.3127	79.9	100	20.5
99-2524	CUF-C104-010	0.2056	121.6	100	21.8
99-2525	CUF-C104-013	0.3344	74.8	100	31.9
99-2526	CUF-C104-014	0.2446	102.2	100	32.5

RPD = Relative Percent Difference (between sample and duplicate/replicate)

"Sample weight" used for the process blank is an average weight of the samples.

Notes:

- 1) "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- 2) The low calibration standard is defined as the estimated quantitation limit (EQL) for the reported results and assumes non-complex aqueous matrices. Actual detection limits or quantitation limits for specific sample matrices may be determined, if requested.
- 3) Routine precision and bias is typically $\pm 15\%$ or better for non-complex aqueous samples that are free of interference.

Q.C. Comments:

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

Working Blank Spike/Process Blank Spike: Process Blank Spike recovery is 112%, well within the acceptance criteria of 80% to 120%.

Matrix Spiked Sample: A matrix spike was prepared for the samples submitted under this ASR. However, the concentration of the matrix spike processed and analyzed with this batch of samples was too low in concentration relative to the high concentration of mercury in the samples measured. As a result, matrix spike recovery could not be assessed.

Duplicate: No duplicates were prepared.

System Blank/Processing Blanks: A system blank was process during the analysis of the sample. All reportable sample concentrations were many times greater than that measured in the system blank or in the processing/dilution blank.

Quality Control Calibration Verification Check Standards: Over 4 mid-range verification standards were analyzed throughout the analysis run. All were within the acceptance criteria of 80% to 120% recovery for the verification standard.

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

99-2527
 10/20/99

Client : KP Brooks

Cognizant Scientist: JR Greenwood

Date : 10/20/99

Concur: T Traing-le

Date : 10/20/99

Procedures: PNL-ALO-420/421, 417

Measured Activities (uCi/g)

ALO ID Client ID	Gross Alpha Error %	Sr-90 Error %	Am-241 Error %	Cm-243+ Cm-244 Error %	Cm-242 Error %
99-2527PB Process Blank	<3.E-5	<5.E-5	<4.E-7	<5.E-7	<3.E-7
99-2527PB DUP Process Blank	<2.E-5	<5.E-5	<2.E-7	<2.E-7	1.67E-7 40%
99-2527 CUF-C104-005	2.95E-4 5%	<3.E-2	4.62E-6 11%	2.93E-7 40%	<2.E-7
99-2527 DUP CUF-C104-005	3.34E-4 5%	<3.E-2	5.37E-6 11%	2.80E-7 53%	<2.E-7
RPD	12%		15%	5%	
99-2527 Rep CUF-C104-005	3.22E-4 5%		6.09E-6 9%	<2.E-7	<8.E-8
99-2528 CUF-C104-011	2.27E-4 6%	<3.E-2	1.88E-6 17%	<3.E-7	<2.E-7
99-2528 REP CUF-C104-011		<3.E-2			
99-2529 CUF-C104-016	5.35E-4 4%	<3.E-2	5.32E-6 8%	3.82E-7 34%	<7.E-8
99-2529 DUP CUF-C104-016	4.86E-4 4%	<3.E-2	5.60E-6 10%	<4.E-7	<3.E-7
RPD	10%		5%		

Measured Activities (uCi/g)

ALO ID Client ID	Gross Alpha Error %	Sr-90 Error %	Am-241 Error %	Cm-243+	Cm-242
				Cm-244 Error %	Error %
99-2530 CUF-C104-017	3.07E-4 6%	<3.E-2	1.65E-6 19%	<5.E-7	<2.E-7
99-2530 DUP CUF-C104-017	2.72E-4 6%	<3.E-2	3.28E-6 13%	<3.E-7	<2.E-7
RPD	12%		66%		
99-2531 CUF-C104-018	3.84E-4 5%	<3.E-2	6.68E-5 5%	6.77E-7 26%	1.88E-7 50%
99-2531 DUP CUF-C104-018	3.46E-4 5%	<3.E-2	6.32E-5 5%	5.27E-7 31%	2.26E-7 48%
RPD	10%		6%	25%	18%
99-2532 CUF-C104-021	1.95E-4 7%	<3.E-2	1.94E-6 25%	<2.E-7	<2.E-7
99-2532 DUP CUF-C104-021	1.75E-4 7%	<3.E-2	2.07E-6 16%	<2.E-7	<2.E-7
RPD	11%		6%		
99-2533 CUF-C104-022	1.24E-4 9%	<3.E-2	3.56E-7 41%	<2.E-7	<2.E-7
99-2533 DUP CUF-C104-022	1.35E-4 9%	<2.E-2	<3.E-7	<3.E-7	<8.E-8
RPD	8%				
99-2534 CUF-C104-019	2.66E-4 6%	<2.E-2	1.36E-6 17%	<3.E-7	<5.E-8
99-2534 DUP CUF-C104-019	3.47E-4 5%	<2.E-2	2.13E-6 16%	<3.E-7	<3.E-7
RPD	26%		44%		
Blank	<3.E-5	<2.E-4	<3.E-7	<2.E-7	3.10E-7 28%
Blank spike	101%	95%	105%		
Sample spike	81%	102%	104%		

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

99-2527
 9/29/99

Client : KP Brooks

Cognizant Scientist:

JR Greenwood

Date :

9/29/99

Concur :

T Trang-le

Date :

9/29/99

Procedure: PNL-ALO-450

Gamma Energy Analysis
 Measured Activities (uCi/g)

ALO ID Client ID	Co-60 Error %	Cs-137 Error %	Sb-125 Error %	Sn-126 Error %	Eu-152 Error %	Eu-154 Error %	Eu-155 Error %
99-2527 CUF-C104-005	1.21E-2 3%	9.62E+0 2%	<2.E-2	<6.E-3	<1.E-2	<8.E-4	<2.E-2
99-2527 DUP CUF-C104-005	1.24E-2 3%	9.74E+0 2%	<2.E-2	<6.E-3	<1.E-2	<7.E-4	<2.E-2
RPD	2%	1%					
99-2528 CUF-C104-011	7.64E-4 8%	2.14E+0 2%	<6.E-3	<8.E-4	<4.E-3	<4.E-4	<5.E-3
99-2529 CUF-C104-016	5.72E-3 3%	4.81E+0 2%	<9.E-3	<3.E-3	<6.E-3	<3.E-4	<7.E-3
99-2529 DUP CUF-C104-016	5.80E-3 3%	4.83E+0 2%	<9.E-3	<3.E-3	<6.E-3	<4.E-4	<7.E-3
RPD	1%	0%					
99-2530 CUF-C104-017	1.58E-3 4%	1.56E+0 2%	<4.E-3	<2.E-3	<2.E-3	<8.E-5	<2.E-3
99-2530 DUP CUF-C104-017	1.59E-3 4%	1.58E+0 2%	<4.E-3	<2.E-3	<2.E-3	<2.E-4	<2.E-3
RPD	1%	1%					
99-2531 CUF-C104-018	6.79E-4 4%	6.90E-1 2%	<2.E-3	<3.E-4	<8.E-4	<2.E-4	<2.E-3
99-2531 DUP CUF-C104-018	6.63E-4 5%	6.94E-1 2%	<2.E-3	<3.E-4	<8.E-4	<1.E-4	<2.E-3
RPD	2%	1%					

Gamma Energy Analysis
Measured Activities (uCi/g)

ALO ID Client ID	Co-60 Error %	Cs-137 Error %	Sb-125 Error %	Sn-126 Error %	Eu-152 Error %	Eu-154 Error %	Eu-155 Error %
99-2532 CUF-C104-021	4.99E-4 10%	1.65E+0 2%	<6.E-3	<2.E-3	<3.E-3	<3.E-4	<4.E-3
99-2532 DUP CUF-C104-021	5.33E-4 9%	1.50E+0 2%	<5.E-3	<2.E-3	<3.E-3	<3.E-4	<4.E-3
RPD	7%	10%					
99-2533 CUF-C104-022	2.76E-4 13%	9.25E-1 2%	<4.E-3	<6.E-4	<3.E-3	<3.E-4	<4.E-3
99-2533 DUP CUF-C104-022	2.58E-4 14%	9.34E-1 2%	<4.E-3	<5.E-4	<3.E-3	<3.E-4	<4.E-3
RPD	7%	1%					
99-2534 CUF-C104-019	4.28E-3 3%	3.51E+0 2%	<7.E-3	<3.E-3	<4.E-3	<4.E-4	<5.E-3
99-2534 DUP CUF-C104-019	4.22E-3 3%	3.49E+0 2%	<7.E-3	<3.E-3	<4.E-3	<3.E-4	<5.E-3
RPD	1%	1%					

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

99-2527
11/10/1999

Client : KP Brooks

Cognizant Scientist: B. K. Fickum

Date : 11/10/99

Concur : M. J. Hill

Date : 11-22-99

Density Determination

<u>ALO ID</u> <u>Client ID</u>	<u>Density</u> <u>g/mL</u>
99-2527 CUF-C104-005	1.0602
99-2528 CUF-C104-011	1.0948
99-2529 CUF-C104-016	1.0318
99-2530 CUF-C104-017	1.0071
99-2531 CUF-C104-018	1.0003
99-2532 CUF-C104-021	1.0440
99-2533 CUF-C104-022	1.0125
99-2534 CUF-C104-019	1.0212
99-2535 CUF-C104-015	1.0066

Used Class A volumetric flasks, except for sample 99-2535 where a 2-mL volumetric flask was used.

^{10 ml}
Mull 11-12-99

Battelle PNNL/RPG/Inorganic Analysis --- TOC/TIC Report

Client: K. Brooks
 ACL Numbers: 99-2527 to 99-2534
 Analyst: MJ Steele

Charge Code/Project: W48489 / 29953
 ASR Number: 5500
 Analysis Date: November 18-19, 1999

Procedure: PNL-ALO-381, "Direct Determination of TC, TOC, and TIC in Radioactive Sludges and Liquids by Hot Persulfate Method"

M&TE: Carbon System (WA92040); Balance (360-06-01-023).

Final Results:

Lab Number	Sample ID	Vol (ml)	TIC (µg C/ml)	TIC RPD (%)	TOC (µg C/ml)	TOC RPD (%)	TC (µg C/ml)	TC RPD (%)
99-2527	CUF-C104-005	0.20	2,260		2,300		4,560	
99-2527 Dup	CUF-C104-005 Dup	0.20	2,180	4	2,330	2	4,510	1
99-2528	CUF-C104-011	0.20	270		<90		270	
99-2528 Dup	CUF-C104-011 Dup	0.20	290	7	<90	n/a	290	7
99-2529	CUF-C104-016	0.40	980		980		1,960	
99-2529 Dup	CUF-C104-016 Dup	0.40	1,010	2	980	0	1,990	1
99-2530	CUF-C104-017	0.40	320		240		560	
99-2530 Dup	CUF-C104-017 Dup	0.40	320	1	260	10	580	5
99-2530 Spike	CUF-C104-017 Spike	0.30	102%		92%		98%	
99-2531	CUF-C104-018	0.40	130		95		225	
99-2531 Dup	CUF-C104-018 Dup	0.60	160	17	150	n/a	310	29
99-2532	CUF-C104-021	0.40	290		190		480	
99-2532 Dup	CUF-C104-021 Dup	0.40	350	19	160	n/a	510	7
99-2533	CUF-C104-022	0.40	180		170		350	
99-2533 Dup	CUF-C104-022 Dup	0.60	200	9	120	n/a	320	11
99-2534	CUF-C104-019	0.20	6,310		4,040		10,350	
99-2534 Dup	CUF-C104-019 Dup	0.025	6,370	1	3,530	14	9,900	5

RPD = Relative Percent Difference (between sample and duplicate/replicate)

The analysis of the subject samples submitted under ASR 5500 was performed by the hot persulfate wet oxidation method. The hot persulfate method uses acid decomposition for TIC and acidic potassium persulfate oxidation at 92-95°C for TOC, all on the same sample, with TC being the sum of the TIC and TOC.

The table above shows the results, rounded to two to three significant figures. The raw data bench sheets and calculation work sheets showing all calculations are attached. All sample results are corrected for average percent recovery of system calibration standards and are also corrected for contribution from the blank

Q.C. Comments:

The TIC standard is calcium carbonate and TOC standard is α -Glucose (the certificates of purity are attached). 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) in either solid or liquid form.

Battelle PNNL/RPG/Inorganic Analysis --- TOC/TIC Report

The QC for the methods involves calibration blanks, system calibration standards, sample duplicates, and one matrix spike per matrix type.

Calibration Standards: The QC system calibration standards were all within acceptance criteria, with the average recovery being 100.2% for TIC and 100.0% for TOC.

Calibration Blanks: The five calibration blanks run at the beginning and middle of the analysis run were acceptable, averaging 12.5 μgC for TIC and 60.8 for μgC for TOC. The standard deviation calculated from the calibration blanks is lower than the estimated method detection limit for both TIC and TOC.

Duplicates: No actual sample duplicates were provided to the laboratory for analysis. However, the relative percent differences (RPD) between replicates are within the acceptance criteria of 20%, except for the summed TC for sample CUF-C104-018. The TOC results for this sample are very near the MDL and the variability in the TOC results impacted the RPD for the TC.

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. The matrix spike for this sample recovered at 102% for TIC and 91% for TOC, well within the 75% to 125% recovery acceptance criteria.

General Comments:

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- Routine precision and bias are typically $\pm 15\%$ or better for non-complex samples that are free of interferences.
- The estimated quantitation limit (EQL) is defined as 5 times the MDL. Results less than 5 times the MDL have higher uncertainties, and RPDs are not calculated for any results less than 5 times the MDL.
- Some results may be reported as less than (" $<$ ") values. These less than values represent the sample MDL (method detection limit), which is the system MDL adjusted for the volume of sample used for the analysis. The system MDL is based on the attached pooled historical blank data. The evaluation and calculation of the system MDL is included in the data package.

Report Prepared by:

MW

Date 11-19-99

Review/Approval by:

D. B. Adams

Date 11-22-99

Archive Information:

Files: ASR 5500 Liquid Brooks.doc

ASR 5500 Brooks - Solids&Liquids.xls

PNNL Radiochemical Processing Group: TOC/TIC/TC Calculation Review** Report - Hot Persulfate Method PNL-ALO-38

Client: K. Brooks
 Project: 29953
 Work Pkg: W48489 (CMC K88409)
 Analyzed: 11/18-19/1999
 ASR: 5500

Analyzer M&TE: WA92040 -- 701
 Balance M&TE: 360-06-01-023

TOC STD: Glucose CSM-6007>>> 40.00% Carbon <<[G]
 TIC STD: CaCO3 CMS-139285>>> 11.99% Carbon <<[C]

		Raw TIC (ug C)	Raw TOC (ug C)
Blanks:	Calibration blank (start of batch)(11/18/99)	11.8	65.9
	Calibration blank (start of batch)(11/18/99)	10.0	68.9
	Calibration blank (end of batch)(11/18/99)	12.6	65.8
	Calibration blank (start of batch)(11/19/99)	12.3	66.8
	Calibration blank (end of batch)(11/19/99)	15.9	36.8

TIC	TOC	
12.5	60.8	<<< Average (ug C)
2.1	13.5	<<< Std Dev (ug C)
2.16	5.8	<<< Pooled Std Dev (ug C)
6.5	17.3	<<< Method Det. Limit (ug C)

Total Inorganic Carbon (TIC)					
	[A] Raw TIC (ug)	[B] Blk (ug)	[D] Std wt (g)	TIC % Rec	
Standards:	Calibration Standard (start of batch)	1448	13	0.01180	101.5
	Calibration Standard (start of batch)	1322	13	0.01100	99.3
	Calibration blank (end of batch)(11/18/99)	970	13	0.00800	99.8
	Calibration Standard (start of batch)(11/19/99)	2096	13	0.01740	99.9
	Calibration blank (end of batch)(11/19/99)	3116	13	0.02570	100.7
[L] Average TIC % Rec >>>>				100.2	

Total Organic Carbon (TOC)					
	[E] Raw TOC (ug)	[F] Blk (ug)	[H] Std wt (g)	TOC % Rec	
Standards:	Calibration Standard (start of batch)	1148	61	0.00260	104.5
	Calibration Standard (start of batch)	2299	61	0.00550	101.7
	Calibration blank (end of batch)(11/18/99)	1150	61	0.00280	97.2
	Calibration Standard (start of batch)(11/19/99)	1395	61	0.00330	101.1
	Calibration blank (end of batch)(11/19/99)	1208	61	0.00300	95.6
[P] Average TOC % Rec >>>>				100.0	

Formulas:	Standard TIC % Recovery = ((A-B)/((C/100)*D))*10 ⁻⁶ *100	Matrix Spike Recoveries: TIC % Recovery = (((Q-R)/(L/100))-S*T)*100/U TOC % Recovery = (((Q-R)/(P/100))-S*T)*100/U TC % Recovery = (((Q ^{TIC} -R ^{TIC})/(L/100))-V ^{TIC})+(((Q ^{TOC} -R ^{TOC})/(P/100))-V ^{TOC}))*100/U ^{TIC+TOC}
	Standard TOC % Recovery = ((E-F)/((G/100)*H))*10 ⁻⁶ *100	
	Sample TIC (ug C/ml) = (I-J)/(K*L/100)	
	Sample TOC (ug C/ml) = (M-N)/(O*P/100)	

Comments: Due to the precision carried in the spreadsheet, some results may appear to be slightly off due to rounding.
 The Pooled SD is the averaged SD for a recent list of 12 sample batches. MDL is based upon the Pooled SD. MDL = 3 x pooled SD.
 If either the Sample or Duplicate are < 5x mdl, then the RPD is not calculated and displayed as "n/a".
 TIC and TOC are measured; TC is the sum of the TIC and TOC results.

PNNL Radiochemical Processing Group: TOC/TIC/TC Calculations **Review** Report - Hot Persulfate Method PNL-ALO-381

Client: K. Brooks
 Project: 29953
 Work Pkg: W48489 (CMC K88409)
 Analyzed: 11/18-19/1999
 ASR: 5500

Analyzer M&TE: WA92040 -- 701

Balance M&TE: 360-06-01-023

TOC STD: Glucose CSM-6007>>> 40.00% Carbon <<[G]

TIC STD: CaCO3 CMS-139285>>> 11.99% Carbon <<[C]

Sample Results

ACL Number	Client Sample ID	[I] Raw TIC (ug C)	[J] Blk (ug C)	[K] Sam Vol(ml)	TIC (ug C/ml)	TIC RPD (%)	[M] Raw TOC (ug C)	[N] Blk (ug C)	[O] Sam Vol(ml)	TOC (ug C/ml)	TOC RPD (%)	TC (ug C/ml)	TC RPD (%)
99-2527	CUF-C104-005	465	13	0.20	2,257		520	61	0.20	2,295		4,552	
99-2527 Dup	CUF-C104-005	449	13	0.20	2,177	4	527	61	0.20	2,330	2	4,507	1
99-2528	CUF-C104-011	67	13	0.20	272		73	61	0.20	61 (<mdl)		272	
99-2528 Dup	CUF-C104-011	71	13	0.20	292	7	61	61	0.20	1 (<mdl)	n/a	292	7
99-2529	CUF-C104-016	407	13	0.40	984		453	61	0.40	980		1,964	
99-2529 Dup	CUF-C104-016	416	13	0.40	1,006	2	453	61	0.40	980	0	1,986	1
99-2530	CUF-C104-017	139	13	0.40	316		156	61	0.40	238		553	
99-2530 Dup	CUF-C104-017	140	13	0.40	318	1	166	61	0.40	263	10	581	5
99-2530 Spike	CUF-C104-017	1124	13	0.30	see below		792	61	0.30	see below		see below	
99-2531	CUF-C104-018	65	13	0.40	131		99	61	0.40	95		226	
99-2531 Dup	CUF-C104-018	106	13	0.60	155	17	150	61	0.60	149	n/a	304	29
99-2532	CUF-C104-021	129	13	0.40	291		135	61	0.40	185		476	
99-2532 Dup	CUF-C104-021	153	13	0.40	350	19	124	61	0.40	158	n/a	508	7
99-2533	CUF-C104-022	85	13	0.40	181		129	61	0.40	170		351	
99-2533 Dup	CUF-C104-022	132	13	0.60	199	9	130	61	0.60	115	n/a	314	11
99-2534	CUF-C104-019	1278	13	0.20	6,313		870	61	0.20	4,044		10,357	
99-2534 Dup	CUF-C104-019	172	13	0.025	6,365	1	149	61	0.025	3,526	14	9,891	5

Matrix Spike Results

ACL Number	[Q] Raw MS (ug C)	[R] MS Blk (ug C)	[S] Sam (ug C/g)	[T] MS Sam Vol(ml)	[V] Sample (ug C)	Spike wt (g)	[U] Spike (ug C)	MS % Recovery
99-2530 Spike	TIC Recovery	1124	12.5	316	0.30	95	0.0083	995 101.9 TIC
	TOC Recovery	792	60.8	238	0.30	71	0.0018	720 91.6 TOC
	Total Carbon Recovery (TIC + TOC)						1715	97.6 TC

Preparer/date: MW thru 11-19-99

Reviewer/date: [Signature] 11-2-2-99



Battelle

Pacific Northwest Laboratories

Project Number

Internal Distribution

Date September 20, 1999
To K.P.Books
From Tom Farmer
Subject ICP/MS Analysis of Submitted Samples
(ALO#99-2527 through 99-2534)

329/4 File
LSO Project File
Mike Urie

Pursuant to your request, the 20 samples that you submitted for analysis were analyzed by ICPMS for ^{99}Tc . The results of this analysis are reported on the attached page.

An Amersham ^{99}Tc was used to generate the calibration curve. An independent Amersham ^{99}Tc standard was used as the continuing calibration verification (CCV) standard. Unless otherwise specified, the overall uncertainty of the values is conservatively estimated at $\pm 10\%$, and is based on the precision between consecutive analytical runs as well as the accuracy of the CCV standard results.

The ^{99}Tc values reported assume that the Ru present is exclusively fission-product Ru, and therefore does not have an isotope at m/z 99; i.e., everything observed at m/z 99 is due to ^{99}Tc . The fingerprint we're seeing for Ru is obviously not natural, and is consistent with that observed in previous tank waste analyses. Approximate ^{101}Ru concentrations are provided for your information.

If you have any questions regarding this analysis, feel free to call me at 372-0700 or James Bramson at 372-0624

K.P. Brooks Tc-99 Analysis

September 20, 1999

J.P. Brooks
9/30/99

Results are reported in ng analyte/ml (ppb) of original solution.
The uncertainty of the results is estimated at $\pm 10\%$.

Sample Number	Client Number	ICP/MS Number	Tc-99 ng/ml	*Ru-101 ng/ml
1%HNO3		9a15a1	<1	
1%HNO3		9a15a6	<1	
1%HNO3		9a15a22	<1	
1%HNO3		9a15a39	<1	
99-2527 Blank		9a15a7	<1	0.6
99-2527 Blank Dup.		9a15a8	<1	0.7
99-2527 Spike		9a15a9	<1	0.7
99-2527 Spike Dup.		9a15a10	<1	0.5
99-2527	CUF-C104-005	9a15a11	234	530
99-2527 Dup.	CUF-C104-005	9a15a12	238	530
99-2528	CUF-C104-011	9a15a14	22 \pm 3	30
99-2528 Dup.	CUF-C104-011	9a15a15	20.1	21
99-2529	CUF-C104-016	9a15a16	117	250
99-2529 Dup.	CUF-C104-016	9a15a17	122	250
99-2530	CUF-C104-017	9a15a18	37.6	69
99-2530 Dup.	CUF-C104-017	9a15a20	37.8	76
99-2531	CUF-C104-018	9a15a19	22.1	38
99-2531 Dup.	CUF-C104-018	9a15a13	21.3	35
99-2532	CUF-C104-021	9a15a23	55.4	11
99-2532 Dup.	CUF-C104-021	9a15a24	49.4	16
99-2533	CUF-C104-022	9a15a25	27 \pm 4	17
99-2533 Dup.	CUF-C104-022	9a15a26	29.8	8
99-2534	CUF-C104-019	9a15a27	91.3	200
99-2534 Dup.	CUF-C104-019	9a15a28	83.5	190
99-2534 + spike	CUF-C104-019	9a15a29	200	
Spike Recovery			112%	
2ppb Tc-99		9a15a4	1.89	
2ppb Tc-99		9a15a40	2.08	
5ppb Tc-99		9a15a21	5.01	
20ppb Co		9a15a41	<1	

*Based on Response of Indium, for information only.

DATA REVIEW

Reviewed by: *C.J. Farmer*

Date: *30 Sep 99* Pages: *1 of 1*

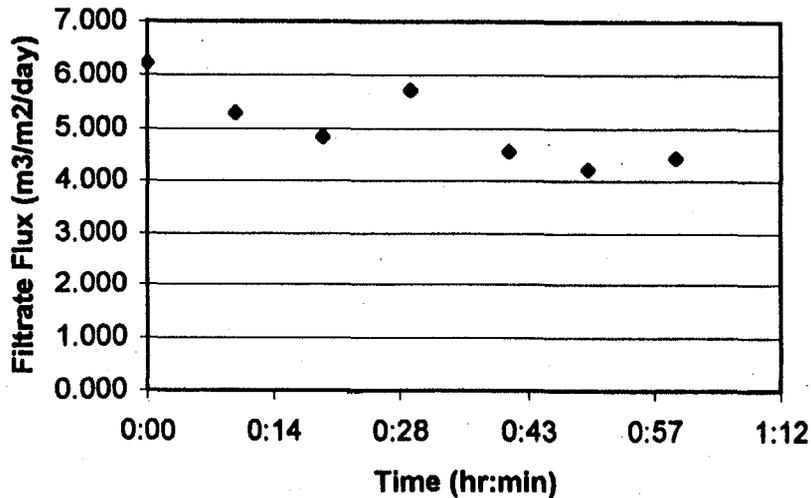
Appendix D: Crossflow Filtration Raw Data

Appendix D: Crossflow Filtration Raw Data

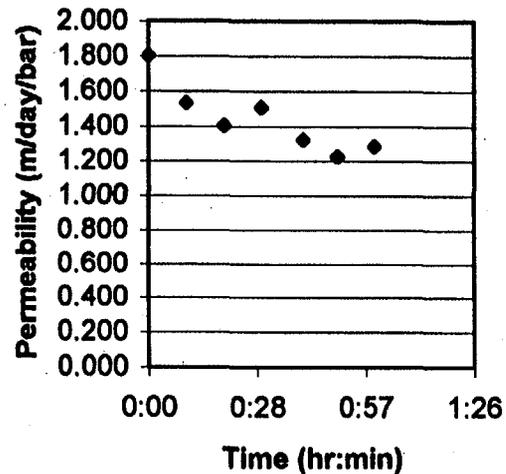
Matrix 1 Condition Number	Time	Total Time Elapsed (Min)	Slurry Loop Flow Rate (gpm)	Filter Outlet Pressure (psig)	Filter Inlet Pressure (psig)	Pressure Drop (psig)	Filtrate Sample Volume (mL)	Time of Collection (Sec)	Filtrate Flow Rate (mL/sec)	Slurry Temp C	Filtrate Flux (m3/m2/day)	Permeability (m/day/bar)
1	0:16	0:00	4.2	NA	50	#VALUE!	40	29.53	1.355	26.1	6.221	1.805
1	0:26	0:10	4.4	NA	50	#VALUE!	40	38.13	1.049	22.8	5.289	1.534
1	0:36	0:20	4.4	NA	50	#VALUE!	40	37.84	1.057	26.2	4.841	1.404
1	0:46	0:30	4.5	NA	55	#VALUE!	40	38	1.053	20.2	5.720	1.508
1	0:57	0:41	4.4	NA	50	#VALUE!	40	42.75	0.936	24	4.559	1.322
1	1:06	0:50	4.5	NA	50	#VALUE!	40	43.1	0.928	26.4	4.227	1.226
1	1:16	1:00	0	NA	50	#VALUE!	40	48.69	0.822	20.4	4.438	1.287

RAW Average Slurry Flow = 3.77 Average Pressure = 50.71429 Average Flow = 1.028 Average Flux = 4.846
 Filtrate

C-104 Sludge Flux vs. Time at 50.7 psig and 3.8 gpm



C-104 Sludge Permeability vs. Time at 50.7 psig and 3.8 gpm



Matrix 1 Condition Number	Time	Total Time Elapsed (Min)	Slurry Loop Flow Rate (gpm)	Filter Outlet Pressure (psig)	Filter Inlet Pressure (psig)	Pressure Drop (psig)	Filtrate Sample Volume (mL)	Time of Collection (Sec)	Filtrate Flow Rate (mL/sec)	Slurry Temp C	Filtrate Flux (m ³ /m ² /day)	Permeability (m/day/bar)
2	1:33	0:00	4.2	29.5	32	2.5	40	32.82	1.219	25.1	5.756	2.715
2	1:43	0:10	4.15	29	32	3	40	43.32	0.923	23	4.629	2.201
2	1:53	0:20	4.25	29	32	3	40	42.13	0.949	21	5.041	2.397
2	2:03	0:30	4.35	28	31	3	40	42.09	0.950	25.2	4.476	2.201
2	2:17	0:44	4	31	34	3	40	41	0.976	20.2	5.301	2.366
2	2:30	0:57	4.4	29	31	2	40	45.32	0.883	23.1	4.412	2.133
2	2:35	1:02	4.55	29	32	3	40	43.16	0.927	25.1	4.377	2.082

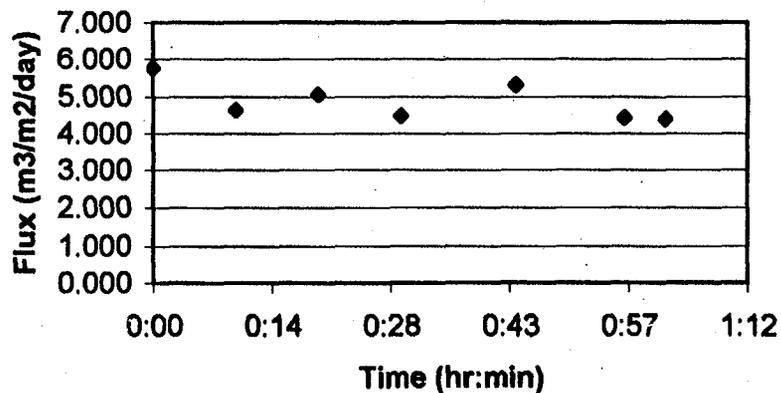
Average Pressure = 30.60714

Average Flow = 0.975 Average Flux = 4.706
Filtrate

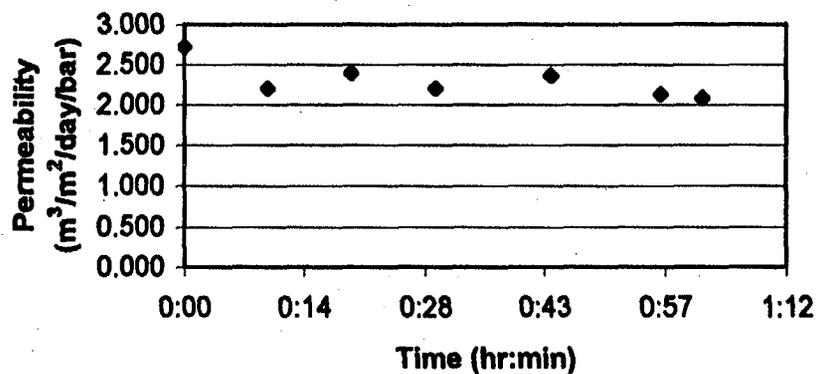
RAW

Average Slurry Flow = 4.27

**C-104 Sludge Flux vs. Time
at 30.6 psig and 4.3 gpm**



**C-104 Sludge Permeability vs. Time at
30.6 psig and 4.3 gpm**

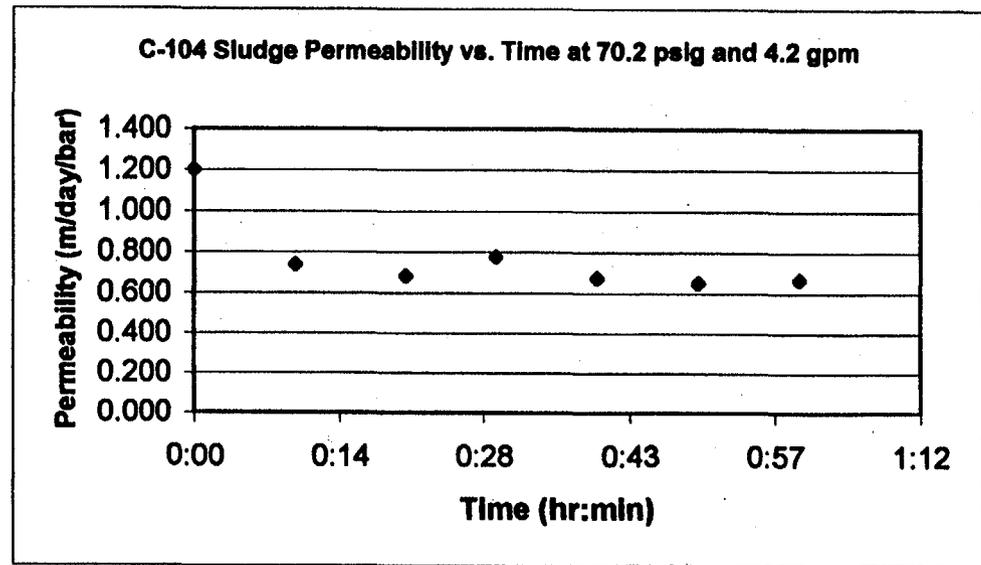
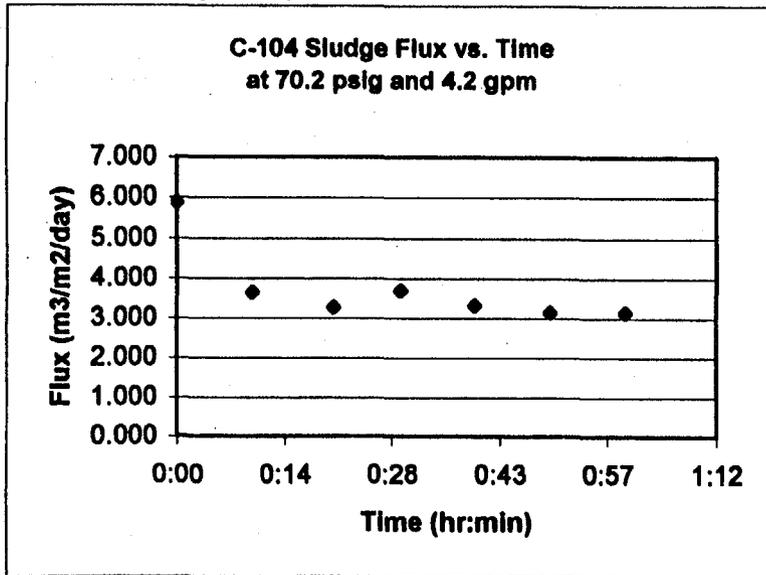


Matrix 1 Condition Number	Time	Total Time Elapsed (Min)	Slurry Loop Flow Rate (gpm)	Filter Outlet Pressure (psig)	Filter Inlet Pressure (psig)	Pressure Drop (psig)	Filtrate Sample Volume (mL)	Time of Collection (Sec)	Filtrate Flow Rate (mL/sec)	Slurry Temp C	Filtrate Flux (m3/m2/day)	Permeability (m/day/bar)	
3	2:50	0:00	3.88	70	72		2	40	34.9	1.146	22.2	5.878	1.201
3	3:00	0:10	4.35	70	73		3	40	50.34	0.795	26.2	3.639	0.738
3	3:11	0:21	4.35	68	71		3	40	58.28	0.686	24.8	3.269	0.682
3	3:20	0:30	4.1	68	70		2	40	56.84	0.704	21.4	3.693	0.776
3	3:30	0:40	4.1	70	73		3	40	57.46	0.696	24.8	3.316	0.673
3	3:40	0:50	4.2	69	72		3	40	58	0.690	26.3	3.150	0.648
3	3:50	1:00	4.25	67	70		3	40	62.96	0.635	23.4	3.149	0.667

Average Pressure = 70.21429

Average Flow = 0.765 Average Flux = 3.369
Filtrate

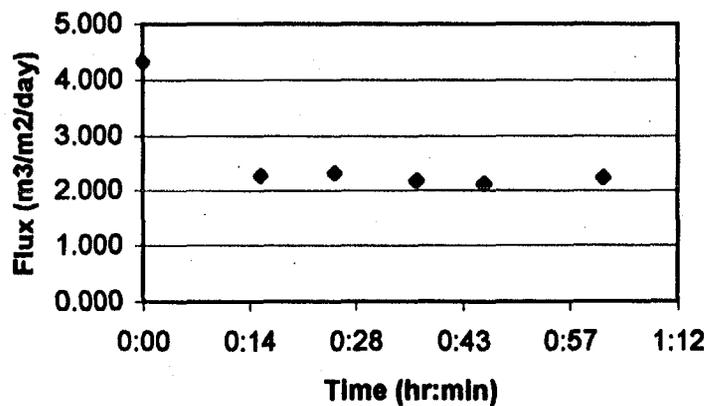
RAW Average Slurry Flow 4.18



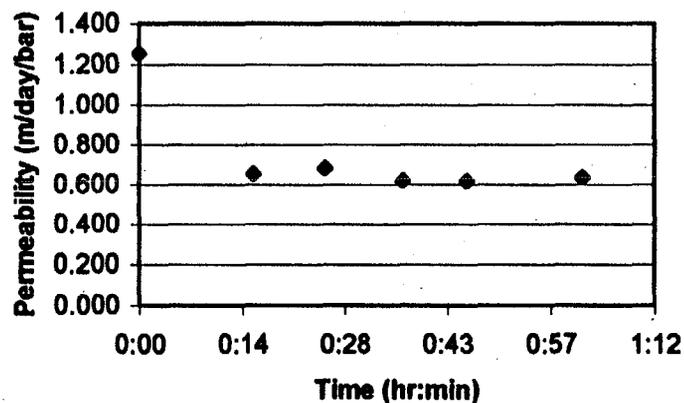
Matrix 1 Condition Number	Time	Total Time Elapsed (Min)	Slurry Loop Flow Rate (gpm)	Filter Outlet Pressure (psig)	Filter Inlet Pressure (psig)	Pressure Drop (psig)	Filtrate Sample Volume (mL)	Time of Collection (Sec)	Filtrate Flow Rate (mL/sec)	Slurry Temp C	Filtrate Flux (m ³ /m ² /day)	Permeability (m/day/bar)	
4	4:08	0:00	3.1	49	51		2	40	44.68	0.895	24.3	4.325	1.255
4	4:24	0:16	3.05	50	51		1	40	90.75	0.441	22	2.274	0.653
4	4:34	0:26	3.1	48	50		2	40	92.16	0.434	20.9	2.311	0.684
4	4:45	0:37	3.05	50	52		2	40	88.16	0.454	24.5	2.180	0.620
4	4:54	0:46	2.95	50	50		0	40	94.72	0.422	22.9	2.123	0.616
4	5:10	1:02	3.35	50	52		2	40	94.59	0.423	21.2	2.232	0.635

RAW Average Slurry Flow = 3.10 Average Pressure = 50.25 Average Flow = 0.511 Average Flux = 2.224
Filtrate

C-104 Sludge Flux vs. Time at 50.3 psig and 3.1 gpm



C-104 Sludge Permeability vs. Time at 50.3 psig and 3.1 gpm



Matrix 1 Condition Number	Time	Total Time Elapsed (Min)	Slurry Loop Flow Rate (gpm)	Filter Outlet Pressure (psig)	Filter Inlet Pressure (psig)	Pressure Drop (psig)	Filtrate Sample Volume (mL)	Time of Collection (Sec)	Filtrate Flow Rate (mL/sec)	Slurry Temp C	Filtrate Flux (m ³ /m ² /day)	Permeability (m/day/bar)
5	5:36	0:00	5.15	48	51	3	40	34.1	1.173	21.8	6.086	1.783
5	5:46	0:10	5.25	49	52	3	40	37.06	1.079	24.7	5.156	1.481
5	5:58	0:22	5.25	47	50	3	40	41.41	0.966	25.1	4.562	1.364
5	6:08	0:32	5.2	47	51	4	40	44.81	0.893	21.1	4.726	1.399
5	6:18	0:42	5.2	47	51	4	40	39.78	1.006	24	4.899	1.450
5	6:29	0:53	5.15	48	51	3	40	39.53	1.012	25.9	4.673	1.369
5	6:39	1:03	5.3	47.5	51	3.5	40	45.22	0.885	21.5	4.629	1.363

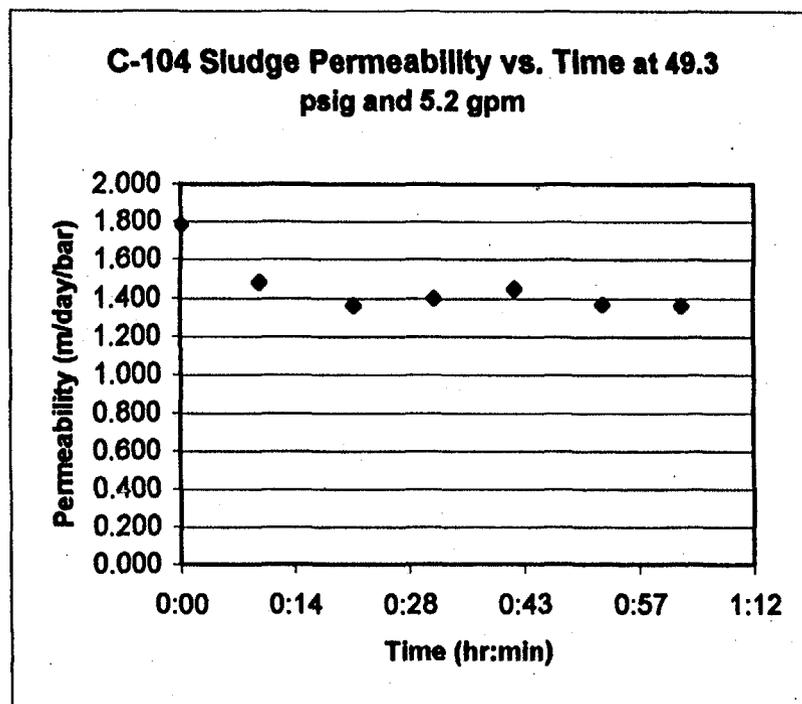
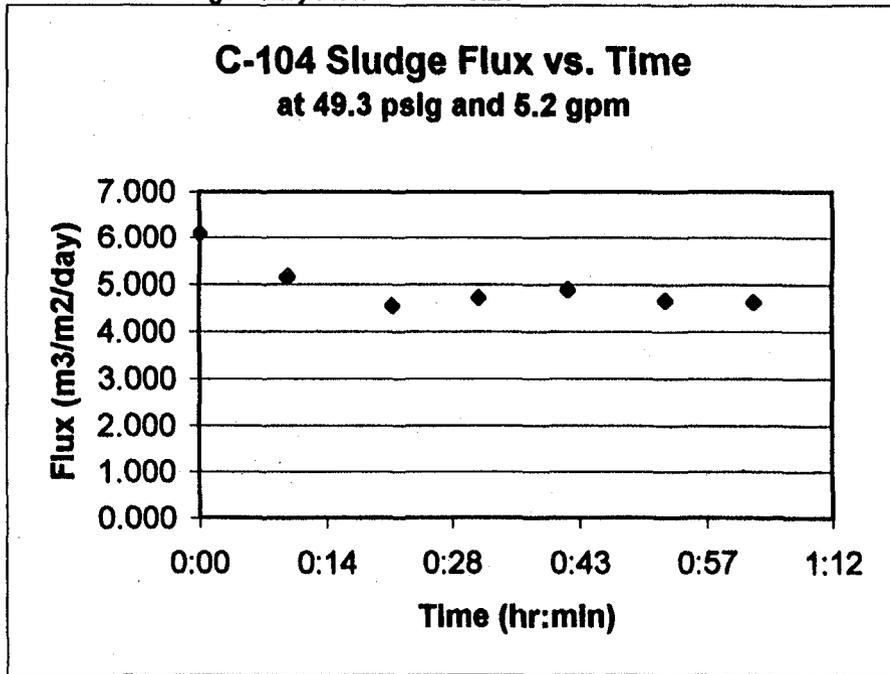
Average Pressure = 49.32143

Average Flow = 1.002 Average Flux = 4.774

RAW

Average Slurry Flow = 5.21

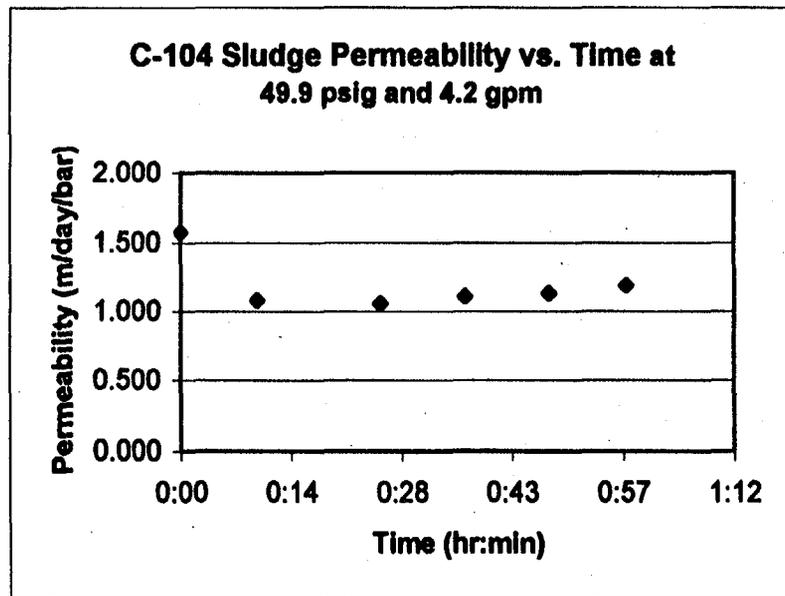
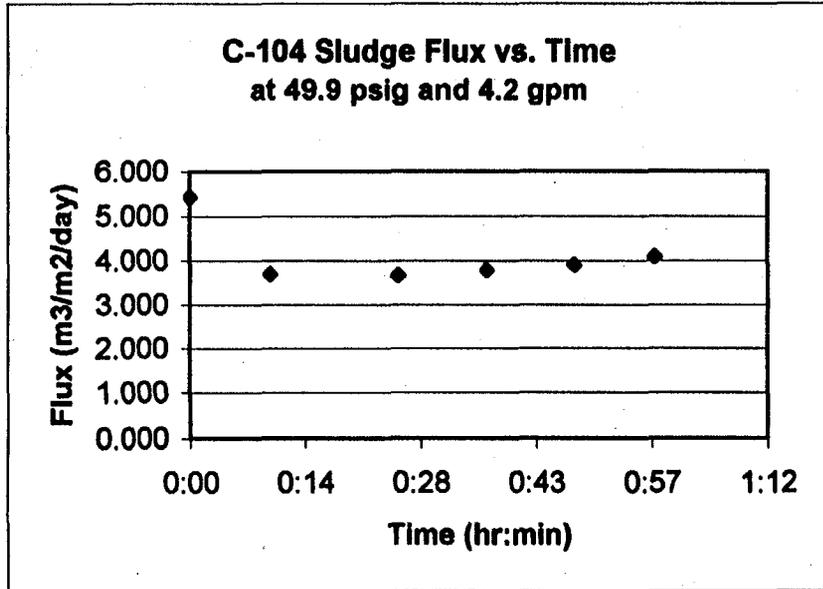
Filtrate



Matrix 1 Condition Number	Time	Total Time Elapsed (Min)	Slurry Loop Flow Rate (gpm)	Filter Outlet Pressure (psig)	Filter Inlet Pressure (psig)	Pressure Drop (psig)	Filtrate Sample Volume (mL)	Time of Collection (Sec)	Filtrate Flow Rate (mL/sec)	Slurry Temp C	Filtrate Flux (m ³ /m ² /day)	Permeability (m/day/bar)
6	6:52	0:00	4.3	49	51	2	40	36.71	1.090	23.3	5.415	1.571
6	7:02	0:10	4.25	48	51	3	40	48.88	0.818	26.8	3.686	1.080
6	7:18	0:26	4.1	49	52	3	40	57.56	0.695	21.2	3.668	1.054
6	7:29	0:37	4.25	48	51	3	40	49.69	0.805	25.4	3.770	1.105
6	7:40	0:48	4.3	49	51	2	40	51.03	0.784	23.3	3.896	1.130
6	7:50	0:58	4.1	49	51	2	40	52.1	0.768	20.9	4.088	1.186

RAW Average Slurry Flow = 4.22 Average Pressure = 49.91667

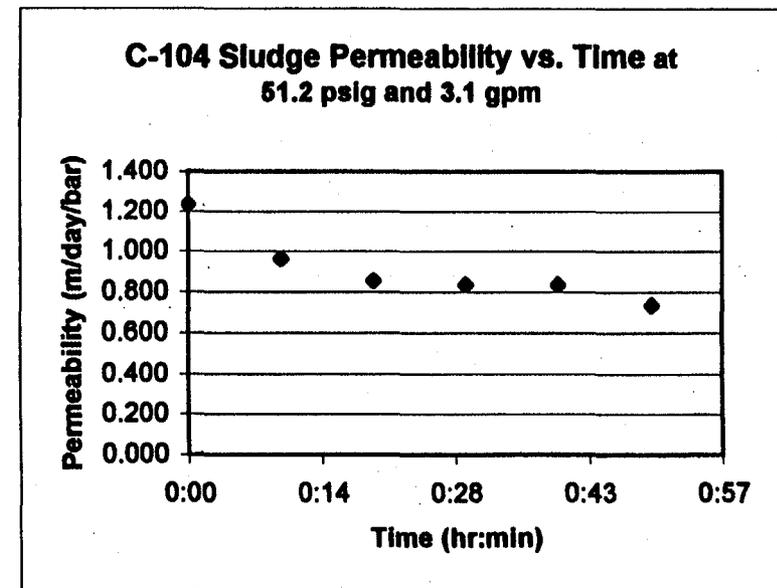
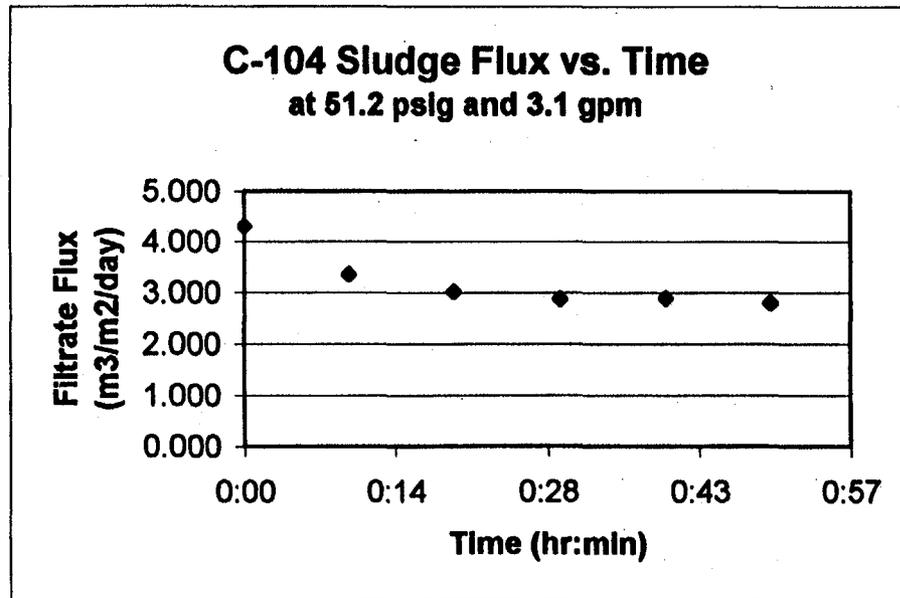
Average Flow = 0.827 Average Flux = 3.822
Filtrate



Matrix 1 Condition Number	Time	Total Time Elapsed (Min)	Slurry Loop Flow Rate (gpm)	Filter Outlet Pressure (psig)	Filter Inlet Pressure (psig)	Pressure Drop (psig)	Filtrate Sample Volume (mL)	Time of Collection (Sec)	Filtrate Flow Rate (mL/sec)	Slurry Temp C	Filtrate Flux (m ³ /m ² /day)	Permeability (m/day/bar)	
7	8:05	0:00	3.15	50	51		1	40	42.75	0.936	26.1	4.297	1.234
7	8:15	0:10	3.05	50	51		1	40	63.59	0.629	20.9	3.349	0.962
7	8:25	0:20	2.9	50	52		2	40	68.32	0.585	22.2	3.003	0.854
7	8:35	0:30	3.2	49	51		2	40	64.5	0.620	25.8	2.872	0.833
7	8:45	0:40	3.1	49	51		2	40	71.5	0.559	22.1	2.877	0.835
7	8:55	0:50	3.1	51	59		8	40	74.69	0.536	21.7	2.786	0.735

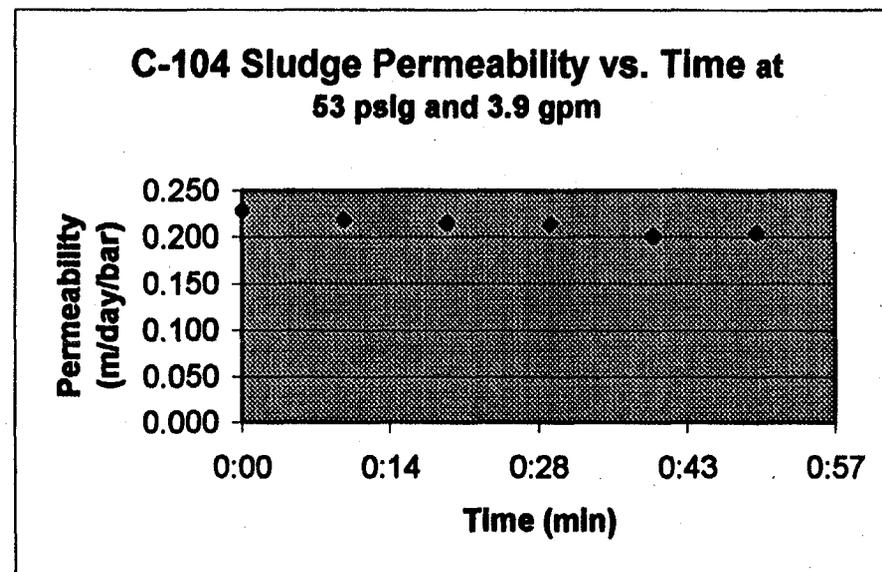
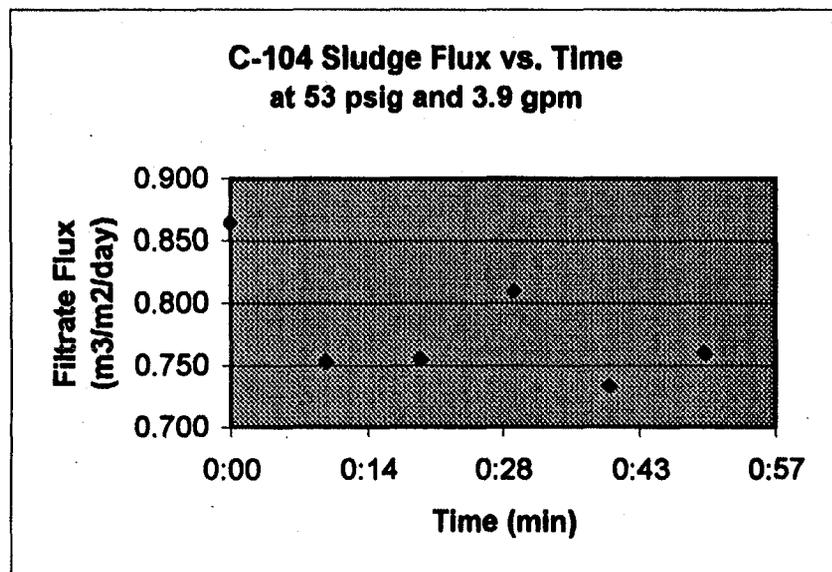
RAW Average Slurry Flow = 3.08 Average Pressure = 51.16667

Average Flow = 0.644 Average Flux = 2.978
Filtrate



Matrix 2 Condition Number	Time	Total Time Elapsed (Min)	Slurry Loop Flow Rate (gpm)	Filter Outlet Pressure (psig)	Filter Inlet Pressure (psig)	Pressure Drop (psig)	Filtrate Sample Volume (mL)	Time of Collection (Sec)	Filtrate Flow Rate (mL/sec)	Slurry Temp C	Filtrate Flux (m3/m2/day)	Permeability (m/day/bar)
1	12:26	0:00	3.6	NA	55	#VALUE!	10	54.84	0.182	25	0.864	0.228
1	12:36	0:10	4	NA	50	#VALUE!	10	70.09	0.143	21.2	0.753	0.218
1	12:46	0:20	4	NA	51	#VALUE!	10	63.25	0.158	24.7	0.755	0.215
1	12:56	0:30	3.8	NA	55	#VALUE!	10	58.32	0.171	25.1	0.810	0.214
1	13:06	0:40	3.9	NA	53	#VALUE!	10	71.75	0.139	21.3	0.734	0.201
1	13:16	0:50	3.9	NA	54	#VALUE!	10	65.97	0.152	23	0.760	0.204

RAW Average Slurry Flow = 3.87 Average Pressure = 53 Average Flow = 0.158 Average Flux = 0.762
 Filtrate



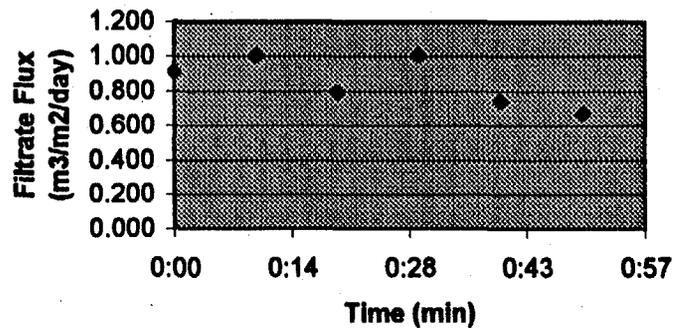
Matrix 2 Condition Number	Time	Total Time Elapsed (Min)	Slurry Loop Flow Rate (gpm)	Filter Outlet Pressure (psig)	Filter Inlet Pressure (psig)	Pressure Drop (psig)	Filtrate Sample Volume (mL)	Time of Collection (Sec)	Filtrate Flow Rate (mL/sec)	Slurry Temp C	Filtrate Flux (m3/m2/day)	Permeability (m/day/bar)
2	2:51	0:00	4.1	NA	35	#VALUE!	10	57.41	0.174	21.4	0.914	0.379
2	3:01	0:10	3.5	NA	37	#VALUE!	10	46.78	0.214	25.2	1.007	0.395
2	3:11	0:20	4.2	NA	30	#VALUE!	10	65.07	0.154	22	0.793	0.383
2	3:21	0:30	3.8	NA	35	#VALUE!	10	52.47	0.191	21.1	1.009	0.418
2	3:31	0:40	3.9	NA	35	#VALUE!	10	64.31	0.155	24.7	0.743	0.308
2	3:41	0:50	4.3	NA	30	#VALUE!	10	71.41	0.140	24.6	0.671	0.324

Average Pressure = 33.66667

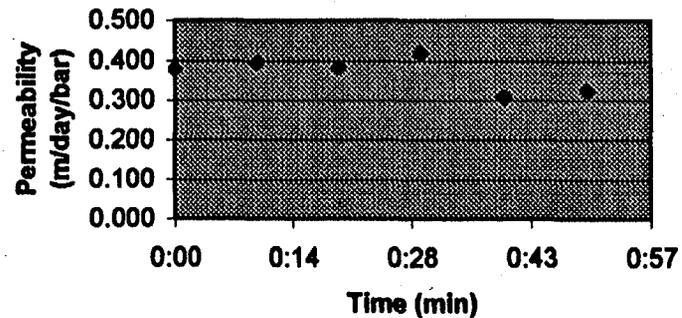
Average Flow = 0.171 Average Flux = 0.844

RAW Average Slurry Flow = 3.97

**C-104 Sludge Flux vs. Time
at 33.6 psig and 4.0 gpm**



**C-104 Sludge Permeability vs. Time at
33.6 psig and 4.0 gpm**



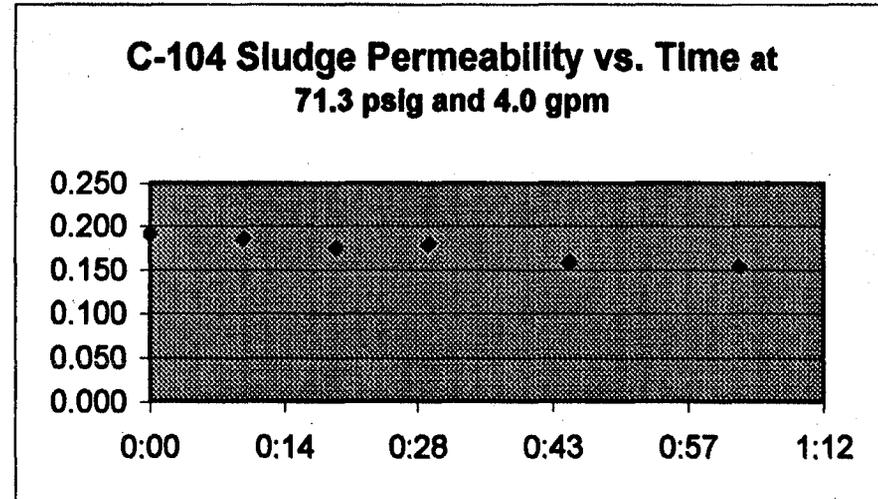
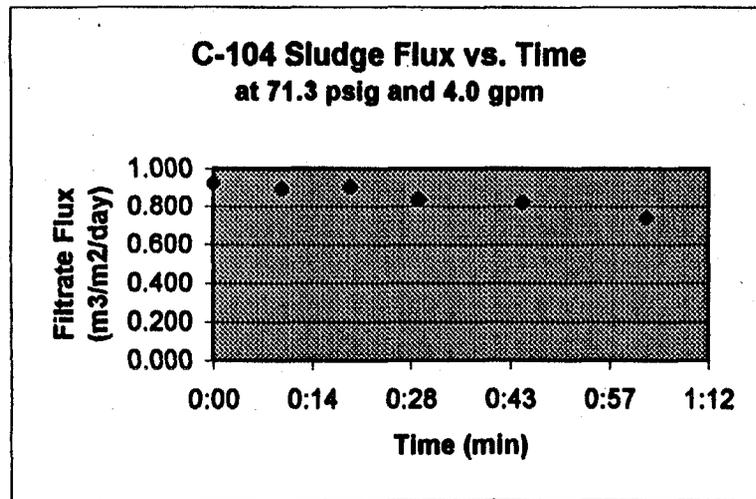
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3	4:02	0:00	3.9	NA	70	#VALUE!	10	45.69	0.219	29.1	0.925	0.192
3	4:12	0:10	4	NA	70	#VALUE!	10	49.94	0.200	27.1	0.894	0.185
3	4:22	0:20	3.8	NA	75	#VALUE!	10	52.91	0.189	24.7	0.903	0.175
3	4:32	0:30	4.2	NA	68	#VALUE!	10	57.93	0.173	24.1	0.839	0.179
3	4:47	0:45	4.3	NA	75	#VALUE!	10	56.09	0.178	26	0.821	0.159
3	5:05	1:03	4.1	NA	70	#VALUE!	10	65.85	0.152	23.9	0.742	0.154

Average Pressure = 71.33333

Average Flow = 0.185 Average Flux = 0.840

RAW

Average Slurry Flow = 4.05



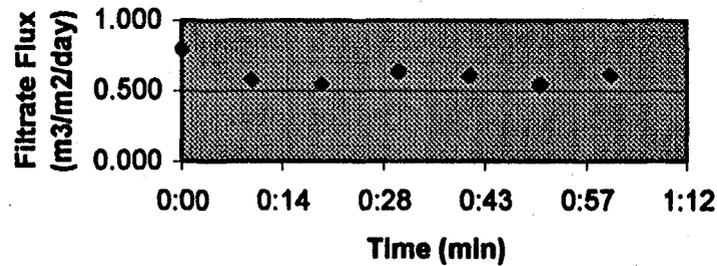
Matrix 2 Condition Number	Time	Total Time Elapsed (Min)	Slurry Loop Flow Rate (gpm)	Filter Outlet Pressure (psig)	Filter Inlet Pressure (psig)	Pressure Drop (psig)	Filtrate Sample Volume (mL)	Time of Collection (Sec)	Filtrate Flow Rate (mL/sec)	Slurry Temp C	Filtrate Flux (m3/m2/day)	Permeability (m/day/bar)
4	5:43	0:00	3.1	NA	50	#VALUE!	10	66.66	0.150	21.2	0.792	0.230
4	5:53	0:10	3.15	NA	53	#VALUE!	10	78.69	0.127	26.5	0.577	0.158
4	6:03	0:20	3.1	NA	50	#VALUE!	10	84.28	0.119	26	0.546	0.159
4	6:14	0:31	3.1	NA	55	#VALUE!	10	83	0.120	21.4	0.632	0.167
4	6:24	0:41	3.1	NA	54	#VALUE!	10	84.19	0.119	22.4	0.606	0.163
4	6:34	0:51	3.15	NA	50	#VALUE!	10	83.47	0.120	26.6	0.543	0.157
4	6:44	1:01	3.2	NA	55	#VALUE!	10	81.07	0.123	23.5	0.610	0.161

Average Pressure = 52.42857

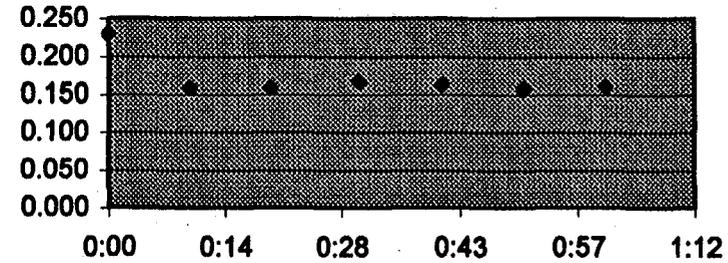
Average Flow = 0.125 Average Flux = 0.586

RAW Average Slurry Flow = 3.13

**C-104 Sludge Flux vs. Time
at 52.4 psig and 3.1 gpm**

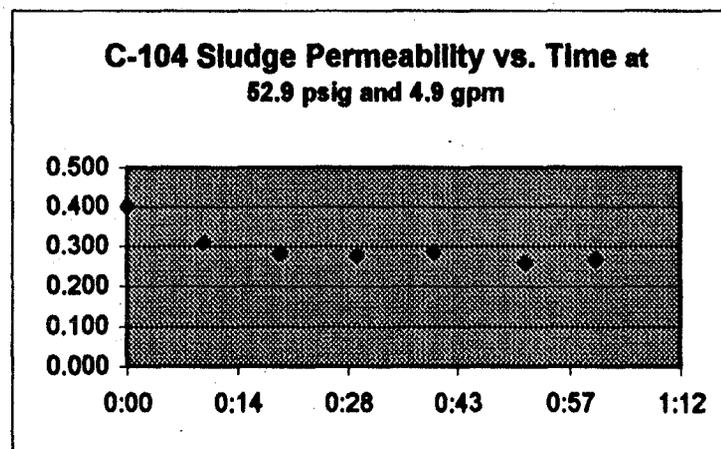
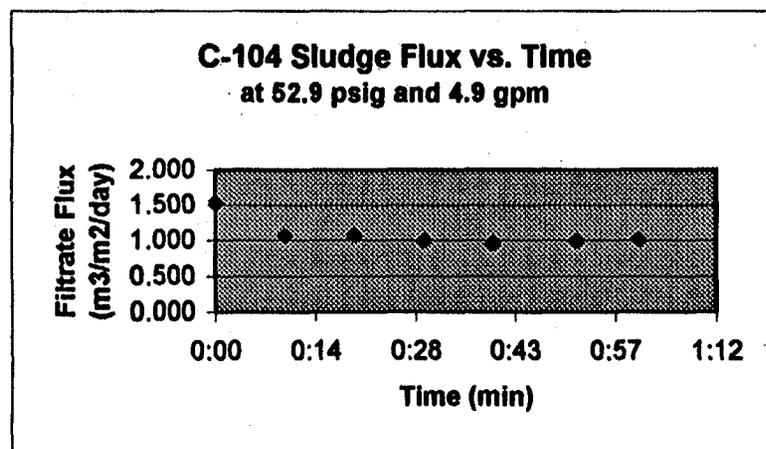


**C-104 Sludge Permeability vs. Time at
52.4 psig and 3.1 gpm**



Matrix 2 Condition Number	Time	Total Time Elapsed (Min)	Slurry Loop Flow Rate (gpm)	Filter Outlet Pressure (psig)	Filter Inlet Pressure (psig)	Pressure Drop (psig)	Filtrate Sample Volume (mL)	Time of Collection (Sec)	Filtrate Flow Rate (mL/sec)	Slurry Temp C	Filtrate Flux (m ³ /m ² /day)	Permeability (m/day/bar)
5	7:19	0:00	5	NA	55	#VALUE!	10	33.38	0.300	22.6	1.519	0.401
5	7:29	0:10	5.1	NA	50	#VALUE!	10	45.22	0.221	24.5	1.062	0.308
5	7:39	0:20	5	NA	55	#VALUE!	10	44.72	0.224	24.7	1.068	0.282
5	7:49	0:30	5	NA	52	#VALUE!	10	51.25	0.195	22.6	0.989	0.276
5	7:59	0:40	5	NA	48	#VALUE!	10	51.35	0.195	24.1	0.946	0.286
5	8:11	0:52	4.7	NA	55	#VALUE!	10	45.85	0.218	26.9	0.980	0.258
5	8:20	1:01	4.8	NA	55	#VALUE!	10	49.06	0.204	23.4	1.010	0.266

RAW Average Slurry Flow = 4.93 Average Pressure = 52.85714 Average Flow = 0.222 Average Flux = 1.009

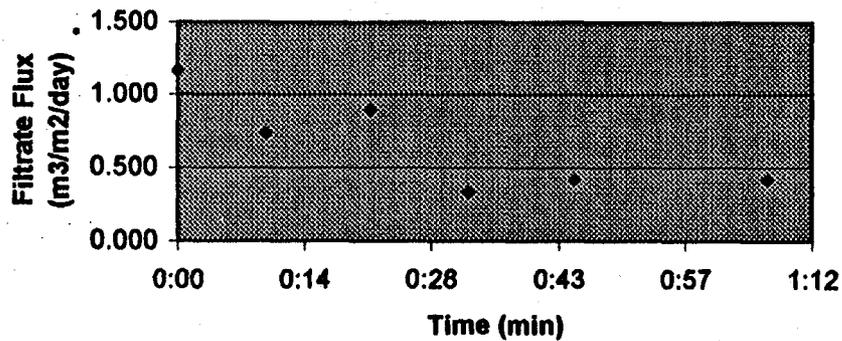


Matrix 2 Condition Number	Time	Total Time Elapsed (Min)	Slurry Loop Flow Rate (gpm)	Filter Outlet Pressure (psig)	Filter Inlet Pressure (psig)	Pressure Drop (psig)	Filtrate Sample Volume (mL)	Time of Collection (Sec)	Filtrate Flow Rate (mL/sec)	Slurry Temp C	Filtrate Flux (m3/m2/day)	Permeability (m/day/bar)
6	8:48	0:00	4.1	NA	50	#VALUE!	10	37.69	0.265	27.8	1.162	0.337
6	8:58	0:10	3.9	NA	52	#VALUE!	10	68.2	0.147	23.1	0.733	0.204
6	9:10	0:22	3.9	NA	50	#VALUE!	10	57.6	0.174	22.2	0.890	0.258
6	9:21	0:33	3.8	NA	55	#VALUE!	10	132.72	0.075	27.7	0.331	0.087
6	9:33	0:45	4.1	NA	50	#VALUE!	10	126.28	0.079	21	0.420	0.122
6	9:55	1:07	4.3	NA	48	#VALUE!	10	111.96	0.089	25.2	0.421	0.127

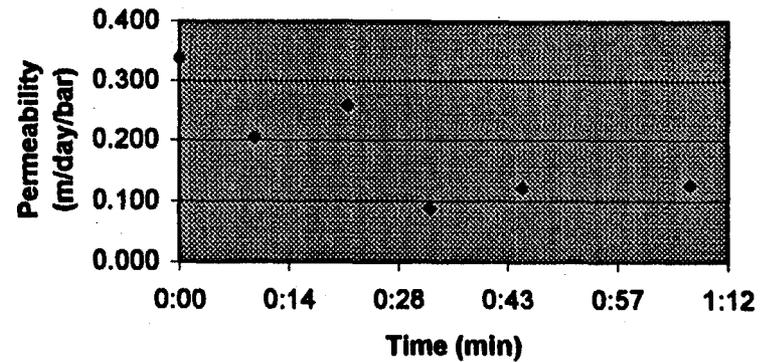
Average Pressure = 50.83333 Average Flow = 0.138 Average Flux = 0.559

RAW Average Slurry Flow = 4.02

**C-104 Sludge Flux vs. Time
at 50.8 psig and 4.0 gpm**



**C-104 Sludge Permeability vs. Time at
50.8 psig and 4.0 gpm**



Date: 8/26/1988 - 8/28/88

Tank Filter	C104 Mott 0.1 micron - L	NOTES Sheet #7	Test Number Dewatering #1	Time	Chiller Temp C	Shurry Temp C	Shurry Rate (gpm)	Filter Outlet Pressure (psig)	Permeate Pressure (psig)	Flow Rate (gpm)	Time of Collection (Sec)	Filter Flow Rate (mL/Sec)	Temperature (C)	Filter Flux (m ³ /m ² /day)	Permeability (mD/psia)	Filter Flux (gpm/ft ²)	
																	Test Conditions
				23:11	23.4	21.8	4.7 NA	1	1	48	10	19.78	21.8	2.623	0.846	0.044713	
				23:20	23.5	20.5	4.8 NA	1	1	60	10	20.500	26.5	2.336	0.877	0.039808	
				23:30	23.2	22.7	4.1 NA	1	1	48	10	33.81	26.7	1.332	0.450	0.022708	
				23:43	23.8	22.8	6.1 NA	1	1	48	10	44.19	22.7	1.144	0.346	0.018604	
				Average Shurry Flow =			4.43										
				Average Pressure =			47.25										
				Average Filtrate Flow =			6.28										
				Average Permeability =			1.88										0.00
				Average Permeability =			0.87										0.01
				1/1/00	23:58	25.1	4.8 NA	1	1	60	10	15.08	24.8	3.181	0.823	0.042228	
				1/2/00	0:08	27.1	4.8 NA	1	1	60	10	25.63	26.8	1.778	0.616	0.030328	
				1/2/00	0:18	24.8	6.1 NA	1	1	48	10	38.34	23.7	1.248	0.370	0.021289	
				1/2/00	0:22	23.7	6 NA	1	1	60	10	41.34	22.8	1.218	0.383	0.020773	
				Average Shurry Flow =			4.86										
				Average Pressure =			49.75										
				Average Filtrate Flow =			6.29										
				Average Permeability =			1.98										
				Average Permeability =			0.84										
				0:36	27	27.2	6 NA	1	1	60	10	17.44	27.2	2.664	0.741	0.049538	
				0:54	24	22.8	4.8 NA	1	1	48	10	26.8	22.8	1.769	0.624	0.030186	
				1:05	24.8	24.8	5 NA	1	1	48	10	32.66	24.8	1.422	0.433	0.024816	
				Average Shurry Flow =			4.63										
				Average Pressure =			48.33										
				Average Filtrate Flow =			6.41										
				Average Permeability =			1.63										
				Average Permeability =			0.87										
				1:24	22.7	20.9	4.78 NA	1	1	60	10	18.72	20.9	2.844	0.826	0.046488	
				1:36	26	28.1	4.8 NA	1	1	61	10	29.28	26.1	1.748	0.487	0.029781	
				1:51	26.1	25.1	4.8 NA	1	1	60	10	44	25.1	1.073	0.311	0.016288	
				Average Shurry Flow =			4.66										
				Average Pressure =			46.33										
				Average Filtrate Flow =			1.98										
				Average Permeability =			1.81										
				Average Permeability =			0.84										
				2:14	24.7	24.5	4.8 NA	1	1	60	10	23.78	24.5	2.023	0.687	0.03448	
				2:48	26.8	28	4.8 NA	1	1	60	10	48.22	28	0.868	0.288	0.014888	
				3:16	24.4	23.8	4.3 NA	1	1	60	10	86.22	23.8	0.808	0.148	0.004881	
				Average Shurry Flow =			4.69										
				Average Pressure =			66.88										
				Average Filtrate Flow =			6.26										
				Average Permeability =			1.18										
				Average Permeability =			0.34										
				3:25	24.8	25	4.8 NA	1	1	60	10	37.81	25	1.322	0.264	0.022641	
				3:42	27.8	27.8	4.8 NA	1	1	60	10	66.88	27.8	0.863	0.182	0.011304	
				3:52	26.3	25.2	4.8 NA	1	1	61	10	70.31	25.2	0.870	0.181	0.01142	
				4:04	23.3	22.4	4.4 NA	1	1	60	10	84.78	22.4	0.838	0.186	0.009178	
				4:18	28	28.2	4.3 NA	1	1	48	10	84.18	28.2	0.818	0.182	0.008778	
				4:28	25.2	24.1	4.8 NA	1	1	48	10	172.36	24.1	0.282	0.083	0.004806	
				4:52	27.8	27.1	4.18 NA	1	1	48	6	71.83	27.1	0.312	0.082	0.006318	
				Average Shurry Flow =			4.48										
				Average Pressure =			48.71										
				Average Filtrate Flow =			3.96										
				Average Permeability =			0.81										
				Average Permeability =			0.18										
				5:22	26.4	24.2	4.8 NA	1	1	48	10	48.84	24.2	0.880	0.288	0.018878	
				5:42	22.7	21.8	4.1 NA	1	1	60	10	66.33	21.8	0.789	0.227	0.013814	
				5:56	27.8	27.7	4.8 NA	1	1	48	10	71.16	27.7	0.617	0.183	0.010326	
				6:06	25	23.7	4.8 NA	1	1	48	10	80.83	23.7	0.842	0.184	0.008243	
				6:20	25.2	25.1	4.2 NA	1	1	48	10	109.47	25.1	0.448	0.133	0.007834	
				6:28	25.1	24	4.2 NA	1	1	48	6	88.8	24	0.368	0.106	0.006083	
				6:43	25.8	25.4	4.2 NA	1	1	60	6	73.19	25.4	0.320	0.085	0.006464	
				Average Shurry Flow =			4.47										
				Average Pressure =			48.14										
				Average Filtrate Flow =			3.88										
				Average Permeability =			0.66										
				Average Permeability =			0.36										
				Average Permeability =			0.17										

Dewatering #2
 Dewatering #3
 Dewatering #4
 Dewatering #5
 Dewatering #6
 Dewatering #7
 Dewatering #8

Had to permeate both.
 1.85 Done
 8/26/1988 Sheet #8
 Changed bottles @ -2:40
 Mott 0.1 micron - L
 No Backpulses
 Lyndale Jagoda
 Kitson Trouble took over about 3:07 AM
 Backpulsed twice

**Appendix E: Physical Properties Test
Instruction**

Appendix E: Physical Properties Test Instruction

PNNL Test Instruction

Document No.: BNFL-TP-29953-051
Rev. No.: 0

Title: BNFL Ultrafiltration Physical Testing: C-104

Work Location: Radiochemical Processing
Laboratory

Page 1 of 18

Author: Paul Bredt

Effective Date: Upon Final Approval
Supersedes Date: New

Use Category Identification: Reference

Identified Hazards:

- Radiological
- Hazardous Materials
- Physical Hazards
- Hazardous Environment
- Other:

Required Reviewers:

Technical Reviewer

Are One-Time Modifications Allowed to this Test Instruction? Yes No

NOTE: If Yes, then modifications are not anticipated to impact safety. For documentation requirements of a modification see SBMS or the controlling Project QA Plan as appropriate.

On-The Job Training Required? Yes or No

FOR REVISIONS:

Is retraining to this procedure required? Yes No

Does the OJT package associated with this procedure require revision to reflect procedure changes?
 Yes No N/A

Approval

Signature

Date

Author

Paul Bredt

8/24/99

Technical Reviewer

D. G. E. Schubert

8/24/99

PR Bredt
August 23, 1999

Test Instruction: TI-29953-051
Page 2 of 18

BNFL Ultrafiltration Physical Testing: C-104

This test instruction defines work to be conducted on slurry samples collected during the ultrafiltration of materials from tanks 241-C-104. The samples are to be ultrafiltered to concentrate the solids in the slurry. Under this test instruction, samples of the initial feed and concentrated slurry will be collected and analyzed for select rheological and physical properties.

This test instruction provides details regarding the implementation of Technical Procedure 29953-010. Client expectations for successful achievement of project data needs have already been established via the ultrafiltration test specifications provided to Battelle by BNFL. This test instruction is an internal mechanism for the cognizant scientist, to communicate to staff specifics on procedure implementation.

Signature Paul Bredt Date 3/10/99

General Instructions:

- 1) Keep the sample in a sealed container as much as possible to prevent it from drying.
- 2) Sign and date the bottom of each page when the requested analyses are complete.
- 3) Cross-contamination between samples and contamination of samples from outside sources must be minimized at each step. Use new tools and bottles for each sample as much as possible. Those tools which are reused should be washed and rinsed prior to reuse.

M&TE List:

_____ Balance 1:

Calib ID 388-86-01-020 Calib Exp Date 8/2000
Location C-Cell

_____ Balance 2:

Calib ID _____ Calib Exp Date _____
Location _____

_____ Other 1: Digital thermometer

Calib ID 2731 Calib Exp Date 5-00
Location 601

_____ Other 2:

Calib ID _____ Calib Exp Date _____
Location _____

Signature Pal Bredt Date 3/10/00

Initial C-104 Viscosity Testing

- 1) If not performed in the last 30 days, analyze one standard between 10 and 100 cP for shear stress as a function of shear rate at 25°C from 0 to approximately 300 s⁻¹. Print out a copy of the rheogram and attach to this test instruction.

Viscometer M5-6 ^{1A 8/24/99} Location 601
Viscosity 49.9 cP Lot 102298 Manufacturer Brookfield
File name 082499a Date analyzed 8/24/99

- 2) Analyze the C-104 initial slurry provided by the ultrafiltration staff for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C from 0 to approximately 300 s⁻¹. Print out a copy of the rheograms and attach to this test instruction. Record the time and date the sample was collected from the ultrafiltration apparatus.

Time Collected _____ Date Collected 8/24/99
C-104 File name 082499B Date analyzed 8/24/99
re-run of same sample 082499C
C-104 Duplicate File name 082499D Date analyzed 8/24/99
re-run of same sample 082499E

10:20pt

Signature Paul Bredt Date 3/10/00

Initial C-104 Physical Properties

1) Record the identification number for the sample of C-104 initial slurry provided by the ultrafiltration task.
Sample ID CUF-C104-002 (initial ~50% solids loading)

2) Thoroughly agitate the sample provided by the Ultrafiltration task and transfer ~8 ml of each in duplicate into preweighed graduated 10 ml centrifuge cones. These cones need to be rate for use at ~105°C. Weigh the loaded centrifuge cones.

Test tube support
tare = 47.628g

	C104A			C104B	
Total	<u>28.403</u>	g	Total	<u>28.108</u>	g
Tare	<u>19.872</u>	g	Tare	<u>19.779</u>	g
Slurry	<u>8.531</u>	g	Slurry	<u>8.329</u>	g

3) Agitate the cones and record the time and date.

Time 14:38 Date 10/13/99

4) One hour after agitation, record the volume of the total sample and the volume of settled solids.

Date 10/13/99 Time 15:44

	C104A			C104B	
Total	<u>8.0</u>	ml	Total	<u>8.0</u>	ml
Solids	<u>6.0</u>	ml	Solids	<u>5.4</u>	ml
Liquid	<u>2.0</u>	ml	Liquid	<u>2.6</u>	ml

5) Before the end of the shift (day 1), record the volume of the total sample and the volume of settled solids.

Date _____ Time _____

END

	C104A			C104B	
Total	_____	ml	Total	_____	ml
Solids	_____	ml	Solids	_____	ml
Liquid	_____	ml	Liquid	_____	ml

C-Cell Balance #
388-06-01-020

Calib. Expires 8-2000

200g → 200.001g
100g → 100.001g

Signature [Signature] Date 10/22/99

[Signature]

- 6) At the start of the next day (day 2), record the volume of the total sample and the volume of settled solids.

Date 10/14/99 Time 08:30

C104A		C104B	
Total	<u>7.95</u> ml	Total	<u>7.80</u> ml
Solids	<u>2.6</u> ml	Solids	<u>2.4</u> ml
Liquid	<u>5.35</u> ml	Liquid	<u>5.4</u> ml

- 7) At the end of the day (day 2), record the volume of the total sample and the volume of settled solids.

Date 10/14/99 Time 14:45

C104A		C104B	
Total	<u>7.95</u> ml	Total	<u>7.8</u> ml
Solids	<u>2.6</u> ml	Solids	<u>2.3</u> ml
Liquid	<u>5.35</u> ml	Liquid	<u>5.5</u> ml

- 8) At the start of the next day (day 3), record the volume of the total sample and the volume of settled solids.

Date 10/15/99 Time 10:00 AM

C104A		C104B	
Total	<u>7.9</u> ml	Total	<u>7.8</u> ml
Solids	<u>2.5</u> ml	Solids	<u>2.2</u> ml
Liquid	<u>5.4</u> ml	Liquid	<u>5.6</u> ml

Friday

- 9) At the end of the day (day 3), record the volume of the total sample and the volume of settled solids.

Date 10/15/99 Time 13:45

C104A		C104B	
Total	<u>7.4</u> ml	Total	<u>7.8</u> ml
Solids	<u>2.5</u> ml	Solids	<u>2.2</u> ml
Liquid	<u>5.4</u> ml	Liquid	<u>5.6</u> ml

Signature [Signature] Date 10/15/99

6 (weekend) rgs
left over

- 10) At the beginning of the next day (day 4), record the volume of the total sample and the volume of settled solids.

Date 10/18/99 Time 10:30

C104A		C104B	
Total	<u>7.95</u> ml	Total	<u>7.80</u> ml
Solids	<u>2.50</u> ml	Solids	<u>2.15</u> ml
Liquid	<u>5.45</u> ml	Liquid	<u>5.65</u> ml

Monday

- 11) Transfer a sample of the settled supernatant into a preweighed graduated cylinder and record the mass and volume of the supernatant. Return the supernatant to the centrifuge cone.

C104A		C104B	
Total	<u>30.399</u> g	Total	<u>30.819</u> g
Tare	<u>25.446</u> g	Tare	<u>25.559</u> g
Liquid	<u>4.953</u> g	Liquid	<u>5.260</u> g
Liquid	<u>4.92</u> ml	Liquid	<u>5.15</u> ml

Tuesday
10/19/99
rgs

- 12) Centrifuge the cones at $\sim 1000 \times g$ for one hour. Reweigh the centrifuge cones and record the volume of the total sample and volume of centrifuged solids.

Before Centrifuge
28.090

C104A		C104B	
Total	<u>28.076</u> g	Total	<u>27.487</u> g
Tare	<u>19.872</u> g	Tare	<u>19.779</u> g
Sample	<u>8.204</u> g	Sample	<u>8.208</u> g
Total	<u>7.60</u> ml	Total	<u>7.70</u> ml
Solids	<u>~1.0</u> ml	Solids	<u>0.75</u> ml
Liquid	<u>6.60</u> ml	Liquid	<u>6.95</u> ml

Before Cent.

- 13) Decant as much of the centrifuged supernatant as possible to a preweighed graduated cylinder and record the mass and volume of the supernatant. Weigh the solids left in the centrifuge cone.

C104A		C104B	
Liquid+Grad	<u>32.300</u> g	Liquid+Grad	<u>32.645</u> g
Grad	<u>25.446</u> g	Grad	<u>25.559</u> g
Liquid	<u>6.854</u> g	Liquid	<u>7.086</u> g
Liquid	<u>6.78</u> ml	Liquid	<u>6.92</u> ml
Solids+cone	<u>21.241</u> g	Solids+cone	<u>20.844</u> g
Cone	<u>19.872</u> g	Cone	<u>19.779</u> g
Solids	<u>1.369</u> g	Solids	<u>1.120</u> g

Signature *[Signature]* Date 10/20/99

- 14) Transfer the decanted supernatant to preweighed 20 ml vials with lids rated to 105°C. Weigh the loaded beakers.

	C104AL	
Total	<u>23.438</u>	g
Tare	<u>16.678</u>	g
Supernatant	<u>6.760</u>	g

	C104BL	
Total	<u>23.895</u>	g
Tare	<u>16.896</u>	g
Supernatant	<u>6.999</u>	g

- 15) Air dry the solids and liquids overnight to minimize splattering during the next drying step. Consult the cognizant scientist on the use of a heat lamp or other drying technique to speed up this preliminary drying.
- 16) Transfer the solids and liquids to an oven at 105°C for 24 hours.
- 17) Remove the solids and liquids from the oven and cap the vials. Allow the vial to cool ~1 hour and reweigh.

	C104AL	
Total	<u>16.889</u>	g
Tare	<u>16.678</u>	g
sample	<u>0.211</u>	g

	C104BL	
Total	<u>17.110</u>	g
Tare	<u>16.896</u>	g
sample	<u>0.214</u>	g

CowE

	C104A	
Total	<u>20.544</u>	g
Tare	<u>19.872</u>	g
sample	<u>0.672</u>	g

	C104B	
Total	<u>20.316</u>	g
Tare	<u>19.779</u>	g
sample	<u>0.537</u>	g

Signature

PR Bredt

Date

10/27/99

Intermediate C-104 Viscosity Testing

- 1) Analyze the C-104 Intermediate slurry provided by the ultrafiltration staff for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C from 0 to approximately 300 s⁻¹. Print out a copy of the rheograms and attach to this test instruction. Record the time and date the sample was collected from the ultrafiltration apparatus.

Time Collected 9:00 am

Date Collected 8/26/99

Intermediate C-104
Dup on run 1

File name 082699A
082699B

Date analyzed 8/26/99

Intermediate C-104 Duplicate
Dup on run 2

File name 082699C
082699D

Date analyzed 8/26/99

C-104 mF Slurry was then diluted and reanalyzed
at 12:00 on 8/26/99

run 1 082699AE 8/26/99

run 1 Dup ~~082699B2~~ 082699F

run 1 Dup 2 082699F2

run 2 082699G ← note on printout was "run 1 Dup"

run 2 Dup 082699H

run 2 Dup 2 082699I

Signature Pat Bredt

Date 8/26/99

C-104 Intermediate Physical Properties

- 1) Record the identification number for the sample of C-104 Intermediate slurry provided by the ultrafiltration task.

Sample ID CUF-C104-006 (high solids loading)

- 2) Thoroughly agitate the sample provided by the Ultrafiltration task and transfer ~8 ml of each in duplicate into preweighed graduated 10 ml centrifuge cones. These cones need to be rate for use at ~105°C. Weigh the loaded centrifuge cones.

	C104C			C104D	
Total	<u>27.659</u>	g	19.731g	Total	_____ g
Tare	<u>19.731</u>	g	19.731g	Tare	<u>20.170</u> g
Slurry	<u>7.928</u>	g		Slurry	_____ g

This sample was very thick but still hard to transfer with spatula or pipet. Transferred as much as possible to "C" vial. Not enough sample for duplicate

- 3) Agitate the cones and record the time and date.

Time 14:40 Date 10/13/99

- 4) One hour after agitation, record the volume of the total sample and the volume of settled solids.

Date 10/13/99 Time 15:44

Not enough sample for duplicate

	C104C			C104D	
Total	<u>~6.5</u>	ml		Total	_____ ml
Solids	<u>6.5</u>	ml		Solids	_____ ml
Liquid	_____	ml		Liquid	_____ ml

- 5) Before the end of the shift (day 1), record the volume of the total sample and the volume of settled solids.

Date _____ Time End

	C104C			C104D	
Total	_____	ml		Total	_____ ml
Solids	_____	ml		Solids	_____ ml
Liquid	_____	ml		Liquid	_____ ml

A fair amt of sludge stayed on side.

C-104
See A, B samples for Balance info. & wt ck data
ryj

Signature [Signature] Date 10/13/99

CuF-C104-006

- 6) At the start of the next day (day 2), record the volume of the total sample and the volume of settled solids.

Date 10/14/99 Time 08:30

	C104C		C104D	
Total	<u>6.6</u> ml	Total	_____ ml	
Solids	<u>6.3</u> ml	Solids	_____ ml	
Liquid	<u>.2</u> ml	Liquid	_____ ml	

Note enough
Sample
for Duplicate
RgB
10/13/99

- 7) At the end of the day (day 2), record the volume of the total sample and the volume of settled solids.

Date 10/14/99 Time 14:50

	C104C		C104D	
Total	<u>6.5</u> ml	Total	_____ ml	
Solids	<u>6.3</u> ml	Solids	_____ ml	
Liquid	<u>.2</u> ml	Liquid	_____ ml	

- 8) At the start of the next day (day 3), record the volume of the total sample and the volume of settled solids.

Date 10/15/99 Time 10:00

	C104C		C104D	
Total	<u>6.2</u> ml	Total	_____ ml	
Solids	<u>6.0</u> ml	Solids	_____ ml	
Liquid	<u>.2</u> ml	Liquid	_____ ml	

Friday

- 9) At the end of the day (day 3), record the volume of the total sample and the volume of settled solids.

Date 10/15/99 Time 15:45

	C104C		C104D	
Total	<u>6.3</u> ml	Total	_____ ml	
Solids	<u>6.1</u> ml	Solids	_____ ml	
Liquid	<u>.2</u> ml	Liquid	_____ ml	

* Sludge on side of test tube above minus cu

Signature RgB Date 10/15/99

10) At the beginning of the next day (day 4), record the volume of the total sample and the volume of settled solids.

Date 10/18/99 Time 10:35

	C104C		C104D	
Total	<u>6.25</u> ml	Total	_____ ml	<i>Note enough sample for duplicate</i> <i>ryj</i>
Solids	<u>6.10</u> ml	Solids	_____ ml	
Liquid	<u>.15</u> ml	Liquid	_____ ml	

11) Transfer a sample of the settled supernatant into a preweighed graduated cylinder and record the mass and volume of the supernatant. Return the supernatant to the centrifuge cone.

	C104C		C104D
Total	_____ g	Total	_____ g
Tare	_____ g	Tare	_____ g
Liquid	_____ g	Liquid	_____ g
Liquid	_____ ml	Liquid	_____ ml

Not possible

12) Centrifuge the cones at ~1000 x g for one hour. Reweigh the centrifuge cones and record the volume of the total sample and volume of centrifuged solids.

Start centrifuge
10/20/99
@ 9:50
11:05

	C104C		C104D
Total	<u>27.494</u> g	Total	_____ g
Tare	<u>19.731</u> g	Tare	_____ g
Sample	<u>7.763</u> g	Sample	_____ g
Total	<u>6.45</u> ml	Total	_____ ml
Solids	<u>~3.00</u> ml	Solids	_____ ml
Liquid	<u>3.45</u> ml	Liquid	_____ ml

Sludge still on side of test tube above Aquasana membrane.
ryj

13) Decant as much of the centrifuged supernatant as possible to a preweighed graduated cylinder and record the mass and volume of the supernatant. Weigh the solids left in the centrifuge cone.

	C104C		C104D
Liquid+Grad	<u>28.790</u> g	Liquid+Grad	_____ g
Grad	<u>25.304</u> g	Grad	_____ g
Liquid	<u>3.426</u> g	Liquid	_____ g
Liquid	<u>3.48</u> ml 6.78 <i>ryj 10/20/99</i>	Liquid	_____ ml
Solids+cone	<u>24.028</u> g <i>d = 0.984</i>	Solids+cone	_____ g
Cone	<u>19.731</u> g	Cone	_____ g
Solids	<u>4.297</u> g	Solids	_____ g

Signature *ryj* Date 10/20/99

CUF-C104-006

- 14) Transfer the decanted supernatant to preweighed 20 ml vials with lids rated to 105°C. Weigh the loaded beakers.

	C104CL	
Total	<u>20.332</u>	g
Tare	<u>16.983</u>	g 16.983
Supernatant	<u>3.349</u>	g

	C104DL	
Total	_____	g
Tare	_____	g
Supernatant	_____	g

Not enough sample for duplicate

- 15) Air dry the solids and liquids overnight to minimize splattering during the next drying step. Consult the cognizant scientist on the use of a heat lamp or other drying technique to speed up this preliminary drying.
- 16) Transfer the solids and liquids to an oven at 105°C for 24 hours.
- 17) Remove the solids and liquids from the oven and cap the vials. Allow the vial to cool ~1 hour and reweigh.

	C104CL	
Total	<u>17.016</u>	g
Tare	<u>16.983</u>	g
sample	<u>0.033</u>	g

	C104DL	
Total	_____	g
Tare	_____	g
sample	_____	g

lowe

	C104C	
Total	<u>21.672</u>	g
Tare	<u>19.731</u>	g
sample	<u>1.941</u>	g

	C104D	
Total	_____	g
Tare	_____	g
sample	_____	g

Signature *[Signature]* Date 10/27/99

Final C-104 Viscosity Testing

- 1) Analyze the C-104 Final slurry provided by the ultrafiltration staff for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C from 0 to approximately 300 s⁻¹. Print out a copy of the rheograms and attach to this test instruction. Record the time and date the sample was collected from the ultrafiltration apparatus.

Time Collected _____ Date Collected _____
Final C-104 File name _____ Date analyzed _____
Final C-104 Duplicate File name _____ Date analyzed _____

See attached
Files
PRB
1/21/00

Signature Paul Bredt Date 1/21/00

PR Bredt
August 23, 1999

C-104 Final Physical Properties

C-104-009 (E, F) were ~~more~~
thicker than C10 samples, ~~stirred~~ stirred to mix ~~sample~~
then, we poured the grey sludge directly (Drozzed off the last drops that flowed.)
in the E vial to about 8ml mark, Not enough sample to transfer to a duplicate vial.

Test Instruction: TI-29953-051
Page 15 of 18

1) Record the identification number for the sample of C-104 Final slurry provided by the ultrafiltration task.

Sample ID CUF-C104-009 (medium solids loading)
C-104 Final Sample Rheology (Final material)

2) Thoroughly agitate the sample provided by the Ultrafiltration task and transfer ~8 ml of each in duplicate into preweighed graduated 10 ml centrifuge cones. These cones need to be rate for use at ~105°C. Weigh the loaded centrifuge cones.

Cone

C104E
Total 29.180 g
Tare 19.894 g
Slurry 9.286 g

C104F
Total 20.51 g
Tare 20.078 g
Slurry 0.432 g

29.492 g
20.078 g
9.414 g
G, H 29.652 g
H 20.037 g
9.615 g

3) Agitate the cones and record the time and date.

Time _____ Date _____

4) One hour after agitation, record the volume of the total sample and the volume of settled solids.

Date 10/13/99 Time 15:45

C104E
Total ~8.0 ml
Solids 8.0 ml
Liquid _____ ml

C104F
Total _____ ml
Solids _____ ml
Liquid _____ ml

G 8.3 ml
H 8.4 ml
8.3 ml
8.4 ml

5) Before the end of the shift (day 1), record the volume of the total sample and the volume of settled solids.

Date _____ Time ND

C104E
Total _____ ml
Solids _____ ml
Liquid _____ ml

C104F
Total _____ ml
Solids _____ ml
Liquid _____ ml

G _____ ml
H _____ ml

A fair amount of sludge was above the volume mark (settled volume should be higher)

No visible ~~white~~ liquid layer on any sample
Date 10/13/99 vrg

See A, B, Prepare C-104 for Balance & Wt Calc Data

Signature vrg Date 10/13/99

vrg

PR Bredt
August 23, 1999

CUF-C104-009 (E, F)

Test Instruction: TI-29953-051
Page 16 of 18

C-104 Final Sample (G, H)

- 6) At the start of the next day (day 2), record the volume of the total sample and the volume of settled solids.

Date 10/14/99 Time 8:30 AM

*

C104E	
Total	<u>8.0</u> ml
Solids	<u>7.7</u> ml
Liquid	<u>0.3</u> ml

~~C104F~~ ^{Cancelled}
~~Total~~ ml
~~Solids~~ ml
~~Liquid~~ ml

C104 G	
Total	<u>8.1</u> ml
Solids	<u>8.1</u> ml
Liquid	<u>0</u> ml

C104 H	
Total	<u>8.4</u> ml
Solids	<u>8.2</u> ml
Liquid	<u>0.2</u> ml

- 7) At the end of the day (day 2), record the volume of the total sample and the volume of settled solids.

Date 10/14/99 Time 14:50

C104E	
Total	<u>8.0</u> ml
Solids	<u>7.5</u> ml
Liquid	<u>0.5</u> ml

~~C104F~~
~~Total~~ ml
~~Solids~~ ml
~~Liquid~~ ml

C-104 G	
Total	<u>8.1</u> ml
Solids	<u>8.0</u> ml
Liquid	<u>0.1</u> ml

C104 H	
Total	<u>8.2</u> ml
Solids	<u>8.0</u> ml
Liquid	<u>0.2</u> ml

- 8) At the start of the next day (day 3), record the volume of the total sample and the volume of settled solids.

Date 10/15/99 Time 10:00

C104E	
Total	<u>8.0</u> ml
Solids	<u>7.5</u> ml
Liquid	<u>0.5</u> ml

~~C104F~~
~~Total~~ ml
~~Solids~~ ml
~~Liquid~~ ml

C-104 G	
Total	<u>8.0</u> ml
Solids	<u>7.9</u> ml
Liquid	<u>0.1</u> ml

C-104 H	
Total	<u>8.15</u> ml
Solids	<u>8.0</u> ml
Liquid	<u>0.15</u> ml

- 9) At the end of the day (day 3), record the volume of the total sample and the volume of settled solids.

Date 10/15/99 Time 15:45

C104E	
Total	<u>8.0</u> ml
Solids	<u>7.7</u> ml
Liquid	<u>0.3</u> ml

~~C104F~~
~~Total~~ ml
~~Solids~~ ml
~~Liquid~~ ml

C-104 G	
Total	<u>8.0</u> ml
Solids	<u>7.9</u> ml
Liquid	<u>0.1</u> ml

C104 H	
Total	<u>8.15</u> ml
Solids	<u>8.0</u> ml
Liquid	<u>0.15</u> ml

* Lots of material smeared on side above ~ minus axis!

10/14/99 K9

Signature

[Signature]

Date 10/15/99

PR Bredt
August 23, 1999

CUF-C104-009 (E, F)
C104 Final sample (G, H)

Test Instruction: TI-29953-051
Page 17 of 18

10) At the beginning of the next day (day A), record the volume of the total sample and the volume of settled solids.
6 - left over the weekend

Date 10/18/99 Time 10:30 *Cancelled Duplicate*

C104E		C104F		C104 G		C104 H	
Total	<u>7.90</u> ml	Total	_____ ml	<u>8.00</u>	ml	<u>8.15</u>	ml
Solids	<u>7.60</u> ml	Solids	_____ ml	<u>7.90</u>		<u>8.0</u>	
Liquid	<u>0.3</u> ml	Liquid	_____ ml	<u>0.1</u>	↓	<u>0.15</u>	↓

11) Transfer a sample of the settled supernatant into a preweighed graduated cylinder and record the mass and volume of the supernatant. Return the supernatant to the centrifuge cone.

C104E		C104F		C-104 G		C-104 H	
Total	_____ g	Total	_____ g	_____ g		_____ g	
Tare	_____ g	Tare	_____ g	_____ g		_____ g	
Liquid	_____ g	Liquid	_____ g	_____ g		_____ g	
Liquid	_____ ml	Liquid	_____ ml	_____ ml		_____ ml	

Not possible due to high solids

12) Centrifuge the cones at ~1000 x g for one hour. Reweigh the centrifuge cones and record the volume of the total sample and volume of centrifuged solids.

Start centrifuge 10/20/99 @ 9:50 stopped @ 11:05

C104E		C104F		C-104 G		C-104 H	
Total	<u>29.078</u> g	Total	_____ g	<u>29.474</u> g		<u>29.639</u> g	
Tare	<u>19.894</u> g	Tare	_____ g	<u>20.028</u> g		<u>20.037</u> g	
Sample	<u>9.184</u> g	Sample	_____ g	<u>9.396</u> g	↓	<u>9.602</u> g	↓
Total	<u>7.85</u> ml	Total	_____ ml	<u>7.95</u> ml		<u>8.25</u> ml	
Solids	<u>~4.05</u> ml	Solids	_____ ml	<u>3.55</u>	↓	<u>3.50</u>	↓
Liquid	<u>3.85</u> ml	Liquid	_____ ml	<u>4.45</u>	↓	<u>4.75</u>	↓

13) Decant as much of the centrifuged supernatant as possible to a preweighed graduated cylinder and record the mass and volume of the supernatant. Weigh the solids left in the centrifuge cone.

C104E		C104F		C104 G		C104 H	
Liquid+Grad	<u>29.280</u> g	Liquid+Grad	_____ g	<u>30.239</u> g		<u>30.421</u> g	
Grad	<u>25.568</u> g	Grad	_____ g	<u>25.773</u> g		<u>25.612</u> g	
Liquid	<u>3.712</u> g	Liquid	_____ g	<u>4.466</u> g		<u>4.809</u> g	
Liquid	<u>3.70</u> ml	Liquid	_____ ml	<u>4.45</u> ml		<u>4.78</u> ml	
Solids+cone	<u>25.310</u> g	Solids+cone	_____ g	<u>24.979</u> g		<u>24.811</u> g	
Cone	<u>19.894</u> g	Cone	_____ g	<u>20.678</u> g		<u>20.037</u> g	
Solids	<u>5.416</u> g	Solids	_____ g	<u>4.901</u> g		<u>4.774</u> g	

Cancelled Duplicate

Following centrifuging:
A little bit of *remained*
Smear substrate material on sides above aqueous medium

Signature RF Wood Date 10/20/99

PR Bredt
August 23, 1999

CuF-C104-009 (E, F)

Test Instruction: TI-29953-051

C-104 Final sample (G, H)

Page 18 of 18

14) Transfer the decanted supernatant to preweighed 20 ml vials with lids rated to 105°C. Weigh the loaded beakers.

	C104EL	C104GL		GL	
Total	<u>20.523</u> g	_____ g	Total	<u>21.282</u> g	<u>21.460</u> g
Tare	<u>16.891</u> g	_____ g	Tare	<u>16.892</u> g	<u>16.730</u> g
Supernatant	<u>3.632</u> g	_____ g	Supernatant	<u>4.390</u> g	<u>4.730</u> g

15) Air dry the solids and liquids overnight to minimize splattering during the next drying step. Consult the cognizant scientist on the use of a heat lamp or other drying technique to speed up this preliminary drying.

16) Transfer the solids and liquids to an oven at 105°C for 24 hours.

17) Remove the solids and liquids from the oven and cap the vials. Allow the vial to cool ~1 hour and reweigh.

	C104EL	C104GL		C104GL	
Total	<u>16.909</u> g	_____ g	Total	<u>16.952</u> g	<u>16.800</u> g
Tare	<u>16.891</u> g	_____ g	Tare	<u>16.892</u> g	<u>16.730</u> g
sample	<u>0.018</u> g	_____ g	sample	<u>0.060</u> g	<u>0.070</u> g

	C104E	C104F		C104G1	
Total	<u>21.929</u> g	_____ g	Total	<u>22.062</u> g	<u>21.967</u> g
Tare	<u>19.894</u> g	_____ g	Tare	<u>20.076</u> g	<u>20.037</u> g
sample	<u>2.035</u> g	_____ g	sample	<u>1.984</u> g	<u>1.930</u> g

Signature

[Handwritten Signature]

Date

10/27/99

**Appendix F: Rheograms for C-104 and
Standards**

Appendix F: Rheograms for C-104 and Standards

Figure 1. 50 cP Standard Brookfield Lot 102298, Analyzed on Haake M5 using MVI Cup and Spindle.

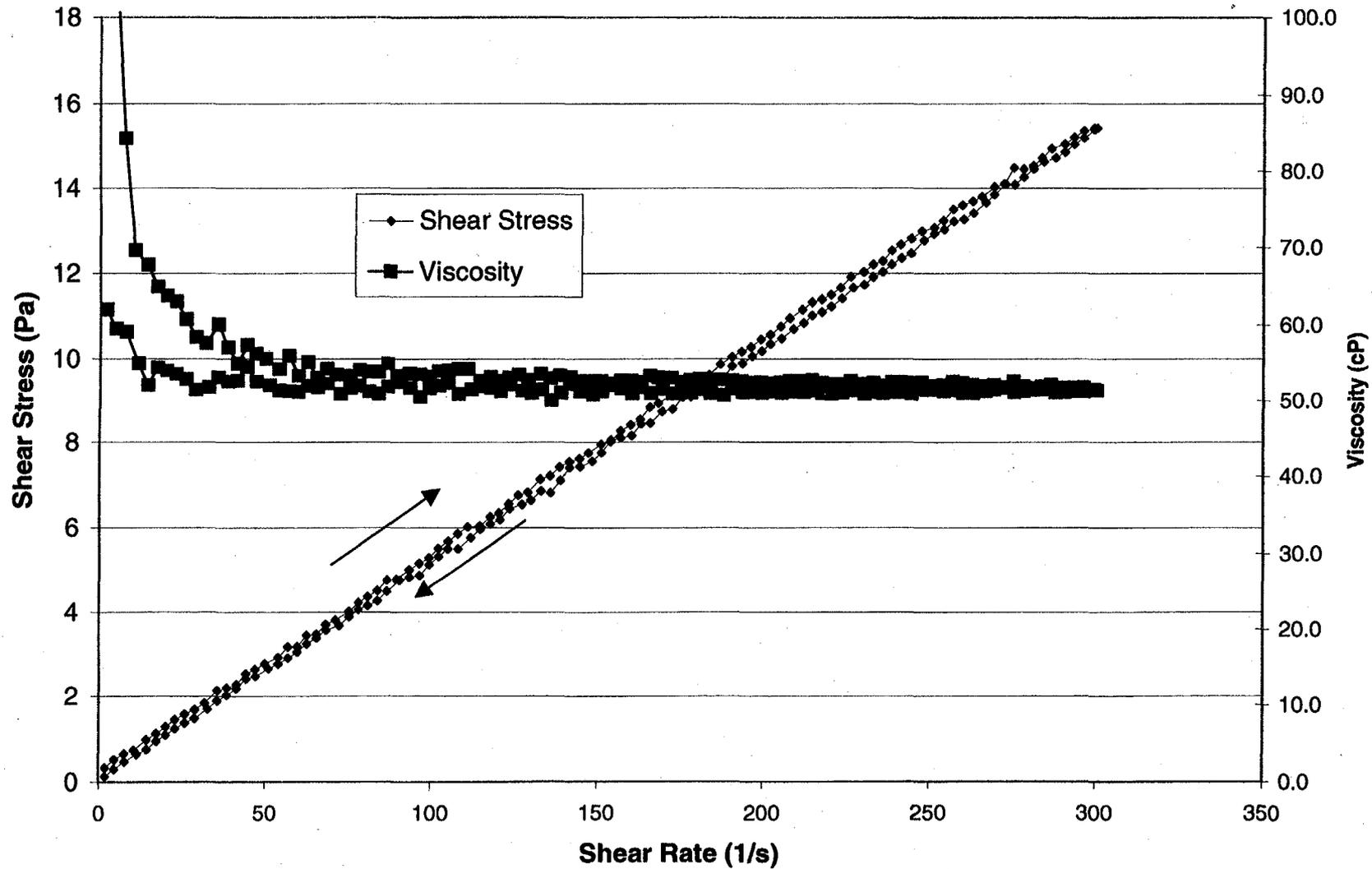


Figure 2. C-104 Initial Slurry Sample 1, First Analysis

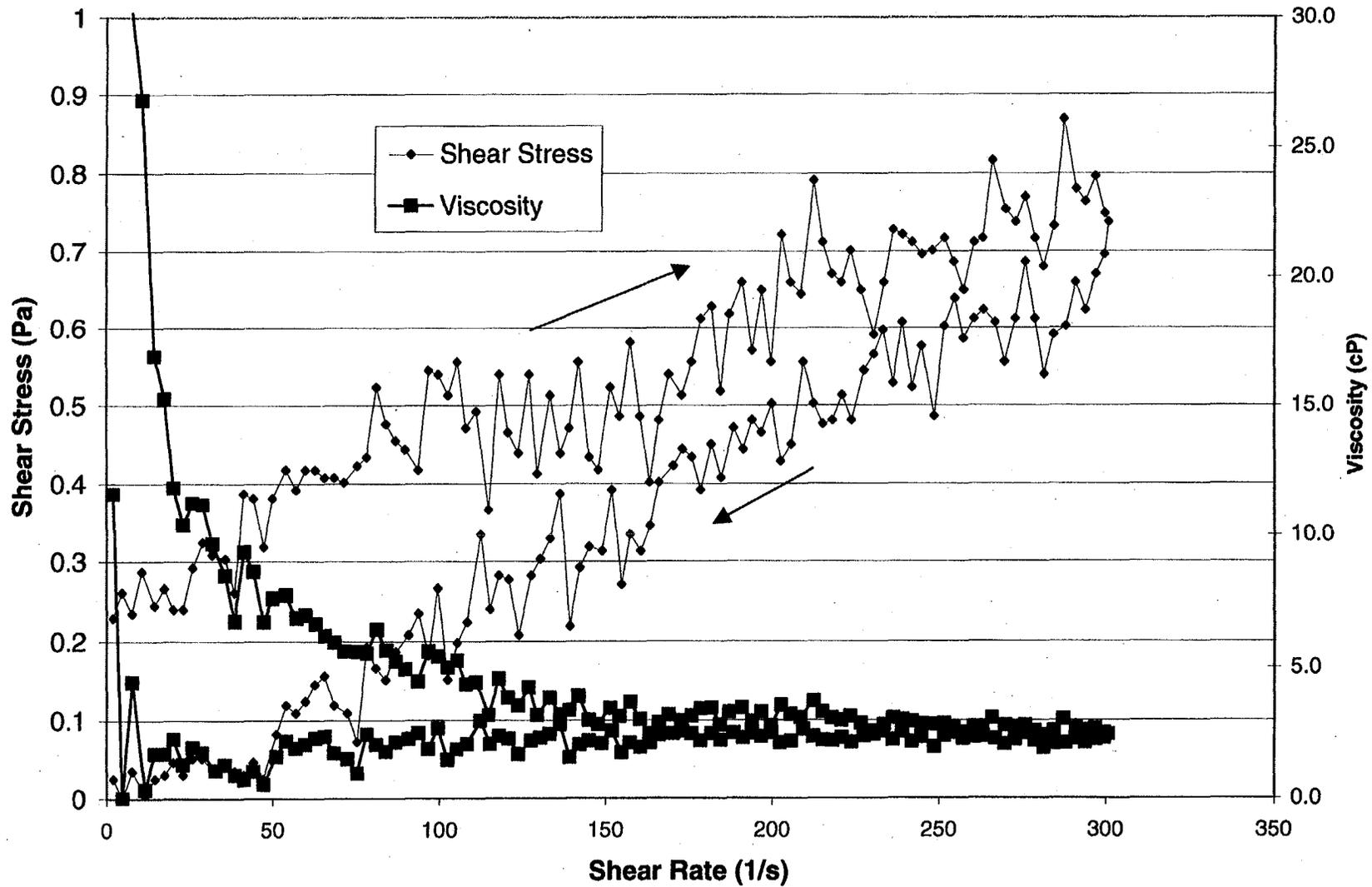


Figure 3. C-104 Initial Slurry Sample 1, Second Analysis

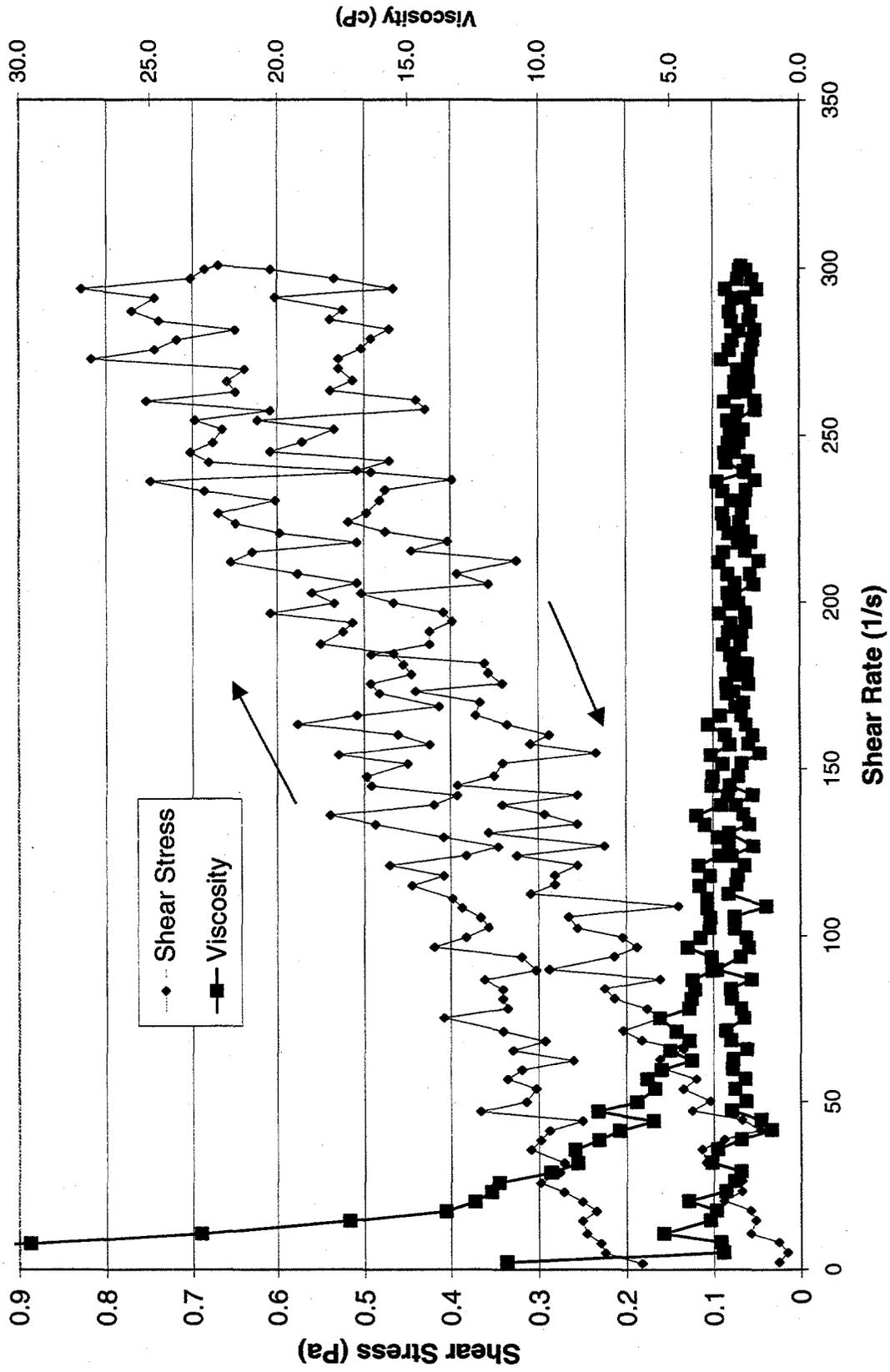


Figure 4. C-104 Initial Slurry Sample 2, First Analysis

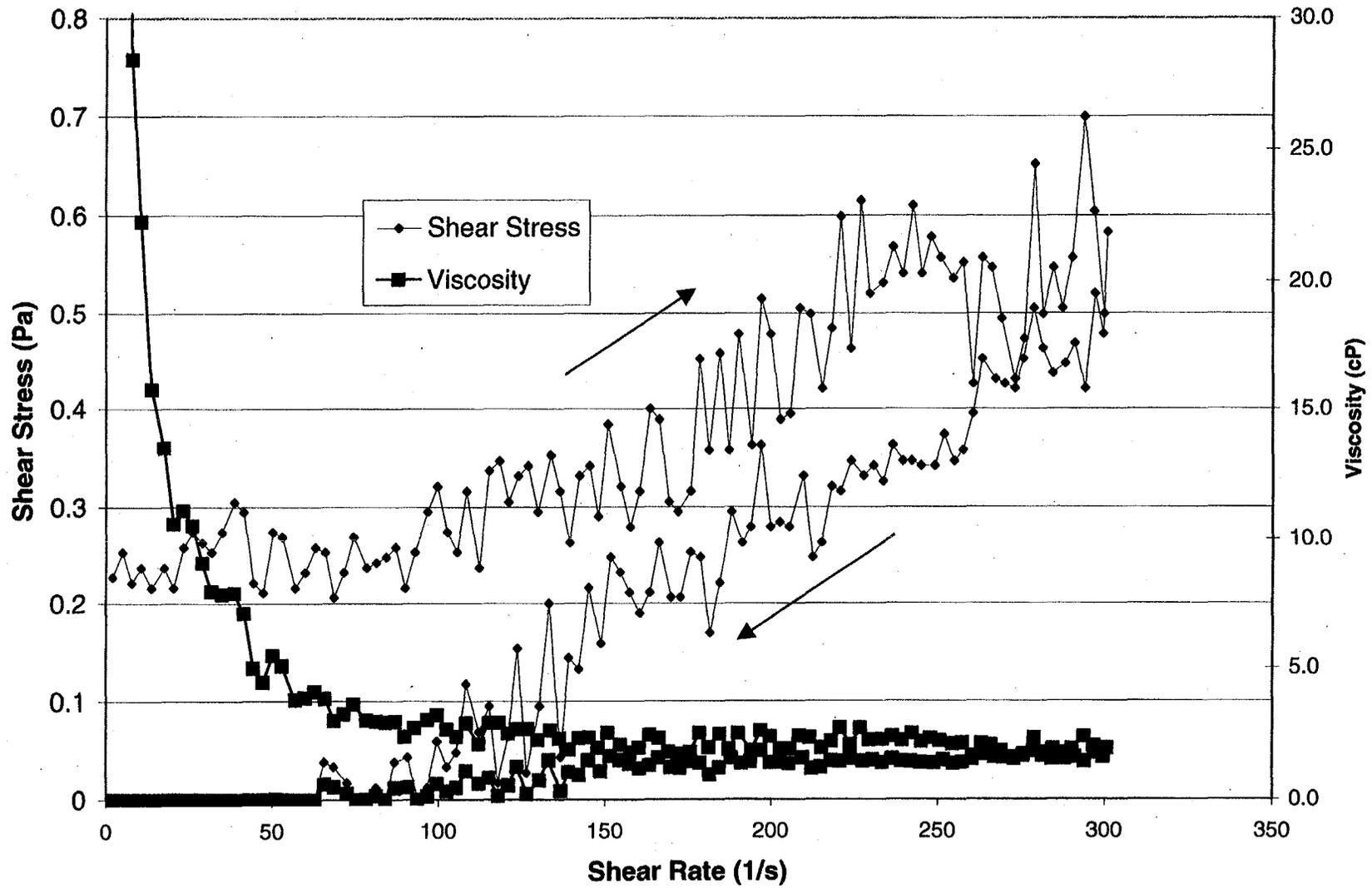


Figure 5. C-104 Initial Slurry Sample 2, Second Analysis

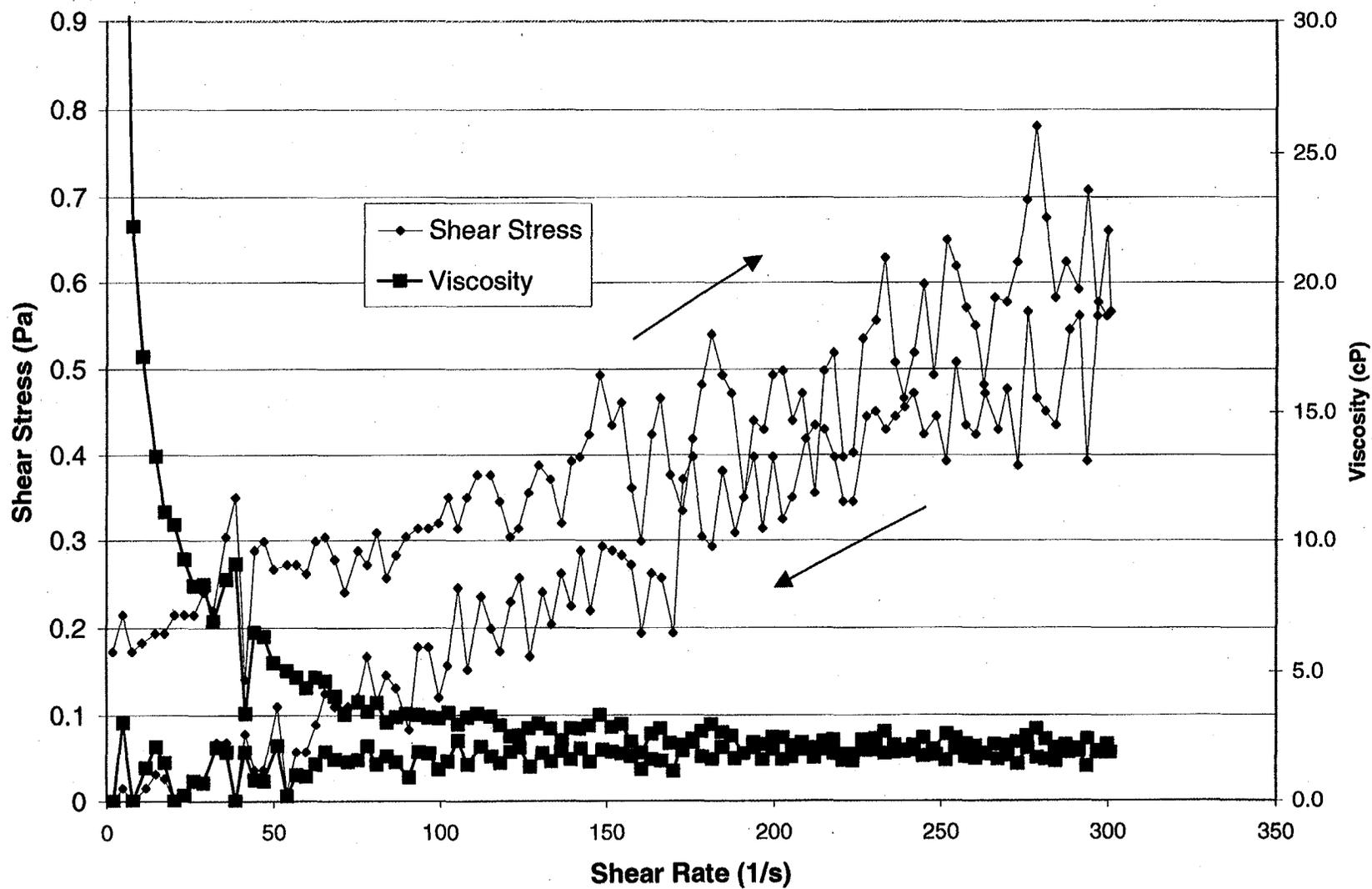


Figure 6. C-104 Washed Slurry Sample 1, First Analysis

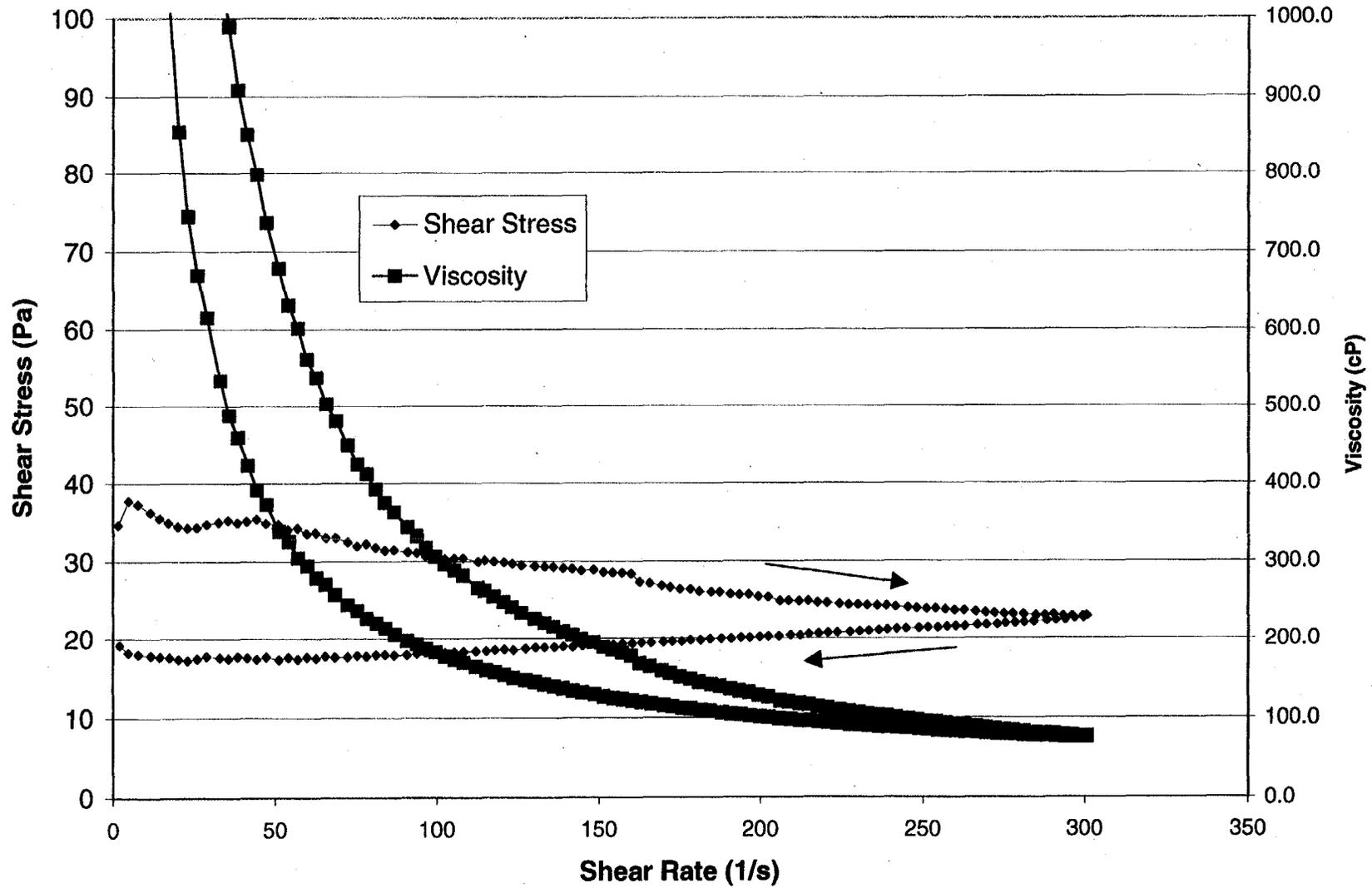


Figure 7. C-104 Washed Slurry Sample 1, Second Analysis

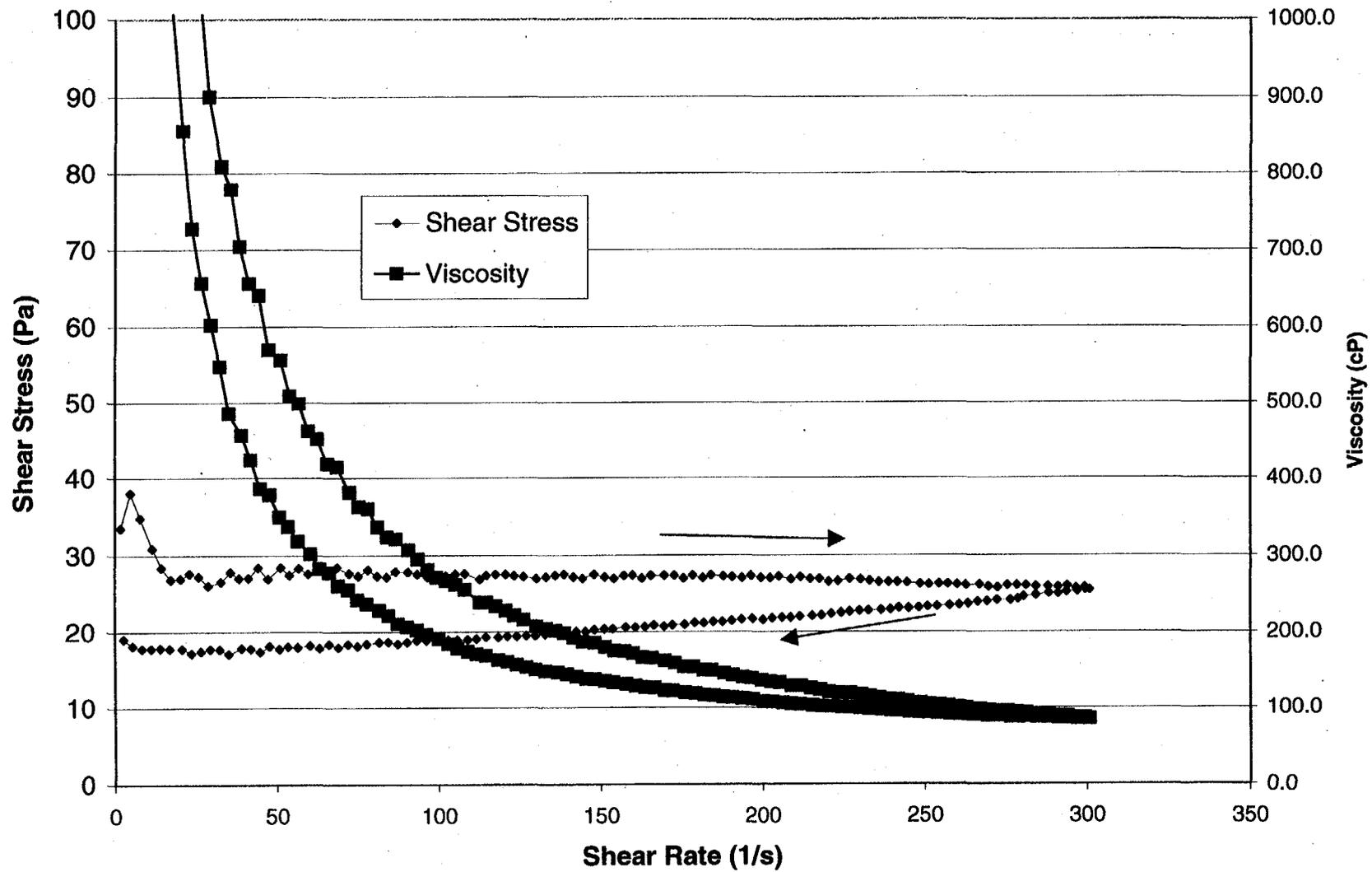


Figure 8. C-104 Washed Slurry Sample 2, First Analysis

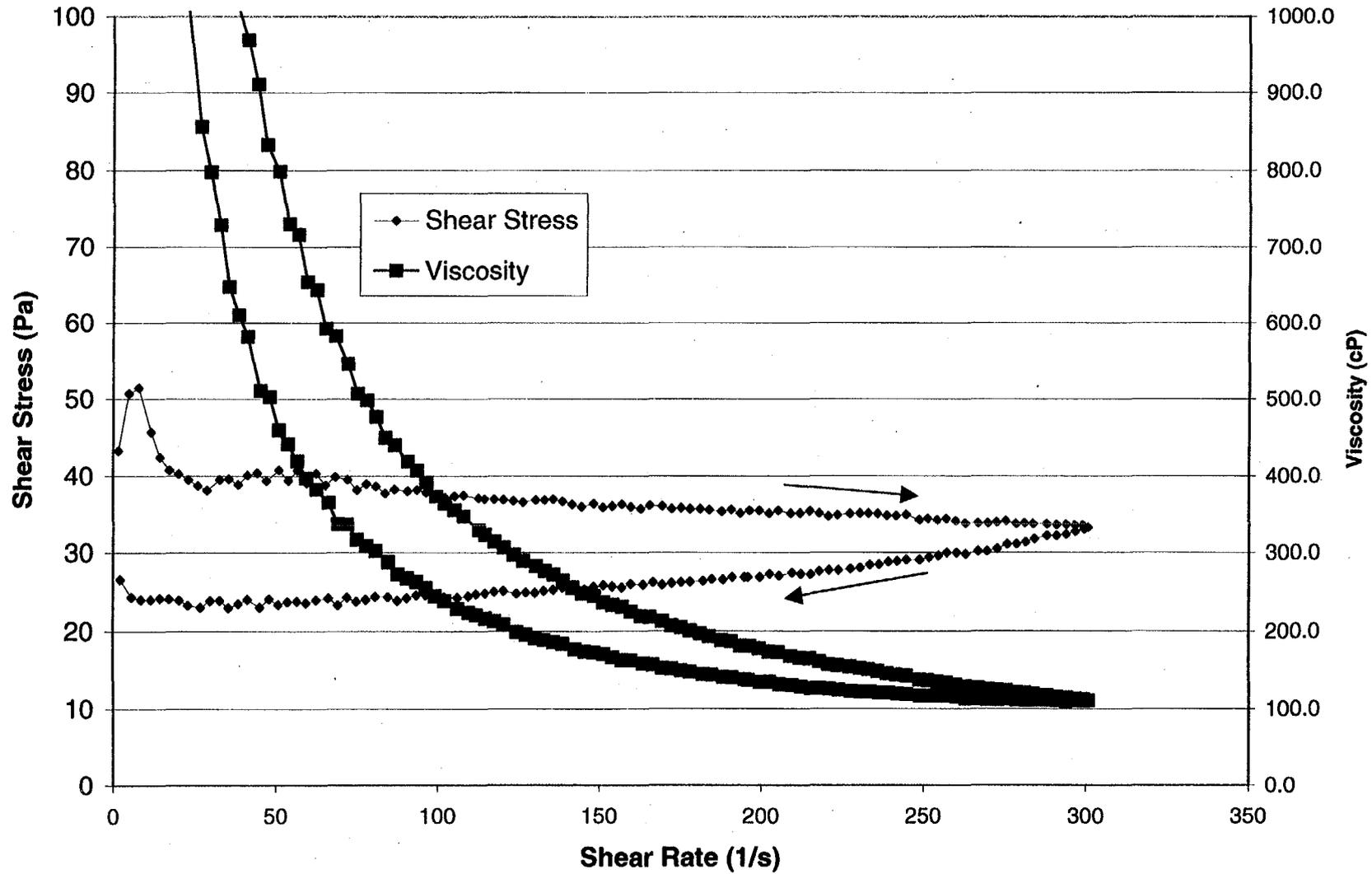


Figure 9. C-104 Washed Slurry Sample 2, Second Analysis

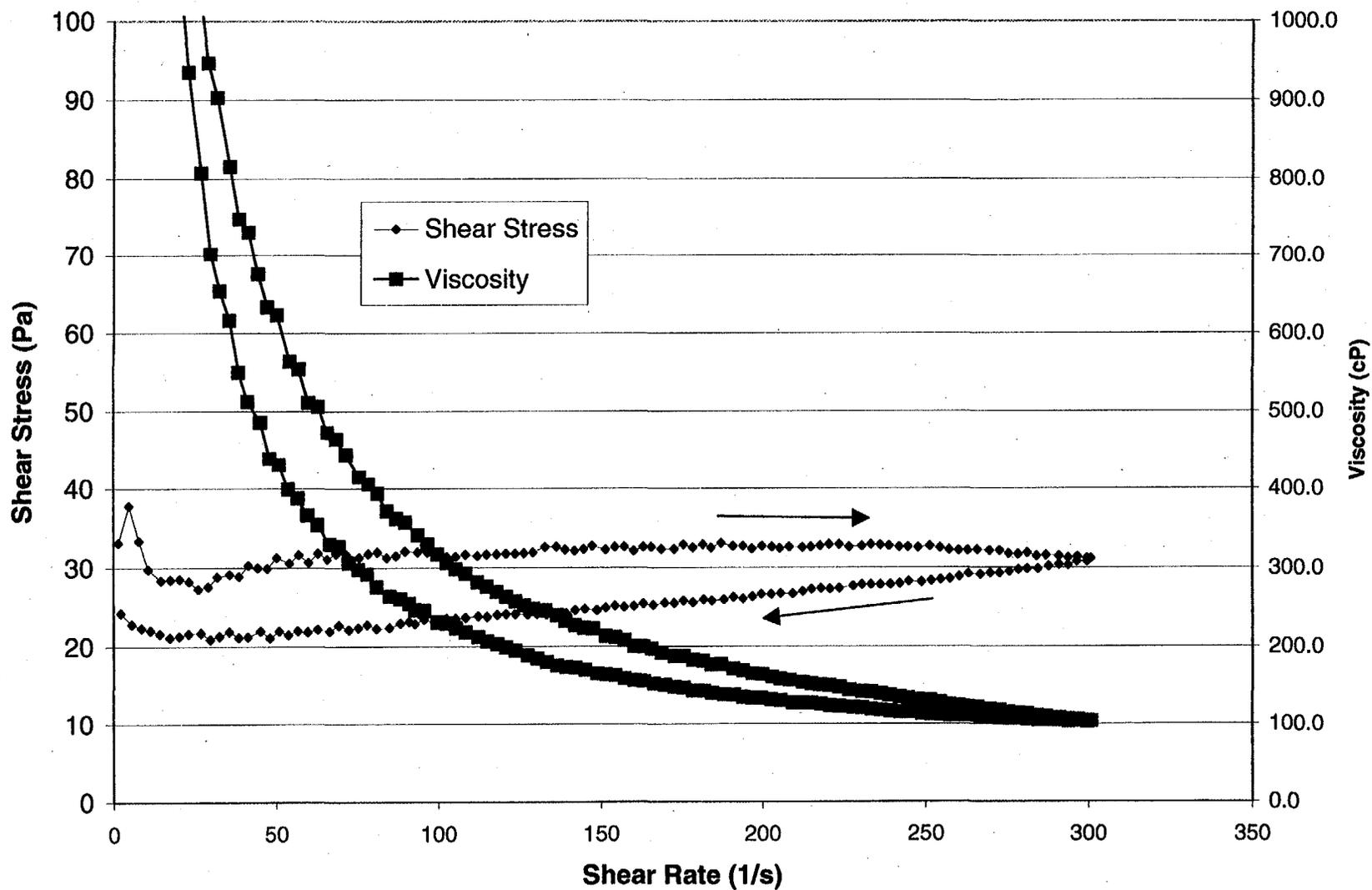


Figure 10. C-104 Reconstituted Slurry Sample 1, First Analysis

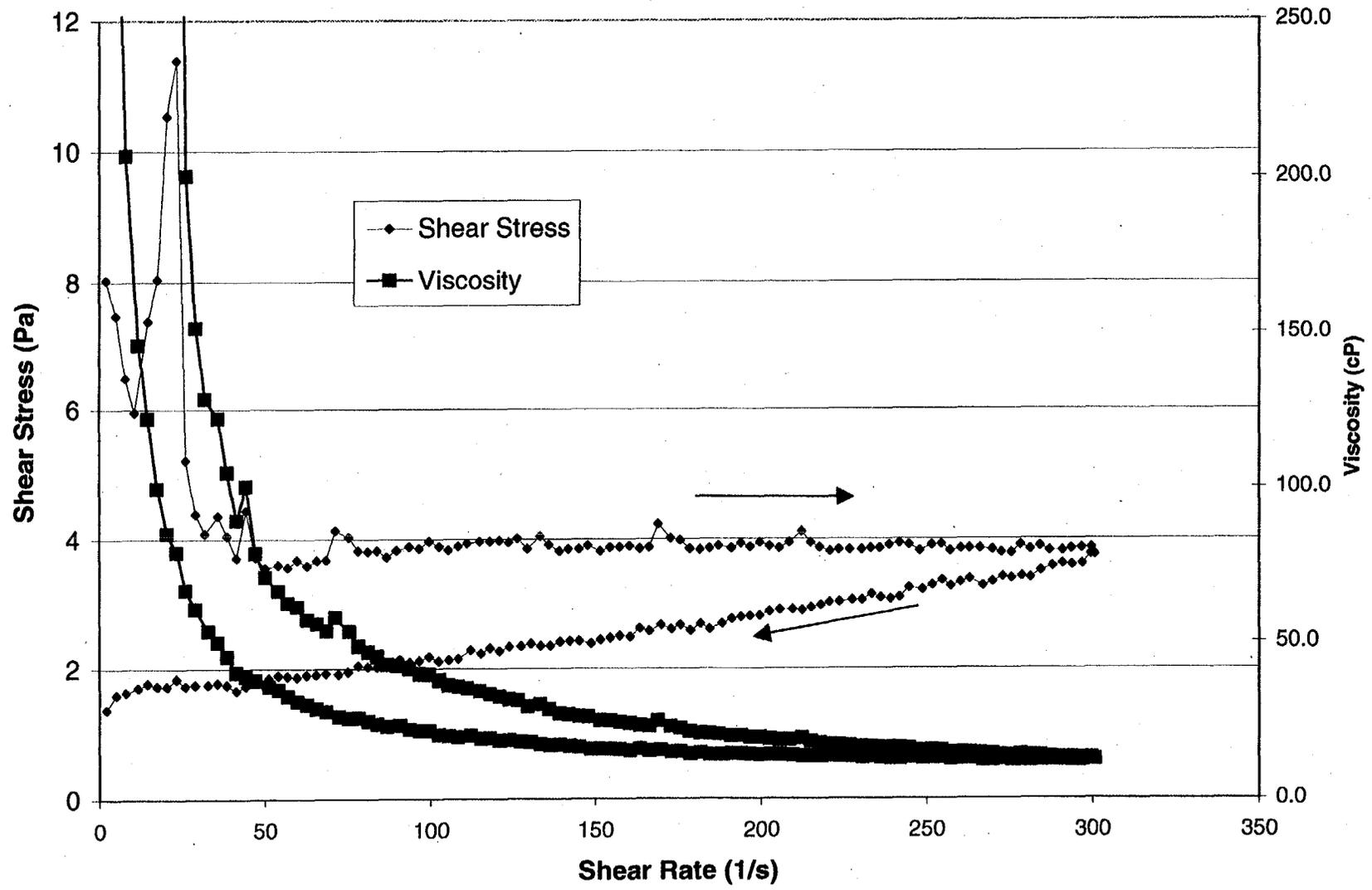


Figure 11. C-104 Reconstituted Slurry Sample 1, Second Analysis

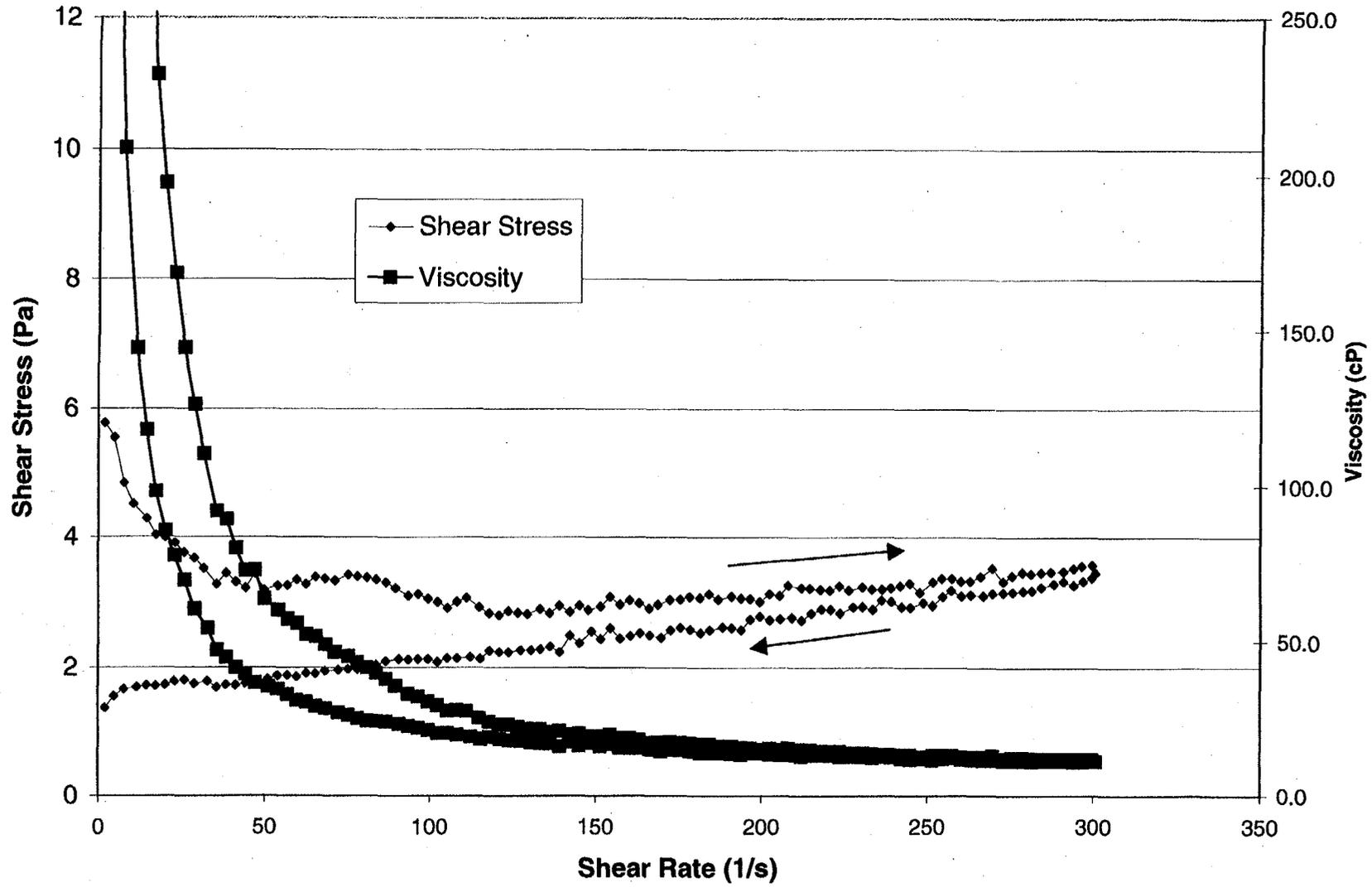


Figure 12. C-104 Reconstituted Slurry Sample 2, First Analysis

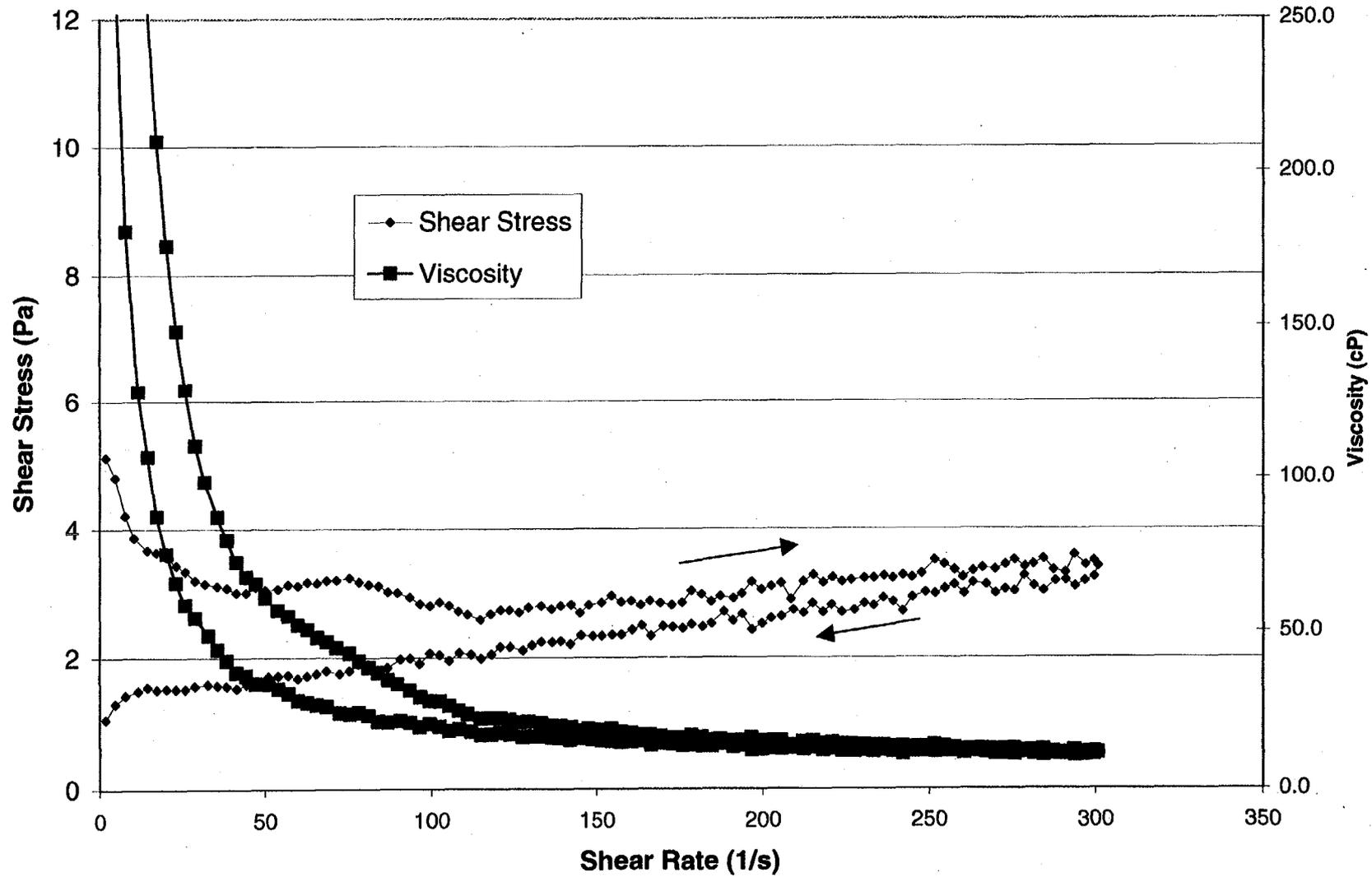
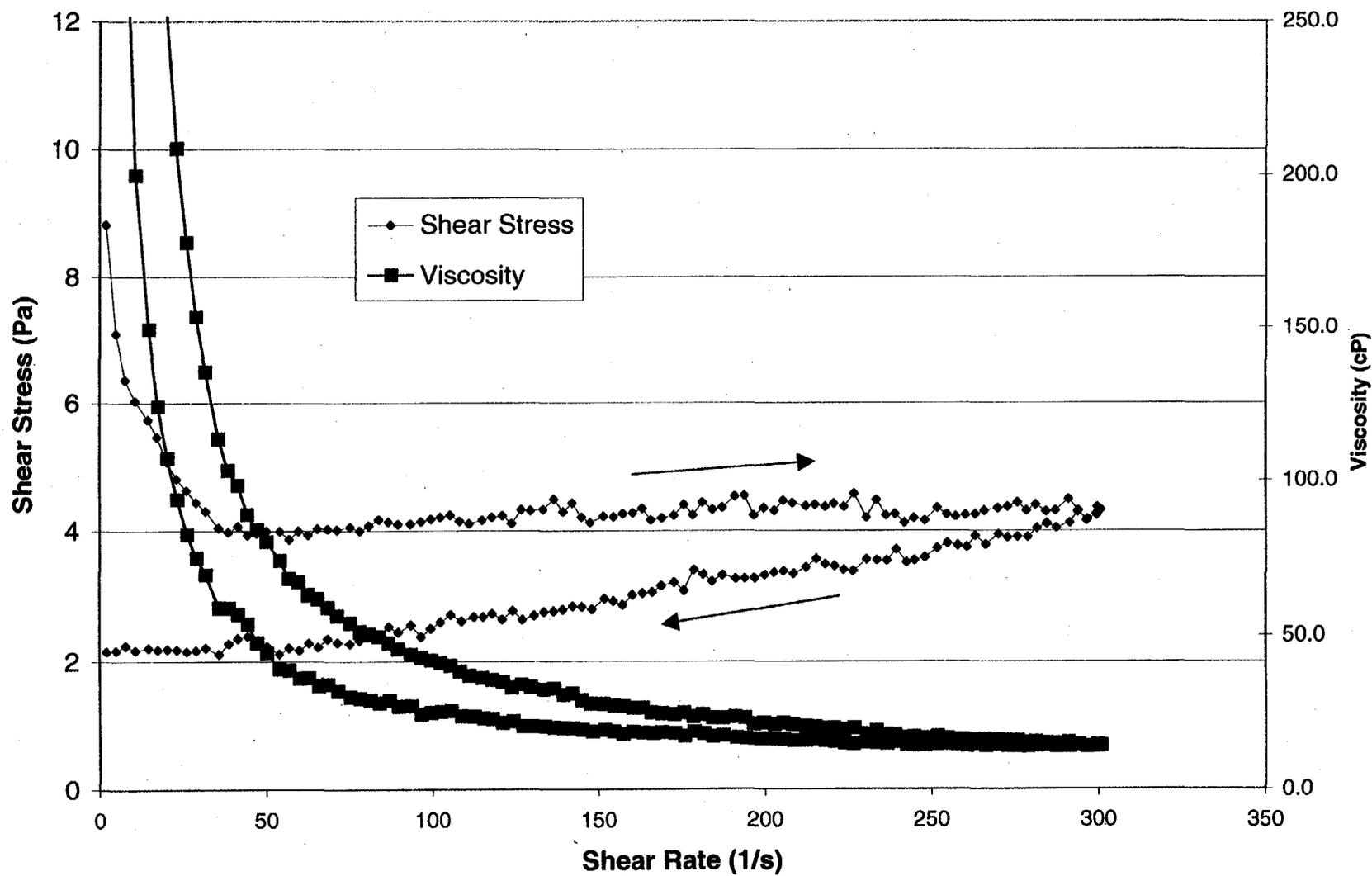
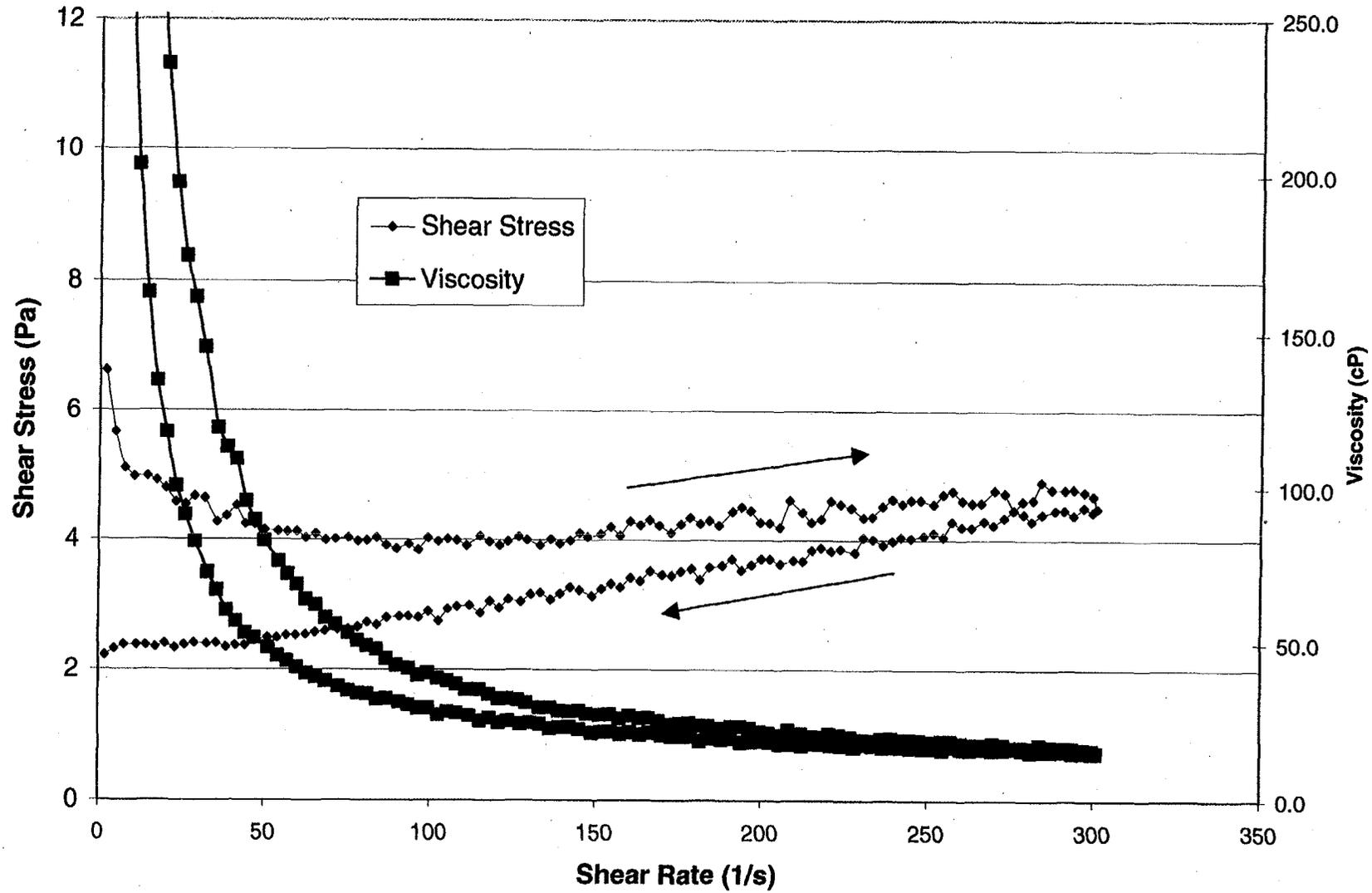


Figure 13. C-104 Reconstituted Slurry Sample 2, Second Analysis



Extra C-104 Diluted Intermediate Slurry 082699: Run 2 Duplicate



Extra C-104 Diluted Intermediate Slurry 082699: Run 2 Second Duplicate

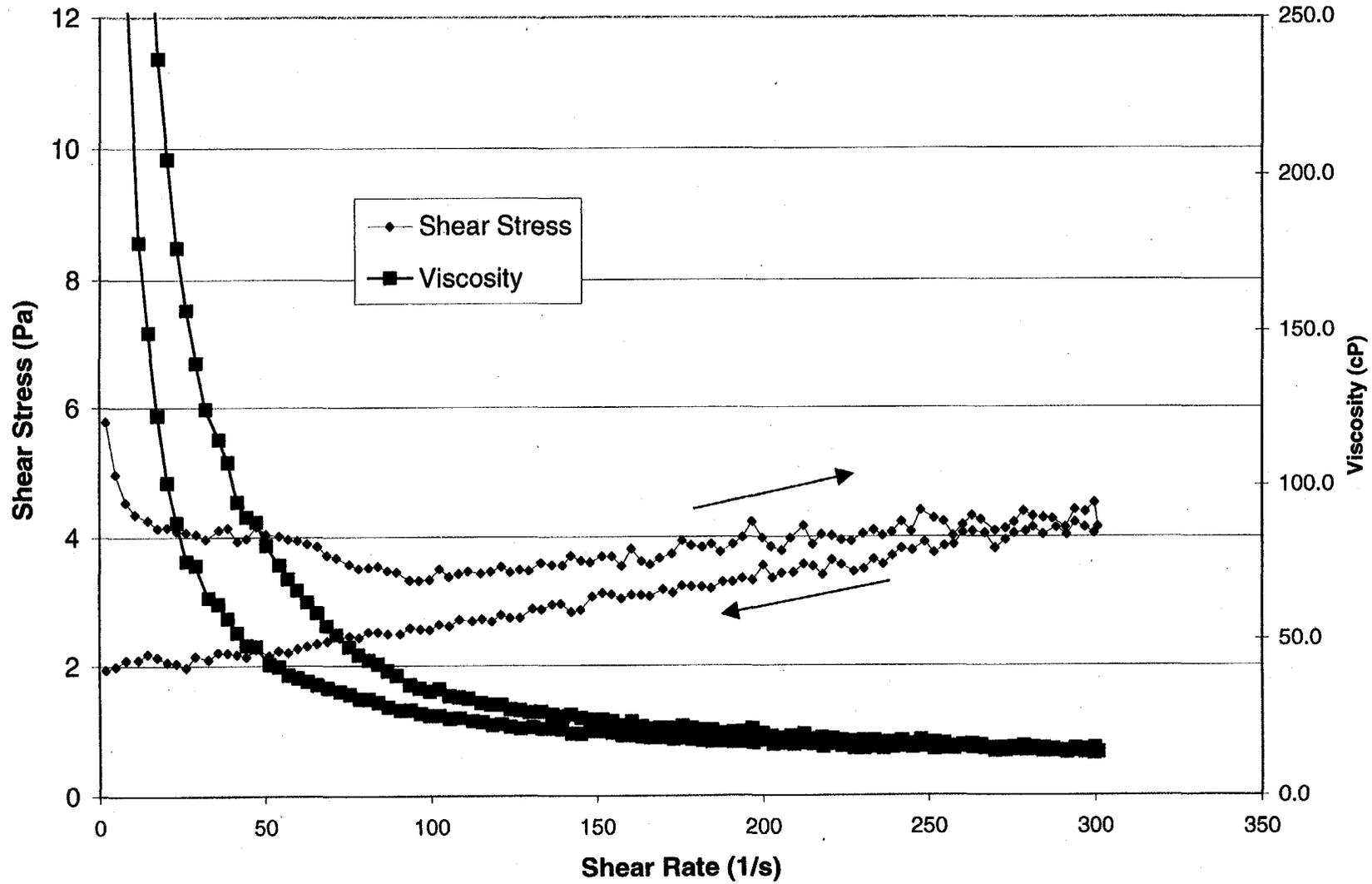


Figure 14. C-104 Final Slurry Sample 1, First Analysis

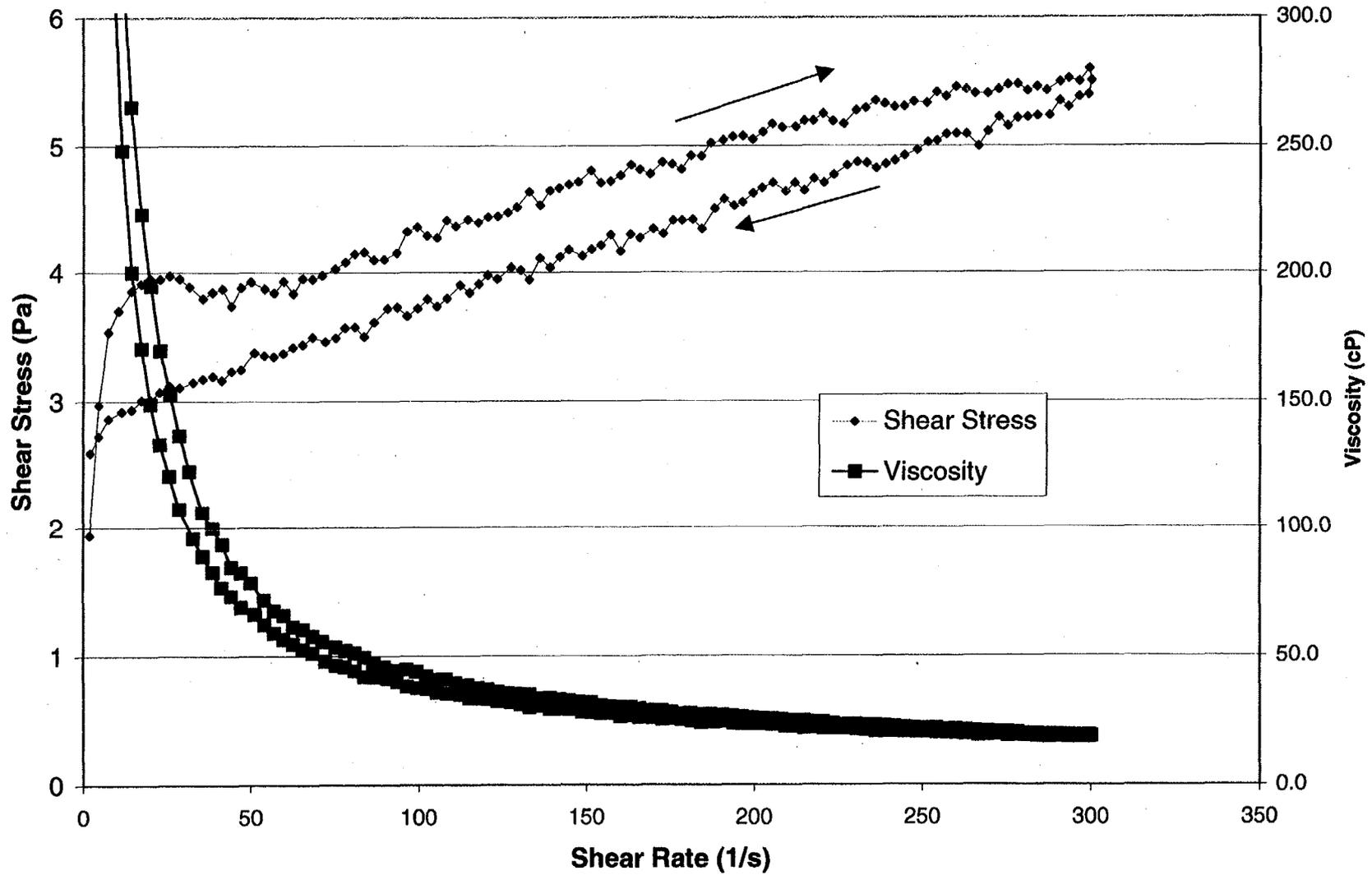


Figure 15. C-104 Final Slurry Sample 1, Second Analysis

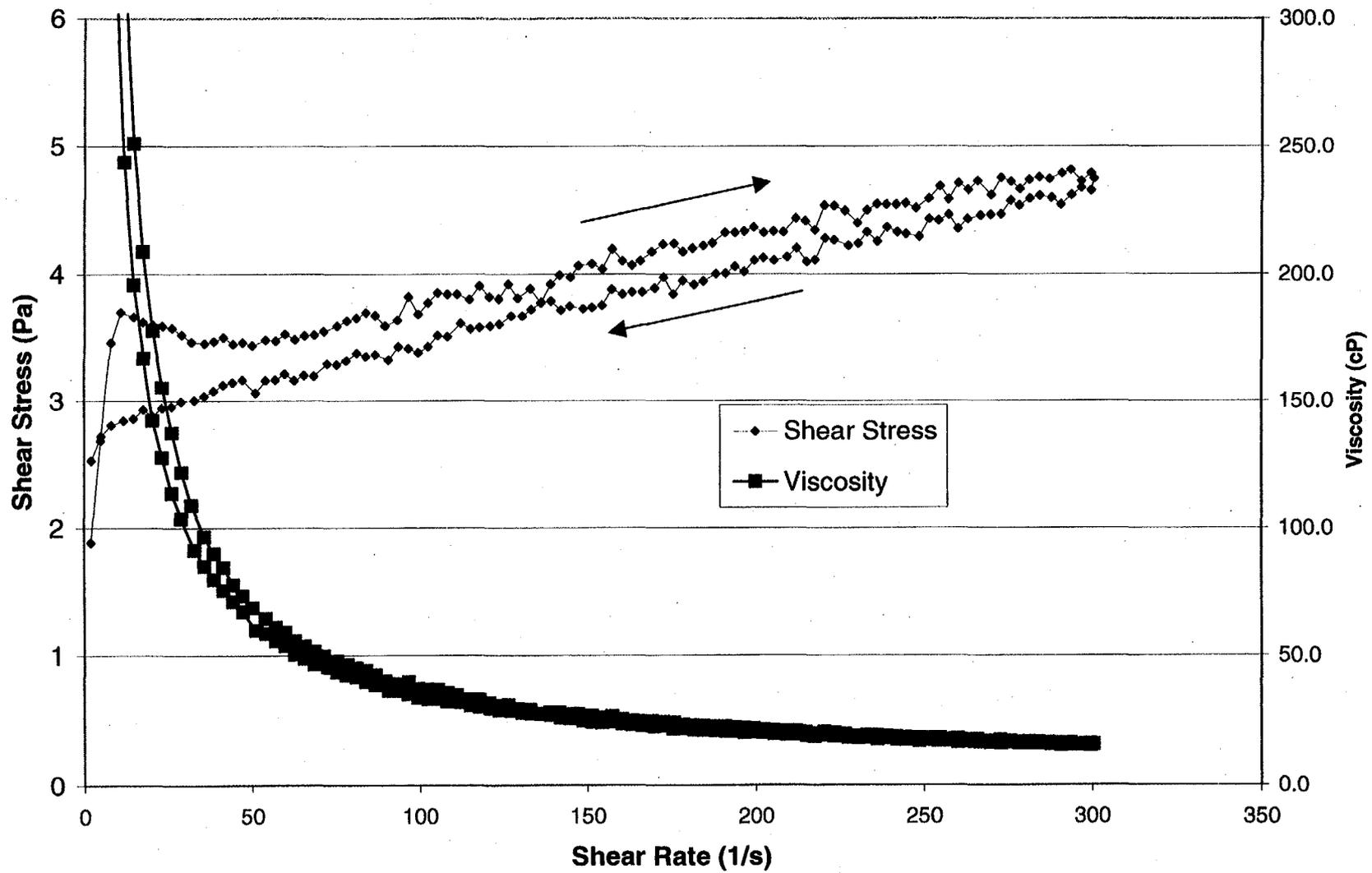
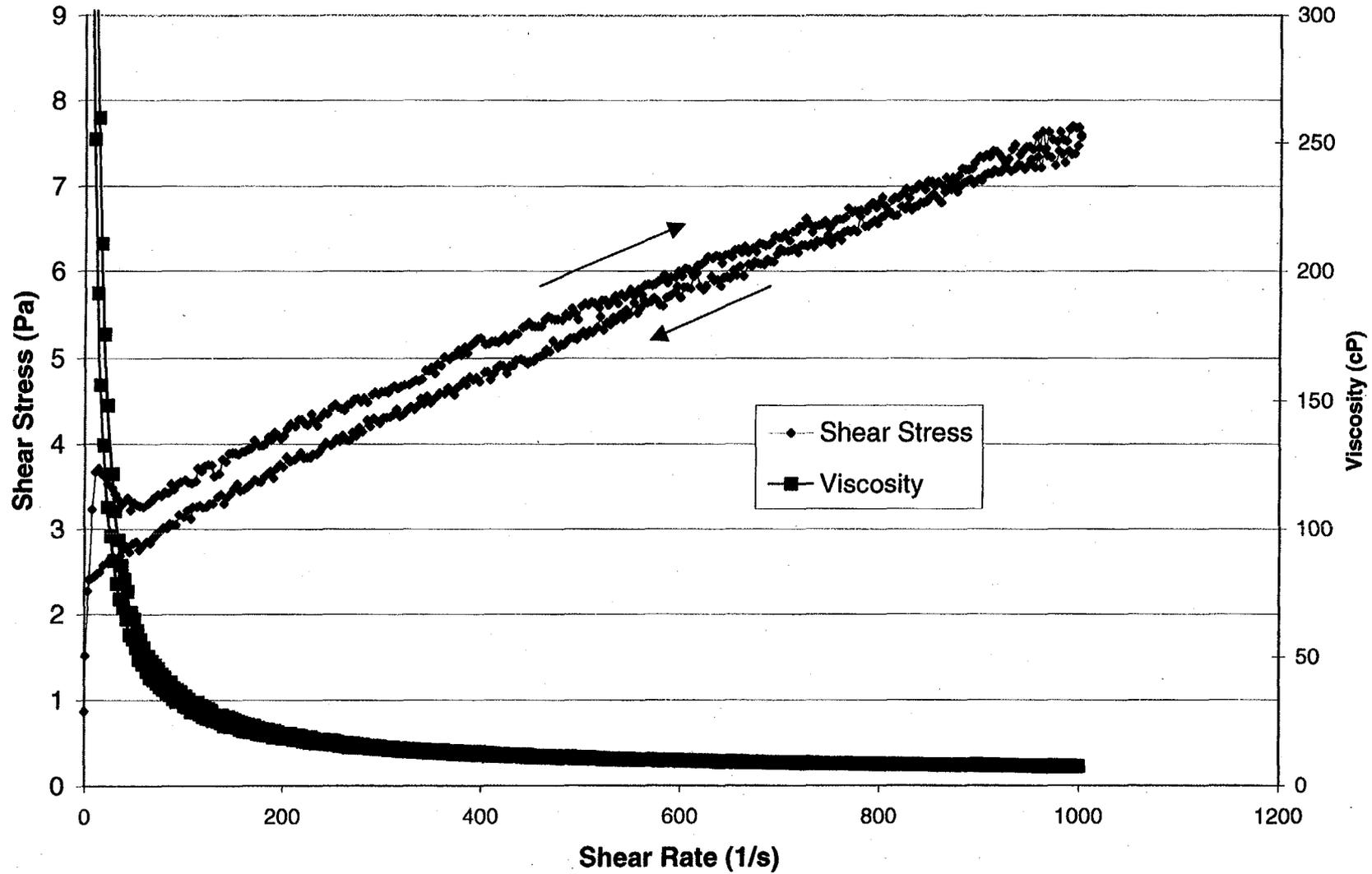


Figure 16. C-104 Final Slurry Sample 1, Third Analysis



Extra C-104 Final Slurry 092199: Sample 1 Run 4

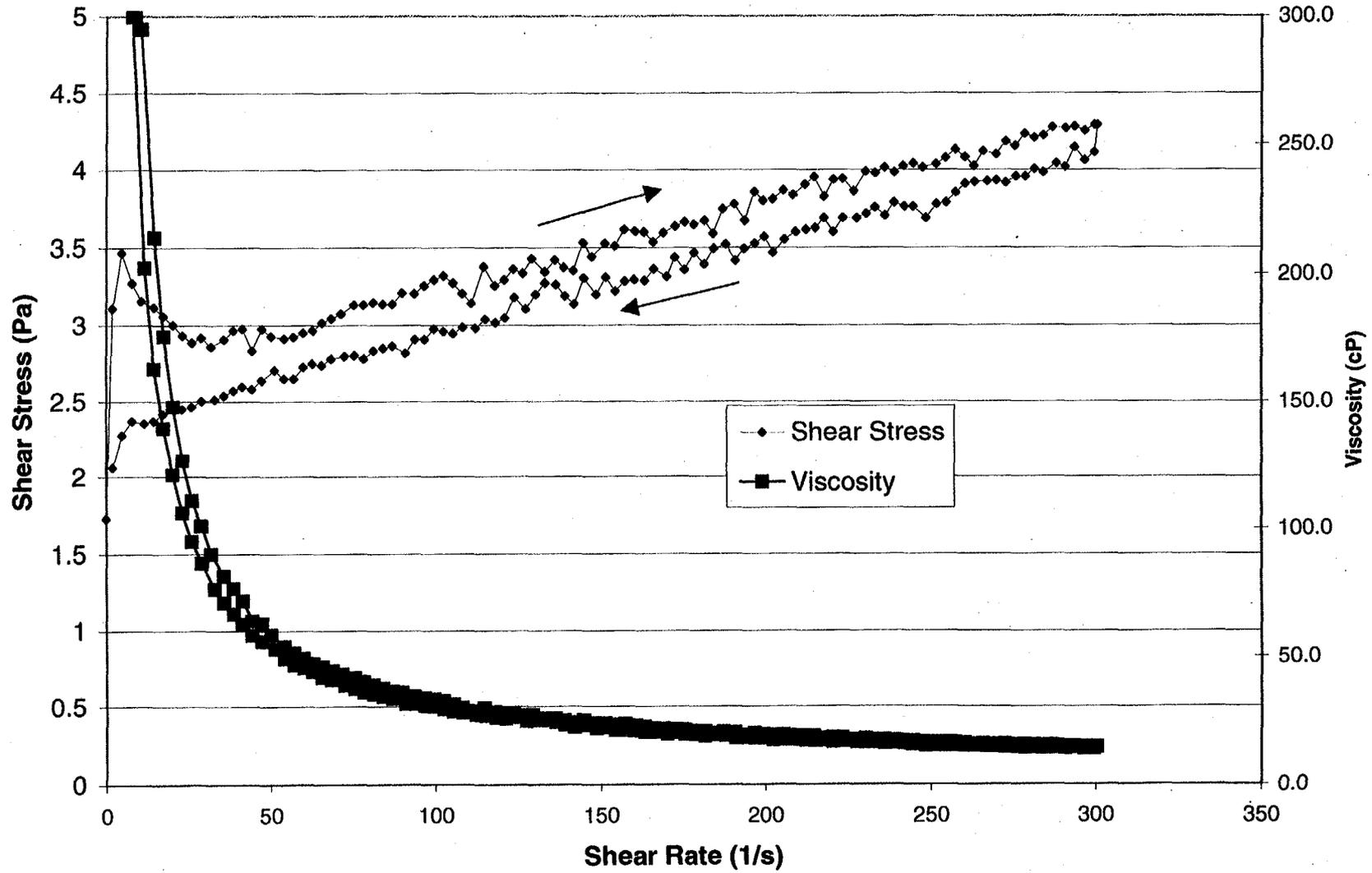


Figure 17. C-104 Final Slurry Sample 2, First Analysis

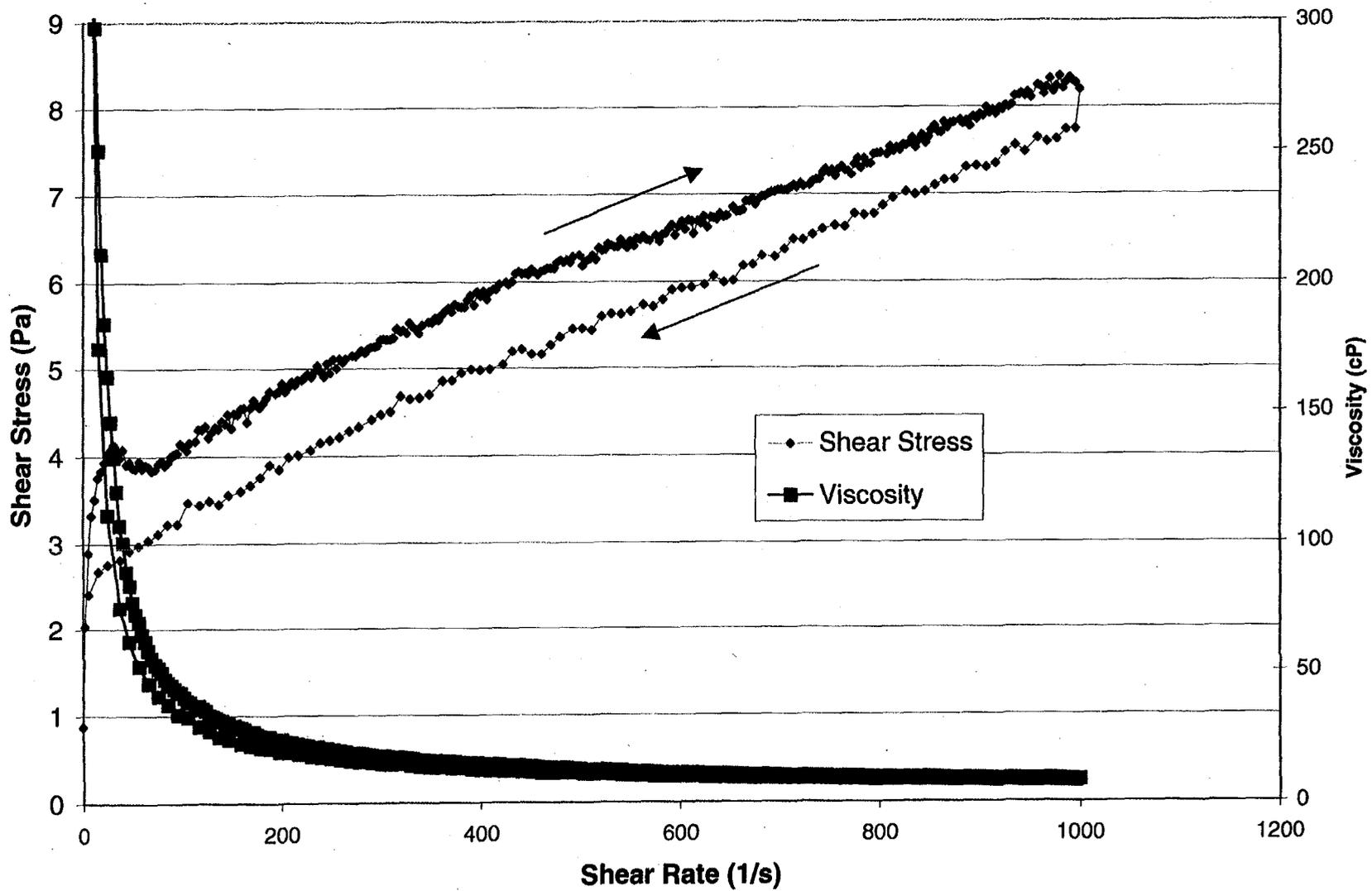
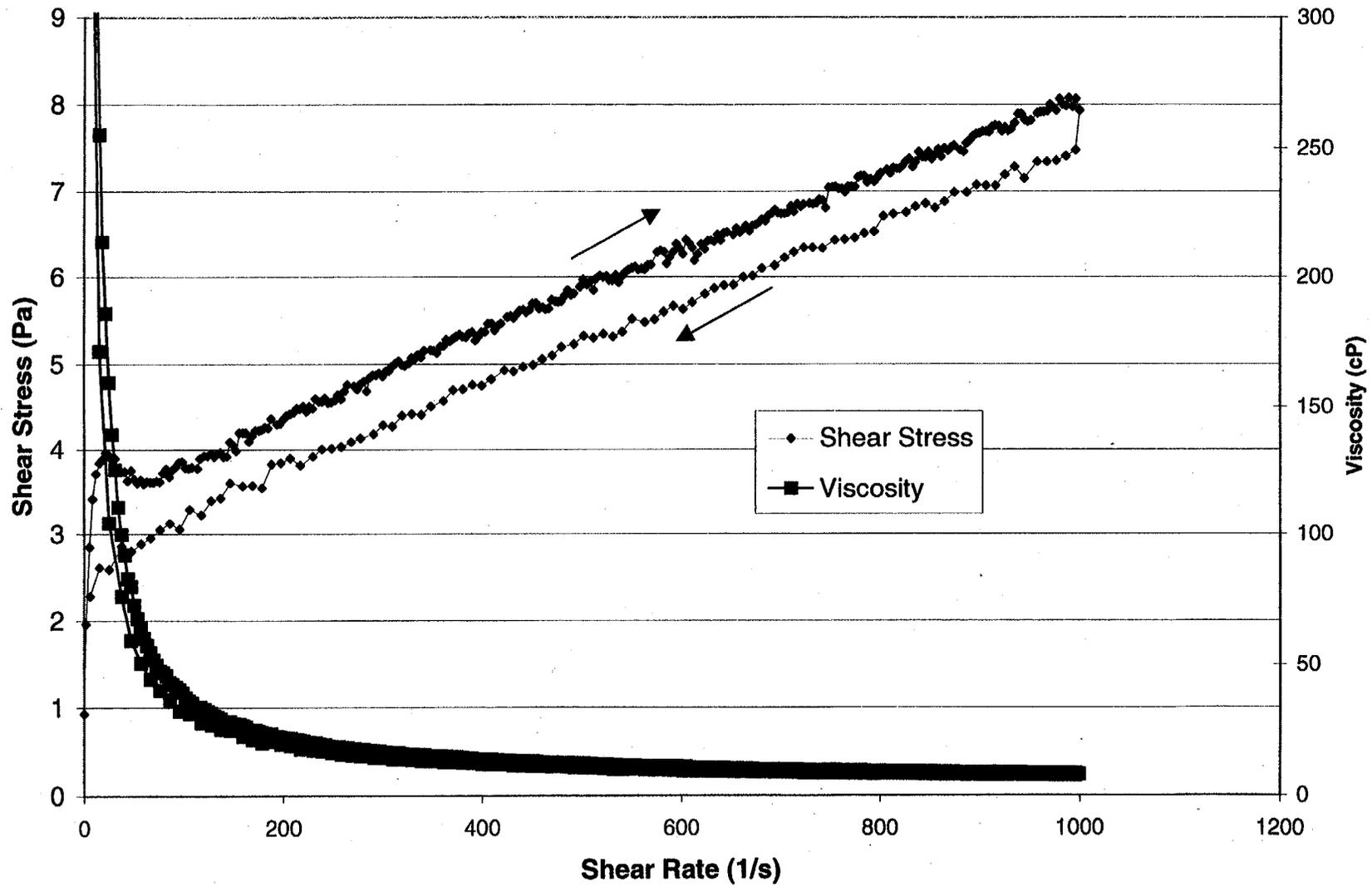
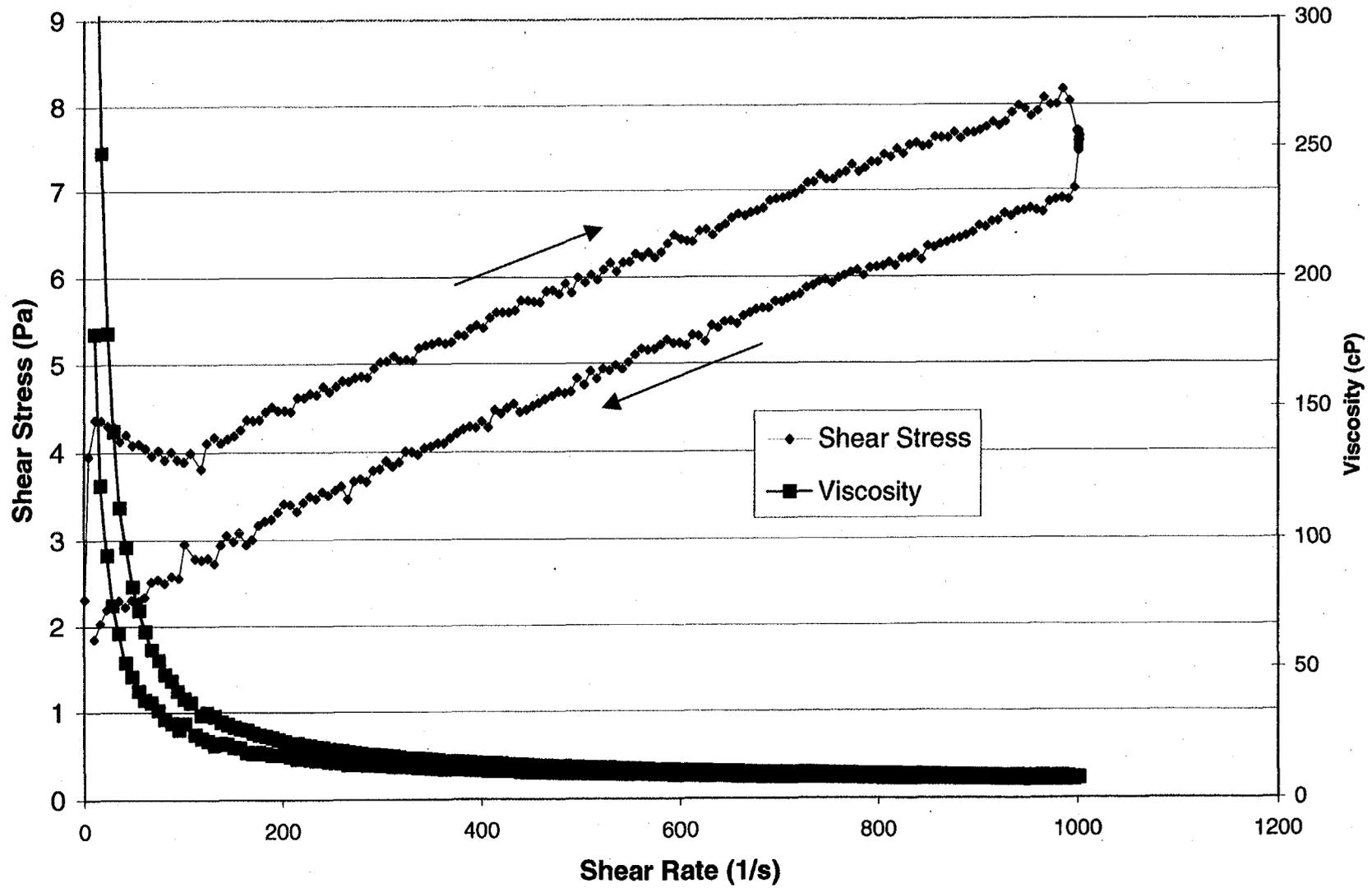


Figure 18. C-104 Final Slurry Sample 2, Second Analysis



Extra C-104 Final Slurry 092199: Sample 2 Run 3



**Appendix G: Key Personnel Affiliated with C-
104 Testing**

Appendix G: Key Personnel Affiliated with C-104 Testing

Name	Responsibility	Telephone/email
Eugene Morrey	Battelle Project Manager	(509) 376-1982 eugene.morrey@pnl.gov
Dean Kurath	Battelle Project Engineer	(509) 376-6752 dean.kurath@pnl.gov
Kriston Brooks	Ultrafiltration Task Manager, Filtration and CUF Testing	(509) 376-2233 kriston.brooks@pnl.gov
Paul Bredt	Rheology and Physical Properties Measurement	(509) 376-3777 paul.bredt@pnl.gov
Stacey Hartley	Statistical Analysis	(509) 372-4945 stacey.hartley@pnl.gov
Mike Urie	Chemical and Radiochemical Analysis	(509) 376-9454 mike.urie@pnl.gov
Ken Rappe	CUF Design and Testing	(509) 372-3918 ken.rappe@pnl.gov
Gita Golcar	Particle Size Distribution Measurement	(509) 372-1967 gita.golcar@pnl.gov
Lynette Jagoda	CUF Testing	(509) 376-9951 lynette.jagoda@pnl.gov
Rick Steele	Hot Cell Operations	(509) 372-0038 rick.steele@pnl.gov

**Appendix H: Particle Size Distribution
Experimental Raw Data**

**Appendix H: Particle Size Distribution Simulant Recipes and
Experimental Raw Data**

**Particle Size Distribution Plots
For NIST Traceable Standards from Duke Scientific Corporation**

Particle Size Analysis

Duke Standard 20um
1 mM KCl

Date: 02/04/00 Meas #: N/A
Time: 13:56 Pres #: N/A

Duke Standard - 20.00 um, lot# 19411
in 1 mM KCl solution

Volume Distribution
X-100

Summary

mv = 20.98
mn = 19.65
ma = 20.50
cs = 0.293
sd = 3.131

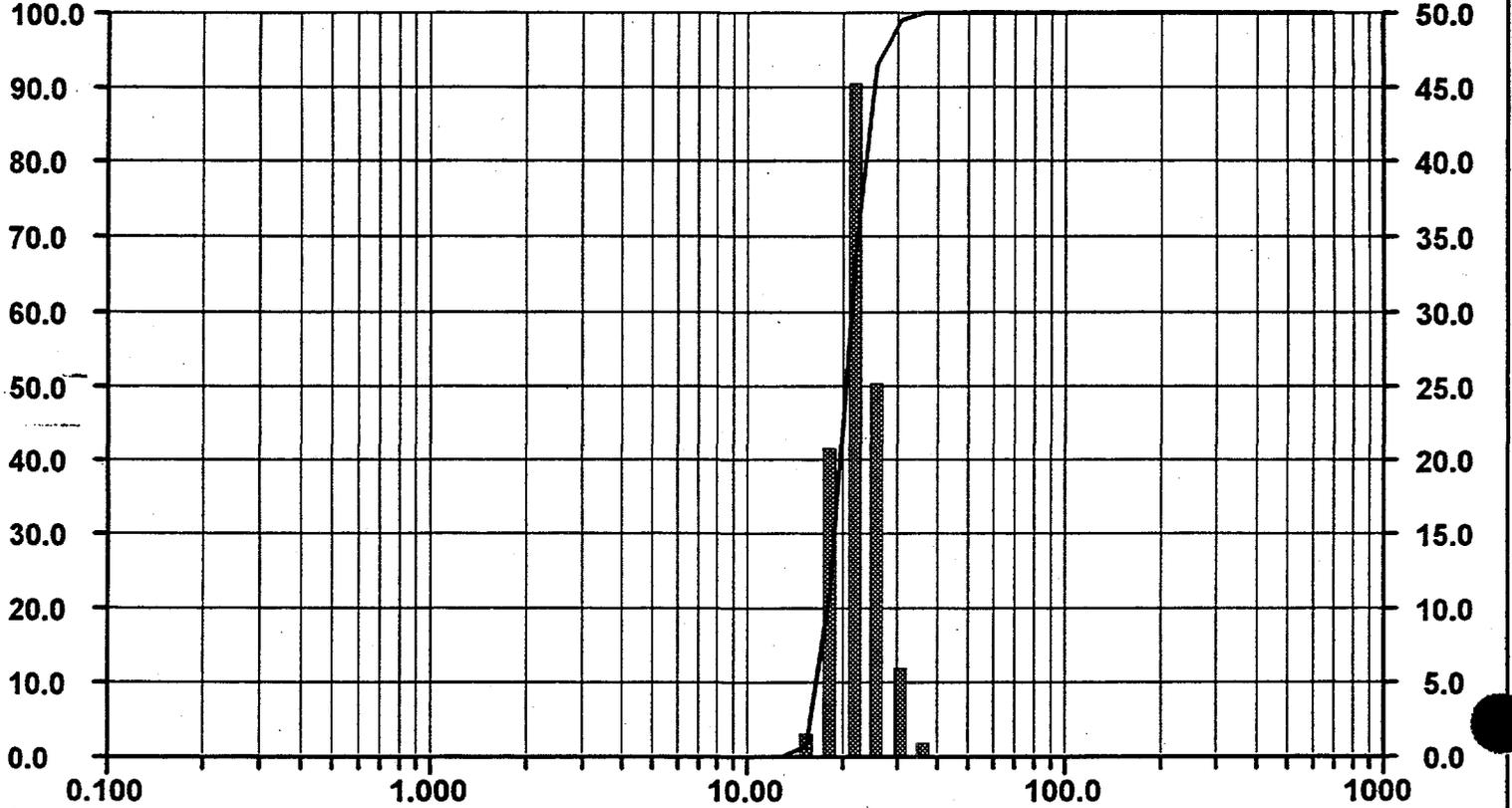
Percentiles

10% = 17.11 60% = 21.34
20% = 18.25 70% = 22.25
30% = 19.09 80% = 23.50
40% = 19.83 90% = 25.35
50% = 20.56 95% = 27.00

Dia Vol% Width

20.56 100% 6.261

%PASS



- Size (microns) -

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	0.00	0.00						
592.0	100.00	0.00	7.778	0.00	0.00						
497.8	100.00	0.00	6.541	0.00	0.00						
418.6	100.00	0.00	5.500	0.00	0.00						
352.0	100.00	0.00	4.625	0.00	0.00						
296.0	100.00	0.00	3.889	0.00	0.00						
248.9	100.00	0.00	3.270	0.00	0.00						
209.3	100.00	0.00	2.750	0.00	0.00						
176.0	100.00	0.00	2.312	0.00	0.00						
148.0	100.00	0.00	1.945	0.00	0.00						
124.5	100.00	0.00	1.635	0.00	0.00						
104.7	100.00	0.00	1.375	0.00	0.00						
88.00	100.00	0.00	1.156	0.00	0.00						
74.00	100.00	0.00	0.972	0.00	0.00						
62.23	100.00	0.00	0.818	0.00	0.00						
52.33	100.00	0.00	0.688	0.00	0.00						
44.00	100.00	0.00	0.578	0.00	0.00						
37.00	100.00	0.92	0.486	0.00	0.00						
31.11	99.08	6.01	0.409	0.00	0.00						
26.16	93.07	25.26	0.344	0.00	0.00						
22.00	67.81	45.38	0.289	0.00	0.00						
18.50	22.43	20.82	0.243	0.00	0.00						
15.56	1.61	1.61	0.204	0.00	0.00						
13.08	0.00	0.00	0.172	0.00	0.00						
11.00	0.00	0.00	0.145	0.00	0.00						

Particle Size Analysis

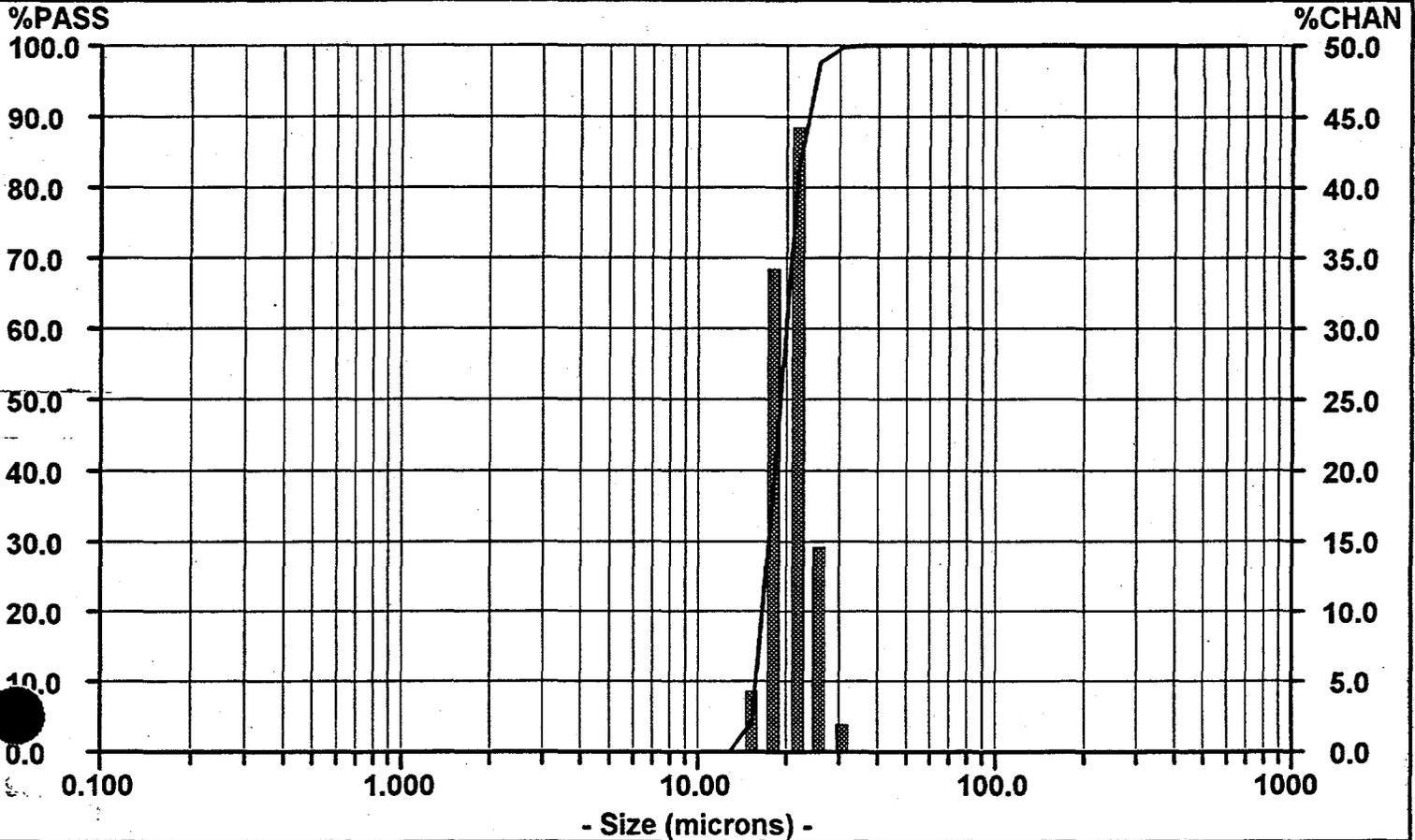
Duke Standard 20um
1 mM KCl

Date: 02/04/0 Meas #: N/A
Time: 13:56 Pres #: N/A

Duke Standard- 20.00 um, lot# 19411
in 1 mM KCl solution

Number Distribution
A-100

Summary	Percentiles		Dia	Vol%	Width
mv = 20.98	10% = 16.20	60% = 19.99	19.29	100%	5.403
mn = 19.65	20% = 17.09	70% = 20.77			
ma = 20.50	30% = 17.87	80% = 21.70			
cs = 0.293	40% = 18.59	90% = 23.20			
sd = 2.702	50% = 19.29	95% = 24.70			



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	0.00	0.00						
592.0	100.00	0.00	7.778	0.00	0.00						
497.8	100.00	0.00	6.541	0.00	0.00						
418.6	100.00	0.00	5.500	0.00	0.00						
352.0	100.00	0.00	4.625	0.00	0.00						
296.0	100.00	0.00	3.889	0.00	0.00						
248.9	100.00	0.00	3.270	0.00	0.00						
209.3	100.00	0.00	2.750	0.00	0.00						
176.0	100.00	0.00	2.312	0.00	0.00						
148.0	100.00	0.00	1.945	0.00	0.00						
124.5	100.00	0.00	1.635	0.00	0.00						
104.7	100.00	0.00	1.375	0.00	0.00						
88.00	100.00	0.00	1.156	0.00	0.00						
74.00	100.00	0.00	0.972	0.00	0.00						
62.23	100.00	0.00	0.818	0.00	0.00						
52.33	100.00	0.00	0.688	0.00	0.00						
44.00	100.00	0.00	0.578	0.00	0.00						
37.00	100.00	0.19	0.486	0.00	0.00						
31.11	99.81	2.08	0.409	0.00	0.00						
26.16	97.73	14.68	0.344	0.00	0.00						
22.00	83.05	44.36	0.289	0.00	0.00						
18.50	38.69	34.24	0.243	0.00	0.00						
15.00	4.45	4.45	0.204	0.00	0.00						
12.00	0.00	0.00	0.172	0.00	0.00						
10.00	0.00	0.00	0.145	0.00	0.00						

Particle Size Analysis

Duke Standard
1 mM KCl

Date: ~~02/04/0~~ ^{02/07/00} Meas #: N/A
Time: 13:34 Pres #: N/A

Duke Standard- 50.4 um, lot# 19213
in 1 mM KCl solution

Summary

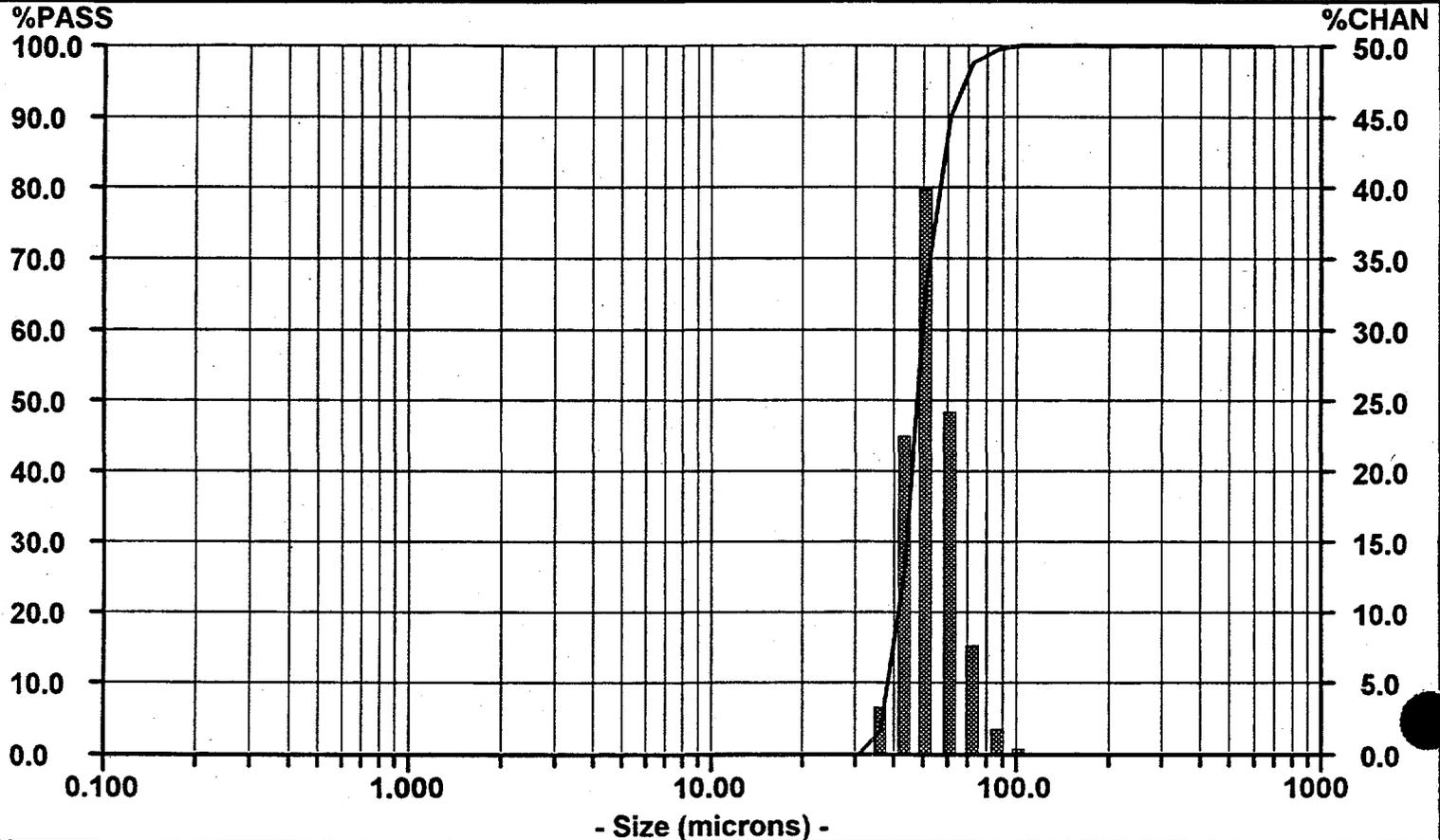
mv = 50.24
mn = 46.05
ma = 48.69
cs = 0.123
sd = 8.623

Percentiles

10% = 39.69 60% = 50.97
20% = 42.55 70% = 53.53
30% = 44.83 80% = 56.91
40% = 46.84 90% = 62.15
50% = 48.83 95% = 67.49

Dia	Vol%	Width
48.83	100%	17.25

Volume Distribution
X-100



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	0.00	0.00						
692.0	100.00	0.00	7.778	0.00	0.00						
497.8	100.00	0.00	6.541	0.00	0.00						
418.6	100.00	0.00	5.500	0.00	0.00						
352.0	100.00	0.00	4.625	0.00	0.00						
296.0	100.00	0.00	3.889	0.00	0.00						
248.9	100.00	0.00	3.270	0.00	0.00						
209.3	100.00	0.00	2.750	0.00	0.00						
176.0	100.00	0.00	2.312	0.00	0.00						
148.0	100.00	0.00	1.945	0.00	0.00						
124.5	100.00	0.00	1.635	0.00	0.00						
104.7	100.00	0.44	1.375	0.00	0.00						
88.00	99.56	1.80	1.156	0.00	0.00						
74.00	97.76	7.63	0.972	0.00	0.00						
62.23	90.13	24.23	0.818	0.00	0.00						
52.33	65.90	39.93	0.688	0.00	0.00						
44.00	25.97	22.66	0.578	0.00	0.00						
37.00	3.31	3.31	0.486	0.00	0.00						
31.11	0.00	0.00	0.409	0.00	0.00						
26.16	0.00	0.00	0.344	0.00	0.00						
22.00	0.00	0.00	0.289	0.00	0.00						
18.50	0.00	0.00	0.243	0.00	0.00						
15.56	0.00	0.00	0.204	0.00	0.00						
13.08	0.00	0.00	0.172	0.00	0.00						
11.00	0.00	0.00	0.145	0.00	0.00						

Particle Size Analysis

Duke Standard
1 mM KCl

Date: 02/04/0 Meas #: N/A
Time: 13:34 Pres #: N/A

Duke Standard- 50.4 um, lot# 19213
in 1 mM KCl solution

Summary

mv = 50.24
mn = 46.05
ma = 48.69
cs = 0.123
sd = 6.993

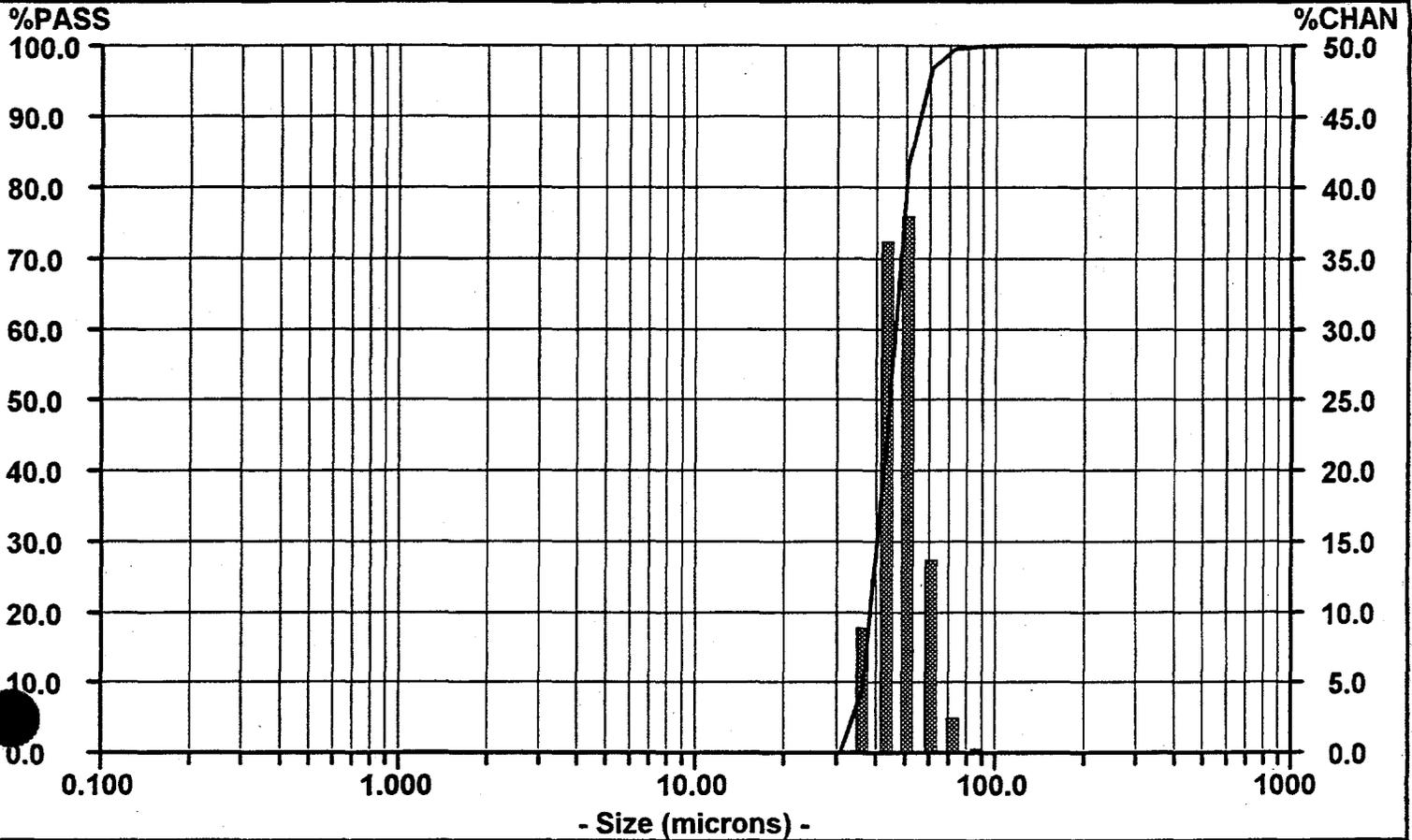
Percentiles

10% = 37.25 60% = 46.78
20% = 39.43 70% = 48.87
30% = 41.29 80% = 51.40
40% = 43.07 90% = 55.49
50% = 44.88 95% = 59.46

Dia Vol% Width

44.88 100% 13.99

Number Distribution
X-100



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	0.00	0.00						
592.0	100.00	0.00	7.778	0.00	0.00						
497.8	100.00	0.00	6.541	0.00	0.00						
418.6	100.00	0.00	5.500	0.00	0.00						
352.0	100.00	0.00	4.625	0.00	0.00						
296.0	100.00	0.00	3.889	0.00	0.00						
248.9	100.00	0.00	3.270	0.00	0.00						
209.3	100.00	0.00	2.750	0.00	0.00						
176.0	100.00	0.00	2.312	0.00	0.00						
148.0	100.00	0.00	1.945	0.00	0.00						
124.5	100.00	0.00	1.635	0.00	0.00						
104.7	100.00	0.05	1.375	0.00	0.00						
88.00	99.95	0.36	1.156	0.00	0.00						
74.00	99.59	2.57	0.972	0.00	0.00						
62.23	97.02	13.73	0.818	0.00	0.00						
52.33	83.29	38.06	0.688	0.00	0.00						
44.00	45.23	36.31	0.578	0.00	0.00						
37.00	8.92	8.92	0.486	0.00	0.00						
31.11	0.00	0.00	0.409	0.00	0.00						
26.16	0.00	0.00	0.344	0.00	0.00						
22.00	0.00	0.00	0.289	0.00	0.00						
18.50	0.00	0.00	0.243	0.00	0.00						
15.56	0.00	0.00	0.204	0.00	0.00						
13.00	0.00	0.00	0.172	0.00	0.00						
11.00	0.00	0.00	0.145	0.00	0.00						

Particle Size Analysis

Duke Standard 301um
1 mM KCl

Date: 02/04/0 Meas #: N/A
Time: 14:40 Pres #: N/A

Duke Standard- 301 um, lot# 19136
in 1 mM KCl solution

Volume Distribution
X-100

Summary

mv = 294.9
mn = 278.1
ma = 289.0
cs = 0.021
sd = 41.39

Percentiles

10% = 245.1 60% = 301.2
20% = 258.9 70% = 314.2
30% = 269.5 80% = 329.8
40% = 279.5 90% = 350.6
50% = 289.9 95% = 371.5

Dia Vol% Width

289.9 100% 82.79

%PASS

100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0

%CHAN

50.0
45.0
40.0
35.0
30.0
25.0
20.0
15.0
10.0
5.0
0.0

0.100 1.000 10.00 100.0 1000

- Size (microns) -

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	0.00	0.00						
592.0	100.00	0.00	7.778	0.00	0.00						
497.8	100.00	0.95	6.541	0.00	0.00						
418.6	99.05	8.44	5.500	0.00	0.00						
352.0	90.61	34.84	4.625	0.00	0.00						
296.0	55.77	43.91	3.889	0.00	0.00						
248.9	11.86	11.29	3.270	0.00	0.00						
209.3	0.57	0.57	2.750	0.00	0.00						
176.0	0.00	0.00	2.312	0.00	0.00						
148.0	0.00	0.00	1.945	0.00	0.00						
124.5	0.00	0.00	1.635	0.00	0.00						
104.7	0.00	0.00	1.375	0.00	0.00						
88.00	0.00	0.00	1.156	0.00	0.00						
74.00	0.00	0.00	0.972	0.00	0.00						
62.23	0.00	0.00	0.818	0.00	0.00						
52.33	0.00	0.00	0.688	0.00	0.00						
44.00	0.00	0.00	0.578	0.00	0.00						
37.00	0.00	0.00	0.486	0.00	0.00						
31.11	0.00	0.00	0.409	0.00	0.00						
26.16	0.00	0.00	0.344	0.00	0.00						
22.00	0.00	0.00	0.289	0.00	0.00						
18.50	0.00	0.00	0.243	0.00	0.00						
15.56	0.00	0.00	0.204	0.00	0.00						
13.08	0.00	0.00	0.172	0.00	0.00						
11.00	0.00	0.00	0.145	0.00	0.00						

Particle Size Analysis

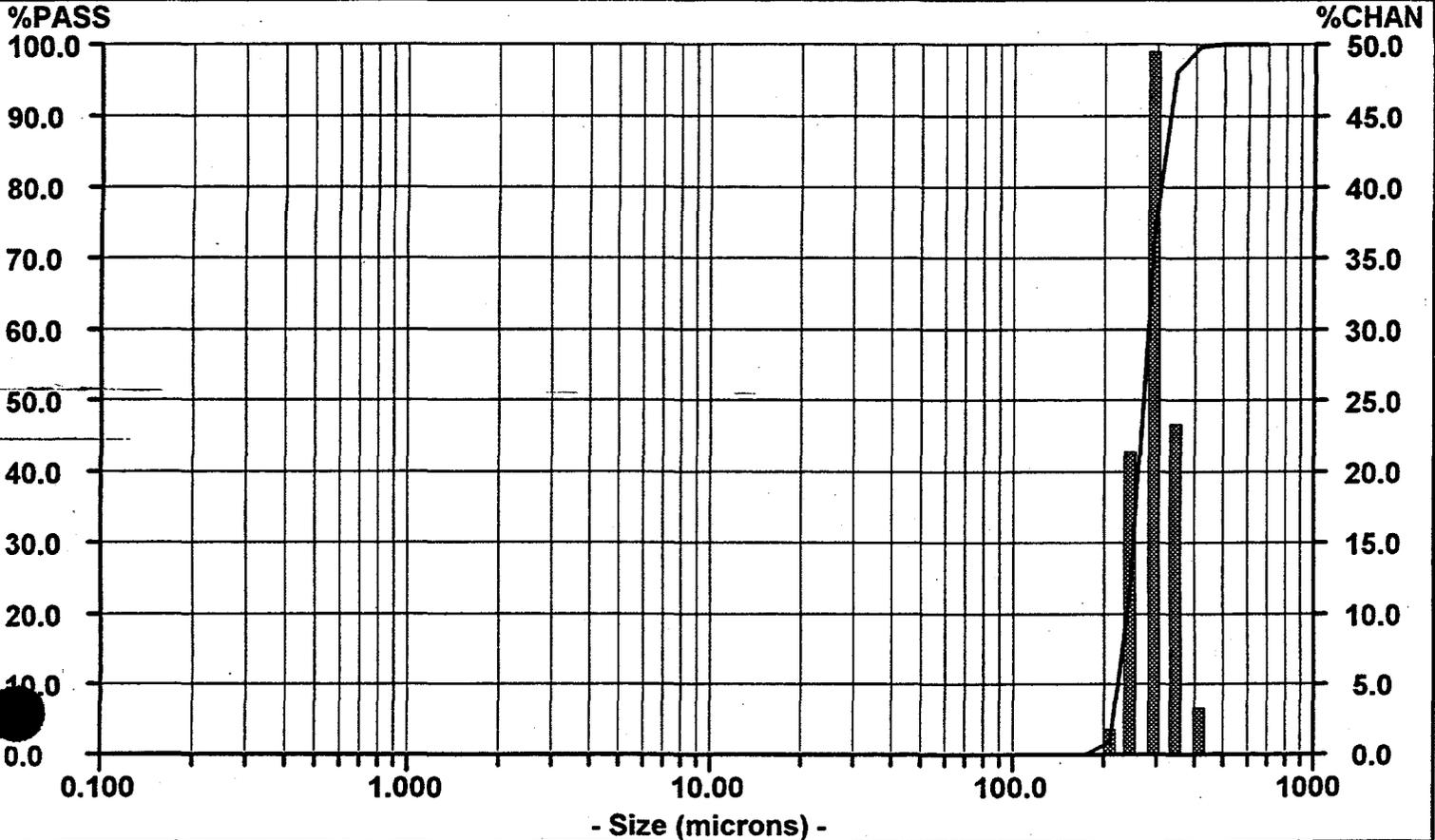
Duke Standard 301um
1 mM KCl

Date: 02/04/0 Meas #: N/A
Time: 14:40 Pres #: N/A

Duke Standard - 301 um, lot# 19136
in 1 mM KCl solution

Number Distribution
-100

Summary	Percentiles		Dia	Vol%	Width
mv = 294.9	10% = 229.7	60% = 282.3	273.2	100%	74.89
mn = 278.1	20% = 244.6	70% = 292.8			
ma = 289.0	30% = 255.3	80% = 306.8			
cs = 0.021	40% = 264.4	90% = 328.5			
sd = 37.45	50% = 273.2	95% = 346.0			



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	0.00	0.00						
592.0	100.00	0.00	7.778	0.00	0.00						
497.8	100.00	0.23	6.541	0.00	0.00						
418.6	99.77	3.37	5.500	0.00	0.00						
352.0	96.40	23.42	4.625	0.00	0.00						
296.0	72.98	49.68	3.889	0.00	0.00						
248.9	23.30	21.48	3.270	0.00	0.00						
209.3	1.82	1.82	2.750	0.00	0.00						
176.0	0.00	0.00	2.312	0.00	0.00						
148.0	0.00	0.00	1.945	0.00	0.00						
124.5	0.00	0.00	1.635	0.00	0.00						
104.7	0.00	0.00	1.375	0.00	0.00						
88.00	0.00	0.00	1.166	0.00	0.00						
74.00	0.00	0.00	0.972	0.00	0.00						
62.23	0.00	0.00	0.818	0.00	0.00						
52.33	0.00	0.00	0.688	0.00	0.00						
44.00	0.00	0.00	0.578	0.00	0.00						
37.00	0.00	0.00	0.486	0.00	0.00						
31.11	0.00	0.00	0.409	0.00	0.00						
26.16	0.00	0.00	0.344	0.00	0.00						
22.00	0.00	0.00	0.289	0.00	0.00						
18.50	0.00	0.00	0.243	0.00	0.00						
16.00	0.00	0.00	0.204	0.00	0.00						
14.00	0.00	0.00	0.172	0.00	0.00						
11.00	0.00	0.00	0.145	0.00	0.00						

Particle Size Distribution Plots
For Initial Slurry: Sample CUF-C104-001
Replicate No: 1

Particle Size Analysis

C104 Slurry
Sample CUP-C104-001

Date: 02/08/99 Meas #: 00032
Time: 11:12 Pres #: 01

C104 Slurry; Sample CUP-C104-001
in #1 Supernatant Simulant
40 ml/sec

Summary

mv = 10.60
mn = 0.393
ma = 1.789
cs = 3.354
sd = 11.09

Percentiles

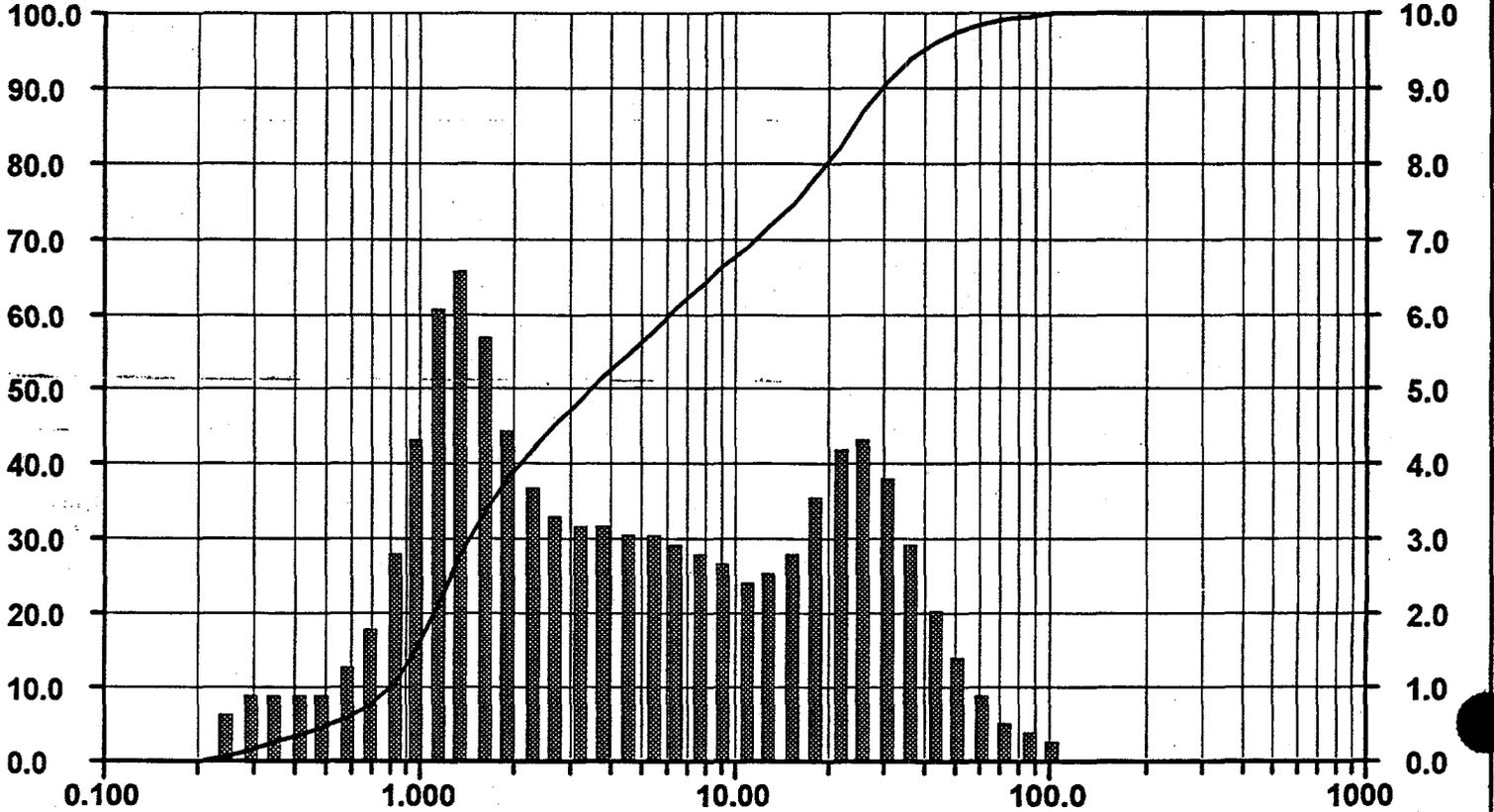
10% = 0.787 60% = 6.162
20% = 1.114 70% = 11.63
30% = 1.455 80% = 19.77
40% = 2.106 90% = 29.70
50% = 3.534 95% = 39.83

Dia Vol% Width

22.58 33% 25.58
1.613 67% 4.098

Volume Distribution

%PASS



- Size (microns) -

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	66.64	2.70						
592.0	100.00	0.00	7.778	63.94	2.90						
497.8	100.00	0.00	6.541	61.04	3.04						
418.6	100.00	0.00	5.500	58.00	3.10						
352.0	100.00	0.00	4.625	54.90	3.14						
296.0	100.00	0.00	3.889	51.76	3.20						
248.9	100.00	0.00	3.270	48.56	3.26						
209.3	100.00	0.00	2.750	45.30	3.36						
176.0	100.00	0.00	2.312	41.94	3.70						
148.0	100.00	0.00	1.945	38.24	4.50						
124.5	100.00	0.00	1.635	33.74	5.73						
104.7	100.00	0.34	1.375	28.01	6.61						
88.00	99.66	0.46	1.156	21.40	6.15						
74.00	99.20	0.65	0.972	15.25	4.49						
62.23	98.55	0.96	0.818	10.76	2.88						
52.33	97.59	1.45	0.688	7.88	1.86						
44.00	96.14	2.14	0.578	6.02	1.33						
37.00	94.00	3.03	0.486	4.69	1.08						
31.11	90.97	3.91	0.409	3.61	0.97						
26.16	87.06	4.40	0.344	2.64	0.95						
22.00	82.66	4.23	0.289	1.69	0.93						
18.50	78.43	3.63	0.243	0.76	0.76						
15.56	74.80	2.99	0.204	0.00	0.00						
13.08	71.81	2.62	0.172	0.00	0.00						
11.00	69.19	2.55	0.145	0.00	0.00						

Particle Size Analysis

C104 Slurry
Sample CUP-C104-001

Date: 02/08/89 Meas #: 00032
Time: 11:12 Pres #: 01

C104 Slurry; Sample CUP-C104-001
in #1 Supernatant Simulant
40 ml/sec

Summary

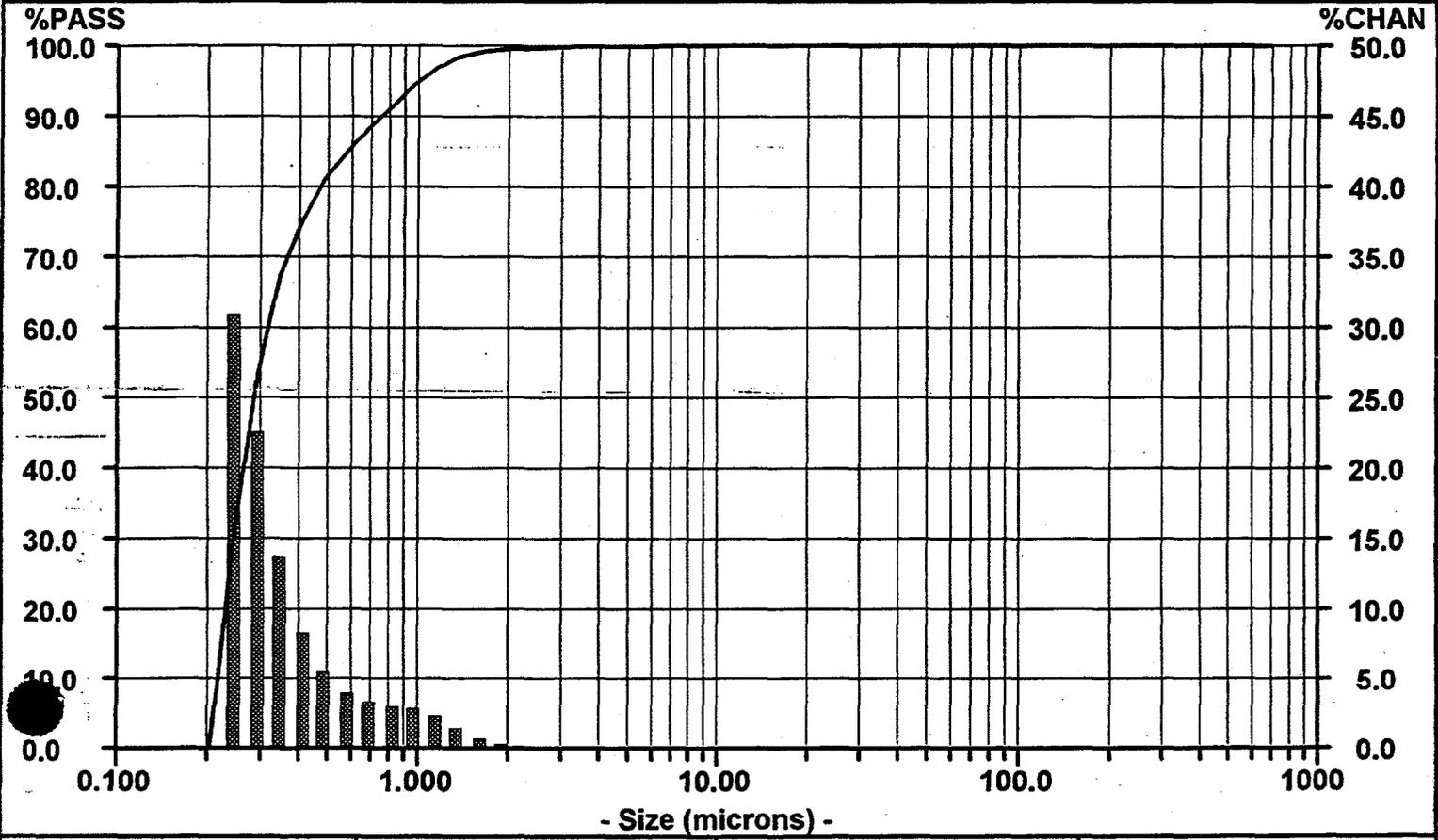
mv = 10.60
mn = 0.393
ma = 1.789
cs = 3.354
sd = 0.159

Percentiles

10% = 0.218 60% = 0.310
20% = 0.230 70% = 0.360
30% = 0.242 80% = 0.465
40% = 0.258 90% = 0.740
50% = 0.280 95% = 0.996

Dia Vol% Width
0.280 100% 0.319

Number Distribution



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	0.01						
418.6	100.00	0.00	5.500	99.99	0.01						
352.0	100.00	0.00	4.625	99.98	0.02						
296.0	100.00	0.00	3.889	99.96	0.03						
248.9	100.00	0.00	3.270	99.93	0.05						
209.3	100.00	0.00	2.750	99.88	0.09						
176.0	100.00	0.00	2.312	99.79	0.18						
148.0	100.00	0.00	1.945	99.61	0.36						
124.5	100.00	0.00	1.635	99.25	0.77						
104.7	100.00	0.00	1.375	98.48	1.49						
88.00	100.00	0.00	1.156	96.99	2.34						
74.00	100.00	0.00	0.972	94.65	2.86						
62.23	100.00	0.00	0.818	91.79	3.09						
52.33	100.00	0.00	0.688	88.70	3.37						
44.00	100.00	0.00	0.578	85.33	4.05						
37.00	100.00	0.00	0.486	81.28	5.51						
31.11	100.00	0.00	0.409	75.77	8.33						
26.16	100.00	0.00	0.344	67.44	13.76						
22.00	100.00	0.00	0.289	53.68	22.63						
18.50	100.00	0.00	0.243	31.05	31.05						
15.00	100.00	0.00	0.204	0.00	0.00						
12.00	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

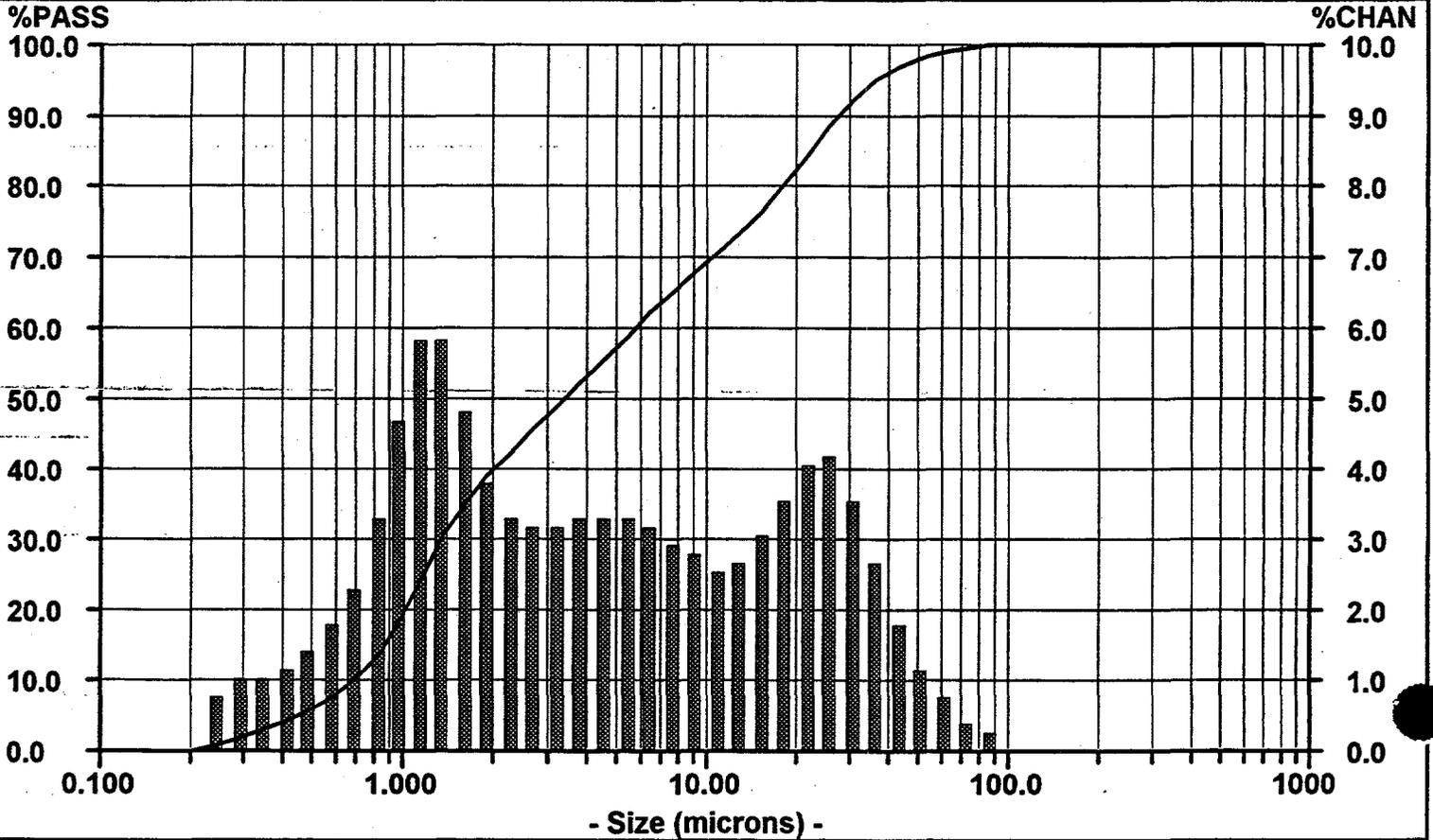
C104 Slurry
Sample CUP-C104-001

Date: 02/08/89 Meas #: 00036
Time: 11:30 Pres #: 01

C104 Slurry; Sample CUP-C104-001
in #1 Supernatant Simulant
60 ml/sec

Volume Distribution

Summary	Percentiles		Dia	Vol%	Width
mv = 9.717	10% = 0.681	60% = 5.807	21.60	32%	23.62
mn = 0.386	20% = 1.020	70% = 10.44	4.546	26%	4.358
ma = 1.646	30% = 1.364	80% = 18.24	1.059	42%	1.136
cs = 3.646	40% = 2.023	90% = 27.68			
sd = 10.33	50% = 3.441	95% = 36.71			



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	68.12	2.84						
592.0	100.00	0.00	7.778	65.28	3.07						
497.8	100.00	0.00	6.541	62.21	3.23						
418.6	100.00	0.00	5.500	58.98	3.30						
352.0	100.00	0.00	4.625	55.68	3.33						
296.0	100.00	0.00	3.889	52.35	3.32						
248.9	100.00	0.00	3.270	49.03	3.25						
209.3	100.00	0.00	2.750	45.78	3.21						
176.0	100.00	0.00	2.312	42.57	3.37						
148.0	100.00	0.00	1.945	39.20	3.95						
124.5	100.00	0.00	1.635	35.25	4.98						
104.7	100.00	0.00	1.375	30.27	5.92						
88.00	100.00	0.38	1.156	24.35	5.91						
74.00	99.62	0.55	0.972	18.44	4.81						
62.23	99.07	0.82	0.818	13.63	3.46						
52.33	98.25	1.25	0.688	10.17	2.45						
44.00	97.00	1.89	0.578	7.72	1.84						
37.00	95.11	2.74	0.486	5.88	1.48						
31.11	92.37	3.63	0.409	4.40	1.28						
26.16	88.74	4.22	0.344	3.12	1.18						
22.00	84.52	4.19	0.289	1.94	1.10						
18.50	80.33	3.69	0.243	0.84	0.84						
15.56	76.64	3.10	0.204	0.00	0.00						
13.08	73.54	2.74	0.172	0.00	0.00						
11.00	70.80	2.68	0.145	0.00	0.00						

02108100 GRG

Particle Size Analysis

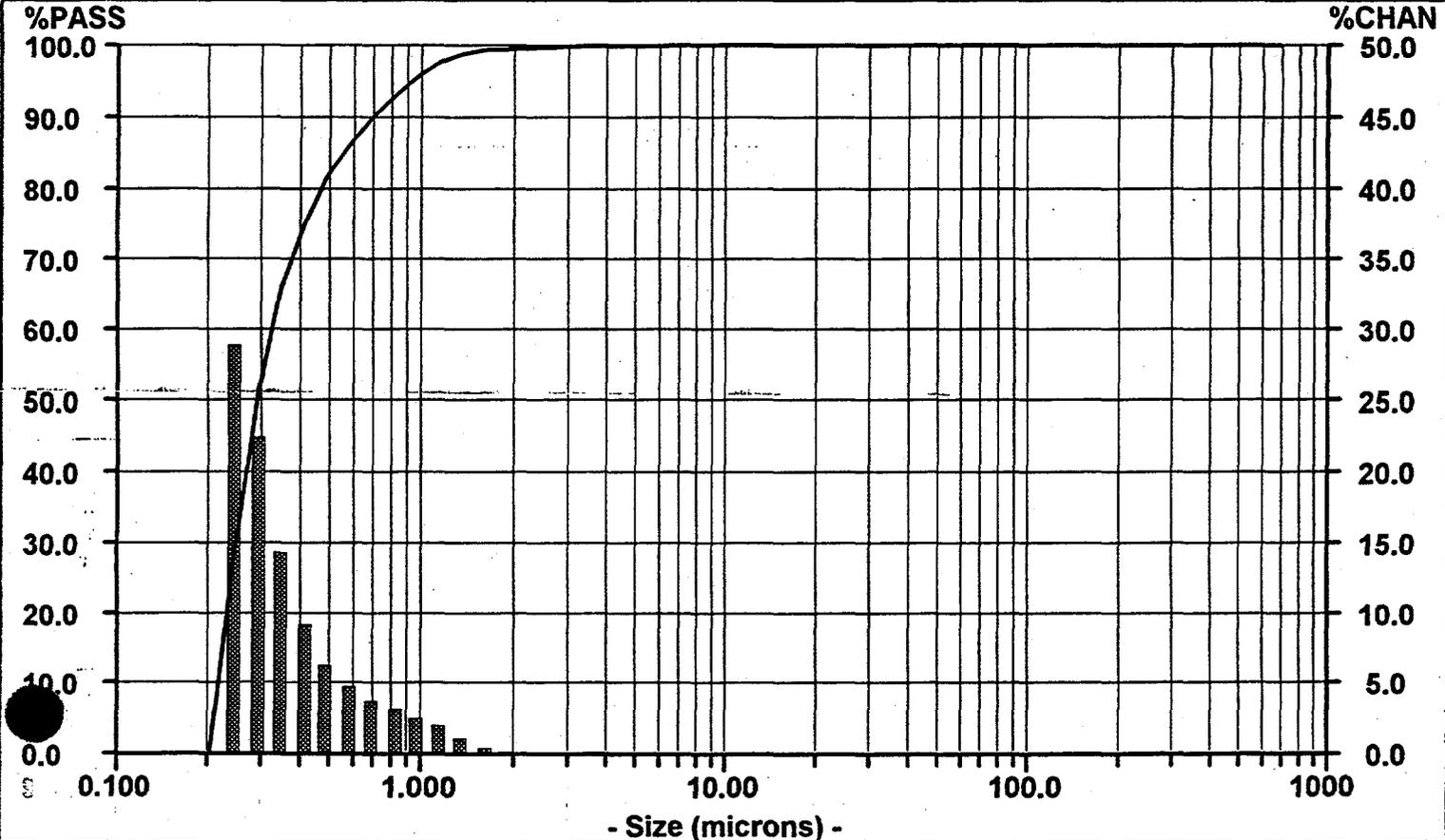
C104 Slurry
Sample CUP-C104-001

Date: 02/08/89 Meas #: 00036
Time: 11:30 Pres #: 01

C104 Slurry; Sample CUP-C104-001
In #1 Supernatant Simulant
60 ml/sec

Summary		Percentiles			Dia	Vol%	Width
mv = 9.717	10% = 0.219	60% = 0.317			0.285	100%	0.300
mn = 0.386	20% = 0.232	70% = 0.368					
ma = 1.646	30% = 0.245	80% = 0.463					
cs = 3.646	40% = 0.262	90% = 0.685					
sd = 0.150	50% = 0.285	95% = 0.917					

Number Distribution



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	0.01						
418.6	100.00	0.00	5.500	99.99	0.01						
352.0	100.00	0.00	4.625	99.98	0.02						
296.0	100.00	0.00	3.889	99.96	0.03						
248.9	100.00	0.00	3.270	99.93	0.05						
209.3	100.00	0.00	2.750	99.88	0.08						
176.0	100.00	0.00	2.312	99.80	0.13						
148.0	100.00	0.00	1.945	99.67	0.27						
124.5	100.00	0.00	1.635	99.40	0.57						
104.7	100.00	0.00	1.375	98.83	1.13						
88.00	100.00	0.00	1.156	97.70	1.90						
74.00	100.00	0.00	0.972	95.80	2.59						
62.23	100.00	0.00	0.818	93.21	3.13						
52.33	100.00	0.00	0.688	90.08	3.74						
44.00	100.00	0.00	0.578	86.34	4.72						
37.00	100.00	0.00	0.486	81.62	6.38						
31.11	100.00	0.00	0.409	75.24	9.28						
26.16	100.00	0.00	0.344	65.96	14.43						
22.00	100.00	0.00	0.289	51.53	22.59						
18.50	100.00	0.00	0.243	28.94	28.94						
14.75	100.00	0.00	0.204	0.00	0.00						
11.00	100.00	0.00	0.172	0.00	0.00						
			0.145	0.00	0.00						

Particle Size Analysis

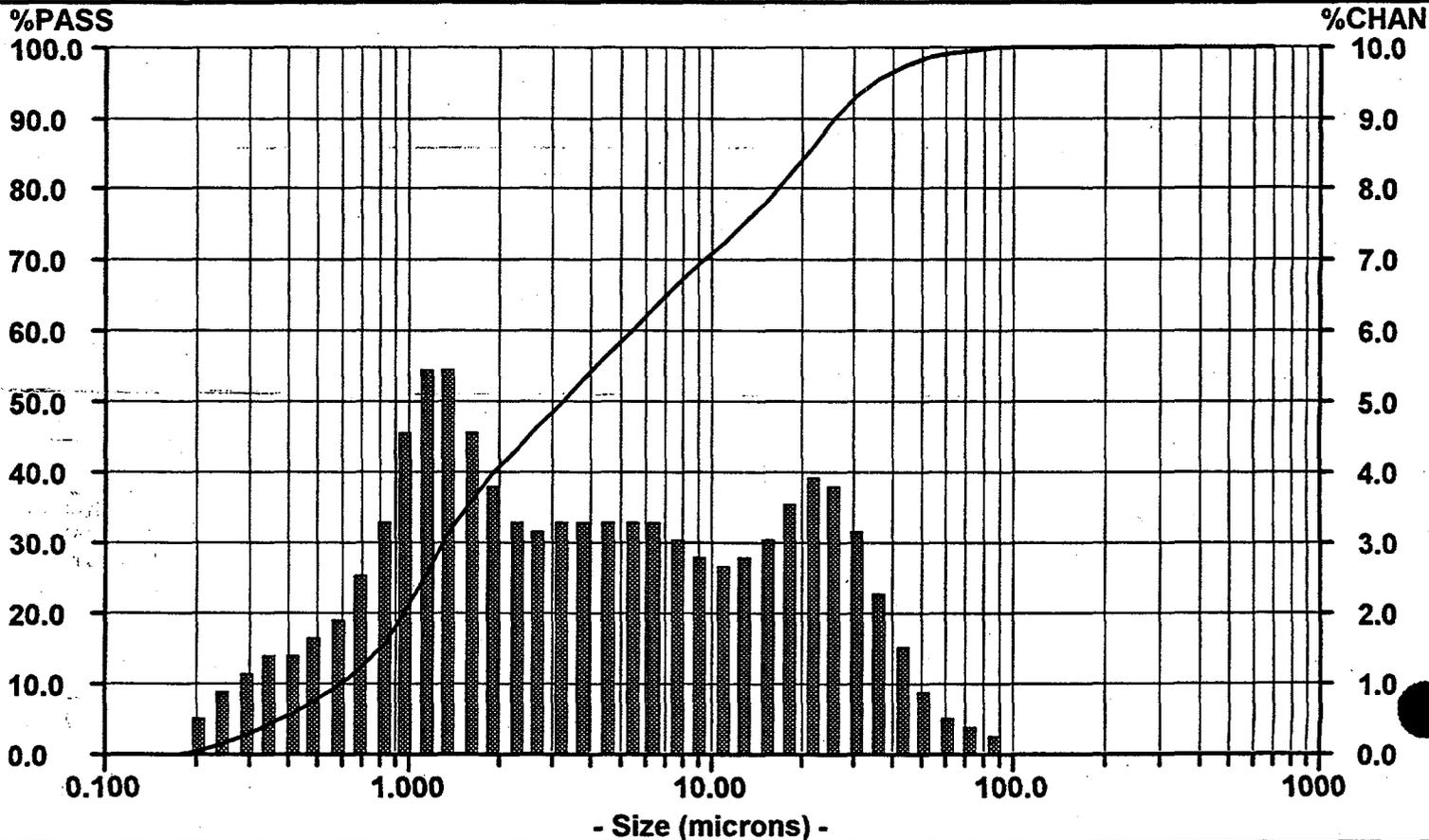
C104 Slurry
Sample CUP-C104-001

Date: 02/08/89 Meas #: 00040
Time: 11:41 Pres #: 01

C104 Slurry; Sample CUP-C104-001
in #1 Supernatant Simulant
Sonicated at 40 W for 90 sec; 60 ml/sec

Volume Distribution

Summary	Percentiles		Dia	Vol%	Width
mv = 9.103	10% = 0.586	60% = 5.434	20.78	30%	22.50
mn = 0.325	20% = 0.955	70% = 9.443	4.560	26%	4.350
ma = 1.477	30% = 1.307	80% = 16.79	1.010	44%	1.190
cs = 4.062	40% = 1.938	90% = 26.00			
sd = 9.616	50% = 3.279	95% = 34.63			



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	69.67	2.93						
592.0	100.00	0.00	7.778	66.74	3.16						
497.8	100.00	0.00	6.641	63.58	3.34						
418.6	100.00	0.00	5.600	60.24	3.42						
352.0	100.00	0.00	4.625	56.82	3.45						
296.0	100.00	0.00	3.889	53.37	3.42						
248.9	100.00	0.00	3.270	49.95	3.32						
209.3	100.00	0.00	2.750	46.63	3.23						
176.0	100.00	0.00	2.312	43.40	3.32						
148.0	100.00	0.00	1.945	40.08	3.81						
124.5	100.00	0.00	1.635	36.27	4.69						
104.7	100.00	0.00	1.375	31.58	5.54						
88.00	100.00	0.33	1.156	26.04	5.52						
74.00	99.67	0.47	0.972	20.52	4.61						
62.23	99.20	0.69	0.818	15.91	3.47						
52.33	98.51	1.06	0.688	12.44	2.62						
44.00	97.45	1.62	0.578	9.82	2.09						
37.00	95.83	2.41	0.486	7.73	1.76						
31.11	93.42	3.28	0.409	5.97	1.56						
26.16	90.14	3.95	0.344	4.41	1.44						
22.00	86.19	4.06	0.289	2.97	1.29						
18.50	82.13	3.69	0.243	1.68	1.01						
15.56	78.44	3.17	0.204	0.67	0.67						
13.08	75.27	2.83	0.172	0.00	0.00						
11.00	72.44	2.77	0.145	0.00	0.00						

02100100 07K19

Particle Size Analysis

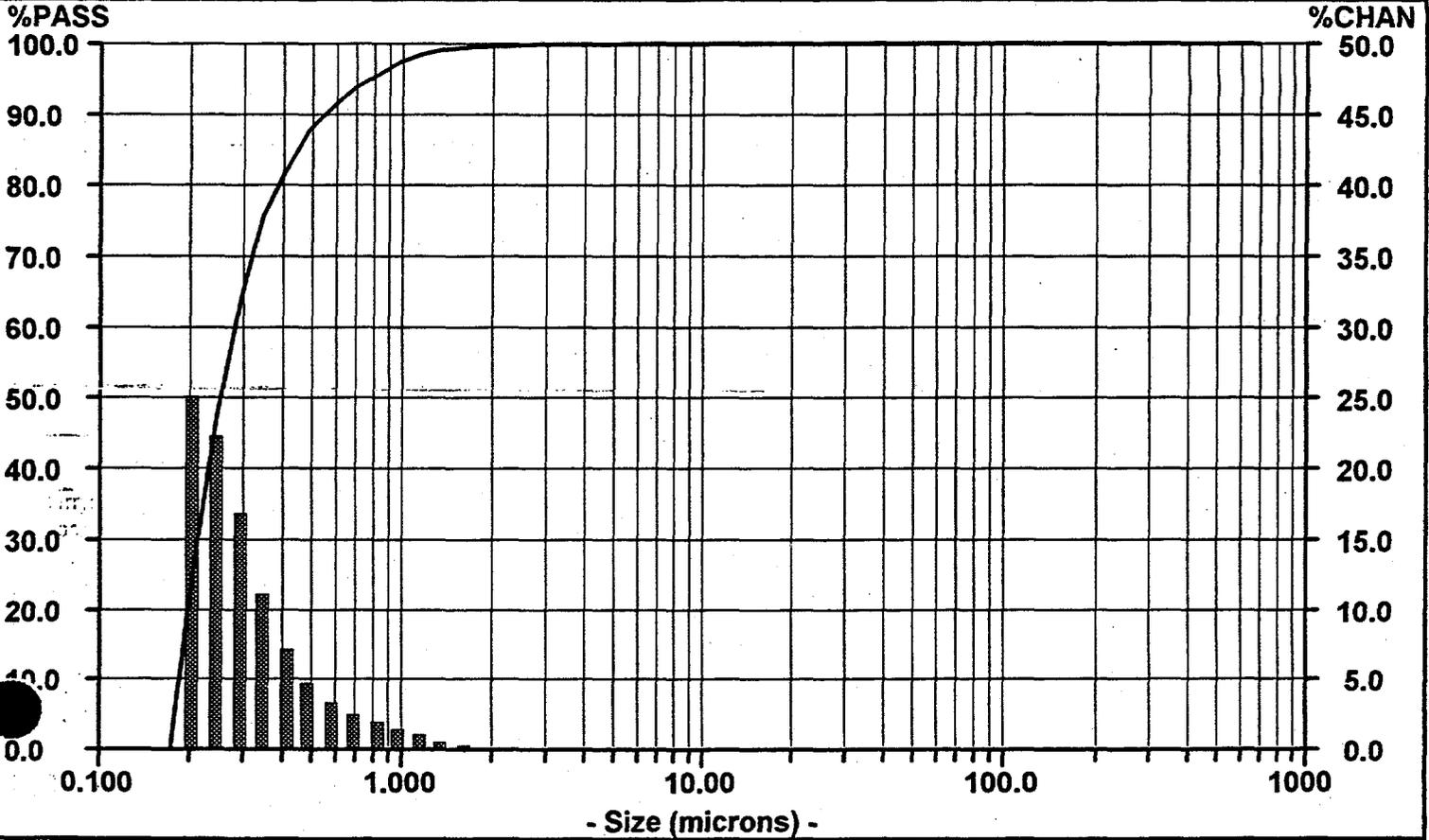
C104 Slurry
Sample CUP-C104-001

Date: 02/08/88 Meas #: 00040
Time: 11:41 Pres #: 01

C104 Slurry; Sample CUP-C104-001
in #1 Supernatant Simulant
Sonicated at 40 W for 90 sec; 60 ml/sec

Summary	Percentiles		Dia	Vol%	Width
mv = 9.103	10% = 0.186	60% = 0.275	0.249	100%	0.228
mn = 0.325	20% = 0.198	70% = 0.312			
ma = 1.477	30% = 0.211	80% = 0.377			
cs = 4.062	40% = 0.229	90% = 0.538			
sd = 0.114	50% = 0.249	95% = 0.755			

Number Distribution



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	0.01						
352.0	100.00	0.00	4.625	99.99	0.01						
296.0	100.00	0.00	3.889	99.98	0.02						
248.9	100.00	0.00	3.270	99.96	0.03						
209.3	100.00	0.00	2.750	99.93	0.05						
176.0	100.00	0.00	2.312	99.88	0.09						
148.0	100.00	0.00	1.945	99.79	0.16						
124.5	100.00	0.00	1.635	99.63	0.34						
104.7	100.00	0.00	1.375	99.29	0.68						
88.00	100.00	0.00	1.156	98.61	1.14						
74.00	100.00	0.00	0.972	97.47	1.59						
62.23	100.00	0.00	0.818	95.88	2.02						
52.33	100.00	0.00	0.688	93.86	2.57						
44.00	100.00	0.00	0.578	91.29	3.44						
37.00	100.00	0.00	0.486	87.85	4.86						
31.11	100.00	0.00	0.409	82.99	7.25						
26.16	100.00	0.00	0.344	75.74	11.29						
22.00	100.00	0.00	0.289	64.45	16.99						
18.50	100.00	0.00	0.243	47.46	22.33						
15.00	100.00	0.00	0.204	25.13	25.13						
11.00	100.00	0.00	0.172	0.00	0.00						
			0.145	0.00	0.00						

02108100 GRG

Particle Size Analysis

C104 Slurry
Sample CUP-C104-001

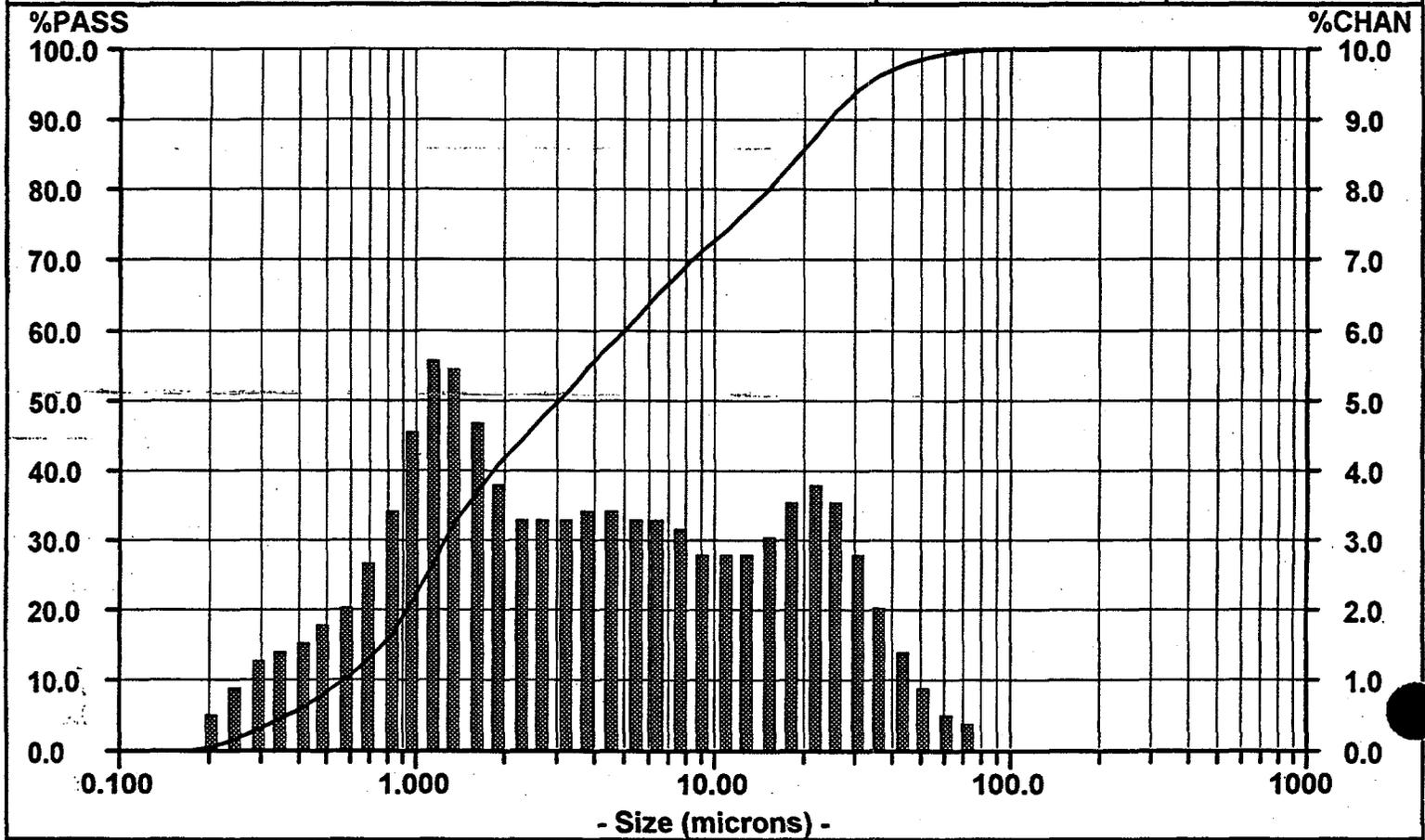
Date: 02/08/89 Meas #: 00044
Time: 11:52 Pres #: 01

C104 Slurry; Sample CUP-C104-001
In #1 Supernatant Simulant
Sonicated at 40 W for 90 sec; 60 ml/sec
2nd sonication

Summary	Percentiles	
mv = 8.474	10% = 0.562	60% = 5.021
mn = 0.325	20% = 0.930	70% = 8.509
ma = 1.428	30% = 1.270	80% = 15.31
cs = 4.202	40% = 1.839	90% = 24.42
sd = 8.878	50% = 3.068	95% = 32.78

Dia	Vol%	Width
20.03	29%	21.66
4.547	27%	4.342
1.003	44%	1.202

Volume Distribution



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	71.41	2.98						
592.0	100.00	0.00	7.778	68.43	3.21						
497.8	100.00	0.00	6.541	65.22	3.39						
418.6	100.00	0.00	5.500	61.83	3.49						
352.0	100.00	0.00	4.625	58.34	3.54						
296.0	100.00	0.00	3.889	54.80	3.52						
248.9	100.00	0.00	3.270	51.28	3.41						
209.3	100.00	0.00	2.750	47.87	3.30						
176.0	100.00	0.00	2.312	44.57	3.39						
148.0	100.00	0.00	1.945	41.18	3.88						
124.5	100.00	0.00	1.635	37.30	4.78						
104.7	100.00	0.00	1.375	32.52	5.59						
88.00	100.00	0.13	1.156	26.93	5.61						
74.00	99.87	0.45	0.972	21.32	4.69						
62.23	99.42	0.64	0.818	16.63	3.55						
52.33	98.78	0.95	0.688	13.08	2.70						
44.00	97.83	1.43	0.578	10.38	2.18						
37.00	96.40	2.11	0.486	8.20	1.86						
31.11	94.29	2.91	0.409	6.34	1.67						
26.16	91.38	3.60	0.344	4.67	1.55						
22.00	87.78	3.84	0.289	3.12	1.38						
18.50	83.94	3.62	0.243	1.74	1.05						
15.56	80.32	3.19	0.204	0.69	0.69						
13.08	77.13	2.89	0.172	0.00	0.00						
11.00	74.24	2.83	0.145	0.00	0.00						

Particle Size Analysis

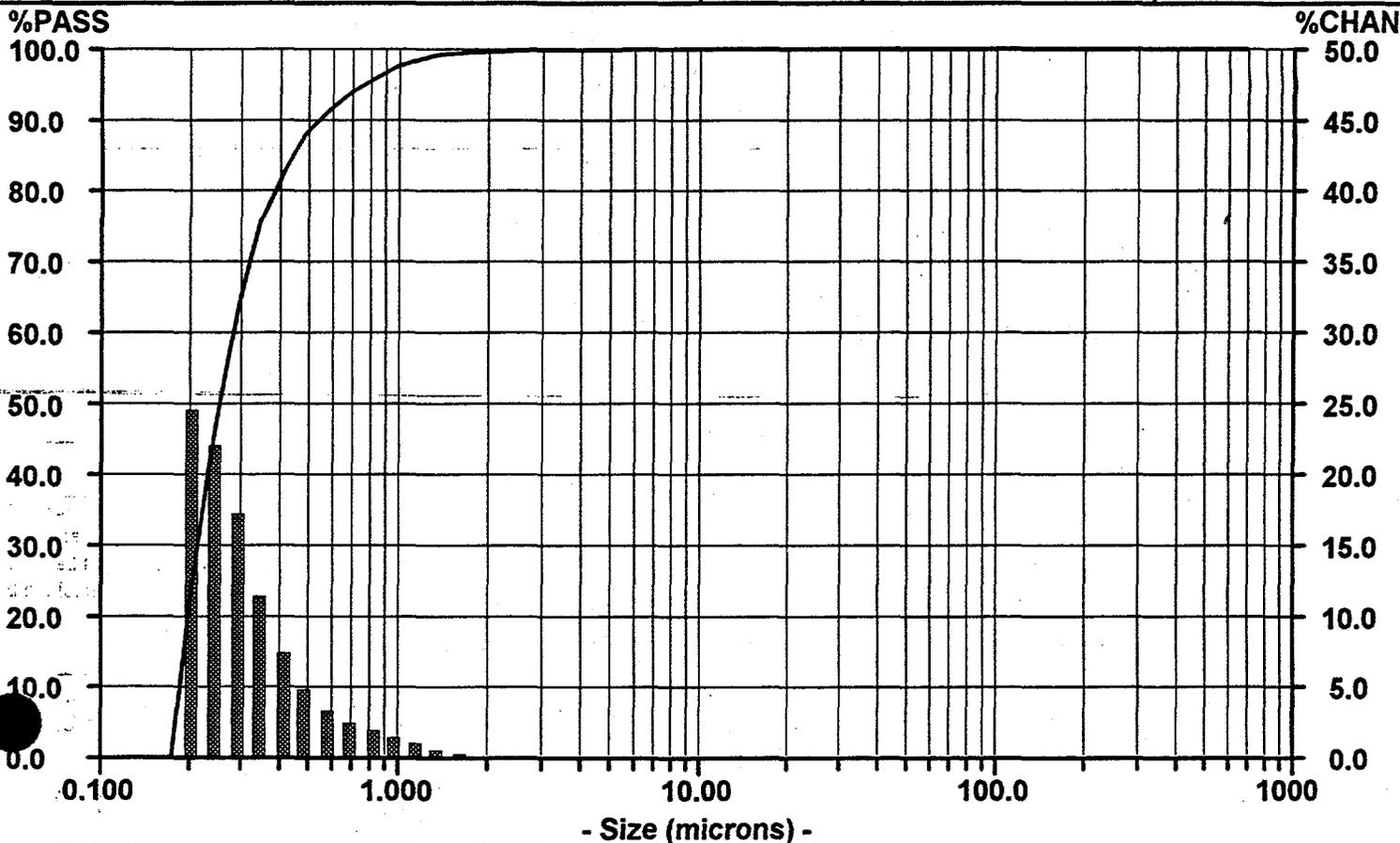
C104 Slurry
Sample CUP-C104-001

02108100 GKW
Date: 02/08/89 Meas #: 00044
Time: 11:52 Pres #: 01

C104 Slurry; Sample CUP-C104-001
in #1 Supernatant Simulant
Sonicated at 40 W for 90 sec; 60 ml/sec
2nd sonication

Summary	Percentiles		Dia	Vol%	Width
mv = 8.474	10% = 0.186	60% = 0.276	0.250	100%	0.225
mn = 0.325	20% = 0.199	70% = 0.312			
ma = 1.428	30% = 0.212	80% = 0.376			
cs = 4.202	40% = 0.230	90% = 0.532			
sd = 0.113	50% = 0.250	95% = 0.745			

Number Distribution



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	0.01						
352.0	100.00	0.00	4.625	99.99	0.01						
296.0	100.00	0.00	3.889	99.98	0.02						
248.9	100.00	0.00	3.270	99.96	0.03						
209.3	100.00	0.00	2.750	99.93	0.05						
176.0	100.00	0.00	2.312	99.88	0.08						
148.0	100.00	0.00	1.945	99.80	0.16						
124.5	100.00	0.00	1.635	99.64	0.33						
104.7	100.00	0.00	1.375	99.31	0.65						
88.0	100.00	0.00	1.156	98.66	1.10						
74.0	100.00	0.00	0.972	97.56	1.55						
62.23	100.00	0.00	0.818	96.01	1.97						
52.33	100.00	0.00	0.688	94.04	2.53						
44.0	100.00	0.00	0.578	91.51	3.43						
37.0	100.00	0.00	0.486	88.08	4.90						
31.11	100.00	0.00	0.409	83.18	7.41						
26.16	100.00	0.00	0.344	75.77	11.60						
22.0	100.00	0.00	0.289	64.17	17.35						
18.50	100.00	0.00	0.243	46.82	22.15						
15.56	100.00	0.00	0.204	24.67	24.67						
13.0	100.00	0.00	0.172	0.00	0.00						
11.0	100.00	0.00	0.145	0.00	0.00						

Particle Size Distribution Plots
For Initial Slurry: Sample CUF-C104-001DUP
Replicate No: 2

Particle Size Analysis

C104 Slurry DUP
Sample CUP-C104-001

Date: 02/08/89 Meas #: 00048
Time: 13:18 Pres #: 01

C104 Slurry DUP; Sample CUP-C104-001
In #1 Supernatant Simulant
40 ml/sec
Duplicate Run

Summary

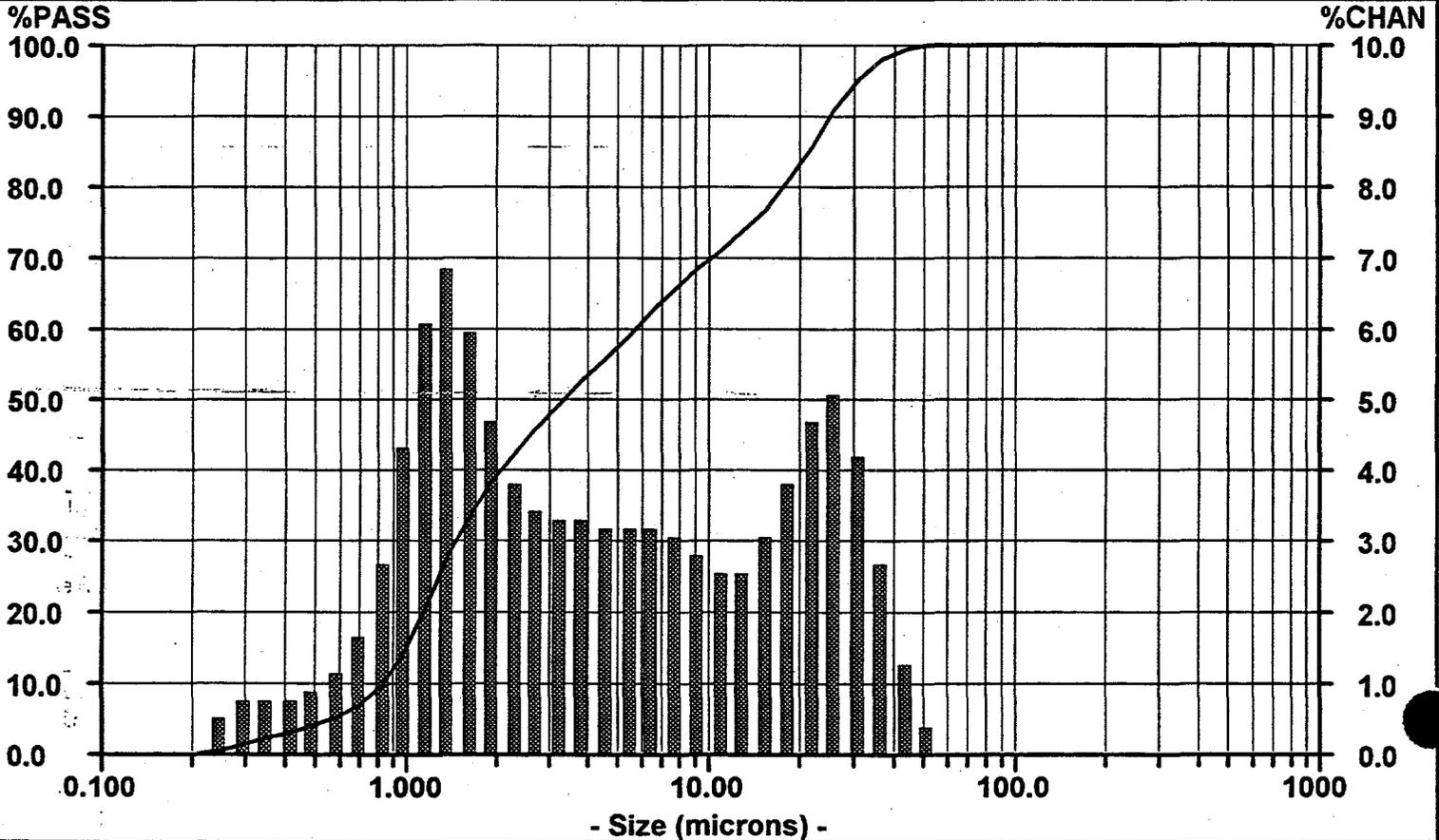
mv = 8.696
mn = 0.407
ma = 1.822
cs = 3.293
sd = 9.829

Percentiles

10% = 0.821 60% = 5.715
20% = 1.138 70% = 10.15
30% = 1.470 80% = 17.82
40% = 2.077 90% = 25.28
50% = 3.379 95% = 30.71

Dia	Vol%	Width
20.89	31%	17.80
5.911	16%	3.482
1.335	53%	1.777

Volume Distribution



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	68.58	2.87						
592.0	100.00	0.00	7.778	65.71	3.16						
497.8	100.00	0.00	6.541	62.56	3.29						
418.6	100.00	0.00	5.500	59.27	3.29						
352.0	100.00	0.00	4.625	55.98	3.28						
296.0	100.00	0.00	3.889	52.70	3.33						
248.9	100.00	0.00	3.270	49.37	3.41						
209.3	100.00	0.00	2.750	45.96	3.57						
176.0	100.00	0.00	2.312	42.39	3.96						
148.0	100.00	0.00	1.945	38.43	4.82						
124.5	100.00	0.00	1.635	33.61	6.08						
104.7	100.00	0.00	1.375	27.53	6.92						
88.00	100.00	0.00	1.156	20.61	6.26						
74.00	100.00	0.00	0.972	14.35	4.44						
62.23	100.00	0.00	0.818	9.91	2.76						
52.33	100.00	0.56	0.688	7.15	1.75						
44.00	99.44	1.38	0.578	5.40	1.23						
37.00	98.06	2.77	0.486	4.17	0.98						
31.11	95.29	4.30	0.409	3.19	0.87						
26.16	90.99	5.17	0.344	2.32	0.84						
22.00	85.82	4.88	0.289	1.48	0.82						
18.50	80.94	3.96	0.243	0.66	0.66						
15.56	76.98	3.11	0.204	0.00	0.00						
13.08	73.87	2.66	0.172	0.00	0.00						
11.00	71.21	2.63	0.145	0.00	0.00						

Particle Size Analysis

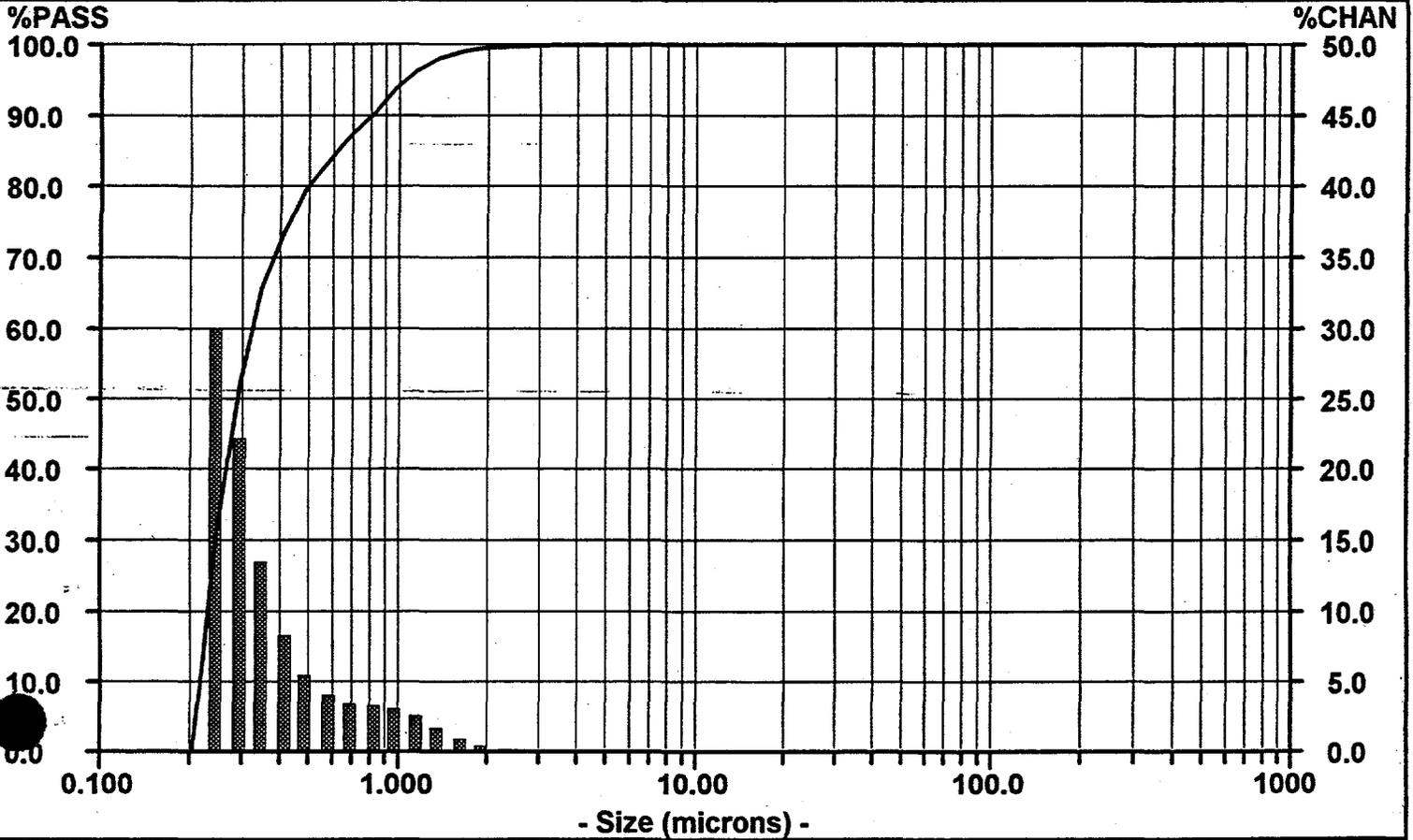
C104 Slurry DUP
Sample CUP-C104-001

02108100
Date: 02/08/89 Meas #: 00048
Time: 13:18 Pres #: 01

C104 Slurry DUP; Sample CUP-C104-001
in #1 Supernatant Simulant
40 ml/sec
Duplicate Run

Summary	Percentiles	Dia	Vol%	Width	
mv = 8.696	10% = 0.219	60% = 0.316	0.283	100%	0.357
mn = 0.407	20% = 0.231	70% = 0.372			
ma = 1.822	30% = 0.243	80% = 0.493			
cs = 3.293	40% = 0.260	90% = 0.790			
sd = 0.178	50% = 0.283	95% = 1.046			

Number Distribution



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	0.01						
418.6	100.00	0.00	5.500	99.99	0.01						
352.0	100.00	0.00	4.625	99.98	0.02						
296.0	100.00	0.00	3.889	99.96	0.04						
248.9	100.00	0.00	3.270	99.92	0.06						
209.3	100.00	0.00	2.750	99.86	0.11						
176.0	100.00	0.00	2.312	99.75	0.21						
148.0	100.00	0.00	1.945	99.54	0.43						
124.5	100.00	0.00	1.635	99.11	0.91						
104.7	100.00	0.00	1.375	98.20	1.74						
88.00	100.00	0.00	1.156	96.46	2.65						
74.00	100.00	0.00	0.972	93.81	3.16						
62.23	100.00	0.00	0.818	90.66	3.30						
52.33	100.00	0.00	0.688	87.36	3.53						
44.00	100.00	0.00	0.578	83.83	4.17						
37.00	100.00	0.00	0.486	79.66	5.57						
31.11	100.00	0.00	0.409	74.09	8.32						
26.16	100.00	0.00	0.344	65.77	13.55						
22.00	100.00	0.00	0.289	52.22	22.21						
18.50	100.00	0.00	0.243	30.01	30.01						
15.55	100.00	0.00	0.204	0.00	0.00						
13.00	100.00	0.00	0.172	0.00	0.00						
11.00	100.00	0.00	0.145	0.00	0.00						

02/08/89 GKG

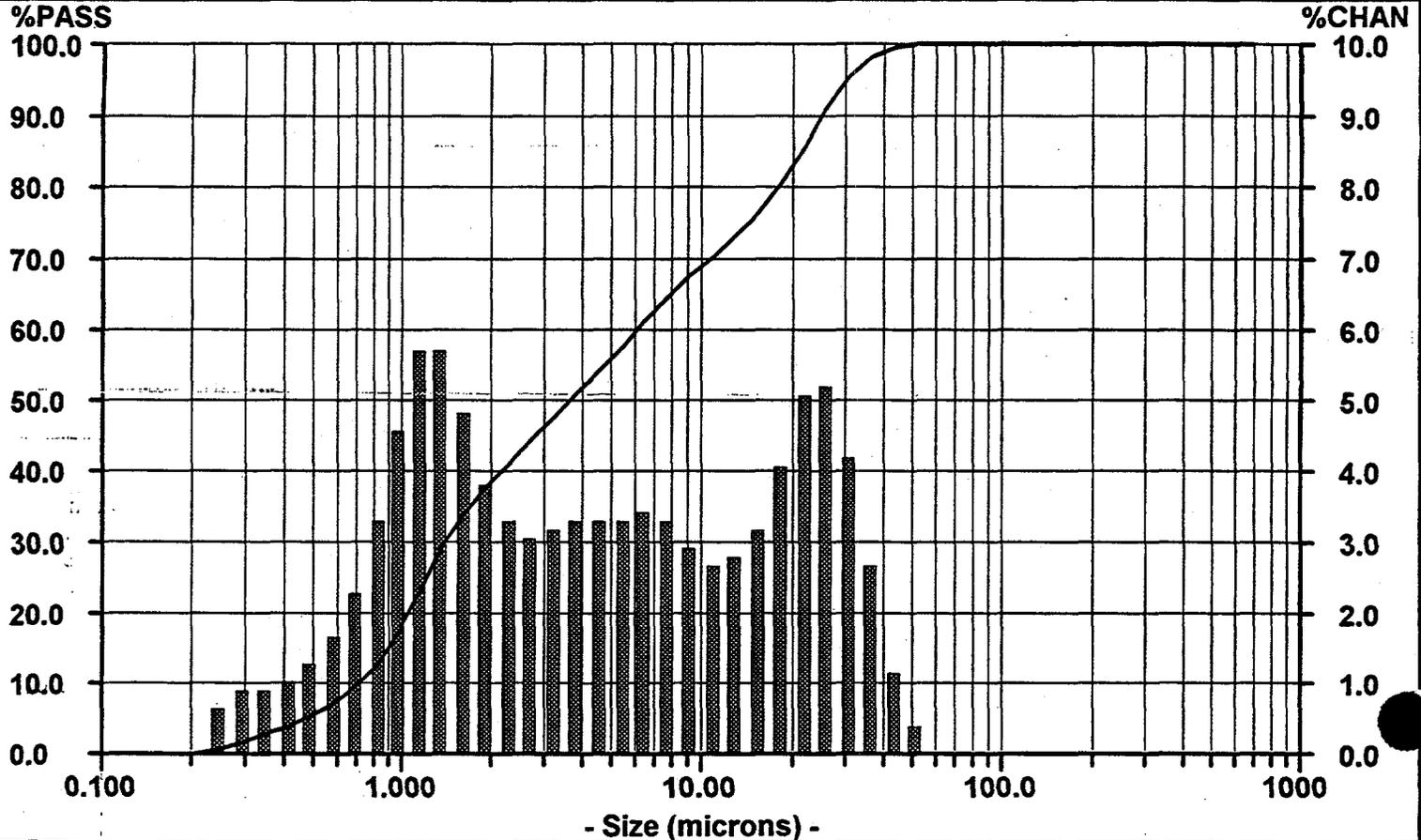
Particle Size Analysis

C104 Slurry DUP
Sample CUP-C104-001

Date: 02/08/89 Meas #: 00062
Time: 13:32 Pres #: 01

C104 Slurry DUP; Sample CUP-C104-001
in #1 Supernatant Simulant
60 ml/sec
Duplicate Run
Volume Distribution

Summary		Percentiles		Dia	Vol%	Width
mv = 8.789	10% = 0.708	60% = 6.140	20.65	32%	17.40	
mn = 0.390	20% = 1.051	70% = 10.66	4.663	26%	4.400	
ma = 1.707	30% = 1.419	80% = 18.03	1.070	42%	1.133	
cs = 3.514	40% = 2.169	90% = 25.17				
sd = 9.913	50% = 3.705	95% = 30.40				



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	67.71	3.06						
592.0	100.00	0.00	7.778	64.65	3.37						
497.8	100.00	0.00	6.541	61.28	3.51						
418.6	100.00	0.00	5.500	57.77	3.47						
352.0	100.00	0.00	4.625	54.30	3.37						
296.0	100.00	0.00	3.889	50.93	3.30						
248.9	100.00	0.00	3.270	47.63	3.23						
209.3	100.00	0.00	2.750	44.40	3.19						
176.0	100.00	0.00	2.312	41.21	3.35						
148.0	100.00	0.00	1.945	37.86	3.91						
124.5	100.00	0.00	1.635	33.95	4.90						
104.7	100.00	0.00	1.375	29.05	5.81						
88.0	100.00	0.00	1.156	23.24	5.75						
74.0	100.00	0.00	0.972	17.49	4.64						
62.23	100.00	0.00	0.818	12.85	3.32						
52.33	100.00	0.49	0.688	9.53	2.34						
44.00	99.51	1.29	0.578	7.19	1.74						
37.00	98.22	2.71	0.486	5.45	1.39						
31.11	95.51	4.36	0.409	4.06	1.19						
26.16	91.15	5.35	0.344	2.87	1.09						
22.00	85.80	5.11	0.289	1.78	1.01						
18.50	80.69	4.15	0.243	0.77	0.77						
15.56	76.54	3.25	0.204	0.00	0.00						
13.08	73.29	2.80	0.172	0.00	0.00						
11.00	70.49	2.78	0.145	0.00	0.00						

Particle Size Analysis

C104 Slurry DUP
Sample CUP-C104-001

Date: 02/08/89 Meas #: 00052
Time: 13:32 Pres #: 01

C104 Slurry DUP; Sample CUP-C104-001

In #1 Supernatant Simulant

Sonicated at 40 W for 90 sec; 60 ml/sec GR6

1st Sonication - Duplicate Sample

2/sec ; Number Distribution

Summary

mv = 8.789
mn = 0.390
ma = 1.707
cs = 3.514
sd = 0.156

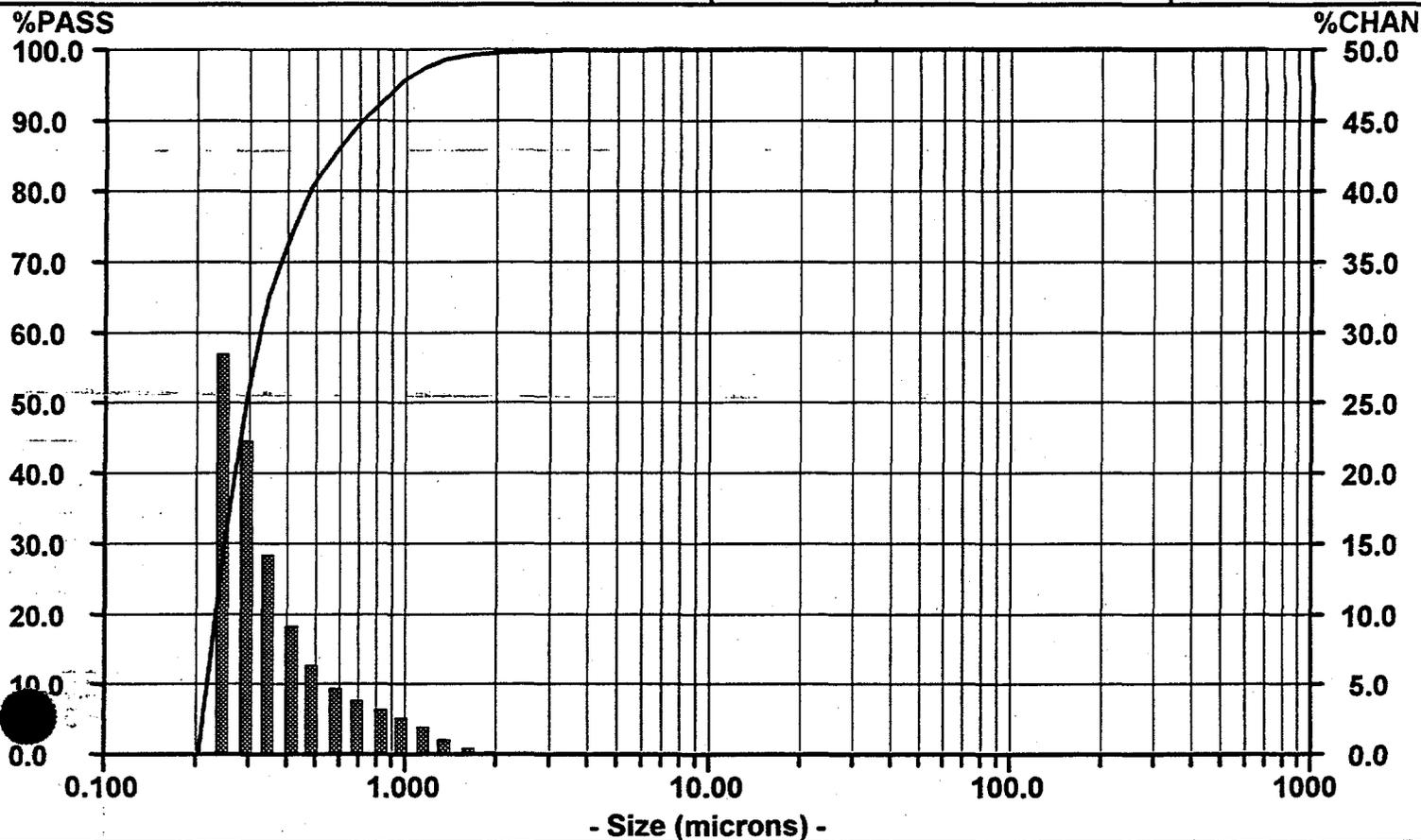
Percentiles

10% = 0.220
20% = 0.232
30% = 0.245
40% = 0.263
50% = 0.287

60% = 0.319
70% = 0.372
80% = 0.472
90% = 0.700
95% = 0.933

Dia Vol% Width

0.287 100% 0.312



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	0.01						
418.6	100.00	0.00	5.500	99.99	0.01						
352.0	100.00	0.00	4.625	99.98	0.02						
296.0	100.00	0.00	3.889	99.96	0.03						
248.9	100.00	0.00	3.270	99.93	0.05						
209.3	100.00	0.00	2.750	99.88	0.08						
176.0	100.00	0.00	2.312	99.80	0.14						
148.0	100.00	0.00	1.945	99.66	0.28						
124.5	100.00	0.00	1.635	99.38	0.60						
104.7	100.00	0.00	1.375	98.78	1.19						
88.00	100.00	0.00	1.156	97.59	1.99						
74.00	100.00	0.00	0.972	95.60	2.69						
62.23	100.00	0.00	0.818	92.91	3.24						
52.33	100.00	0.00	0.688	89.67	3.85						
44.00	100.00	0.00	0.578	85.82	4.81						
37.00	100.00	0.00	0.486	81.01	6.45						
31.11	100.00	0.00	0.409	74.56	9.29						
26.16	100.00	0.00	0.344	65.27	14.35						
22.00	100.00	0.00	0.289	50.92	22.34						
18.50	100.00	0.00	0.243	28.58	28.58						
15.00	100.00	0.00	0.204	0.00	0.00						
12.00	100.00	0.00	0.172	0.00	0.00						
11.00	100.00	0.00	0.145	0.00	0.00						

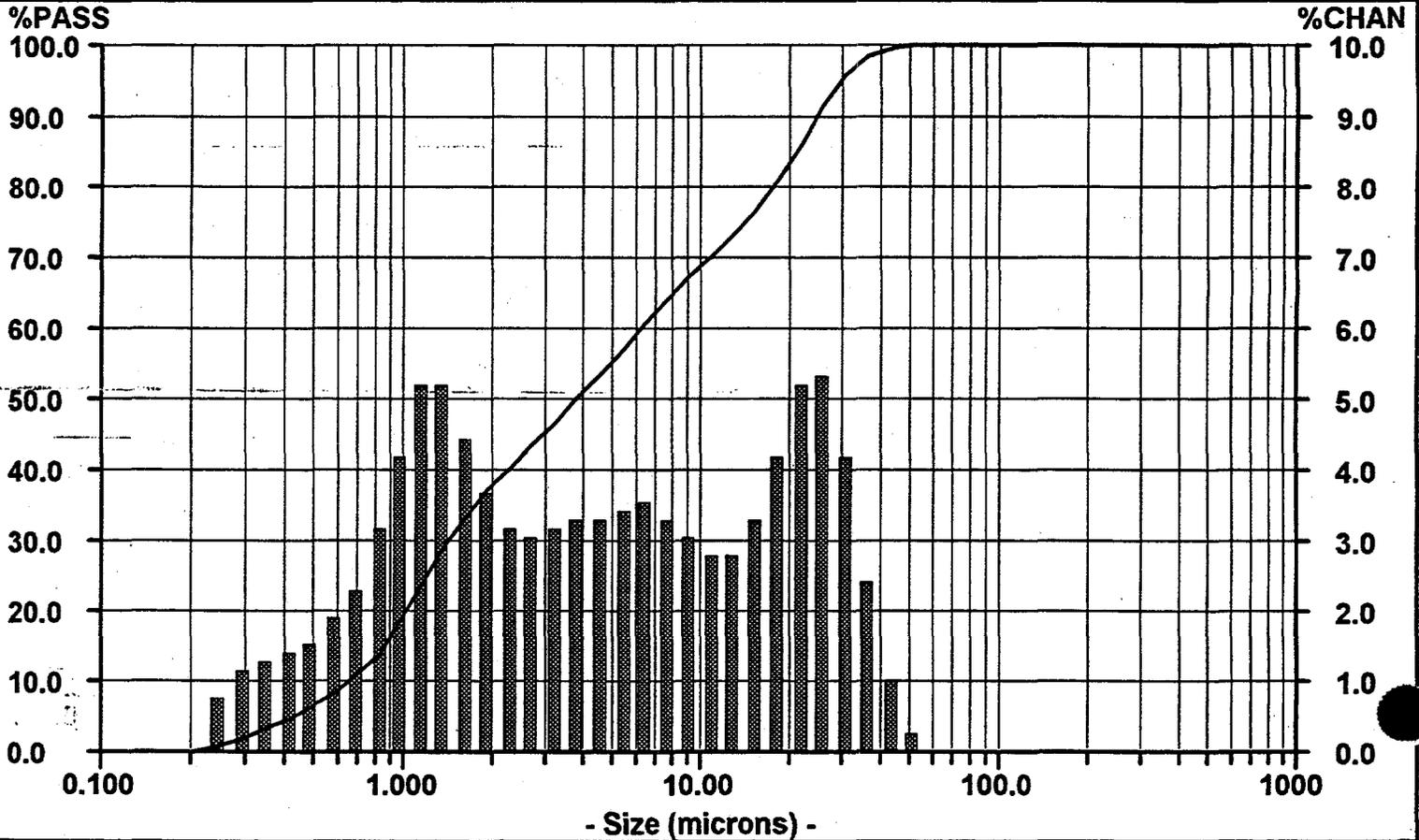
Particle Size Analysis

C104 Slurry DUP
Sample CUP-C104-001

Date: -02/08/89- Meas #: 00056
Time: 13:45 Pres #: 01

C104 Slurry DUP; Sample CUP-C104-001
in #1 Supernatant Simulant
Sonicated at 40 W for 90 sec; 60 ml/sec
1st Sonication—Duplicate Sample
M Volume Distribution

Summary	Percentiles		Dia	Vol%	Width
mv = 8.735	10% = 0.650	60% = 6.306	20.30	33%	16.82
mn = 0.375	20% = 1.027	70% = 10.79	4.698	27%	4.408
ma = 1.650	30% = 1.431	80% = 17.87	1.033	40%	1.187
cs = 3.636	40% = 2.266	90% = 24.75			
sd = 9.803	50% = 3.862	95% = 29.74			



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	67.42	3.17						
592.0	100.00	0.00	7.778	64.25	3.49						
497.8	100.00	0.00	6.541	60.76	3.62						
418.6	100.00	0.00	5.500	57.14	3.56						
352.0	100.00	0.00	4.625	53.58	3.44						
296.0	100.00	0.00	3.889	50.14	3.35						
248.9	100.00	0.00	3.270	46.79	3.25						
209.3	100.00	0.00	2.750	43.54	3.17						
176.0	100.00	0.00	2.312	40.37	3.26						
148.0	100.00	0.00	1.945	37.11	3.70						
124.5	100.00	0.00	1.635	33.41	4.51						
104.7	100.00	0.00	1.375	28.90	5.26						
88.00	100.00	0.00	1.156	23.64	5.23						
74.00	100.00	0.00	0.972	18.41	4.33						
62.23	100.00	0.00	0.818	14.08	3.24						
52.33	100.00	0.39	0.688	10.84	2.43						
44.00	99.61	1.12	0.578	8.41	1.93						
37.00	98.49	2.53	0.486	6.48	1.62						
31.11	95.96	4.28	0.409	4.86	1.44						
26.16	91.68	5.44	0.344	3.42	1.33						
22.00	86.24	5.29	0.289	2.09	1.21						
18.50	80.95	4.34	0.243	0.88	0.88						
15.56	76.61	3.39	0.204	0.00	0.00						
13.08	73.22	2.91	0.172	0.00	0.00						
11.00	70.31	2.89	0.145	0.00	0.00						

02108100 GRG

Particle Size Analysis

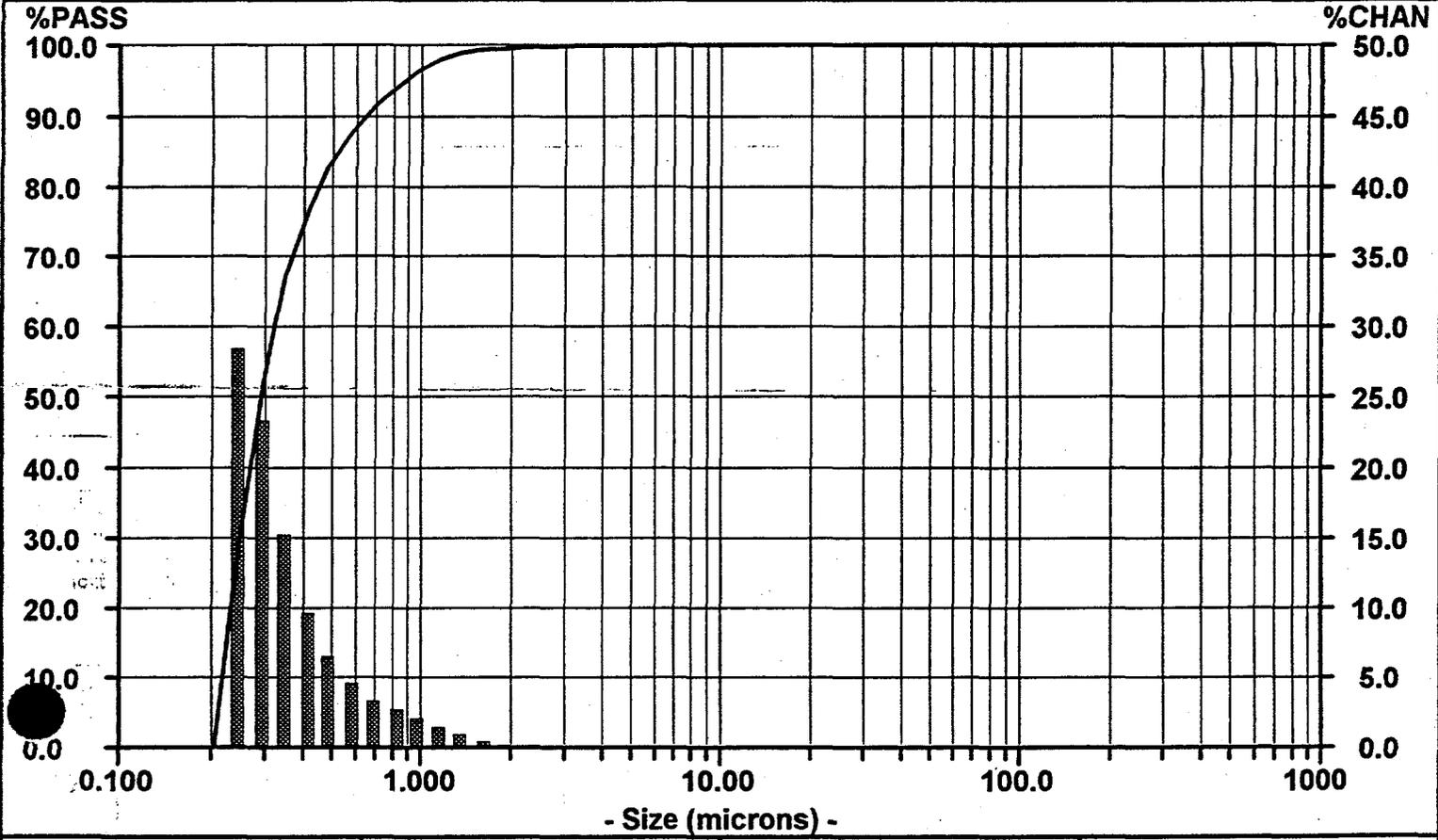
C104 Slurry DUP
Sample CUP-C104-001

Date: 02/08/89 Meas #: 00056
Time: 13:45 Pres #: 01

C104 Slurry DUP; Sample CUP-C104-001
in #1 Supernatant Simulant
Sonicated at 40 W for 90 sec; 60 ml/sec
1st Sonication—Duplicate Sample

Summary	Percentiles		Dia	Vol%	Width
mv = 8.735	10% = 0.220	60% = 0.314	0.285	100%	0.269
mn = 0.375	20% = 0.232	70% = 0.360			
ma = 1.650	30% = 0.246	80% = 0.441			
cs = 3.636	40% = 0.263	90% = 0.634			
sd = 0.134	50% = 0.285	95% = 0.862			

Number Distribution



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	0.01						
418.6	100.00	0.00	5.500	99.99	0.01						
352.0	100.00	0.00	4.625	99.98	0.02						
296.0	100.00	0.00	3.889	99.96	0.03						
248.9	100.00	0.00	3.270	99.93	0.04						
209.3	100.00	0.00	2.750	99.89	0.07						
176.0	100.00	0.00	2.312	99.82	0.12						
148.0	100.00	0.00	1.945	99.70	0.23						
124.5	100.00	0.00	1.635	99.47	0.48						
104.7	100.00	0.00	1.375	98.99	0.94						
88.0	100.00	0.00	1.156	98.05	1.57						
74.0	100.00	0.00	0.972	96.48	2.19						
62.23	100.00	0.00	0.818	94.29	2.75						
52.33	100.00	0.00	0.688	91.54	3.49						
44.0	100.00	0.00	0.578	88.05	4.65						
37.0	100.00	0.00	0.486	83.40	6.55						
31.11	100.00	0.00	0.409	76.85	9.79						
26.16	100.00	0.00	0.344	67.06	15.26						
22.0	100.00	0.00	0.289	51.80	23.32						
18.50	100.00	0.00	0.243	28.48	28.48						
15.56	100.00	0.00	0.204	0.00	0.00						
13.00	100.00	0.00	0.172	0.00	0.00						
11.00	100.00	0.00	0.145	0.00	0.00						

02/08/89

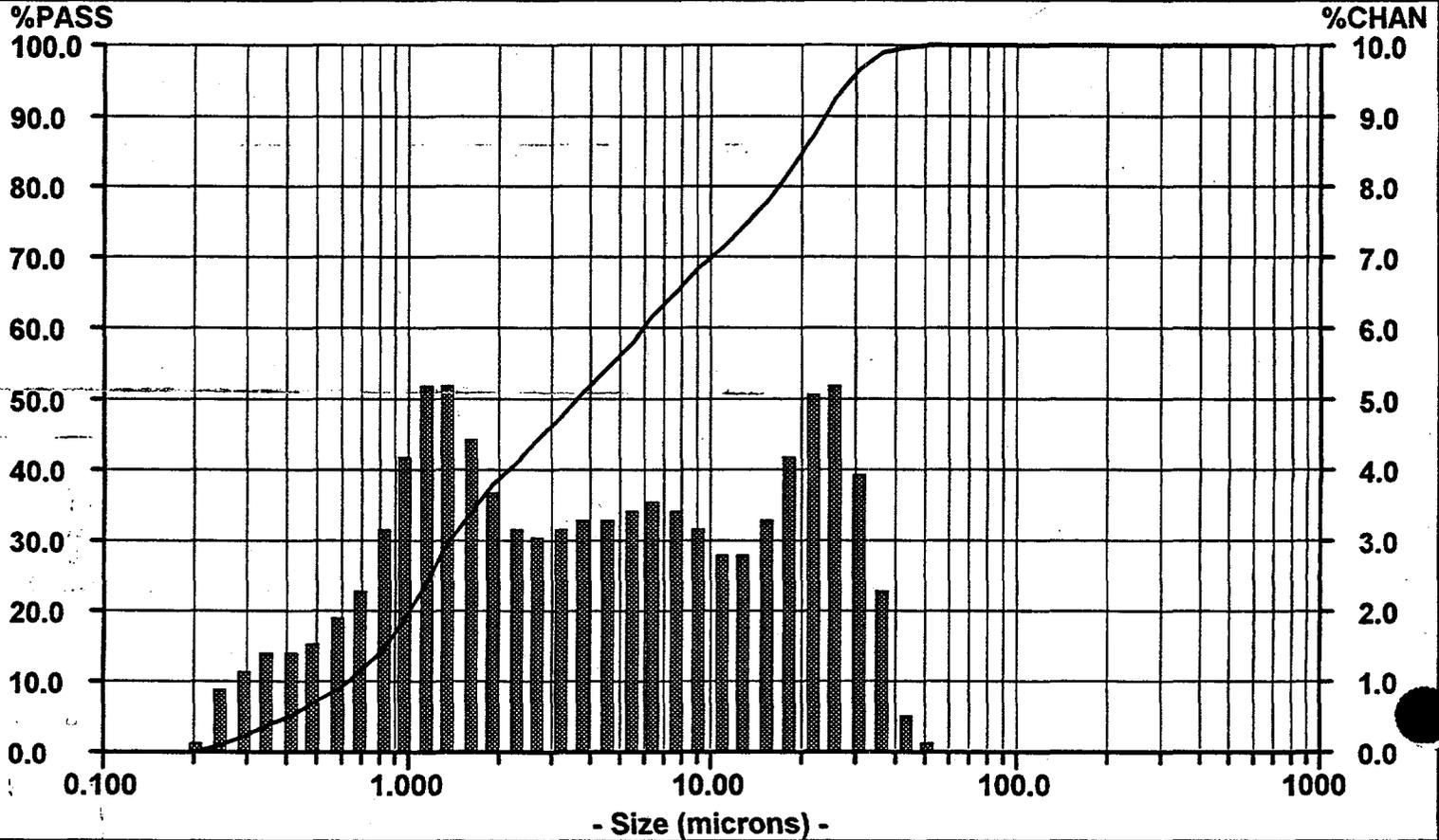
Particle Size Analysis

C104 Slurry DUP
Sample CUP-C104-001

Date: 02/08/89 Meas #: 00060
Time: 14:00 Pres #: 01

C104 Slurry DUP; Sample CUP-C104-001
In #1 Supernatant Simulant
Sonicated at 40 W for 90 sec; 60 ml/sec
2nd Sonication—Duplicate Sample
Volume Distribution

Summary	Percentiles		Dia	Vol%	Width
mv = 8.327	10% = 0.624	60% = 6.001	19.84	31%	16.20
mn = 0.354	20% = 1.004	70% = 10.07	4.704	27%	4.411
ma = 1.582	30% = 1.389	80% = 16.99	1.025	42%	1.197
cs = 3.792	40% = 2.158	90% = 23.91			
sd = 9.400	50% = 3.678	95% = 28.69			



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	68.54	3.21						
592.0	100.00	0.00	7.778	65.33	3.52						
497.8	100.00	0.00	6.541	61.81	3.65						
418.6	100.00	0.00	5.500	58.16	3.59						
352.0	100.00	0.00	4.625	54.57	3.47						
296.0	100.00	0.00	3.889	51.10	3.37						
248.9	100.00	0.00	3.270	47.73	3.27						
209.3	100.00	0.00	2.750	44.46	3.18						
176.0	100.00	0.00	2.312	41.28	3.27						
148.0	100.00	0.00	1.945	38.01	3.73						
124.5	100.00	0.00	1.635	34.28	4.56						
104.7	100.00	0.00	1.375	29.72	5.35						
88.0	100.00	0.00	1.156	24.37	5.30						
74.00	100.00	0.00	0.972	19.07	4.37						
62.23	100.00	0.00	0.818	14.70	3.26						
52.33	100.00	0.22	0.688	11.44	2.45						
44.00	99.78	0.64	0.578	8.99	1.97						
37.00	99.14	2.41	0.486	7.02	1.68						
31.11	96.73	4.08	0.409	5.34	1.50						
26.16	92.65	5.23	0.344	3.84	1.40						
22.00	87.42	5.18	0.289	2.44	1.27						
18.50	82.24	4.33	0.243	1.17	0.95						
15.56	77.91	3.45	0.204	0.22	0.22						
13.08	74.46	2.98	0.172	0.00	0.00						
11.00	71.48	2.94	0.145	0.00	0.00						

02108100 GRG

Particle Size Analysis

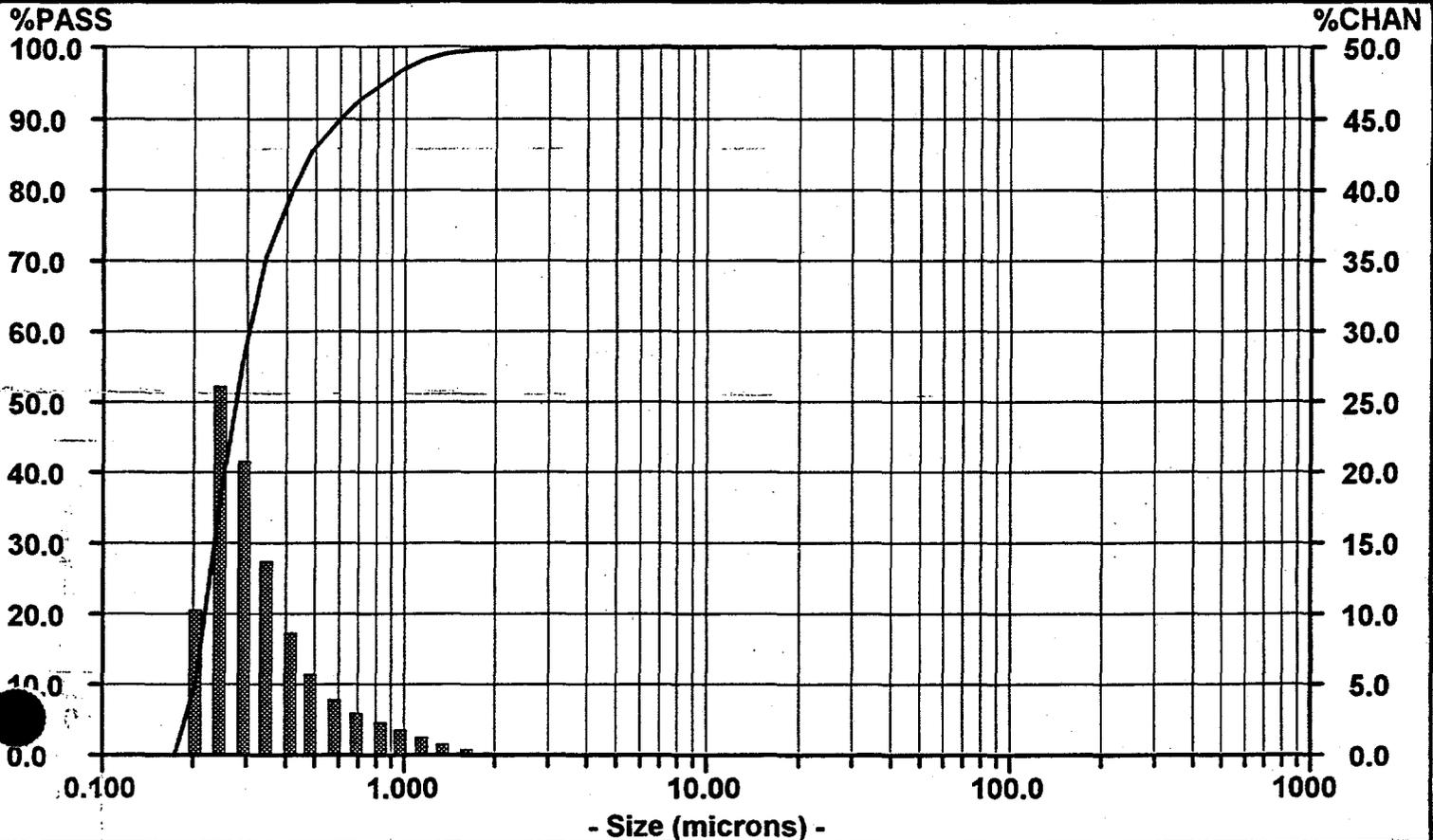
C104 Slurry DUP
Sample CUP-C104-001

Date: 02/08/89 Meas #: 00060
Time: 14:00 Pres #: 01

C104 Slurry DUP; Sample CUP-C104-001
in #1 Supernatant Simulant
Sonicated at 40 W for 90 sec; 60 ml/sec
2nd Sonication—Duplicate Sample

Summary	Percentiles		Dia	Vol%	Width
mv = 8.327	10% = 0.203	60% = 0.297	0.270	100%	0.247
mn = 0.354	20% = 0.219	70% = 0.338			
ma = 1.582	30% = 0.233	80% = 0.411			
cs = 3.792	40% = 0.249	90% = 0.588			
sd = 0.124	50% = 0.270	95% = 0.814			

Number Distribution



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	0.01						
418.6	100.00	0.00	5.500	99.99	0.01						
352.0	100.00	0.00	4.625	99.98	0.01						
296.0	100.00	0.00	3.889	99.97	0.02						
248.9	100.00	0.00	3.270	99.95	0.04						
209.3	100.00	0.00	2.750	99.91	0.06						
176.0	100.00	0.00	2.312	99.85	0.10						
148.0	100.00	0.00	1.945	99.75	0.20						
124.5	100.00	0.00	1.635	99.55	0.41						
104.7	100.00	0.00	1.375	99.14	0.82						
88.00	100.00	0.00	1.156	98.32	1.36						
74.00	100.00	0.00	0.972	96.96	1.89						
62.23	100.00	0.00	0.818	95.07	2.37						
52.33	100.00	0.00	0.688	92.70	3.00						
44.00	100.00	0.00	0.578	89.70	4.05						
37.00	100.00	0.00	0.486	85.65	5.80						
31.11	100.00	0.00	0.409	79.85	8.71						
26.16	100.00	0.00	0.344	71.14	13.71						
22.00	100.00	0.00	0.289	57.43	20.89						
18.50	100.00	0.00	0.243	36.54	26.24						
15.00	100.00	0.00	0.204	10.30	10.30						
11.00	100.00	0.00	0.172	0.00	0.00						
	100.00	0.00	0.145	0.00	0.00						

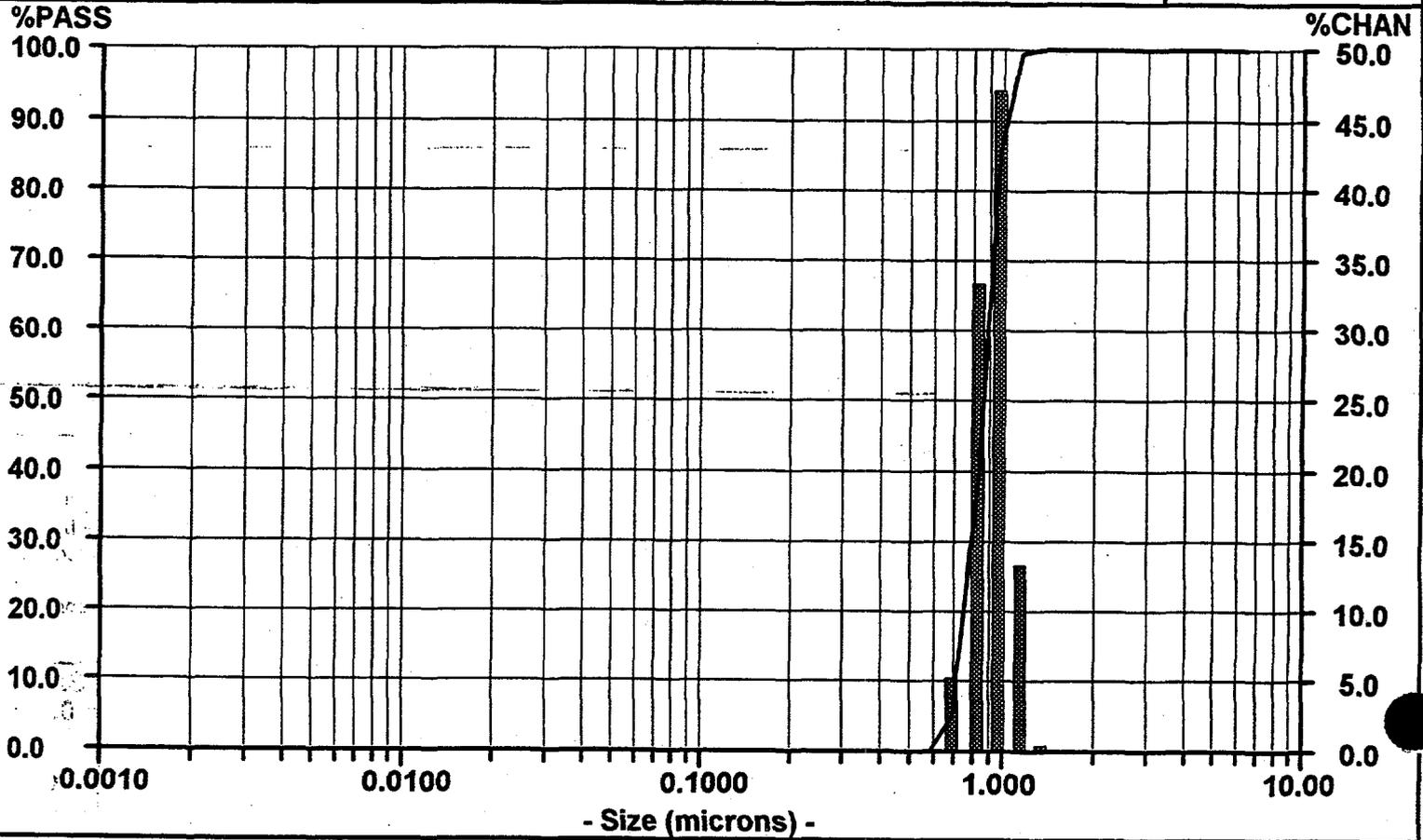
Particle Size Analysis

CUF-C104-001 SON D UP

Date: 02/09/89 Meas #: 00050
Time: 15:18 Pres #: 01

C-104 slurry DUP, sample CUF-C104-001 SON
Sonicated 90 sec
In #1 Supernatant Solution ; Duplicate Sample
UPA
30 seconds
Volume Distribution

Summary		Percentiles			Dia Vol% Width		
mv = .8546	10% = .7130	60% = .8797	.8503	100%	.2255		
mn = .8125	20% = .7545	70% = .9110					
ma = .8403	30% = .7896	80% = .9474					
cs = 7.141	40% = .8212	90% = 1.001					
sd = .1127	50% = .8503	95% = 1.051					



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
6.541	100.00	0.00	0.0859	0.00	0.00						
5.500	100.00	0.00	0.0723	0.00	0.00						
4.625	100.00	0.00	0.0608	0.00	0.00						
3.889	100.00	0.00	0.0511	0.00	0.00						
3.270	100.00	0.00	0.0430	0.00	0.00						
2.750	100.00	0.00	0.0361	0.00	0.00						
2.313	100.00	0.00	0.0304	0.00	0.00						
1.945	100.00	0.00	0.0255	0.00	0.00						
1.635	100.00	0.00	0.0215	0.00	0.00						
1.375	100.00	0.60	0.0181	0.00	0.00						
1.156	99.40	13.48	0.0152	0.00	0.00						
0.9723	85.92	47.16	0.0128	0.00	0.00						
0.8176	38.76	33.48	0.0107	0.00	0.00						
0.6875	5.28	5.28	0.0090	0.00	0.00						
0.5781	0.00	0.00	0.0076	0.00	0.00						
0.4861	0.00	0.00	0.0064	0.00	0.00						
0.4088	0.00	0.00	0.0054	0.00	0.00						
0.3437	0.00	0.00	0.0045	0.00	0.00						
0.2891	0.00	0.00	0.0038	0.00	0.00						
0.2431	0.00	0.00									
0.2044	0.00	0.00									
0.1719	0.00	0.00									
0.1445	0.00	0.00									
0.1215	0.00	0.00									
0.1022	0.00	0.00									

2
3
4
3
2

Particle Size Distribution Plots
For Washed Slurry: Sample CUF-C104-007
Replicate No: 1

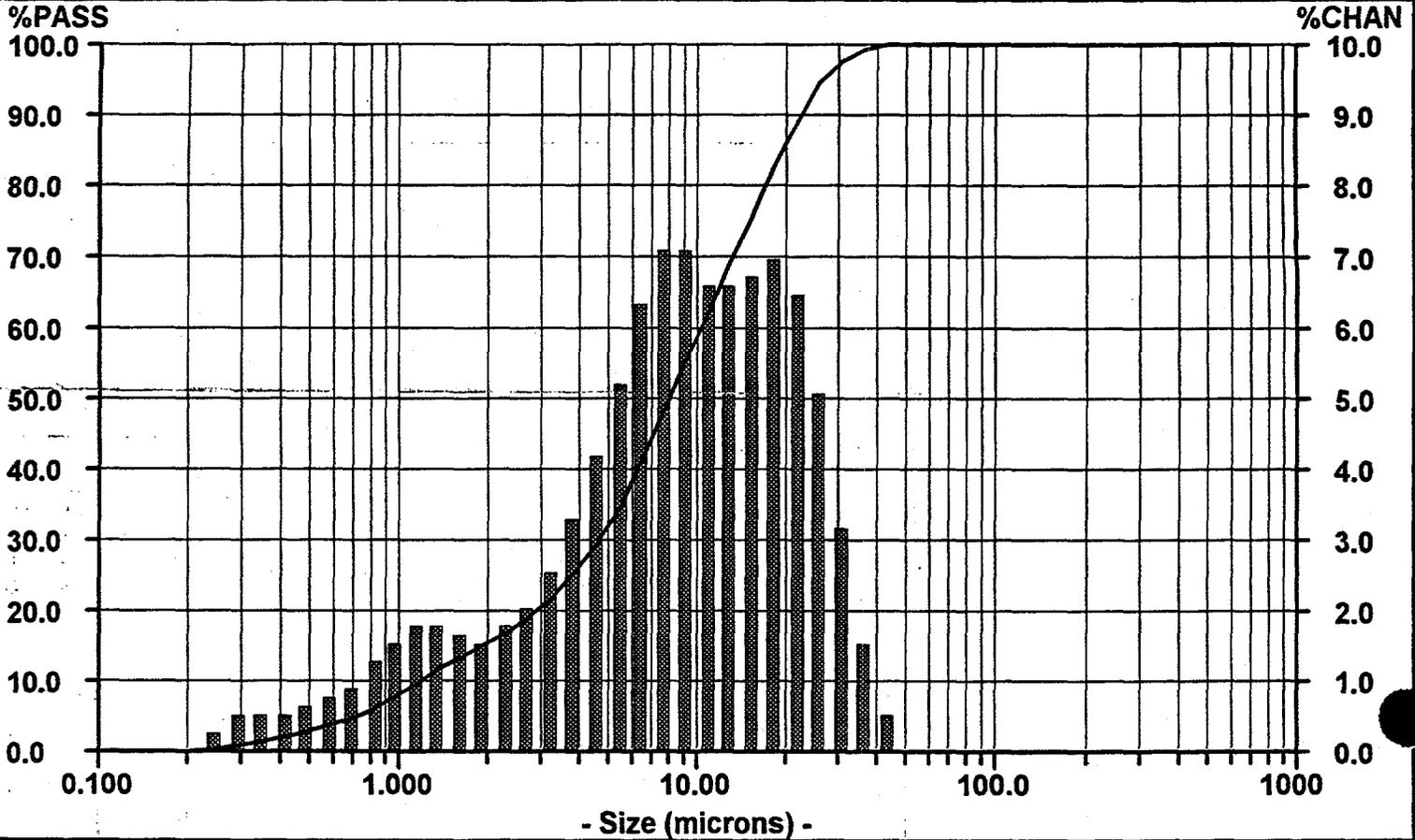
Particle Size Analysis

C104 Slurry DUP
Sample CUP-C104-007

02109100 GML
Date: 02/09/89 Meas #: 00020
Time: 10:28 Pres #: 01

C104 Slurry DUP; Sample CUP-C104-007
in #8 Supernatant Simulant
40 ml/sec
Duplicate Sample
Volume Distribution

Summary	Percentiles	Dia	Vol%	Width	
mv = 10.32	10% = 1.179	60% = 10.37	17.76	38%	12.73
mn = 0.378	20% = 2.938	70% = 13.46	5.986	49%	5.840
ma = 3.036	30% = 4.721	80% = 17.27	0.859	13%	0.925
cs = 1.977	40% = 6.333	90% = 22.42			
sd = 8.472	50% = 8.085	95% = 26.74			



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	55.51	7.11						
592.0	100.00	0.00	7.778	48.40	7.13						
497.8	100.00	0.00	6.541	41.27	6.47						
418.6	100.00	0.00	5.500	34.80	5.38						
352.0	100.00	0.00	4.625	29.42	4.29						
296.0	100.00	0.00	3.889	25.13	3.40						
248.9	100.00	0.00	3.270	21.73	2.68						
209.3	100.00	0.00	2.750	19.05	2.14						
176.0	100.00	0.00	2.312	16.91	1.81						
148.0	100.00	0.00	1.945	15.10	1.69						
124.5	100.00	0.00	1.635	13.41	1.75						
104.7	100.00	0.00	1.375	11.66	1.87						
88.00	100.00	0.00	1.156	9.79	1.86						
74.00	100.00	0.00	0.972	7.93	1.67						
62.23	100.00	0.00	0.818	6.26	1.37						
52.33	100.00	0.00	0.688	4.89	1.08						
44.00	100.00	0.62	0.578	3.81	0.86						
37.00	99.38	1.63	0.486	2.95	0.72						
31.11	97.75	3.24	0.409	2.23	0.64						
26.16	94.51	5.14	0.344	1.59	0.62						
22.00	89.37	6.57	0.289	0.97	0.61						
18.50	82.80	7.06	0.243	0.36	0.36						
15.56	75.74	6.84	0.204	0.00	0.00						
13.08	68.90	6.62	0.172	0.00	0.00						
11.00	62.28	6.77	0.145	0.00	0.00						

Particle Size Analysis

C104 Slurry DUP
Sample CUP-C104-007

02409100 414
Date: 02/09/89 Meas #: 00020
Time: 10:28 Pres #: 01

C104 Slurry DUP; Sample CUP-C104-007

In #8 Supernatant Simulant
60 ml/sec 40 ml/sec GR6

Duplicate Sample

Number Number Distribution

Summary

mv = 10.32
mn = 0.378
ma = 3.036
cs = 1.977
sd = 0.126

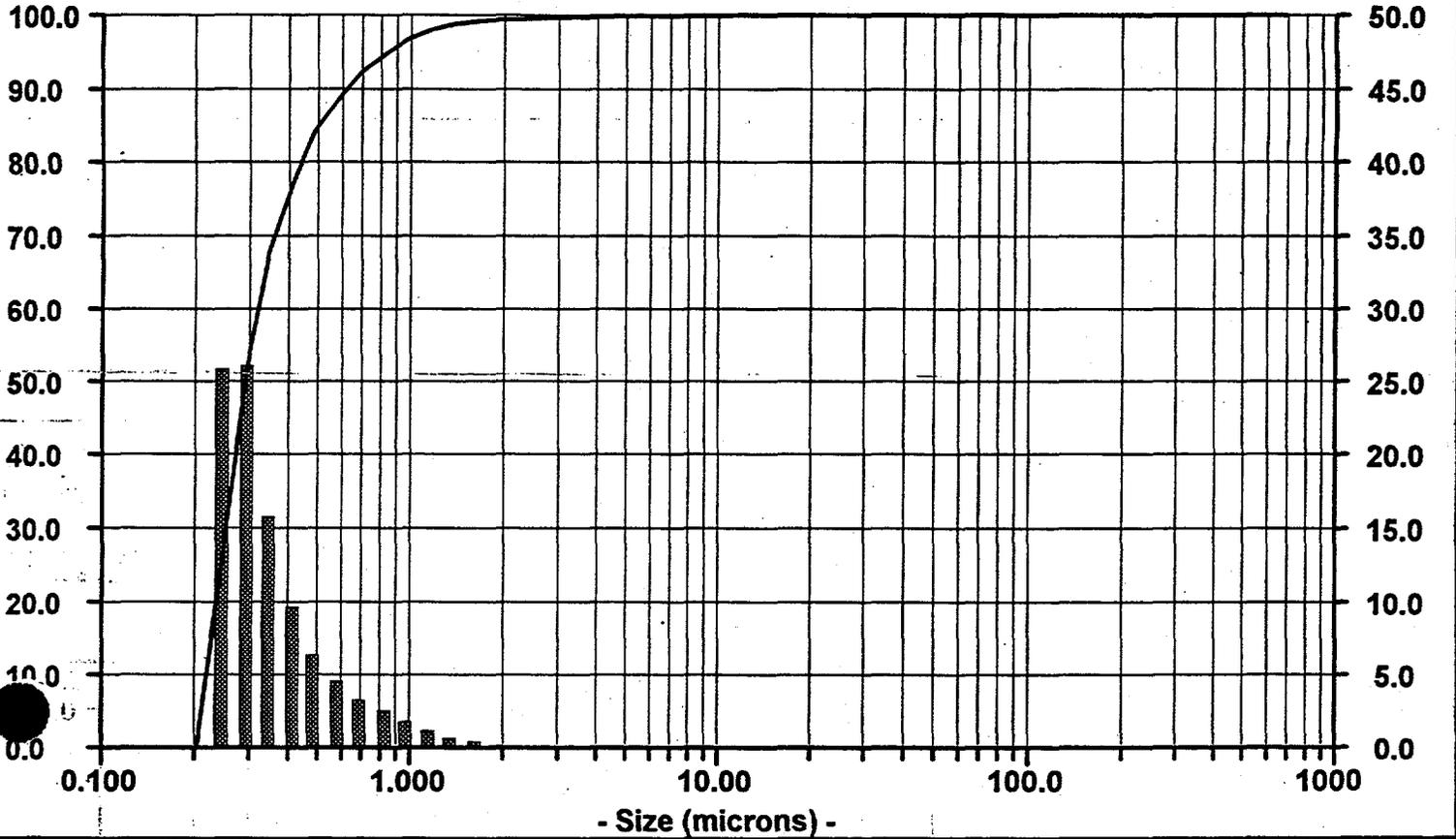
Percentiles

10% = 0.222 60% = 0.311
20% = 0.235 70% = 0.354
30% = 0.249 80% = 0.431
40% = 0.265 90% = 0.609
50% = 0.284 95% = 0.823

Dia Vol% Width

0.284 100% 0.252

%PASS



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	99.99	0.01						
692.0	100.00	0.00	7.778	99.98	0.02						
497.8	100.00	0.00	6.541	99.96	0.02						
418.6	100.00	0.00	5.500	99.94	0.03						
352.0	100.00	0.00	4.625	99.91	0.04						
296.0	100.00	0.00	3.889	99.87	0.06						
248.9	100.00	0.00	3.270	99.81	0.08						
209.3	100.00	0.00	2.750	99.73	0.11						
176.0	100.00	0.00	2.312	99.62	0.15						
148.0	100.00	0.00	1.945	99.47	0.24						
124.5	100.00	0.00	1.635	99.23	0.42						
104.7	100.00	0.00	1.375	98.81	0.75						
88.00	100.00	0.00	1.156	98.06	1.25						
74.00	100.00	0.00	0.972	96.81	1.88						
62.23	100.00	0.00	0.818	94.93	2.60						
52.33	100.00	0.00	0.688	92.33	3.46						
44.00	100.00	0.00	0.578	88.87	4.62						
37.00	100.00	0.00	0.486	84.25	6.49						
31.11	100.00	0.00	0.409	77.76	9.71						
26.16	100.00	0.00	0.344	68.05	15.87						
22.00	100.00	0.00	0.289	52.18	26.21						
18.50	100.00	0.00	0.243	25.97	25.97						
15.50	100.00	0.00	0.204	0.00	0.00						
11.00	100.00	0.01	0.172	0.00	0.00						
			0.145	0.00	0.00						

02/09/99 9:49

Particle Size Analysis

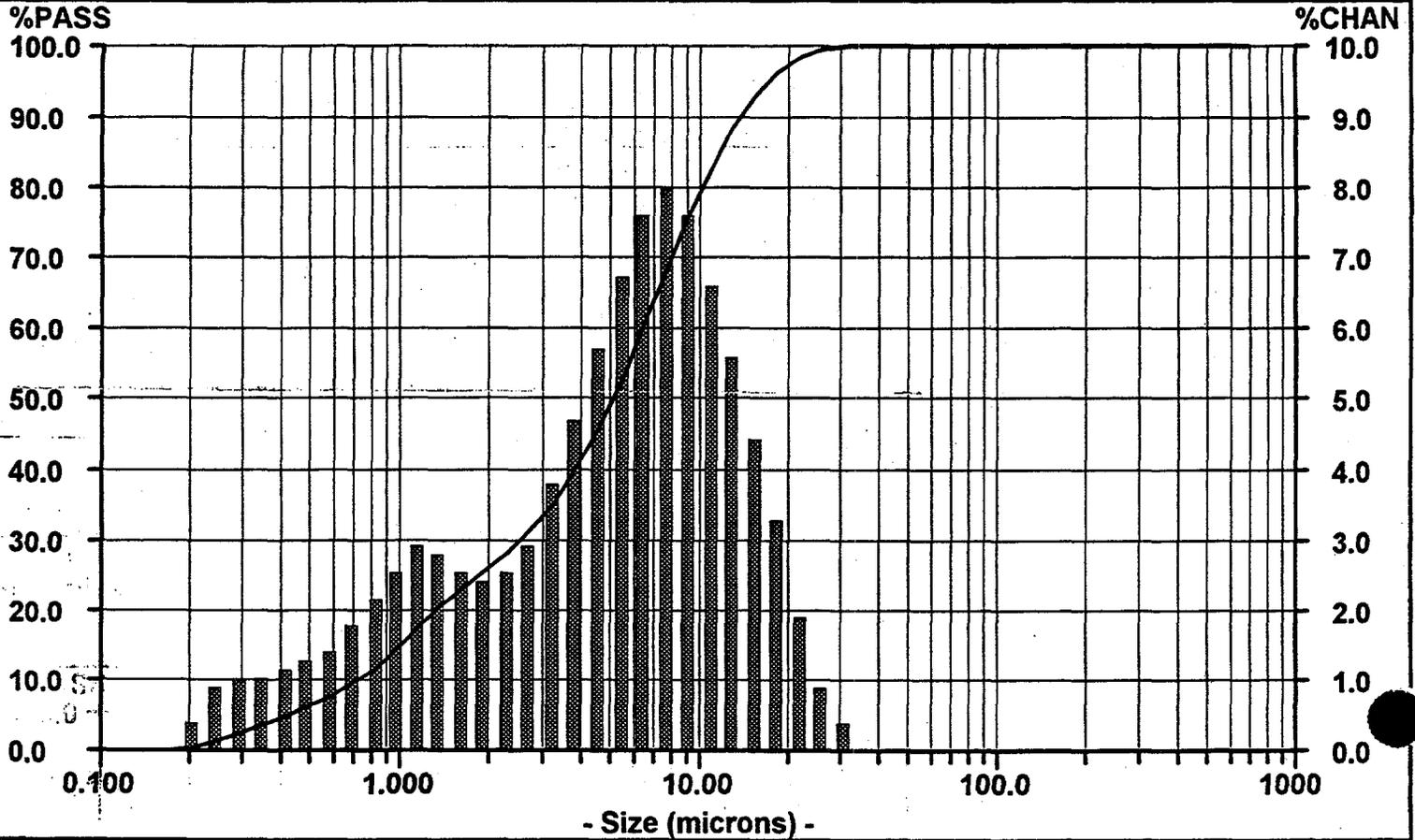
C104 Slurry DUP
Sample CUP-C104-007

Date: ~~02/09/99~~ Meas #: 00024
Time: 10:49 Pres #: 01

C104 Slurry DUP; Sample CUP-C104-007
in #8 Supernatant Simulant
60 ml/sec
Duplicate Sample

Summary	Percentiles	Dia	Vol%	Width	
mv = 6.315	10% = 0.714	60% = 6.489	6.711	77%	9.444
mn = 0.316	20% = 1.343	70% = 8.058	0.805	23%	0.951
ma = 1.859	30% = 2.568	80% = 10.21			
cs = 3.228	40% = 3.890	90% = 13.79			
sd = 5.159	50% = 5.155	95% = 17.10			

Volume Distribution



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	76.05	7.65						
592.0	100.00	0.00	7.778	68.40	8.03						
497.8	100.00	0.00	6.541	60.37	7.69						
418.6	100.00	0.00	5.500	52.68	6.85						
352.0	100.00	0.00	4.625	45.83	5.84						
296.0	100.00	0.00	3.889	39.99	4.82						
248.9	100.00	0.00	3.270	35.17	3.87						
209.3	100.00	0.00	2.750	31.30	3.09						
176.0	100.00	0.00	2.312	28.21	2.62						
148.0	100.00	0.00	1.945	25.59	2.50						
124.5	100.00	0.00	1.635	23.09	2.69						
104.7	100.00	0.00	1.375	20.40	2.96						
88.00	100.00	0.00	1.156	17.44	3.00						
74.00	100.00	0.00	0.972	14.44	2.68						
62.23	100.00	0.00	0.818	11.76	2.21						
52.33	100.00	0.00	0.688	9.55	1.80						
44.00	100.00	0.00	0.578	7.75	1.51						
37.00	100.00	0.00	0.486	6.24	1.33						
31.11	100.00	0.46	0.409	4.91	1.22						
26.16	99.54	1.09	0.344	3.69	1.18						
22.00	98.45	2.09	0.289	2.51	1.12						
18.50	96.36	3.32	0.243	1.39	0.92						
15.56	93.04	4.54	0.204	0.47	0.47						
13.08	88.50	5.69	0.172	0.00	0.00						
11.00	82.81	6.76	0.145	0.00	0.00						

Particle Size Analysis

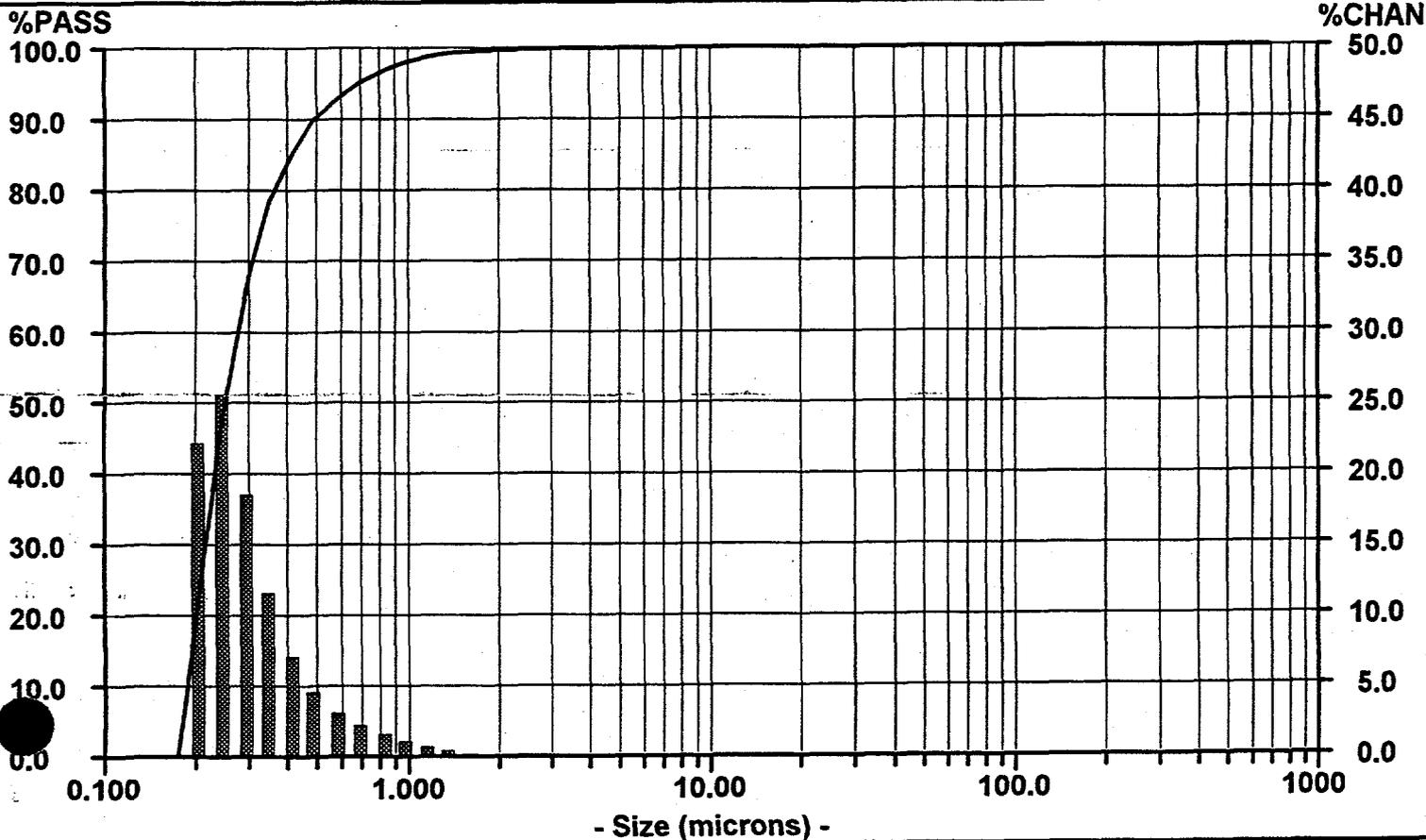
C104 Slurry DUP
Sample CUP-C104-007

Date: 02/09/89 Meas #: 00024
Time: 10:49 Pres #: 01

C104 Slurry DUP; Sample CUP-C104-007
in #8 Supernatant Simulant
60 ml/sec
Duplicate Sample

Summary	Percentiles		Dia	Vol%	Width
mv = 6.315	10% = 0.188	60% = 0.270	0.247	100%	0.196
mn = 0.316	20% = 0.201	70% = 0.302			
ma = 1.859	30% = 0.215	80% = 0.357			
cs = 3.228	40% = 0.230	90% = 0.487			
sd = 0.098	50% = 0.247	95% = 0.669			

Number Distribution



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	0.01						
497.8	100.00	0.00	6.541	99.99	0.01						
418.6	100.00	0.00	5.500	99.98	0.02						
352.0	100.00	0.00	4.625	99.96	0.02						
296.0	100.00	0.00	3.889	99.94	0.03						
248.9	100.00	0.00	3.270	99.91	0.04						
209.3	100.00	0.00	2.750	99.87	0.06						
176.0	100.00	0.00	2.312	99.81	0.08						
148.0	100.00	0.00	1.945	99.73	0.14						
124.5	100.00	0.00	1.635	99.59	0.25						
104.7	100.00	0.00	1.375	99.34	0.46						
88.00	100.00	0.00	1.156	98.88	0.78						
74.00	100.00	0.00	0.972	98.10	1.17						
62.23	100.00	0.00	0.818	96.93	1.62						
52.33	100.00	0.00	0.688	95.31	2.23						
44.00	100.00	0.00	0.578	93.08	3.14						
37.00	100.00	0.00	0.486	89.94	4.64						
31.11	100.00	0.00	0.409	85.30	7.15						
26.16	100.00	0.00	0.344	78.15	11.67						
22.00	100.00	0.00	0.289	66.48	18.61						
18.50	100.00	0.00	0.243	47.87	25.65						
15.55	100.00	0.00	0.204	22.22	22.22						
11.00	100.00	0.00	0.172	0.00	0.00						
7.00	100.00	0.00	0.145	0.00	0.00						

02109100 446

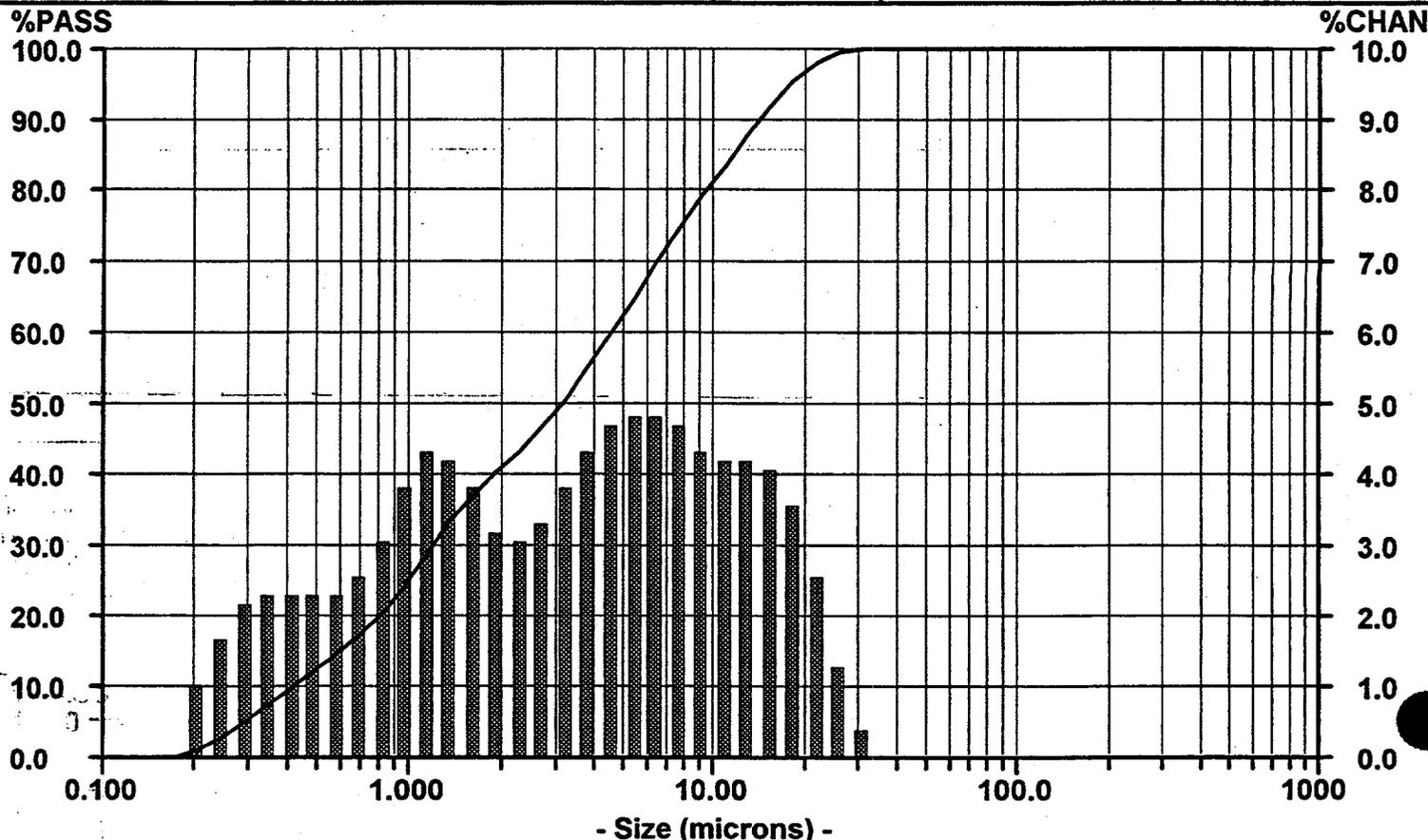
Particle Size Analysis

C104 Slurry DUP
Sample CUP-C104-007

Date: 02/09/89 - Meas #: 00028
Time: 11:01 Pres #: 01

C104 Slurry DUP; Sample CUP-C104-007
in #8 Supernatant Simulant
Sonicated at 40W for 90 sec; 60 ml/sec
Duplicate Sample
Volume distribution

Summary	Percentiles		Dia	Vol%	Width
mv = 5.386	10% = 0.412	60% = 4.638	6.600	60%	11.47
mn = 0.294	20% = 0.798	70% = 6.565	1.001	30%	0.934
ma = 1.220	30% = 1.209	80% = 9.570	0.285	10%	0.158
cs = 4.918	40% = 1.913	90% = 14.36			
sd = 5.310	50% = 3.177	95% = 17.93			



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	79.15	4.48						
592.0	100.00	0.00	7.778	74.67	4.77						
497.8	100.00	0.00	6.541	69.90	4.98						
418.6	100.00	0.00	5.500	64.92	5.00						
352.0	100.00	0.00	4.625	59.92	4.81						
296.0	100.00	0.00	3.889	55.11	4.42						
248.9	100.00	0.00	3.270	50.69	3.89						
209.3	100.00	0.00	2.750	46.80	3.38						
176.0	100.00	0.00	2.312	43.42	3.12						
148.0	100.00	0.00	1.945	40.30	3.28						
124.5	100.00	0.00	1.635	37.02	3.81						
104.7	100.00	0.00	1.375	33.21	4.38						
88.00	100.00	0.00	1.156	28.83	4.45						
74.00	100.00	0.00	0.972	24.38	3.89						
62.23	100.00	0.00	0.818	20.49	3.17						
52.33	100.00	0.00	0.688	17.32	2.66						
44.00	100.00	0.00	0.578	14.66	2.42						
37.00	100.00	0.00	0.486	12.24	2.34						
31.11	100.00	0.45	0.409	9.90	2.36						
26.16	99.55	1.34	0.344	7.54	2.39						
22.00	98.21	2.60	0.289	5.15	2.25						
18.50	95.61	3.69	0.243	2.90	1.76						
15.56	91.92	4.19	0.204	1.14	1.14						
13.08	87.73	4.28	0.172	0.00	0.00						
11.00	83.45	4.30	0.145	0.00	0.00						

Particle Size Analysis

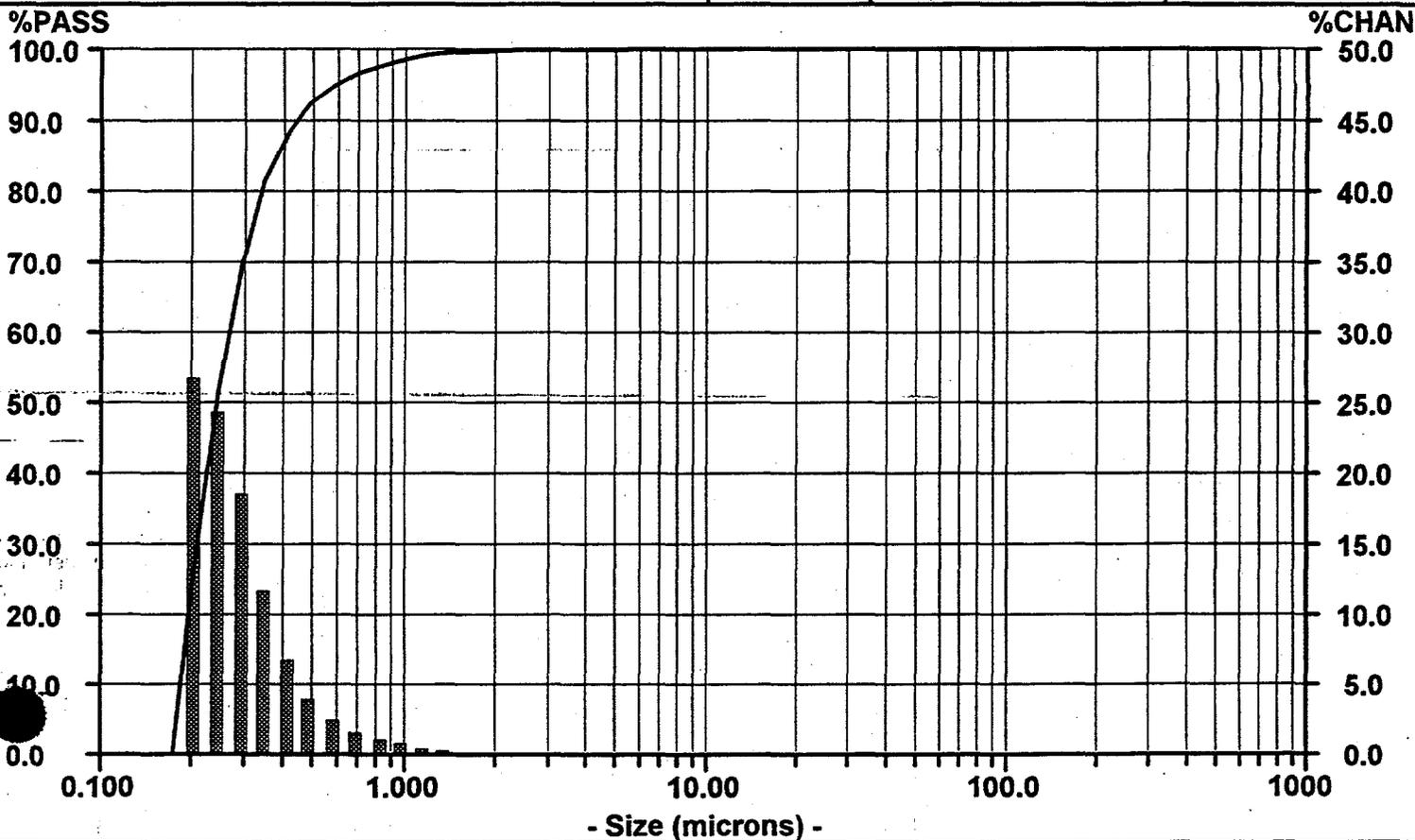
C104 Slurry DUP
Sample CUP-C104-007

Date: 02/09/89 Meas #: 00028
Time: 11:01 Pres #: 01

C104 Slurry DUP; Sample CUP-C104-007
in #8 Supernatant Simulant
Sonicated at 40W for 90 sec; 60 ml/sec
Dup Sonication -- Duplicate Sample

Summary	Percentiles		Dia	Vol%	Width
mv = 5.386	10% = 0.185	60% = 0.262	0.241	100%	0.169
mn = 0.294	20% = 0.197	70% = 0.290			
ma = 1.220	30% = 0.208	80% = 0.334			
cs = 4.918	40% = 0.223	90% = 0.432			
sd = 0.085	50% = 0.241	95% = 0.576			

Number distribution



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	0.01						
352.0	100.00	0.00	4.625	99.99	0.01						
296.0	100.00	0.00	3.889	99.98	0.01						
248.9	100.00	0.00	3.270	99.97	0.02						
209.3	100.00	0.00	2.750	99.95	0.03						
176.0	100.00	0.00	2.312	99.92	0.05						
148.0	100.00	0.00	1.945	99.87	0.09						
124.5	100.00	0.00	1.635	99.78	0.17						
104.7	100.00	0.00	1.375	99.61	0.34						
88.00	100.00	0.00	1.156	99.27	0.57						
74.00	100.00	0.00	0.972	98.70	0.84						
62.23	100.00	0.00	0.818	97.86	1.16						
52.33	100.00	0.00	0.688	96.70	1.64						
44.00	100.00	0.00	0.578	95.06	2.50						
37.00	100.00	0.00	0.486	92.56	4.06						
31.11	100.00	0.00	0.409	88.50	6.89						
26.16	100.00	0.00	0.344	81.61	11.76						
22.00	100.00	0.00	0.289	69.85	18.60						
18.50	100.00	0.00	0.243	51.25	24.42						
15.75	100.00	0.00	0.204	26.83	26.83						
13.75	100.00	0.00	0.172	0.00	0.00						
11.00	100.00	0.00	0.145	0.00	0.00						

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Particle Size Analysis

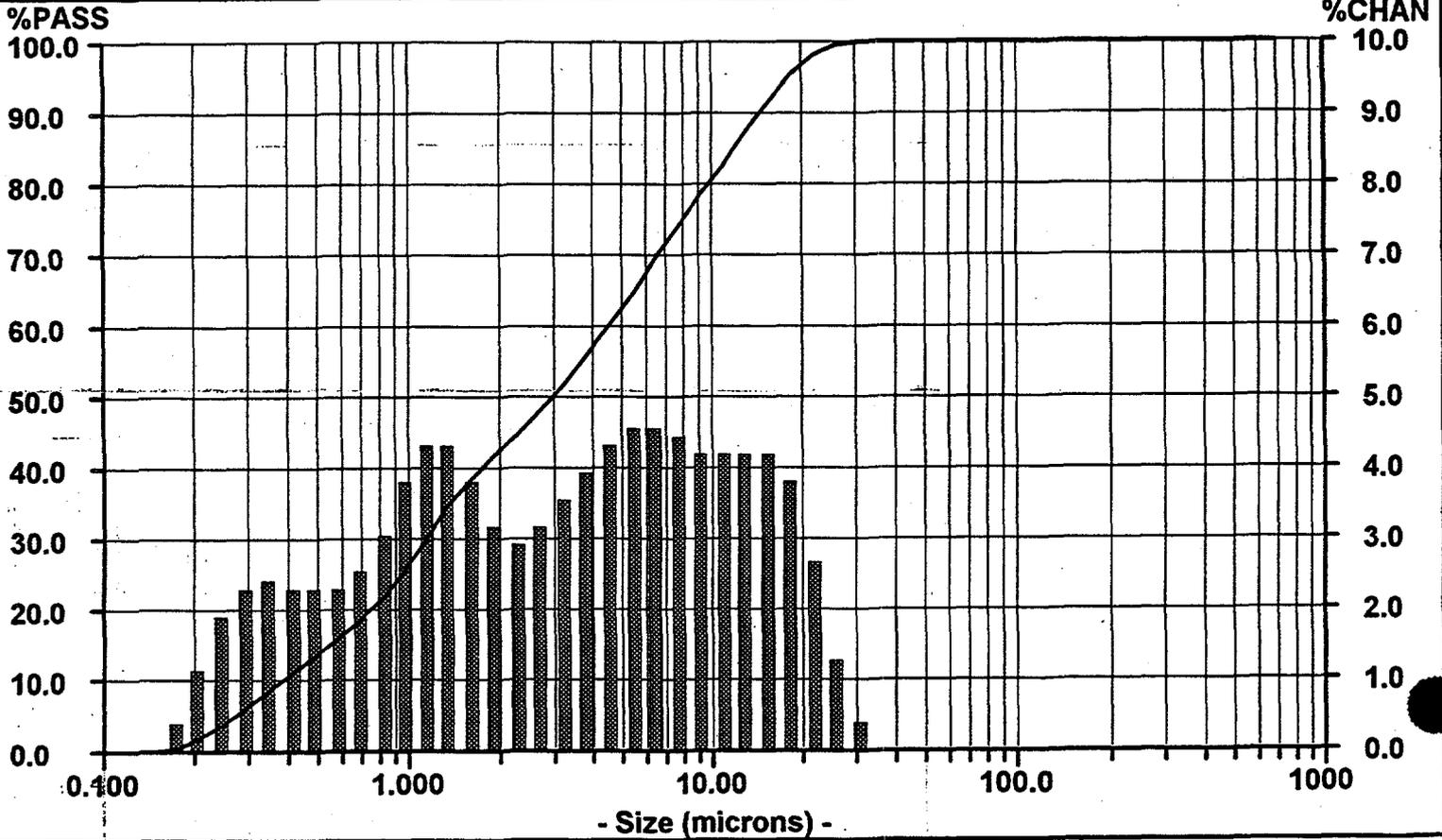
C104 Slurry DUP
Sample CUP-C104-007

Date: 02/09/89 Meas #: 00032
Time: 11:11 Pres #: 01

C104 Slurry DUP; Sample CUP-C104-007
in #8 Supernatant Simulant
Sonicated at 40W for 90 sec; 60 ml/sec
2nd Sonication — Duplicate Sample

Volume Distribution

Summary	Percentiles		Dia	Vol%	Width
mv = 5.385	10% = 0.375	60% = 4.561	14.19	21%	9.018
mn = 0.268	20% = 0.740	70% = 6.634	4.595	37%	4.654
ma = 1.132	30% = 1.144	80% = 9.819	1.002	31%	0.930
cs = 5.301	40% = 1.759	90% = 14.61	0.278	11%	0.150
sd = 5.480	50% = 3.004	95% = 18.08			



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	78.53	4.36						
592.0	100.00	0.00	7.778	74.17	4.54						
497.8	100.00	0.00	6.541	69.63	4.64						
418.6	100.00	0.00	5.500	64.99	4.62						
352.0	100.00	0.00	4.625	60.37	4.43						
296.0	100.00	0.00	3.889	55.94	4.09						
248.9	100.00	0.00	3.270	51.85	3.66						
209.3	100.00	0.00	2.750	48.19	3.25						
176.0	100.00	0.00	2.312	44.94	3.08						
148.0	100.00	0.00	1.945	41.86	3.29						
124.5	100.00	0.00	1.635	38.57	3.85						
104.7	100.00	0.00	1.375	34.72	4.44						
88.00	100.00	0.00	1.156	30.28	4.48						
74.00	100.00	0.00	0.972	25.80	3.89						
62.23	100.00	0.00	0.818	21.91	3.16						
52.33	100.00	0.00	0.688	18.75	2.66						
44.00	100.00	0.00	0.578	16.09	2.44						
37.00	100.00	0.00	0.486	13.65	2.41						
31.11	100.00	0.43	0.409	11.24	2.48						
26.16	99.57	1.36	0.344	8.76	2.57						
22.00	98.21	2.73	0.289	6.19	2.44						
18.50	95.48	3.90	0.243	3.75	1.90						
15.56	91.58	4.39	0.204	1.85	1.28						
13.08	87.19	4.37	0.172	0.57	0.57						
11.00	82.82	4.29	0.145	0.00	0.00						

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Particle Size Analysis

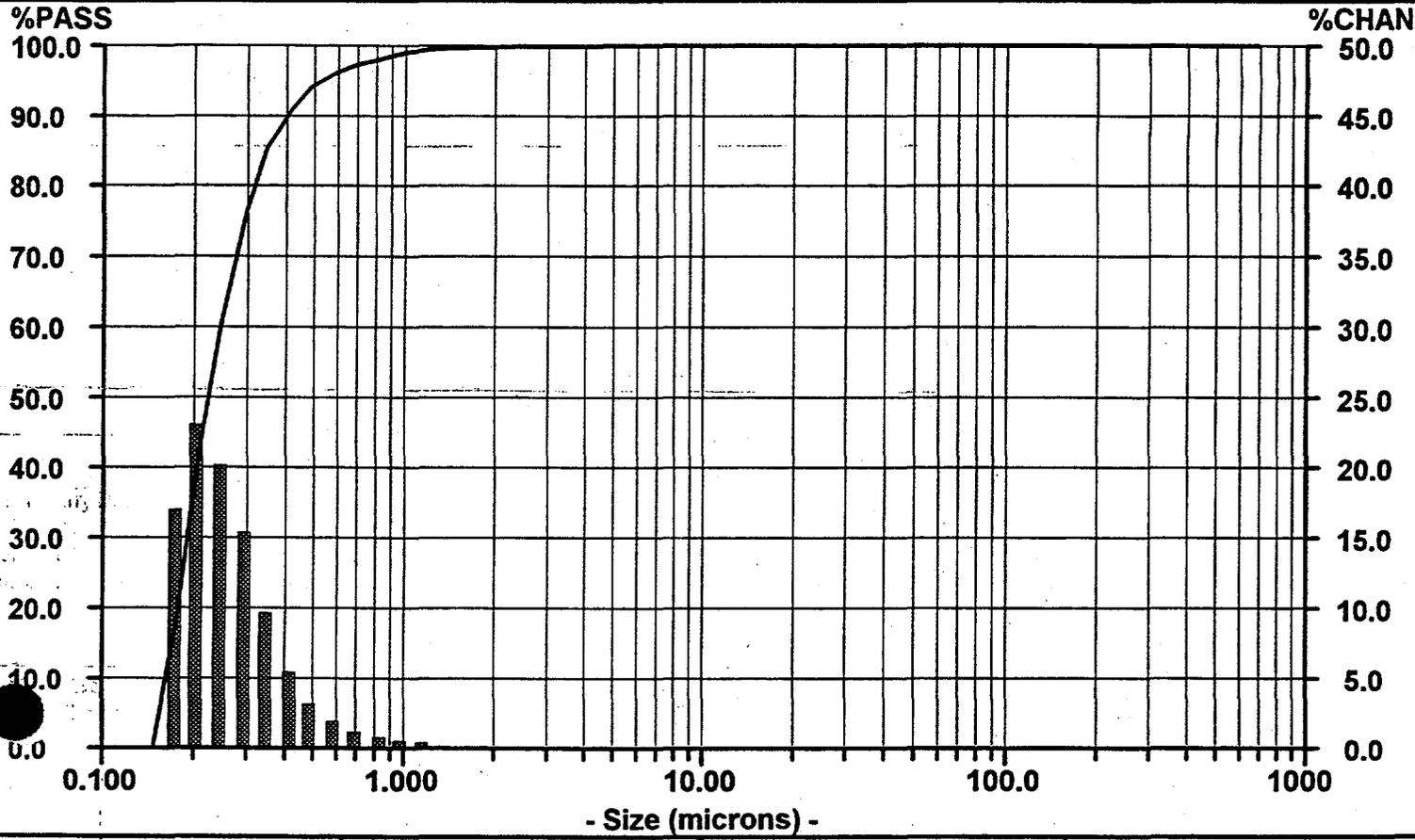
C104 Slurry DUP
Sample CUP-C104-007

Date: 02/09/89 - Meas #: 00032
Time: 11:11 Pres #: 01

C104 Slurry DUP; Sample CUP-C104-007
in #8 Supematant Simulant
Sonicated at 40W for 90 sec; 60 ml/sec
2nd Sonication -- Duplicate Sample

Summary	Percentiles		Dia	Vol%	Width
mv = 5.385	10% = 0.163	60% = 0.242	0.221	100%	0.162
mn = 0.268	20% = 0.176	70% = 0.269			
ma = 1.132	30% = 0.189	80% = 0.308			
cs = 5.301	40% = 0.204	90% = 0.392			
sd = 0.081	50% = 0.221	95% = 0.515			

Number Distribution



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	0.01						
248.9	100.00	0.00	3.270	99.98	0.02						
209.3	100.00	0.00	2.750	99.96	0.02						
176.0	100.00	0.00	2.312	99.94	0.04						
148.0	100.00	0.00	1.945	99.90	0.07						
124.5	100.00	0.00	1.635	99.83	0.13						
104.7	100.00	0.00	1.375	99.70	0.26						
88.00	100.00	0.00	1.156	99.44	0.44						
74.00	100.00	0.00	0.972	99.00	0.65						
62.23	100.00	0.00	0.818	98.35	0.88						
52.33	100.00	0.00	0.688	97.47	1.25						
44.00	100.00	0.00	0.578	96.22	1.93						
37.00	100.00	0.00	0.486	94.29	3.20						
31.11	100.00	0.00	0.409	91.09	5.55						
26.16	100.00	0.00	0.344	85.54	9.70						
22.00	100.00	0.00	0.289	75.84	15.47						
18.50	100.00	0.00	0.243	60.37	20.21						
15.50	100.00	0.00	0.204	40.16	23.11						
13.00	100.00	0.00	0.172	17.05	17.05						
11.00	100.00	0.00	0.145	0.00	0.00						

Particle Size Distribution Plots
For Washed Slurry: Sample CUF-C104-007DUP
Replicate No: 2

Particle Size Analysis

C104 Slurry DUP2
Sample CUP-C104-007

Date: 02/08/89 Meas #: 00036
Time: 11:33 Pres #: 01

C104 Slurry DUP2 ; Sample CUP-C104-007
in #8 Supernatant Simulant
40 ml/sec

Summary

mv = 11.48
mn = 0.344
ma = 2.796
cs = 2.146
sd = 9.530

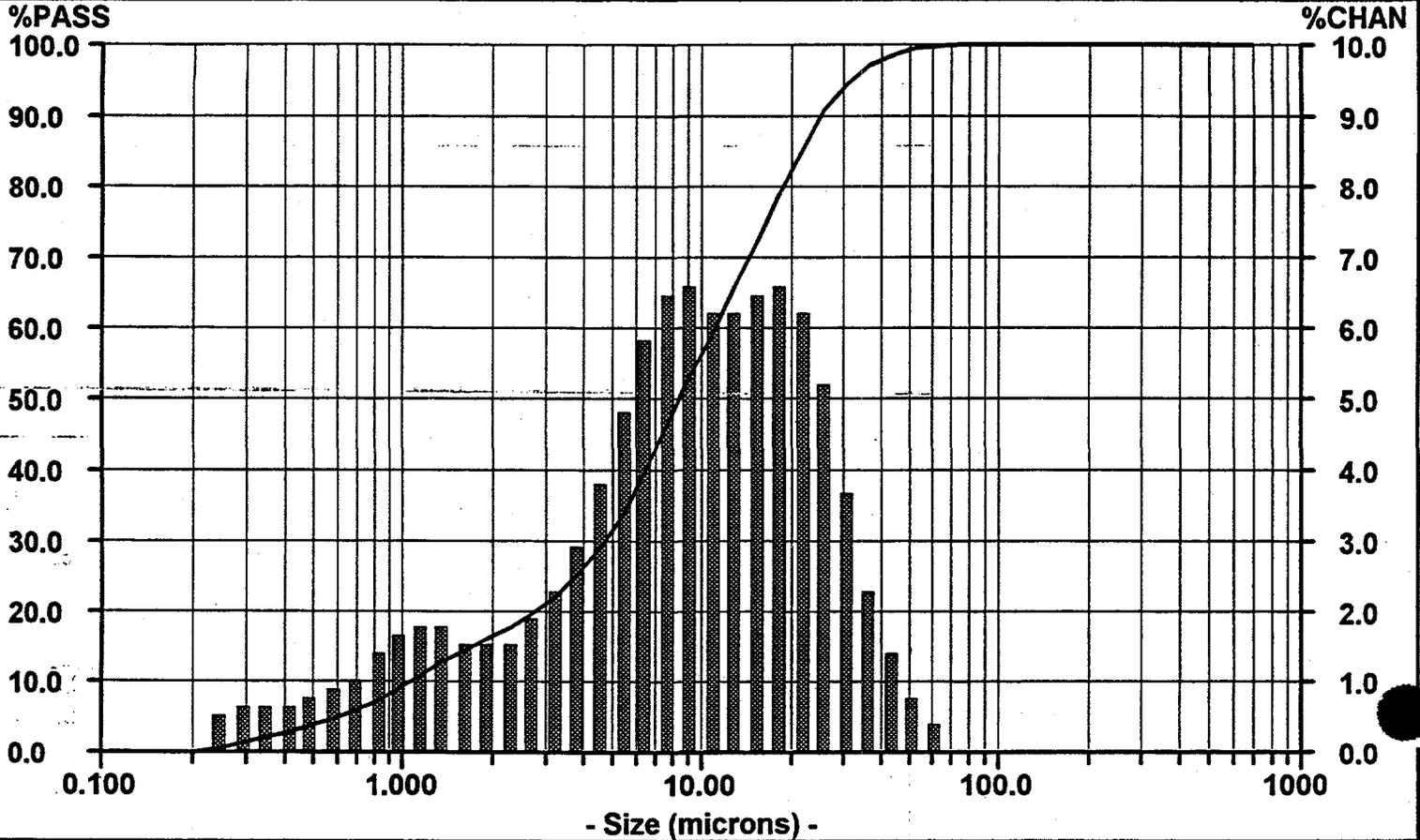
Percentiles

10% = 1.052 60% = 11.12
20% = 2.798 70% = 14.58
30% = 4.768 80% = 18.85
40% = 6.530 90% = 25.29
50% = 8.495 95% = 31.76

Dia Vol% Width

18.75 40% 16.20
6.013 45% 5.878
0.870 14% 0.857
0.246 1% 0.0

Volume Distribution



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	53.23	6.61						
692.0	100.00	0.00	7.778	46.62	6.56						
497.8	100.00	0.00	6.541	40.06	5.93						
418.6	100.00	0.00	5.500	34.13	4.92						
352.0	100.00	0.00	4.625	29.21	3.91						
296.0	100.00	0.00	3.889	25.30	3.08						
248.9	100.00	0.00	3.270	22.22	2.44						
209.3	100.00	0.00	2.750	19.78	1.96						
176.0	100.00	0.00	2.312	17.82	1.68						
148.0	100.00	0.00	1.945	16.14	1.60						
124.5	100.00	0.00	1.635	14.54	1.68						
104.7	100.00	0.00	1.375	12.86	1.83						
88.00	100.00	0.00	1.156	11.03	1.88						
74.00	100.00	0.11	0.972	9.15	1.73						
62.23	99.89	0.46	0.818	7.42	1.46						
52.33	99.43	0.85	0.688	5.96	1.19						
44.00	98.58	1.48	0.578	4.77	0.97						
37.00	97.10	2.44	0.486	3.80	0.83						
31.11	94.66	3.74	0.409	2.97	0.75						
26.16	90.92	5.24	0.344	2.22	0.75						
22.00	85.68	6.39	0.289	1.47	0.78						
18.50	79.29	6.79	0.243	0.69	0.69						
15.56	72.50	6.57	0.204	0.00	0.00						
13.08	65.93	6.32	0.172	0.00	0.00						
11.00	59.61	6.38	0.145	0.00	0.00						

02-107/00 9/06

Particle Size Analysis

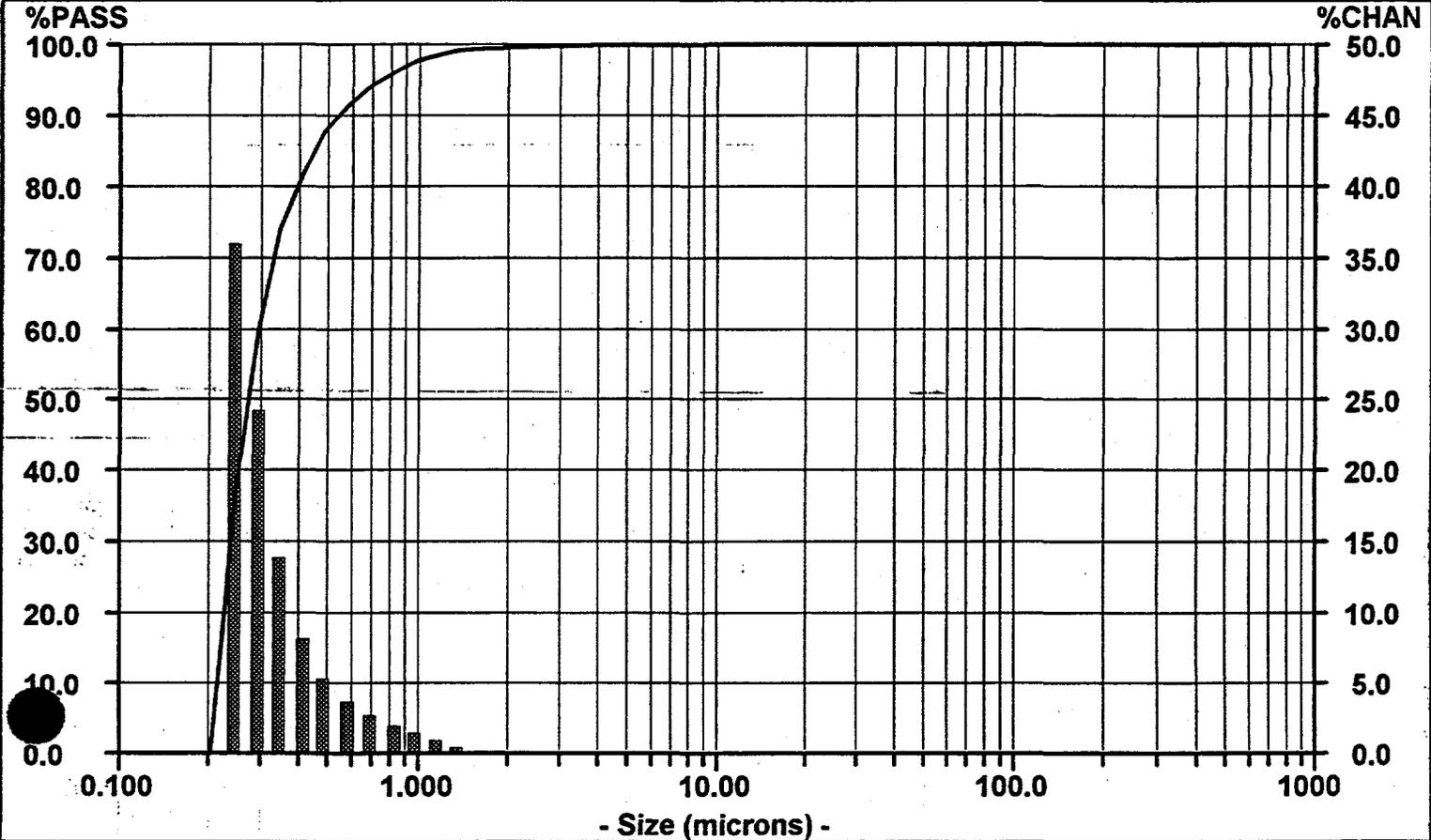
C104 Slurry DUP2
Sample CUP-C104-007

Date: ~~02/09/89~~ Meas #: 00036
Time: 11:33 Pres #: 01

C104 Slurry DUP2 ; Sample CUP-C104-007
in #8 Supernatant Simulant
40 ml/sec

Summary	Percentiles		Dia	Vol%	Width
mv = 11.48	10% = 0.217	60% = 0.288	0.265	100%	0.205
mn = 0.344	20% = 0.227	70% = 0.323			
ma = 2.796	30% = 0.237	80% = 0.385			
cs = 2.146	40% = 0.249	90% = 0.534			
sd = 0.102	50% = 0.265	95% = 0.724			

Number Distribution



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	100.00	0.01						
592.0	100.00	0.00	7.778	99.99	0.01						
497.8	100.00	0.00	6.541	99.98	0.02						
418.6	100.00	0.00	5.500	99.96	0.02						
352.0	100.00	0.00	4.625	99.94	0.03						
296.0	100.00	0.00	3.889	99.91	0.04						
248.9	100.00	0.00	3.270	99.87	0.05						
209.3	100.00	0.00	2.750	99.82	0.07						
176.0	100.00	0.00	2.312	99.75	0.10						
148.0	100.00	0.00	1.945	99.65	0.16						
124.5	100.00	0.00	1.635	99.49	0.29						
104.7	100.00	0.00	1.375	99.20	0.53						
88.00	100.00	0.00	1.156	98.67	0.91						
74.00	100.00	0.00	0.972	97.76	1.41						
62.23	100.00	0.00	0.818	96.35	2.00						
52.33	100.00	0.00	0.688	94.35	2.75						
44.00	100.00	0.00	0.578	91.60	3.77						
37.00	100.00	0.00	0.486	87.83	5.42						
31.11	100.00	0.00	0.409	82.41	8.23						
26.16	100.00	0.00	0.344	74.18	13.89						
22.00	100.00	0.00	0.289	60.29	24.26						
18.50	100.00	0.00	0.243	36.03	36.03						
15.50	100.00	0.00	0.204	0.00	0.00						
13.00	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

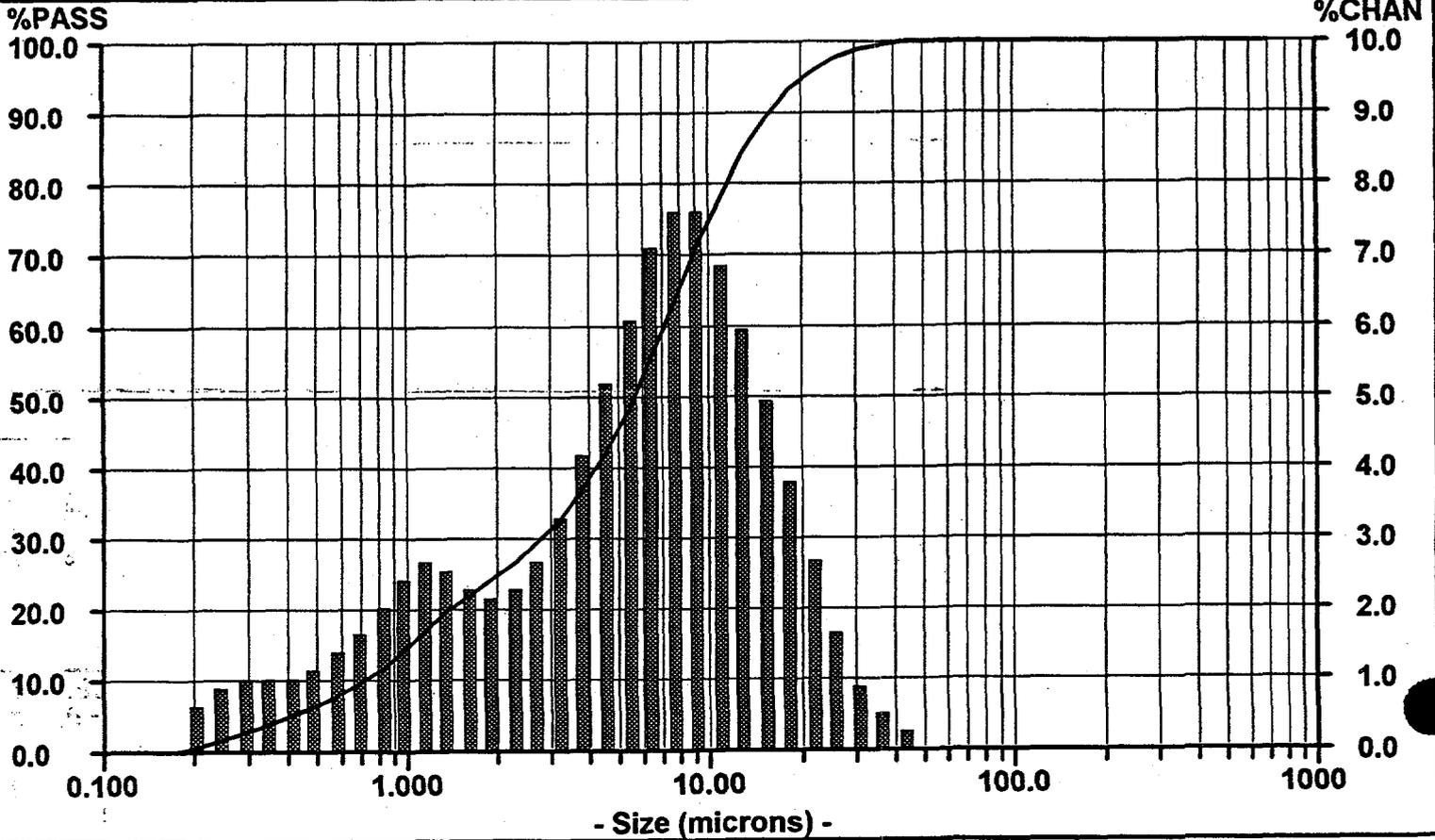
C104 Slurry DUP2
Sample CUP-C104-007

Date: 02/08/89 Meas #: 00040
Time: 11:47 Pres #: 01

C104 Slurry DUP2 ; Sample CUP-C104-007
In #8 Supernatant Simulant
60 ml/sec

Summary	Percentiles		Dia	Vol%	Width
mv = 7.241	10% = 0.704	60% = 7.166	7.340	78%	11.02
mn = 0.296	20% = 1.401	70% = 8.981	0.770	22%	0.955
ma = 1.873	30% = 2.835	80% = 11.53			
cs = 3.203	40% = 4.271	90% = 15.93			
sd = 5.930	50% = 5.668	95% = 20.37			

Volume Distribution
VUP



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	71.28	7.61						
592.0	100.00	0.00	7.778	63.67	7.69						
497.8	100.00	0.00	6.541	55.98	7.16						
418.6	100.00	0.00	5.500	48.82	6.27						
352.0	100.00	0.00	4.625	42.55	5.29						
296.0	100.00	0.00	3.889	37.26	4.34						
248.9	100.00	0.00	3.270	32.92	3.47						
209.3	100.00	0.00	2.750	29.45	2.76						
176.0	100.00	0.00	2.312	26.69	2.33						
148.0	100.00	0.00	1.945	24.36	2.23						
124.5	100.00	0.00	1.635	22.13	2.40						
104.7	100.00	0.00	1.375	19.73	2.66						
88.00	100.00	0.00	1.156	17.07	2.73						
74.00	100.00	0.00	0.972	14.34	2.50						
62.23	100.00	0.00	0.818	11.84	2.11						
52.33	100.00	0.00	0.688	9.73	1.75						
44.00	100.00	0.38	0.578	7.98	1.48						
37.00	99.62	0.65	0.486	6.50	1.29						
31.11	98.97	1.09	0.409	5.21	1.19						
26.16	97.88	1.78	0.344	4.02	1.16						
22.00	96.10	2.76	0.289	2.86	1.13						
18.50	93.34	3.92	0.243	1.73	0.99						
15.56	89.42	5.06	0.204	0.74	0.74						
13.08	84.36	6.09	0.172	0.00	0.00						
11.00	78.27	6.99	0.145	0.00	0.00						

02/07/00 G166

Particle Size Analysis

C104 Slurry DUP2
Sample CUP-C104-007

Date: 02/09/89 Meas #: 00040
Time: 11:47 Pres #: 01

C104 Slurry DUP2 ; Sample CUP-C104-007
in #8 Supematant Simulant
60 ml/sec

Summary

mv = 7.241
mn = 0.296
ma = 1.873
cs = 3.203
sd = 0.088

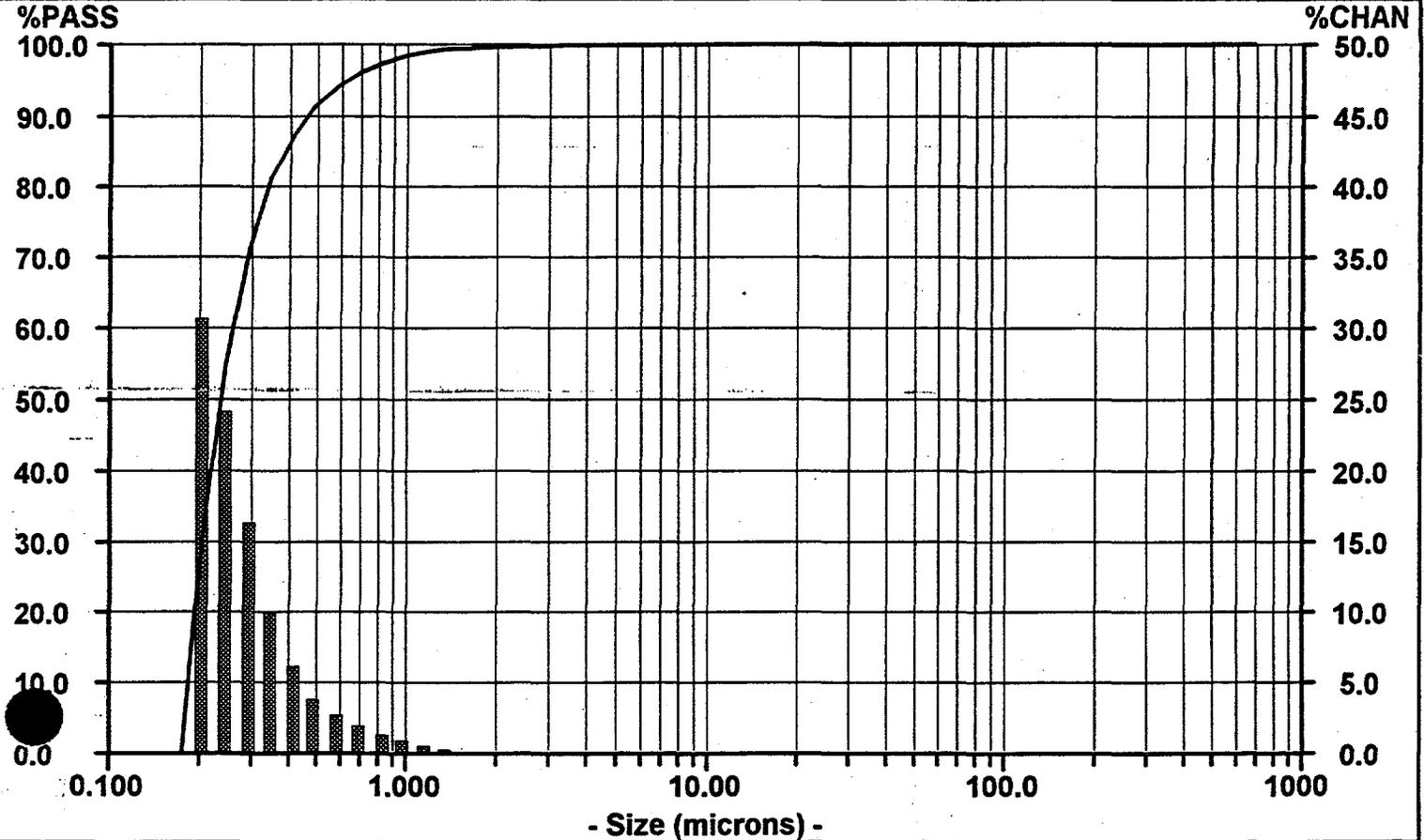
Percentiles

10% = 0.184 60% = 0.255
20% = 0.194 70% = 0.284
30% = 0.203 80% = 0.333
40% = 0.217 90% = 0.450
50% = 0.234 95% = 0.614

Dia Vol% Width

0.234 100% 0.176

Number Distribution



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	0.01						
497.8	100.00	0.00	6.541	99.99	0.01						
418.6	100.00	0.00	5.500	99.98	0.01						
352.0	100.00	0.00	4.625	99.97	0.02						
296.0	100.00	0.00	3.889	99.95	0.03						
248.9	100.00	0.00	3.270	99.92	0.03						
209.3	100.00	0.00	2.750	99.89	0.05						
176.0	100.00	0.00	2.312	99.84	0.07						
148.0	100.00	0.00	1.945	99.77	0.11						
124.5	100.00	0.00	1.635	99.66	0.19						
104.7	100.00	0.00	1.375	99.47	0.36						
88.00	100.00	0.00	1.156	99.11	0.62						
74.00	100.00	0.00	0.972	98.49	0.96						
62.23	100.00	0.00	0.818	97.53	1.36						
52.33	100.00	0.00	0.688	96.17	1.90						
44.00	100.00	0.00	0.578	94.27	2.70						
37.00	100.00	0.00	0.486	91.57	3.95						
31.11	100.00	0.00	0.409	87.62	6.13						
26.16	100.00	0.00	0.344	81.49	10.07						
22.00	100.00	0.00	0.289	71.42	16.48						
18.50	100.00	0.00	0.243	54.94	24.23						
15.50	100.00	0.00	0.204	30.71	30.71						
11.00	100.00	0.00	0.172	0.00	0.00						
			0.145	0.00	0.00						

02409100 SKD

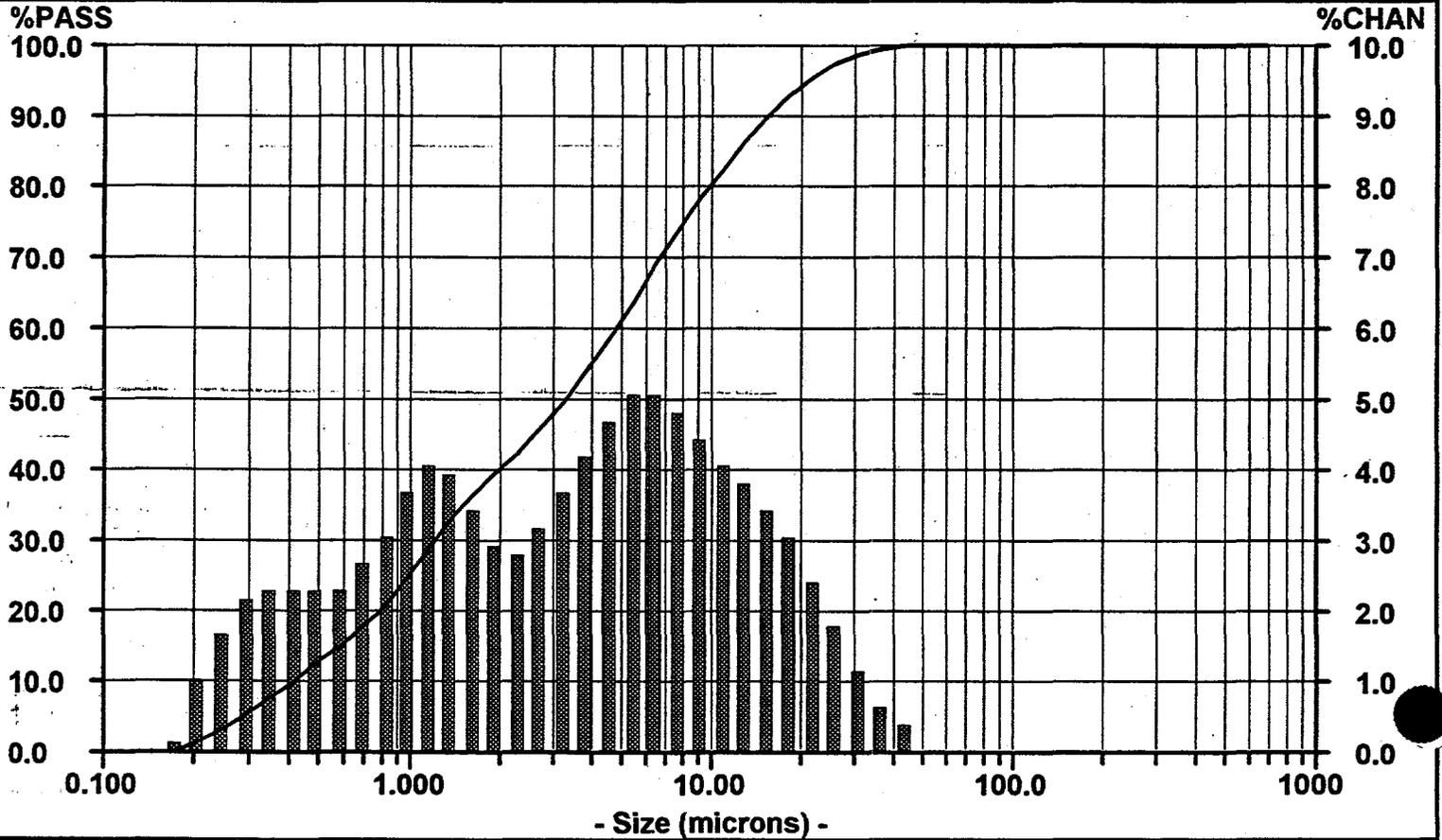
Particle Size Analysis

C104 Slurry DUP2
Sample CUP-C104-007

Date: 02/09/89 Meas #: 00044
Time: 12:01 Pres #: 01

C104 Slurry DUP2 ; Sample CUP-C104-007
in #8 Supematant Simulant
60 ml/sec
1st Sonicare 40W, 90 sec.
Volume Distribution

Summary	Percentiles	Dia	Vol%	Width	
mv = 5.918	10% = 0.404	60% = 4.830	6.714	60%	12.64
mn = 0.280	20% = 0.779	70% = 6.763	0.770	40%	1.083
ma = 1.205	30% = 1.210	80% = 9.854			
cs = 4.978	40% = 1.995	90% = 15.60			
sd = 5.554	50% = 3.340	95% = 20.91			



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	78.47	4.51						
592.0	100.00	0.00	7.778	73.96	4.93						
497.8	100.00	0.00	6.541	69.03	5.15						
418.6	100.00	0.00	5.500	63.88	5.15						
352.0	100.00	0.00	4.625	58.73	4.86						
296.0	100.00	0.00	3.889	53.87	4.37						
248.9	100.00	0.00	3.270	49.50	3.76						
209.3	100.00	0.00	2.750	45.74	3.22						
176.0	100.00	0.00	2.312	42.52	2.95						
148.0	100.00	0.00	1.945	39.57	3.06						
124.5	100.00	0.00	1.635	36.51	3.54						
104.7	100.00	0.00	1.375	32.97	4.08						
88.00	100.00	0.00	1.156	28.89	4.19						
74.00	100.00	0.00	0.972	24.70	3.76						
62.23	100.00	0.00	0.818	20.94	3.16						
52.33	100.00	0.00	0.688	17.78	2.72						
44.00	100.00	0.43	0.578	15.06	2.49						
37.00	99.57	0.76	0.486	12.57	2.40						
31.11	98.81	1.24	0.409	10.17	2.38						
26.16	97.57	1.88	0.344	7.79	2.37						
22.00	95.69	2.57	0.289	5.42	2.22						
18.50	93.12	3.17	0.243	3.20	1.74						
15.56	89.95	3.55	0.204	1.46	1.18						
13.08	86.40	3.82	0.172	0.28	0.28						
11.00	82.58	4.11	0.145	0.00	0.00						

Particle Size Analysis

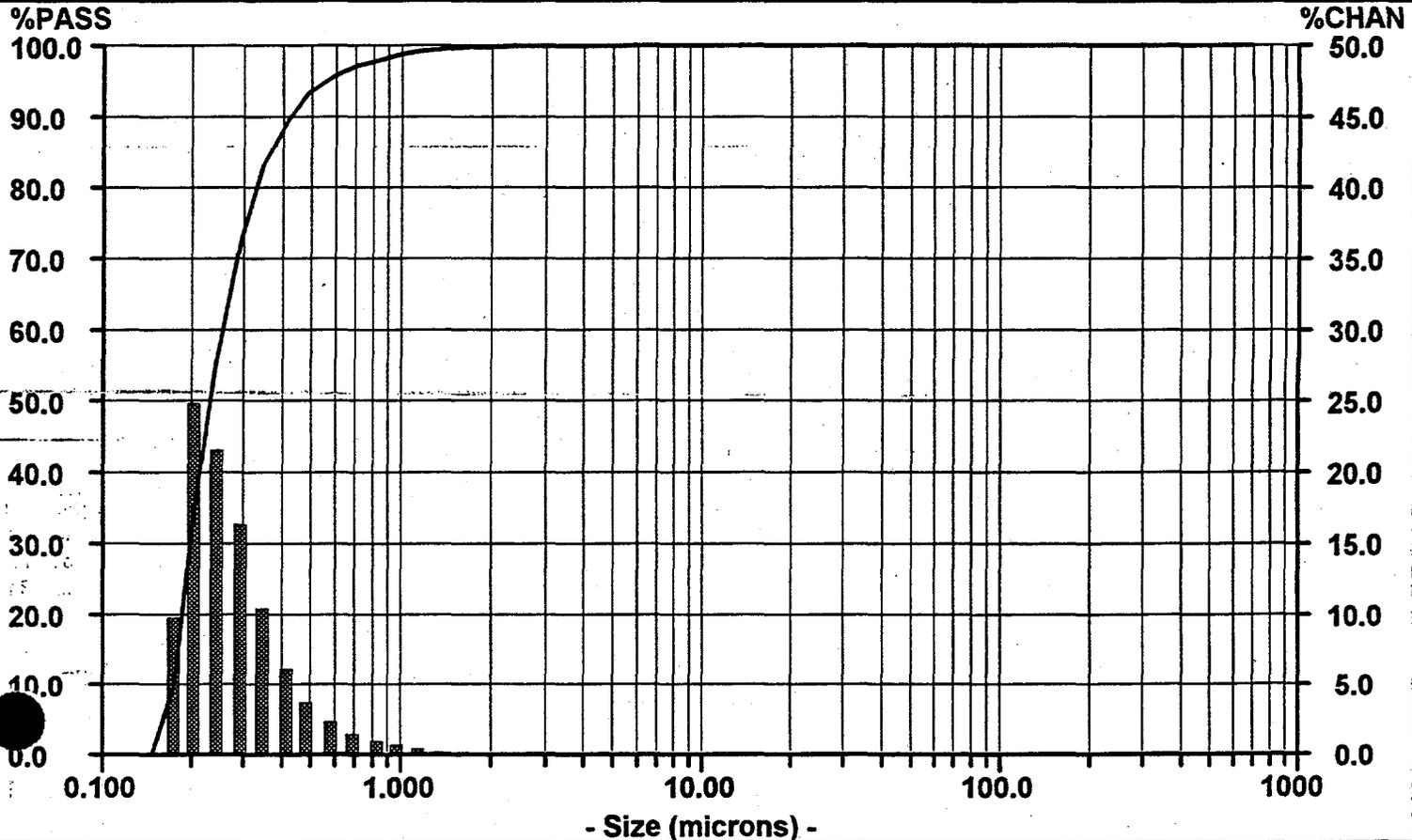
C104 Slurry DUP2
Sample CUP-C104-007

Date: 02/09/89 Meas #: 00044
Time: 12:01 Pres #: 01

C104 Slurry DUP2 ; Sample CUP-C104-007
In #8 Supernatant Simulant
60 ml/sec
1st Sonicate 40W, 90 sec.

Summary	Percentiles	Dia	Vol%	Width	
mv = 5.918	10% = 0.172	60% = 0.252	0.230	100%	0.169
mn = 0.280	20% = 0.185	70% = 0.279			
ma = 1.205	30% = 0.198	80% = 0.322			
cs = 4.978	40% = 0.212	90% = 0.416			
sd = 0.085	50% = 0.230	95% = 0.548			

Number Distribution



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	0.01						
352.0	100.00	0.00	4.625	99.99	0.01						
296.0	100.00	0.00	3.889	99.98	0.01						
248.9	100.00	0.00	3.270	99.97	0.02						
209.3	100.00	0.00	2.750	99.95	0.03						
176.0	100.00	0.00	2.312	99.92	0.04						
148.0	100.00	0.00	1.945	99.88	0.07						
124.5	100.00	0.00	1.635	99.81	0.16						
104.7	100.00	0.00	1.375	99.66	0.28						
88.00	100.00	0.00	1.156	99.38	0.49						
74.00	100.00	0.00	0.972	98.89	0.73						
62.23	100.00	0.00	0.818	98.16	1.03						
52.33	100.00	0.00	0.688	97.13	1.50						
44.00	100.00	0.00	0.578	95.63	2.31						
37.00	100.00	0.00	0.486	93.32	3.74						
31.11	100.00	0.00	0.409	89.58	6.23						
26.16	100.00	0.00	0.344	83.35	10.47						
22.00	100.00	0.00	0.289	72.88	16.48						
18.50	100.00	0.00	0.243	56.40	21.67						
15.56	100.00	0.00	0.204	34.73	24.93						
11.00	100.00	0.00	0.172	9.80	9.80						
7.00	100.00	0.00	0.145	0.00	0.00						

Particle Size Analysis

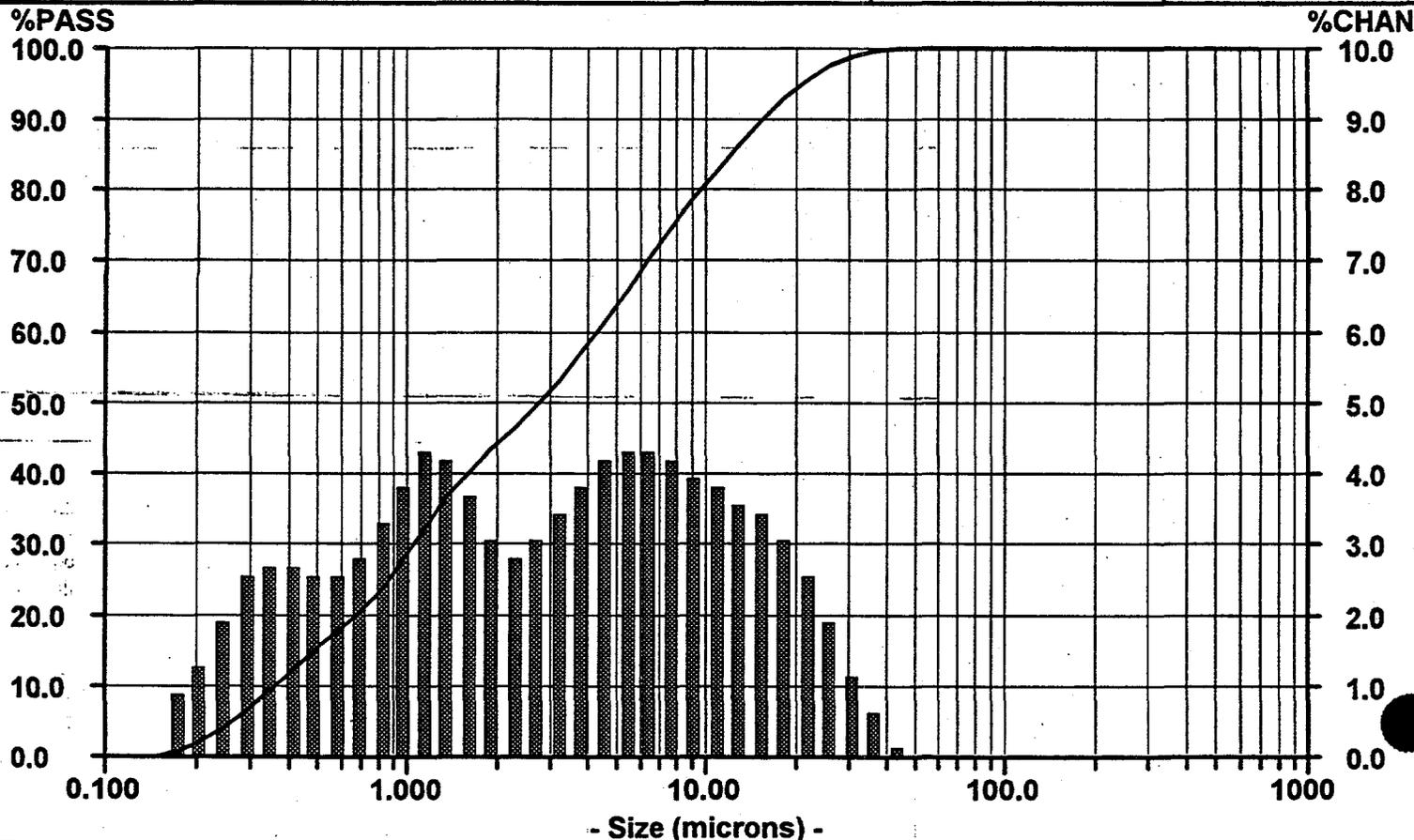
C104 Slurry DUP2
Sample CUP-C104-007

Date: 02/09/89 Meas #: 00048
Time: 12:12 Pres #: 01

C104 Slurry DUP2 ; Sample CUP-C104-007
in #8 Supernatant Simulant
60 ml/sec
2nd Sonicat 40W, 90 sec.

Summary	Percentiles			Dia	Vol%	Width
mv = 5.656	10% = 0.351	60% = 4.337	6.867	56%	13.21	
mn = 0.258	20% = 0.661	70% = 6.395	0.977	31%	0.934	
ma = 1.058	30% = 1.056	80% = 9.686	0.277	13%	0.162	
cs = 5.671	40% = 1.601	90% = 15.56				
sd = 5.560	50% = 2.783	95% = 20.81				

Volume Distribution



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	78.96	4.07						
692.0	100.00	0.00	7.778	74.89	4.31						
497.8	100.00	0.00	6.541	70.58	4.47						
418.6	100.00	0.00	5.500	66.11	4.48						
352.0	100.00	0.00	4.625	61.63	4.31						
296.0	100.00	0.00	3.889	57.32	3.99						
248.9	100.00	0.00	3.270	53.33	3.56						
209.3	100.00	0.00	2.750	49.77	3.16						
176.0	100.00	0.00	2.312	46.61	2.99						
148.0	100.00	0.00	1.945	43.62	3.19						
124.5	100.00	0.00	1.635	40.43	3.75						
104.7	100.00	0.00	1.375	36.68	4.34						
88.00	100.00	0.00	1.156	32.34	4.43						
74.00	100.00	0.00	0.972	27.91	3.95						
62.23	100.00	0.00	0.818	23.96	3.30						
52.33	100.00	0.00	0.688	20.66	2.86						
44.00	100.00	0.27	0.578	17.80	2.69						
37.00	99.73	0.78	0.486	15.11	2.68						
31.11	98.95	1.27	0.409	12.43	2.74						
26.16	97.68	1.91	0.344	9.69	2.79						
22.00	95.77	2.60	0.289	6.90	2.61						
18.50	93.17	3.17	0.243	4.29	2.00						
15.56	90.00	3.51	0.204	2.29	1.38						
13.08	86.49	3.69	0.172	0.91	0.91						
11.00	82.80	3.84	0.145	0.00	0.00						

Particle Size Analysis

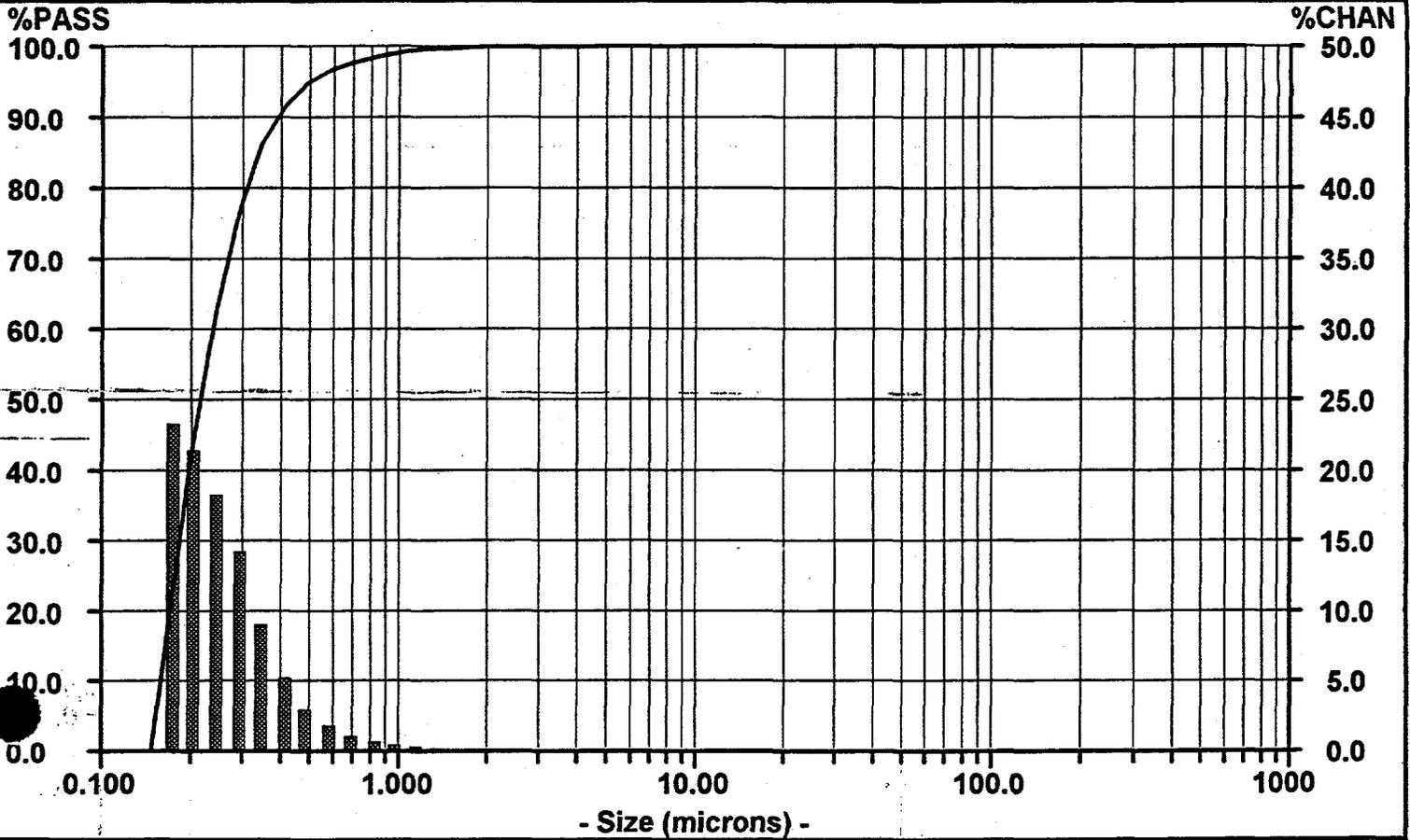
C104 Slurry DUP2
Sample CUP-C104-007

Date: 02/09/89 - Meas #: 00048
Time: 12:12 Pres #: 01

C104 Slurry DUP2 ; Sample CUP-C104-007
in #8 Supematant Simulant
60 ml/sec
2nd Sonicat 40W, 90 sec.

Summary	Percentiles		Dia	Vol%	Width
mv = 5.656	10% = 0.158	60% = 0.236	0.214	100%	0.161
mn = 0.258	20% = 0.168	70% = 0.262			
ma = 1.058	30% = 0.180	80% = 0.302			
cs = 5.671	40% = 0.196	90% = 0.382			
sd = 0.080	50% = 0.214	95% = 0.494			

Number Distribution



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	0.01						
248.9	100.00	0.00	3.270	99.98	0.01						
209.3	100.00	0.00	2.750	99.97	0.02						
176.0	100.00	0.00	2.312	99.95	0.03						
148.0	100.00	0.00	1.945	99.92	0.06						
124.5	100.00	0.00	1.635	99.86	0.11						
104.7	100.00	0.00	1.375	99.75	0.22						
88.00	100.00	0.00	1.156	99.53	0.38						
74.00	100.00	0.00	0.972	99.15	0.56						
62.23	100.00	0.00	0.818	98.59	0.79						
52.33	100.00	0.00	0.688	97.80	1.16						
44.00	100.00	0.00	0.578	96.64	1.83						
37.00	100.00	0.00	0.486	94.81	3.07						
31.11	100.00	0.00	0.409	91.74	5.27						
26.16	100.00	0.00	0.344	86.47	9.06						
22.00	100.00	0.00	0.289	77.41	14.24						
18.50	100.00	0.00	0.243	63.17	18.31						
15.56	100.00	0.00	0.204	44.86	21.42						
13.00	100.00	0.00	0.172	23.44	23.44						
11.00	100.00	0.00	0.145	0.00	0.00						

Particle Size Distribution Plots
For Final Slurry: Sample CUF-C104-012
Replicate No: 1

Particle Size Analysis

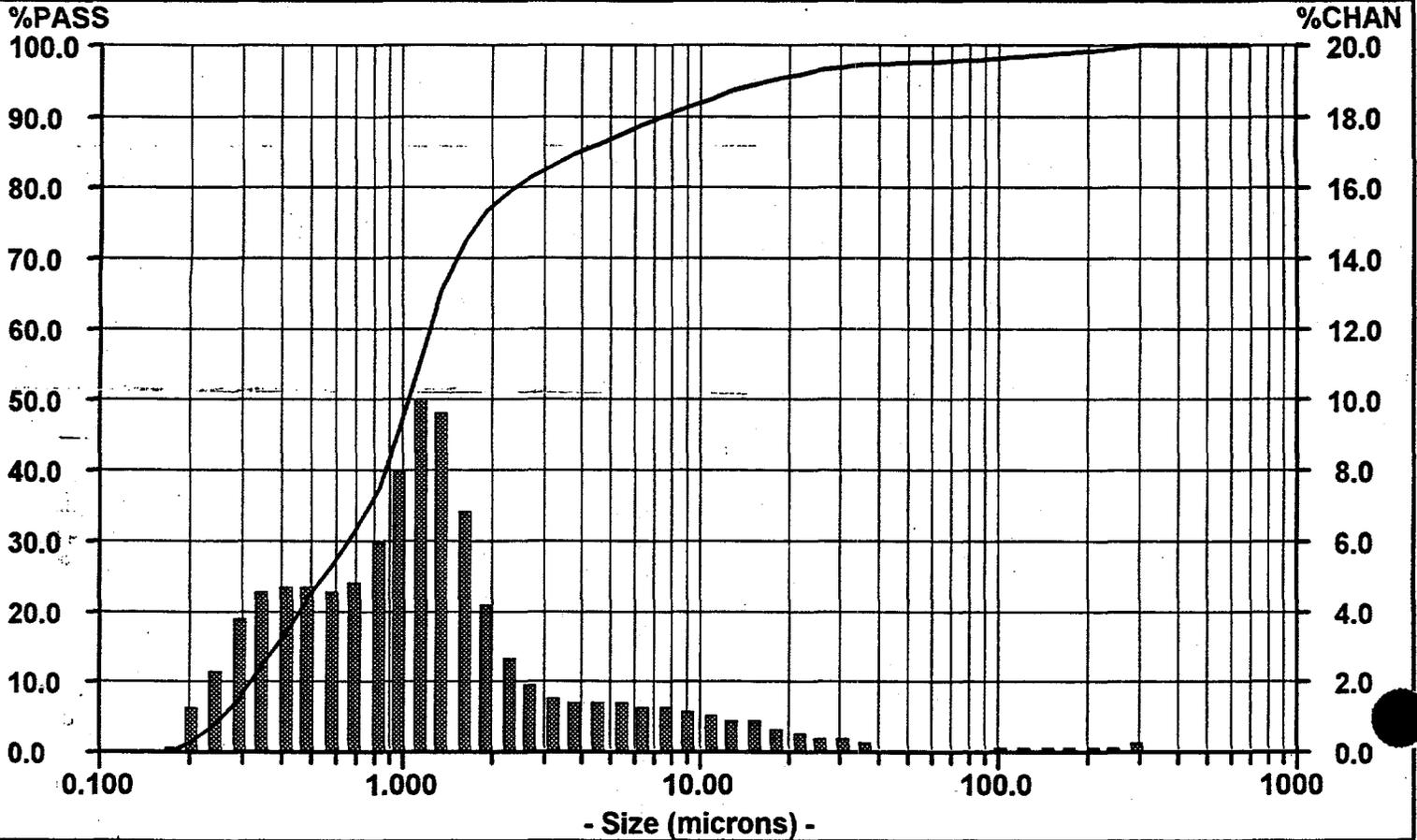
C104 Slurry
Sample CUP-C104-012

Date: 02/08/99 Meas #: 00064
Time: 14:24 Pres #: 01

C104 Slurry; Sample CUP-C104-012
in #12 Supernatant Simulant
40 ml/sec

Summary	Percentiles	Dia	Vol%	Width	
mv = 6.051	10% = 0.314	60% = 1.241	134.4	3%	173.7
mn = 0.304	20% = 0.452	70% = 1.522	1.235	75%	3.423
ma = 0.742	30% = 0.652	80% = 2.397	0.326	22%	0.191
cs = 8.088	40% = 0.866	90% = 7.442			
sd = 1.583	50% = 1.048	95% = 16.74			

Volume Distribution



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	91.57	1.24						
592.0	100.00	0.00	7.778	90.33	1.32						
497.8	100.00	0.00	6.541	89.01	1.37						
418.6	100.00	0.00	5.500	87.64	1.41						
352.0	100.00	0.00	4.625	86.23	1.45						
296.0	100.00	0.34	3.889	84.78	1.54						
248.9	99.66	0.28	3.270	83.24	1.69						
209.3	99.38	0.26	2.750	81.55	2.00						
176.0	99.12	0.26	2.312	79.55	2.72						
148.0	98.86	0.26	1.945	76.83	4.27						
124.5	98.60	0.25	1.635	72.56	6.98						
104.7	98.35	0.22	1.375	65.58	9.74						
88.00	98.13	0.18	1.156	55.84	10.13						
74.00	97.95	0.15	0.972	45.71	8.13						
62.23	97.80	0.13	0.818	37.58	6.02						
52.33	97.67	0.12	0.688	31.56	4.91						
44.00	97.55	0.12	0.578	26.65	4.64						
37.00	97.43	0.36	0.486	22.01	4.73						
31.11	97.07	0.43	0.409	17.28	4.82						
26.16	96.64	0.54	0.344	12.46	4.61						
22.00	96.10	0.66	0.289	7.85	3.80						
18.50	95.44	0.79	0.243	4.05	2.43						
15.56	94.65	0.92	0.204	1.62	1.33						
13.08	93.73	1.03	0.172	0.29	0.29						
11.00	92.70	1.13	0.145	0.00	0.00						

02/08/89 916

Particle Size Analysis

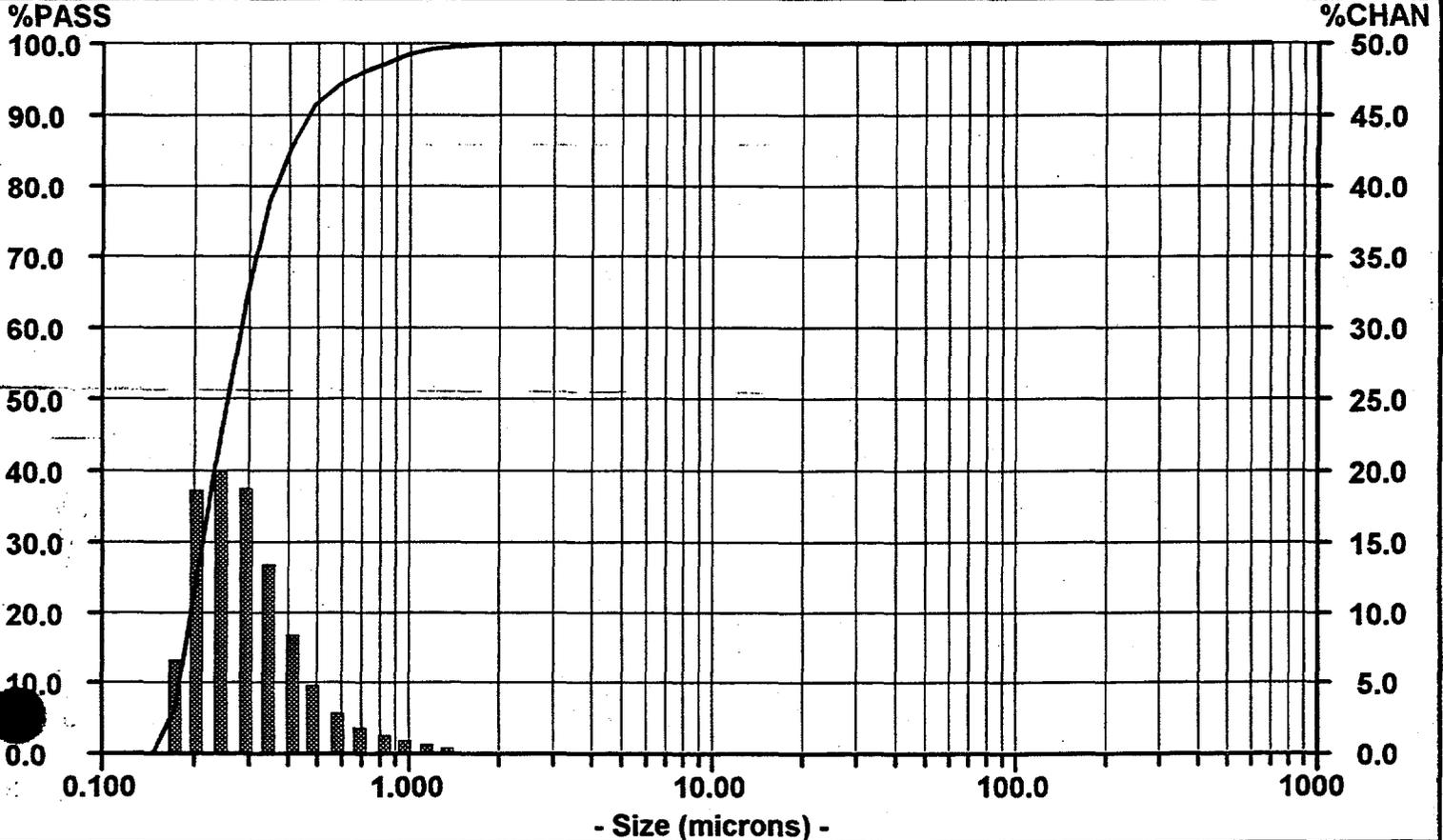
C104 Slurry
Sample CUP-C104-012

Date: 02/08/89 Meas #: 00064
Time: 14:24 Pres #: 01

C104 Slurry; Sample CUP-C104-012
in #12 Supernatant Simulant
40 ml/sec

Summary	Percentiles		Dia	Vol%	Width
mv = 6.051	10% = 0.178	60% = 0.277	0.253	100%	0.196
mn = 0.304	20% = 0.196	70% = 0.308			
ma = 0.742	30% = 0.212	80% = 0.355			
cs = 8.088	40% = 0.232	90% = 0.459			
sd = 0.098	50% = 0.253	95% = 0.615			

Number Distribution



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.260	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	0.00						
296.0	100.00	0.00	3.889	100.00	0.00						
248.9	100.00	0.00	3.270	100.00	0.01						
209.3	100.00	0.00	2.750	99.99	0.01						
176.0	100.00	0.00	2.312	99.98	0.03						
148.0	100.00	0.00	1.945	99.95	0.07						
124.5	100.00	0.00	1.635	99.88	0.19						
104.7	100.00	0.00	1.375	99.69	0.45						
88.0	100.00	0.00	1.156	99.24	0.78						
74.00	100.00	0.00	0.972	98.46	1.06						
62.23	100.00	0.00	0.818	97.40	1.31						
52.33	100.00	0.00	0.688	96.09	1.81						
44.00	100.00	0.00	0.578	94.28	2.87						
37.00	100.00	0.00	0.486	91.41	4.91						
31.11	100.00	0.00	0.409	86.50	8.42						
26.16	100.00	0.00	0.344	78.08	13.58						
22.00	100.00	0.00	0.289	64.50	18.81						
18.50	100.00	0.00	0.243	45.69	20.19						
15.56	100.00	0.00	0.204	25.50	18.73						
13.00	100.00	0.00	0.172	6.77	6.77						
11.00	100.00	0.00	0.145	0.00	0.00						

Particle Size Analysis

C104 Slurry
Sample CUP-C104-012

Date: 02/08/89 Meas #: 00068
Time: 14:42 Pres #: 01

C104 Slurry; Sample CUP-C104-012
in #12 Supernatant Simulant
60 ml/sec

Summary

mv = 2.063
mn = 0.288
ma = 0.631
cs = 9.515
sd = 0.949

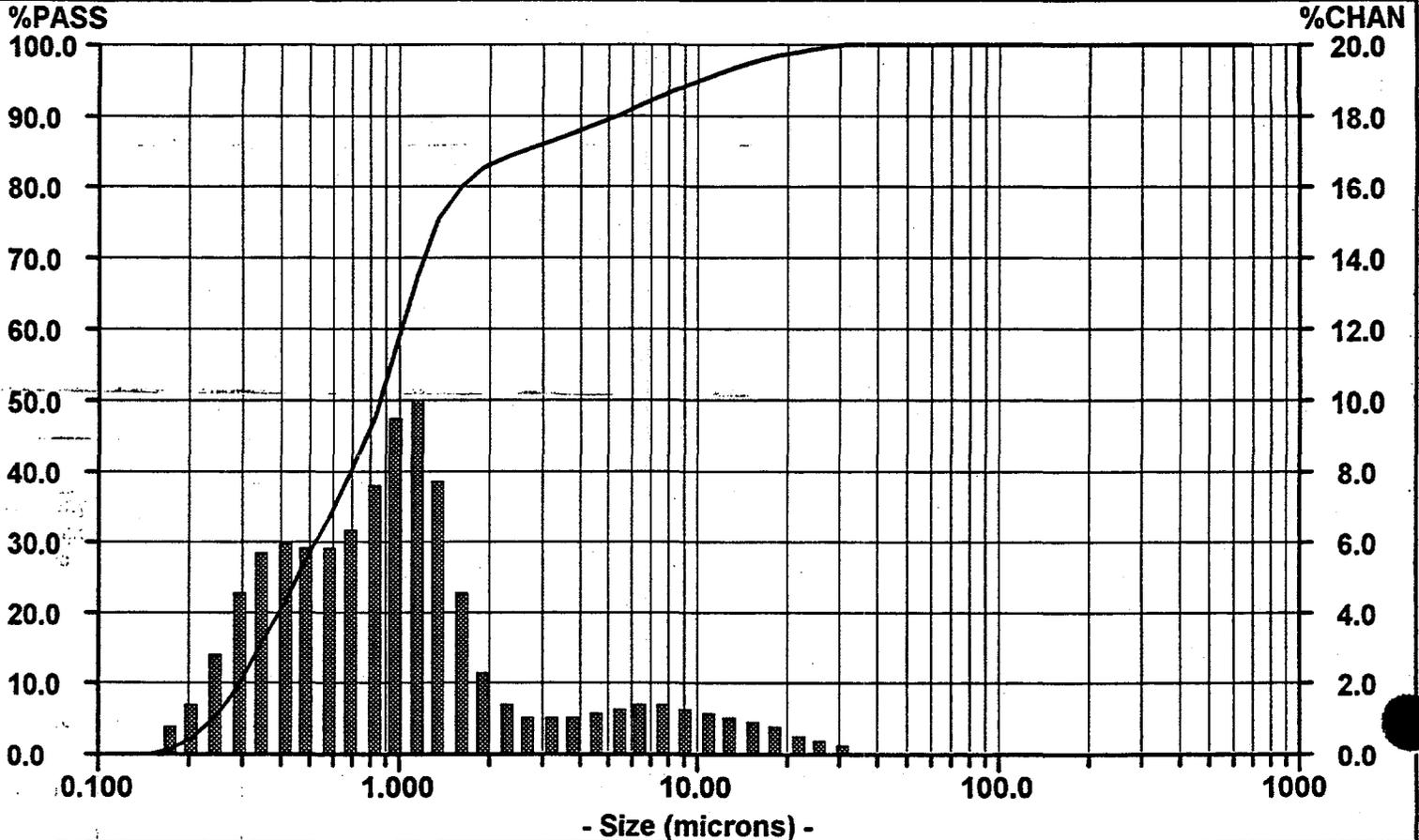
Percentiles

10% = 0.289 60% = 1.012
20% = 0.387 70% = 1.209
30% = 0.519 80% = 1.618
40% = 0.685 90% = 5.310
50% = 0.850 95% = 10.04

Dia Vol% Width

7.417 15% 11.56
0.956 58% 0.768
0.325 27% 0.194

Volume Distribution



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	94.41	1.33						
592.0	100.00	0.00	7.778	93.08	1.40						
497.8	100.00	0.00	6.541	91.68	1.40						
418.6	100.00	0.00	5.500	90.28	1.35						
352.0	100.00	0.00	4.625	88.93	1.26						
296.0	100.00	0.00	3.889	87.67	1.16						
248.9	100.00	0.00	3.270	86.51	1.11						
209.3	100.00	0.00	2.750	85.40	1.17						
176.0	100.00	0.00	2.312	84.23	1.52						
148.0	100.00	0.00	1.945	82.71	2.49						
124.5	100.00	0.00	1.635	80.22	4.65						
104.7	100.00	0.00	1.375	75.57	7.89						
88.00	100.00	0.00	1.156	67.68	10.10						
74.00	100.00	0.00	0.972	57.58	9.61						
62.23	100.00	0.00	0.818	47.97	7.78						
52.33	100.00	0.00	0.688	40.19	6.44						
44.00	100.00	0.00	0.578	33.75	5.94						
37.00	100.00	0.00	0.486	27.81	5.95						
31.11	100.00	0.37	0.409	21.86	6.05						
26.16	99.63	0.50	0.344	15.81	5.78						
22.00	99.13	0.64	0.289	10.03	4.68						
18.50	98.49	0.80	0.243	5.35	2.89						
15.56	97.69	0.96	0.204	2.46	1.67						
13.08	96.73	1.10	0.172	0.89	0.89						
11.00	95.63	1.22	0.145	0.00	0.00						

02/08/88 9125

Particle Size Analysis

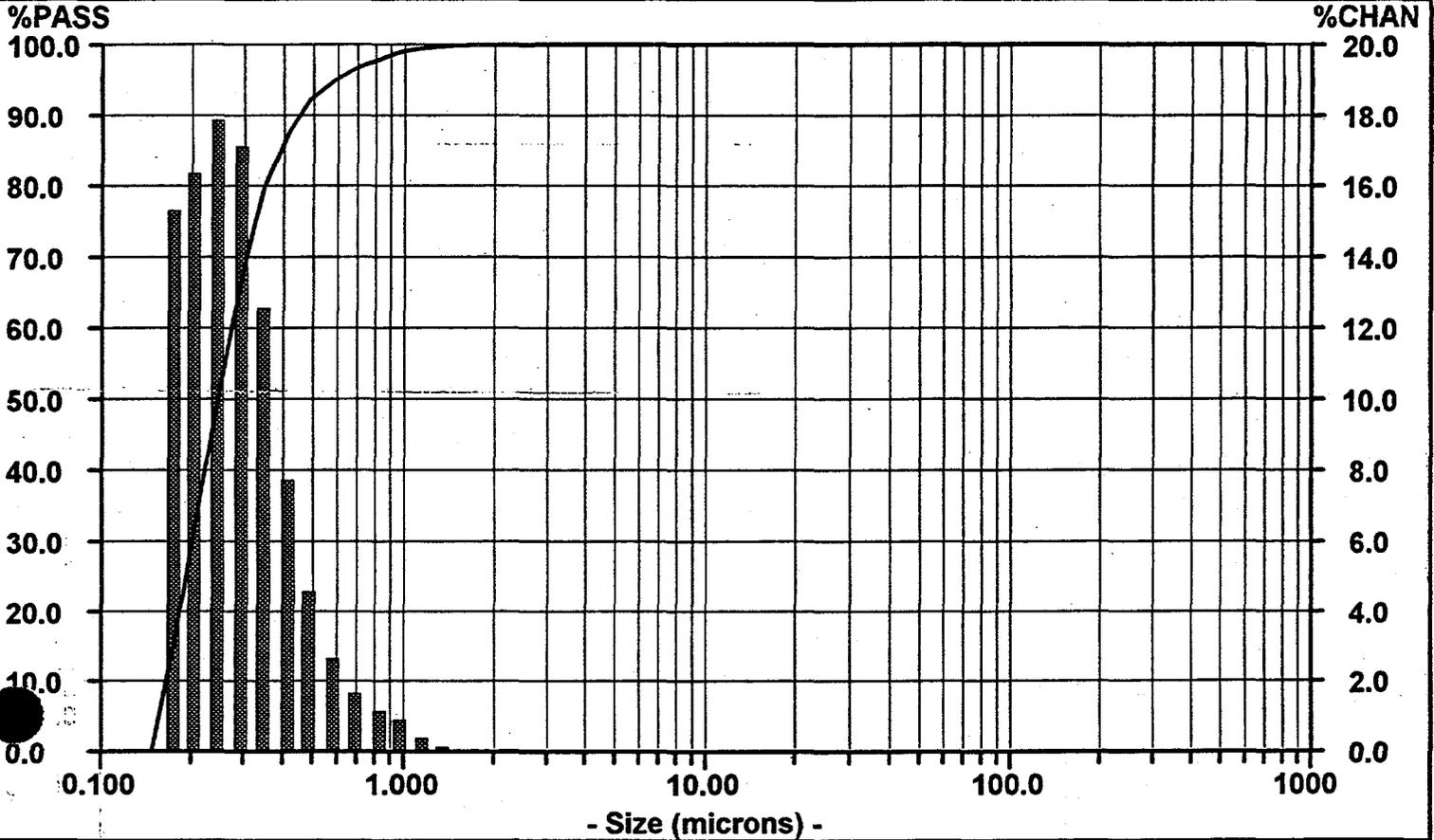
C104 Slurry
Sample CUP-C104-012

Date: 02/08/88 Meas #: 00068
Time: 14:42 Pres #: 01

C104 Slurry; Sample CUP-C104-012
In #12 Supernatant Simulant
60 ml/sec

Summary	Percentiles		Dia	Vol%	Width
mv = 2.063	10% = 0.164	60% = 0.268	0.243	100%	0.200
mn = 0.288	20% = 0.180	70% = 0.299			
ma = 0.631	30% = 0.200	80% = 0.345			
cs = 9.515	40% = 0.221	90% = 0.441			
sd = 0.100	50% = 0.243	95% = 0.576			

Number Distribution



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	0.00						
296.0	100.00	0.00	3.889	100.00	0.00						
248.9	100.00	0.00	3.270	100.00	0.00						
209.3	100.00	0.00	2.750	100.00	0.01						
176.0	100.00	0.00	2.312	99.99	0.01						
148.0	100.00	0.00	1.945	99.98	0.03						
124.5	100.00	0.00	1.635	99.95	0.09						
104.7	100.00	0.00	1.375	99.86	0.27						
88.00	100.00	0.00	1.156	99.59	0.58						
74.00	100.00	0.00	0.972	99.01	0.93						
62.23	100.00	0.00	0.818	98.08	1.27						
52.33	100.00	0.00	0.688	96.81	1.77						
44.00	100.00	0.00	0.578	95.04	2.74						
37.00	100.00	0.00	0.486	92.30	4.60						
31.11	100.00	0.00	0.409	87.70	7.88						
26.16	100.00	0.00	0.344	79.82	12.69						
22.00	100.00	0.00	0.289	67.13	17.26						
18.50	100.00	0.00	0.243	49.87	17.90						
15.75	100.00	0.00	0.204	31.97	16.48						
13.00	100.00	0.00	0.172	15.49	15.49						
11.00	100.00	0.00	0.145	0.00	0.00						

C-100/00 C-10

Particle Size Analysis

C104 Slurry
Sample CUP-C104-012

Date: ~~02/08/89~~ Meas #: 00072
Time: 14:55 Pres #: 01

C104 Slurry; Sample CUP-C104-012
In #12 Supernatant Simulant
Sonicated at 40W for 90 sec; 60 ml/sec
1st sonication

Summary

mv = 2.244
mn = 0.243
ma = 0.513
cs = 11.70
sd = 1.439

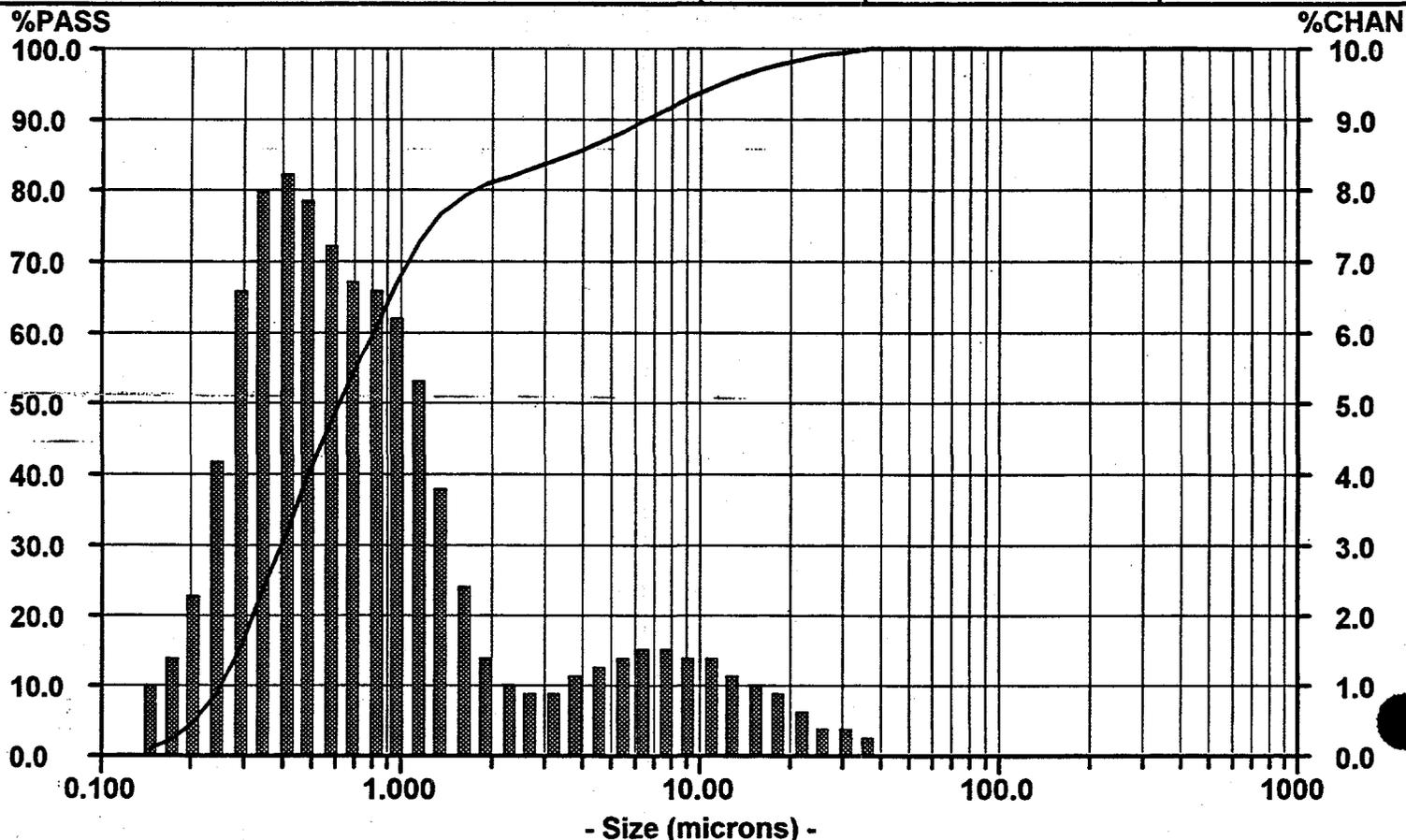
Percentiles

10% = 0.249 60% = 0.796
20% = 0.316 70% = 1.052
30% = 0.390 80% = 1.744
40% = 0.484 90% = 6.548
50% = 0.615 95% = 11.73

Dia Vol% Width

7.320 18% 12.53
0.497 82% 0.748

Volume Distribution



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	93.12	1.52						
592.0	100.00	0.00	7.778	91.60	1.61						
497.8	100.00	0.00	6.541	89.99	1.62						
418.6	100.00	0.00	5.500	88.37	1.54						
352.0	100.00	0.00	4.625	86.83	1.39						
296.0	100.00	0.00	3.889	85.44	1.23						
248.9	100.00	0.00	3.270	84.21	1.09						
209.3	100.00	0.00	2.750	83.12	1.04						
176.0	100.00	0.00	2.312	82.08	1.16						
148.0	100.00	0.00	1.945	80.92	1.58						
124.5	100.00	0.00	1.635	79.34	2.52						
104.7	100.00	0.00	1.375	76.82	3.99						
88.00	100.00	0.00	1.156	72.83	5.49						
74.00	100.00	0.00	0.972	67.34	6.33						
62.23	100.00	0.00	0.818	61.01	6.60						
52.33	100.00	0.00	0.688	54.41	6.86						
44.00	100.00	0.00	0.578	47.55	7.37						
37.00	100.00	0.32	0.486	40.18	7.98						
31.11	99.68	0.45	0.409	32.20	8.31						
26.16	99.23	0.60	0.344	23.89	8.00						
22.00	98.63	0.77	0.289	15.89	6.62						
18.50	97.86	0.95	0.243	9.27	4.28						
15.56	96.91	1.12	0.204	4.99	2.42						
13.08	95.79	1.27	0.172	2.57	1.46						
11.00	94.52	1.40	0.145	1.11	1.11						

02100100 9/24

Particle Size Analysis

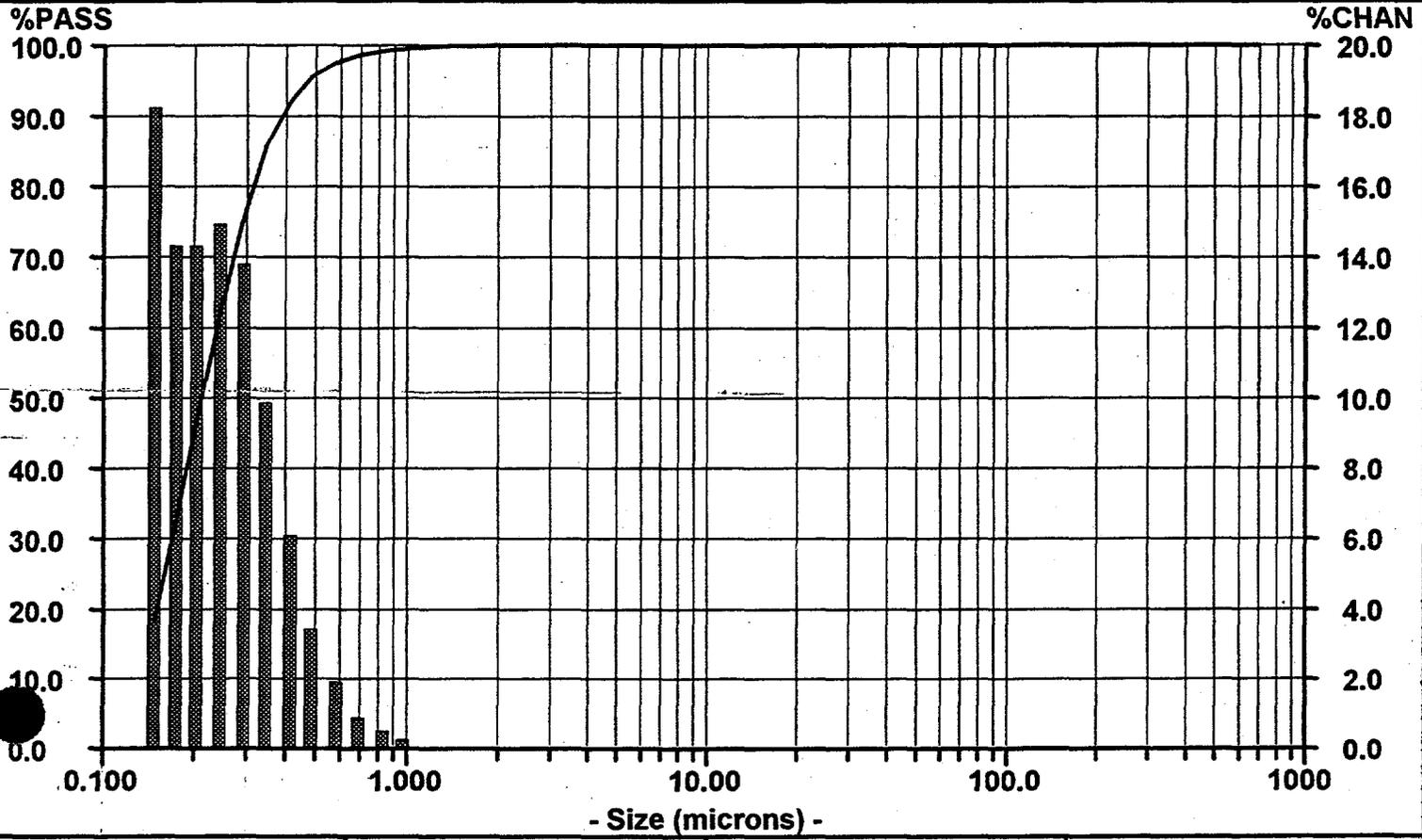
C104 Slurry
Sample CUP-C104-012

Date: 02/08/89 Meas #: 00072
Time: 14:55 Pres #: 01

C104 Slurry; Sample CUP-C104-012
in #12 Supernatant Simulant
Sonicated at 40W for 90 sec; 60 ml/sec
1st sonication

Summary	Percentiles			Dia	Vol%	Width
mv = 2.244	10% = 0.134	60% = 0.237		0.255	67%	0.176
mn = 0.243	20% = 0.147	70% = 0.267		0.145	33%	0.030
ma = 0.513	30% = 0.166	80% = 0.307				
cs = 11.70	40% = 0.187	90% = 0.380				
sd = 0.094	50% = 0.211	95% = 0.465				

Number Distribution



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	0.00						
296.0	100.00	0.00	3.889	100.00	0.00						
248.9	100.00	0.00	3.270	100.00	0.00						
209.3	100.00	0.00	2.750	100.00	0.00						
176.0	100.00	0.00	2.312	100.00	0.00						
148.0	100.00	0.00	1.945	100.00	0.01						
124.5	100.00	0.00	1.635	99.99	0.03						
104.7	100.00	0.00	1.375	99.96	0.08						
88.00	100.00	0.00	1.156	99.88	0.18						
74.00	100.00	0.00	0.972	99.70	0.35						
62.23	100.00	0.00	0.818	99.35	0.61						
52.33	100.00	0.00	0.688	98.74	1.07						
44.00	100.00	0.00	0.578	97.67	1.93						
37.00	100.00	0.00	0.486	95.74	3.50						
31.11	100.00	0.00	0.409	92.24	6.14						
26.16	100.00	0.00	0.344	86.10	9.97						
22.00	100.00	0.00	0.289	76.13	13.86						
18.50	100.00	0.00	0.243	62.27	15.04						
15.56	100.00	0.00	0.204	47.23	14.42						
13.00	100.00	0.00	0.172	32.81	14.42						
11.00	100.00	0.00	0.145	18.39	18.39						

Particle Size Analysis

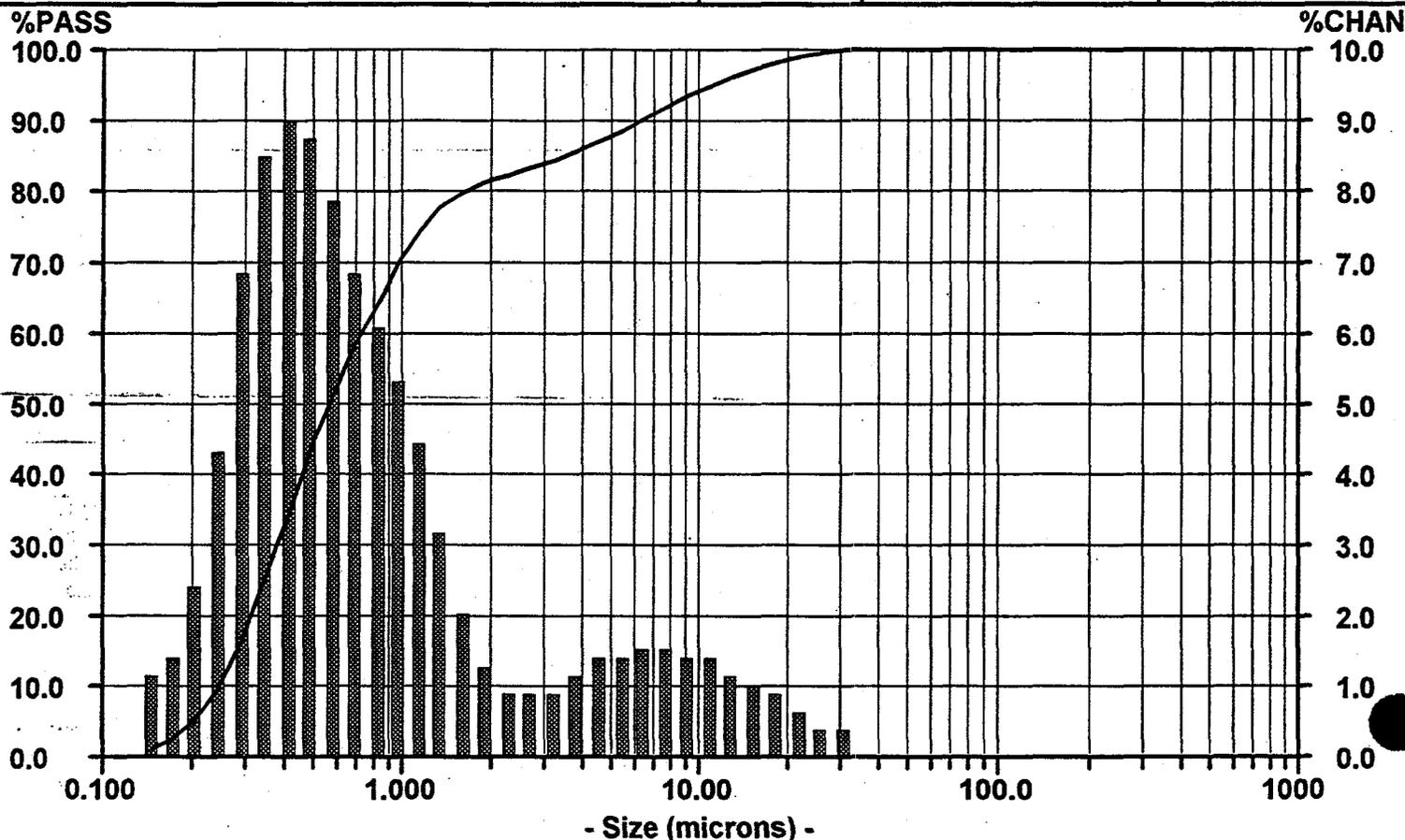
C104 Slurry
Sample CUP-C104-012

Date: 02/08/89 Meas #: 00076
Time: 15:12 Pres #: 01

C104 Slurry; Sample CUP-C104-012
in #12 Supernatant Simulant
Sonicated at 40W for 90 sec; 60 ml/sec
2nd sonication

Summary	Percentiles	Dia	Vol%	Width	
mv = 2.089	10% = 0.244	60% = 0.717	7.143	18%	11.43
mn = 0.242	20% = 0.309	70% = 0.970	0.466	82%	0.679
ma = 0.491	30% = 0.375	80% = 1.652			
cs = 12.22	40% = 0.454	90% = 6.324			
sd = 1.384	50% = 0.560	95% = 11.01			

Volume Distribution



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	93.57	1.57						
592.0	100.00	0.00	7.778	92.00	1.67						
497.8	100.00	0.00	6.541	90.33	1.68						
418.6	100.00	0.00	5.500	88.65	1.58						
352.0	100.00	0.00	4.625	87.07	1.41						
296.0	100.00	0.00	3.889	85.66	1.22						
248.9	100.00	0.00	3.270	84.44	1.07						
209.3	100.00	0.00	2.750	83.37	1.00						
176.0	100.00	0.00	2.312	82.37	1.07						
148.0	100.00	0.00	1.945	81.30	1.39						
124.5	100.00	0.00	1.635	79.91	2.10						
104.7	100.00	0.00	1.375	77.81	3.25						
88.00	100.00	0.00	1.156	74.56	4.51						
74.00	100.00	0.00	0.972	70.05	5.46						
62.23	100.00	0.00	0.818	64.59	6.17						
52.33	100.00	0.00	0.688	58.42	6.96						
44.00	100.00	0.00	0.578	51.46	7.97						
37.00	100.00	0.00	0.486	43.49	8.86						
31.11	100.00	0.40	0.409	34.63	9.19						
26.16	99.60	0.56	0.344	25.44	8.62						
22.00	99.04	0.74	0.289	16.82	6.97						
18.50	98.30	0.93	0.243	9.85	4.49						
15.56	97.37	1.11	0.204	5.36	2.57						
13.08	96.26	1.27	0.172	2.79	1.58						
11.00	94.99	1.42	0.145	1.21	1.21						

Particle Size Analysis

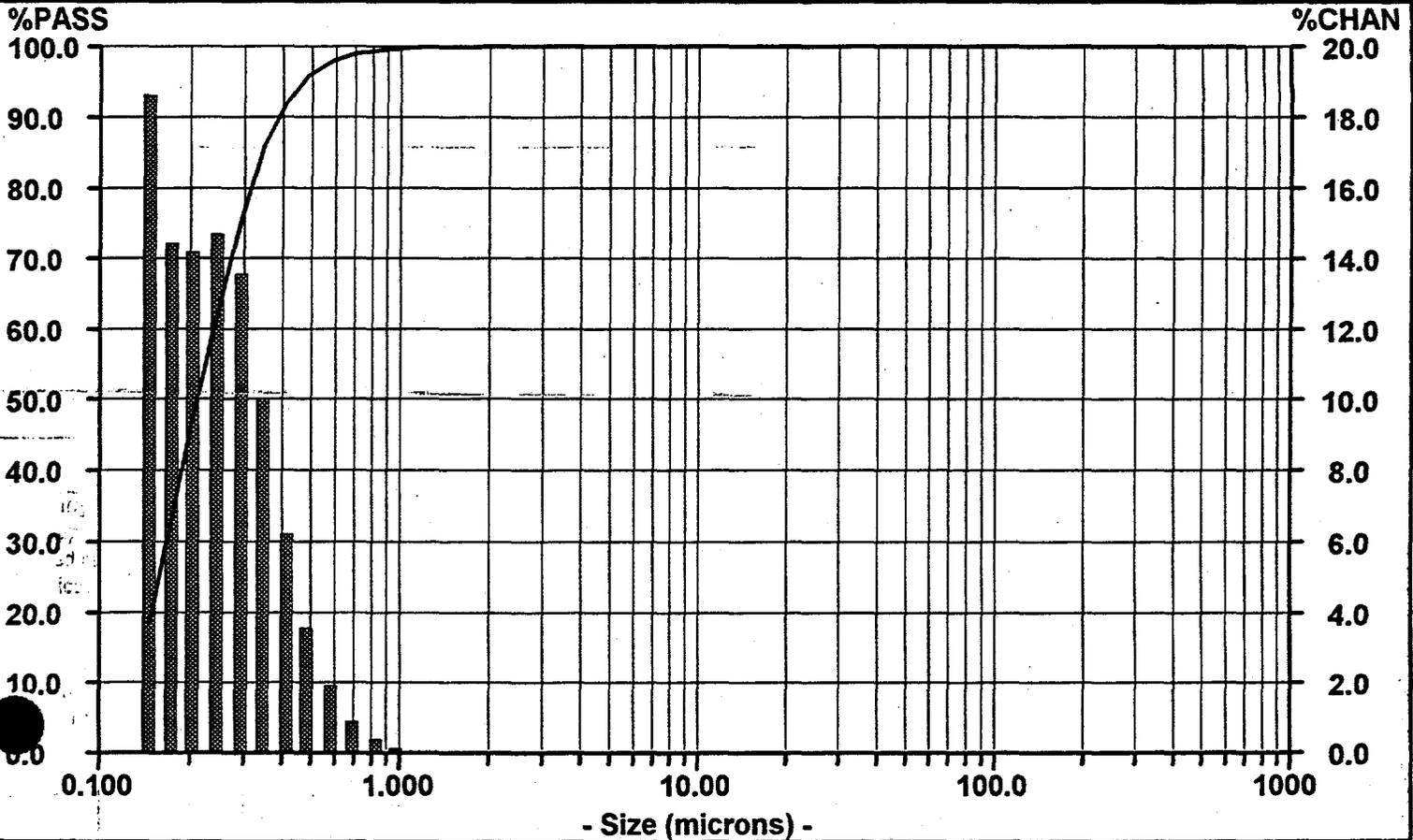
C104 Slurry
Sample CUP-C104-012

Date: ~~02/08/89~~ Meas #: 00076
Time: 15:12 Pres #: 01

C104 Slurry; Sample CUP-C104-012
in #12 Supernatant Simulant
Sonicated at 40W for 90 sec; 60 ml/sec
2nd sonication

Summary	Percentiles		Dia	Vol%	Width
mv = 2.089	10% = 0.134	60% = 0.237	0.256	67%	0.177
mn = 0.242	20% = 0.147	70% = 0.267	0.145	33%	0.030
ma = 0.491	30% = 0.165	80% = 0.308			
cs = 12.22	40% = 0.186	90% = 0.379			
sd = 0.094	50% = 0.210	95% = 0.459			

Number Distribution



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	0.00						
296.0	100.00	0.00	3.889	100.00	0.00						
248.9	100.00	0.00	3.270	100.00	0.00						
209.3	100.00	0.00	2.750	100.00	0.00						
176.0	100.00	0.00	2.312	100.00	0.00						
148.0	100.00	0.00	1.945	100.00	0.01						
124.5	100.00	0.00	1.635	99.99	0.02						
104.7	100.00	0.00	1.375	99.97	0.06						
88.00	100.00	0.00	1.156	99.91	0.14						
74.00	100.00	0.00	0.972	99.77	0.28						
62.23	100.00	0.00	0.818	99.49	0.53						
52.33	100.00	0.00	0.688	98.96	1.01						
44.00	100.00	0.00	0.578	97.95	1.95						
37.00	100.00	0.00	0.486	96.00	3.63						
31.11	100.00	0.00	0.409	92.37	6.34						
26.16	100.00	0.00	0.344	86.03	10.04						
22.00	100.00	0.00	0.289	75.99	13.63						
18.50	100.00	0.00	0.243	62.36	14.74						
15.56	100.00	0.00	0.204	47.62	14.30						
12.50	100.00	0.00	0.172	33.32	14.58						
10.00	100.00	0.00	0.145	18.74	18.74						

Particle Size Distribution Plots
For Final Slurry: Sample CUF-C104-012DUP
Replicate No: 2

Particle Size Analysis

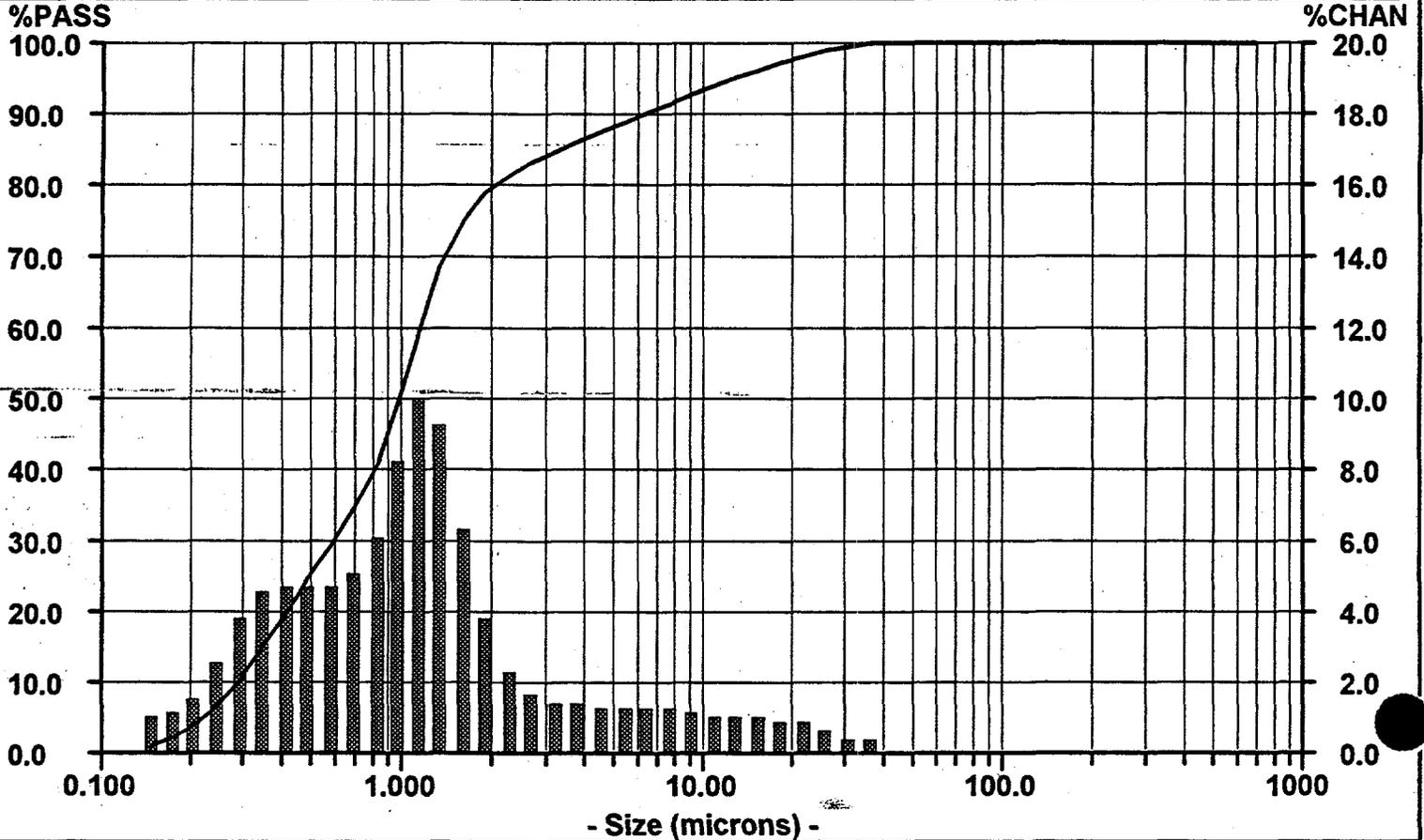
C104 Slurry DUP
Sample CUP-C104-012

Date: 02/08/89 Meas #: 00080
Time: 15:41 Pres #: 01

C104 Slurry DUP; Sample CUP-C104-012
in #12 Supernatant Simulant
40 ml/sec
Duplicate Sample

Summary	Percentiles		Dia	Vol%	Width
mv = 2.511	10% = 0.283	60% = 1.166	1.216	75%	4.155
mn = 0.237	20% = 0.409	70% = 1.412	0.312	25%	0.216
ma = 0.656	30% = 0.586	80% = 2.064			
cs = 9.153	40% = 0.797	90% = 6.334			
sd = 1.322	50% = 0.983	95% = 12.77			

Volume Distribution



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	92.82	1.25						
592.0	100.00	0.00	7.778	91.57	1.32						
497.8	100.00	0.00	6.541	90.25	1.35						
418.6	100.00	0.00	5.500	88.90	1.36						
352.0	100.00	0.00	4.625	87.54	1.38						
296.0	100.00	0.00	3.889	86.16	1.42						
248.9	100.00	0.00	3.270	84.74	1.51						
209.3	100.00	0.00	2.750	83.23	1.76						
176.0	100.00	0.00	2.312	81.47	2.39						
148.0	100.00	0.00	1.945	79.08	3.82						
124.5	100.00	0.00	1.635	75.26	6.42						
104.7	100.00	0.00	1.375	68.84	9.34						
88.0	100.00	0.00	1.156	59.50	10.11						
74.0	100.00	0.00	0.972	49.39	8.36						
62.23	100.00	0.00	0.818	41.03	6.28						
52.33	100.00	0.00	0.688	34.75	5.13						
44.00	100.00	0.00	0.578	29.62	4.79						
37.00	100.00	0.40	0.486	24.83	4.83						
31.11	99.60	0.58	0.409	20.00	4.86						
26.16	99.02	0.77	0.344	15.14	4.62						
22.00	98.25	0.93	0.289	10.52	3.87						
18.50	97.32	1.05	0.243	6.65	2.63						
15.56	96.27	1.11	0.204	4.02	1.67						
13.08	95.16	1.15	0.172	2.35	1.22						
11.00	94.01	1.19	0.145	1.13	1.13						

Particle Size Analysis

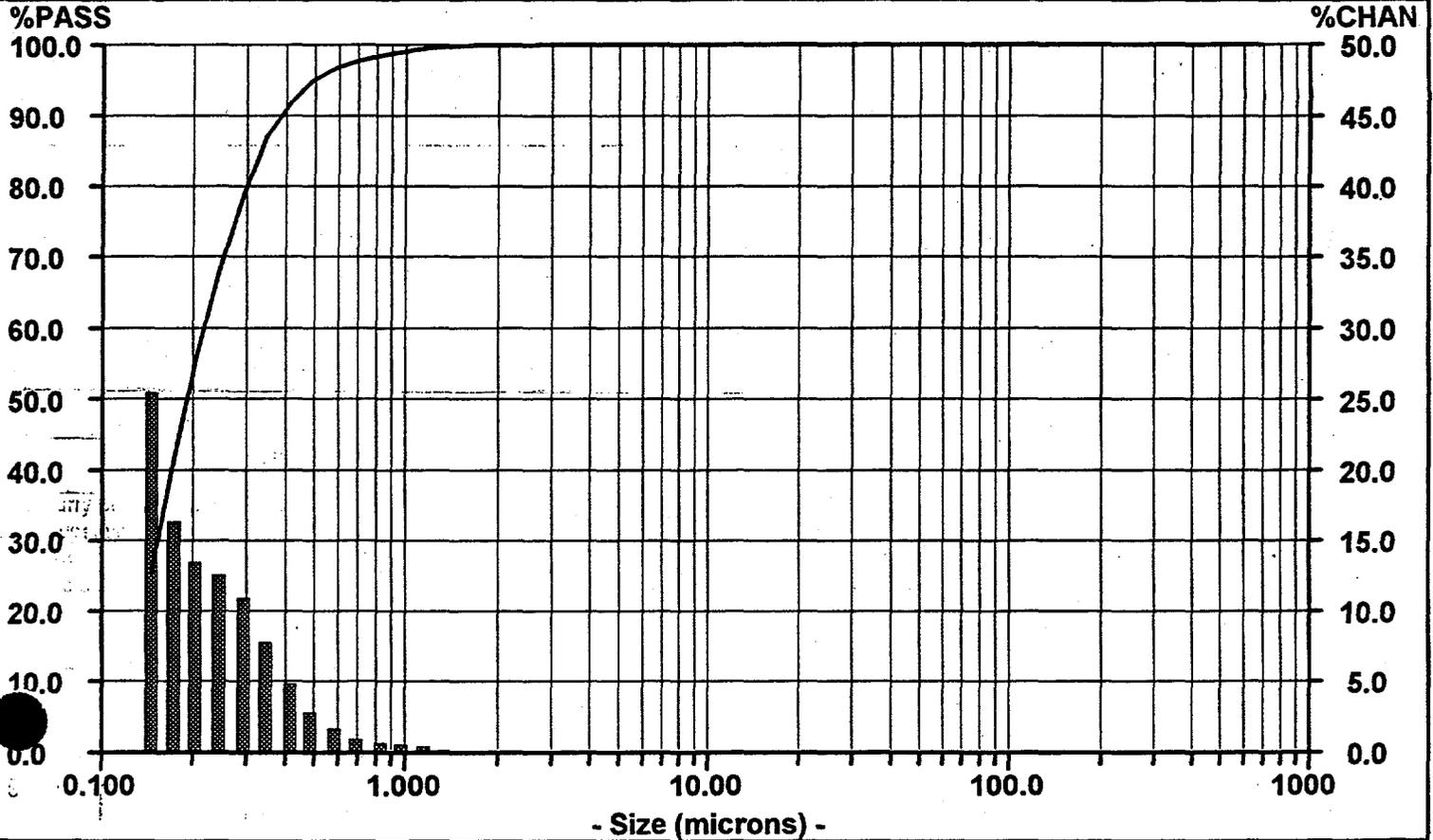
C104 Slurry DUP
Sample CUP-C104-012

Date: 02/08/89 Meas #: 00080
Time: 15:41 Pres #: 01

C104 Slurry DUP; Sample CUP-C104-012
In #12 Supernatant Simulant
50 ml/sec ~~40 ml/sec~~ GRG
Duplicate Sample

Summary	Percentiles		Dia	Vol%	Width
mv = 2.511	10% = 0.131	60% = 0.217	0.190	100%	0.182
mn = 0.237	20% = 0.139	70% = 0.250			
ma = 0.656	30% = 0.150	80% = 0.293			
cs = 9.153	40% = 0.168	90% = 0.377			
sd = 0.091	50% = 0.190	95% = 0.490			

Number Distribution



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	0.00						
296.0	100.00	0.00	3.889	100.00	0.00						
248.9	100.00	0.00	3.270	100.00	0.00						
209.3	100.00	0.00	2.750	100.00	0.01						
176.0	100.00	0.00	2.312	99.99	0.01						
148.0	100.00	0.00	1.945	99.98	0.04						
124.5	100.00	0.00	1.635	99.94	0.10						
104.7	100.00	0.00	1.375	99.84	0.25						
88.00	100.00	0.00	1.156	99.59	0.45						
74.00	100.00	0.00	0.972	99.14	0.63						
62.23	100.00	0.00	0.818	98.51	0.79						
52.33	100.00	0.00	0.688	97.72	1.09						
44.00	100.00	0.00	0.578	96.63	1.71						
37.00	100.00	0.00	0.486	94.92	2.90						
31.11	100.00	0.00	0.409	92.02	4.90						
26.16	100.00	0.00	0.344	87.12	7.86						
22.00	100.00	0.00	0.289	79.26	11.06						
18.50	100.00	0.00	0.243	68.20	12.61						
15.56	100.00	0.00	0.204	55.59	13.58						
13.00	100.00	0.00	0.172	42.01	16.45						
11.00	100.00	0.00	0.145	25.56	25.56						

Particle Size Analysis

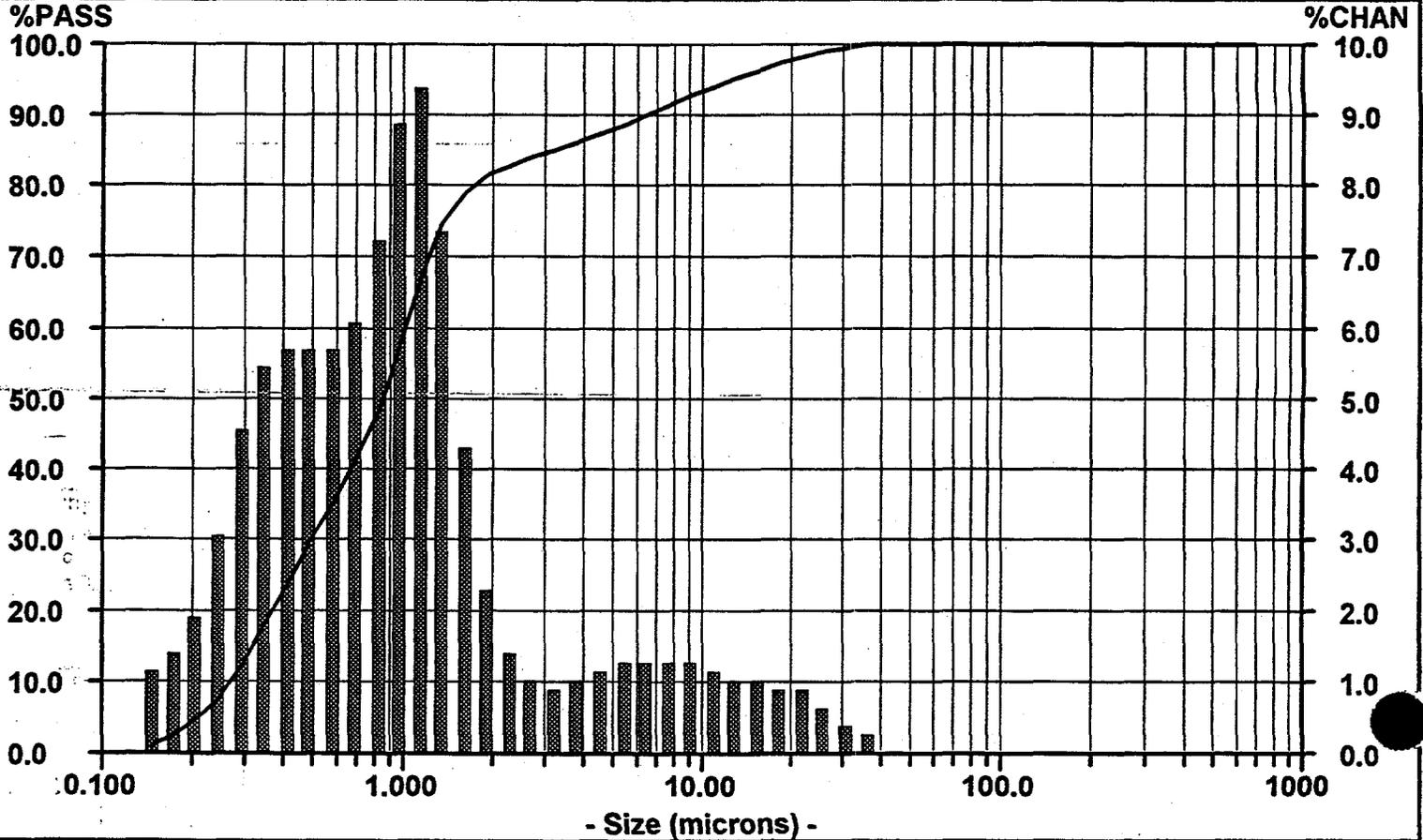
C104 Slurry DUP
Sample CUP-C104-012

Date: 92/08/89 Meas #: 00084
Time: 15:54 Pres #: 01

C104 Slurry DUP; Sample CUP-C104-012
in #12 Supernatant Simulant
60 ml/sec
Duplicate Sample

Summary	Percentiles	Dia	Vol%	Width	
mv = 2.393	10% = 0.266	60% = 1.012	8.317	16%	14.56
mn = 0.236	20% = 0.365	70% = 1.226	0.955	54%	0.776
ma = 0.590	30% = 0.493	80% = 1.735	0.313	30%	0.214
cs = 10.16	40% = 0.662	90% = 6.436			
sd = 1.207	50% = 0.839	95% = 12.60			

Volume Distribution



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.260	92.82	1.32						
592.0	100.00	0.00	7.778	91.50	1.37						
497.8	100.00	0.00	6.541	90.13	1.37						
418.6	100.00	0.00	5.500	88.76	1.31						
352.0	100.00	0.00	4.625	87.45	1.22						
296.0	100.00	0.00	3.889	86.23	1.13						
248.9	100.00	0.00	3.270	85.10	1.07						
209.3	100.00	0.00	2.750	84.03	1.14						
176.0	100.00	0.00	2.312	82.89	1.47						
148.0	100.00	0.00	1.945	81.42	2.39						
124.5	100.00	0.00	1.635	79.03	4.41						
104.7	100.00	0.00	1.375	74.62	7.40						
88.00	100.00	0.00	1.156	67.22	9.45						
74.00	100.00	0.00	0.972	57.77	8.99						
62.23	100.00	0.00	0.818	48.78	7.35						
52.33	100.00	0.00	0.688	41.43	6.15						
44.00	100.00	0.00	0.578	35.28	5.72						
37.00	100.00	0.38	0.486	29.56	5.76						
31.11	99.62	0.56	0.409	23.80	5.83						
26.16	99.06	0.75	0.344	17.97	5.58						
22.00	98.31	0.91	0.289	12.39	4.64						
18.50	97.40	1.03	0.243	7.75	3.12						
15.56	96.37	1.12	0.204	4.63	1.95						
13.08	95.25	1.18	0.172	2.68	1.40						
11.00	94.07	1.25	0.145	1.28	1.28						

Particle Size Analysis

C104 Slurry DUP
Sample CUP-C104-012

Date: ~~02/08/89~~ Meas #: 00084
Time: 15:54 Pres #: 01

C104 Slurry DUP; Sample CUP-C104-012
in #12 Supernatant Simulant
60 ml/sec
Duplicate Sample

Summary

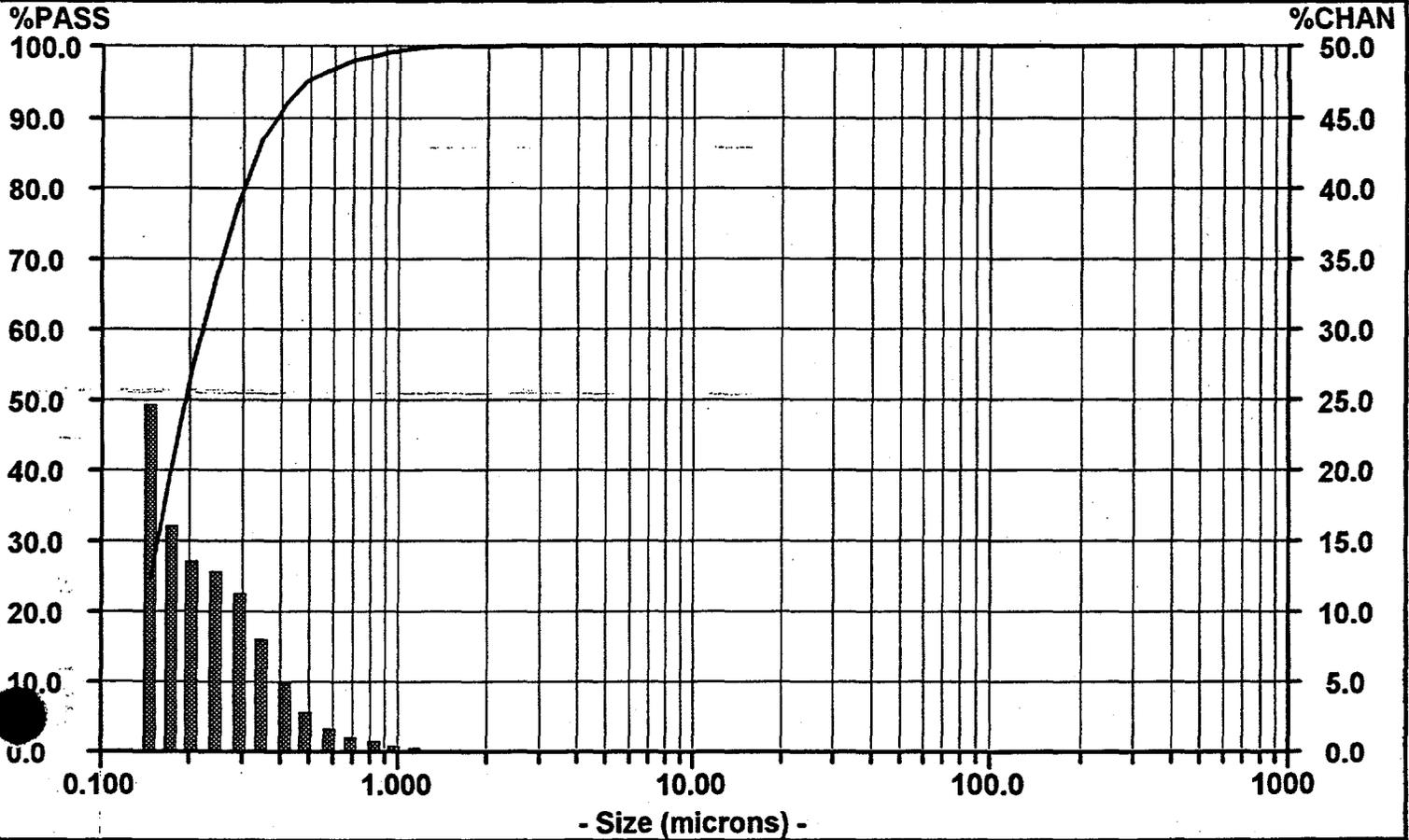
mv = 2.393
mn = 0.236
ma = 0.590
cs = 10.16
sd = 0.091

Percentiles

10% = 0.131 60% = 0.219
20% = 0.140 70% = 0.252
30% = 0.152 80% = 0.295
40% = 0.170 90% = 0.376
50% = 0.192 95% = 0.482

Dia Vol% Width
0.192 100% 0.183

Number Distribution



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	0.00						
296.0	100.00	0.00	3.889	100.00	0.00						
248.9	100.00	0.00	3.270	100.00	0.00						
209.3	100.00	0.00	2.750	100.00	0.00						
176.0	100.00	0.00	2.312	100.00	0.01						
148.0	100.00	0.00	1.945	99.99	0.02						
124.5	100.00	0.00	1.635	99.97	0.06						
104.7	100.00	0.00	1.375	99.91	0.17						
88.00	100.00	0.00	1.156	99.74	0.36						
74.00	100.00	0.00	0.972	99.38	0.58						
62.23	100.00	0.00	0.818	98.80	0.80						
52.33	100.00	0.00	0.688	98.00	1.12						
44.00	100.00	0.00	0.578	96.88	1.76						
37.00	100.00	0.00	0.486	95.12	2.97						
31.11	100.00	0.00	0.409	92.15	5.05						
26.16	100.00	0.00	0.344	87.10	8.16						
22.00	100.00	0.00	0.289	78.94	11.39						
18.50	100.00	0.00	0.243	67.55	12.85						
15.56	100.00	0.00	0.204	54.70	13.62						
13.00	100.00	0.00	0.172	41.08	16.22						
11.00	100.00	0.00	0.145	24.86	24.86						

C21-0100 910

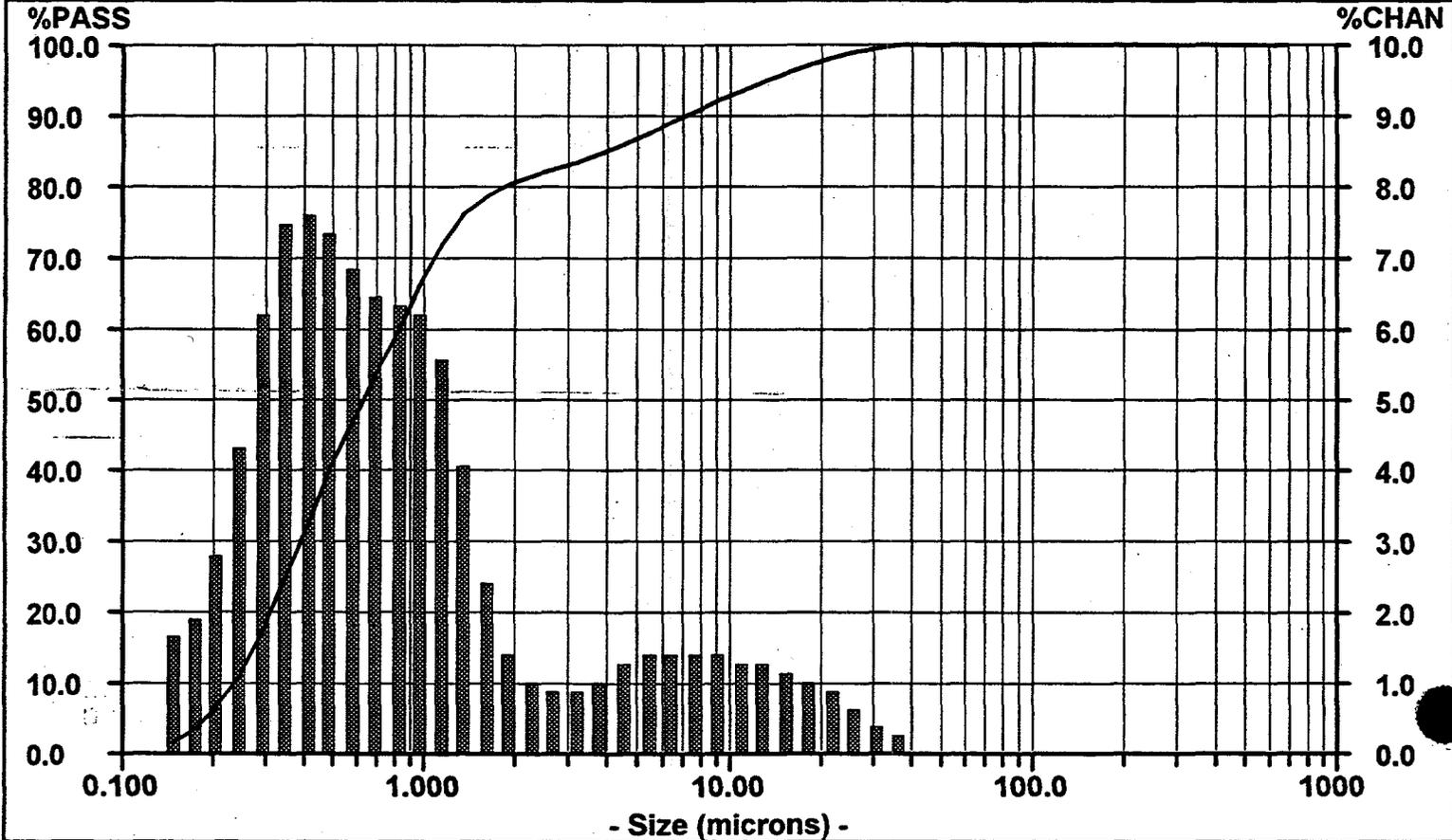
Particle Size Analysis

C104 Slurry DUP
Sample CUP-C104-012

Date: 02/08/89 Meas #: 00088
Time: 16:04 Pres #: 01

C104 Slurry DUP; Sample CUP-C104-012
in #12 Supernatant Simulant
Sonicated at 40W for 90 sec; 60 ml/sec
1st Sonication—Duplicate Sample
Volume Distribution

Summary	Percentiles	Dia	Vol%	Width	
mv = 2.396	10% = 0.234	60% = 0.813	7.814	18%	14.00
mn = 0.226	20% = 0.306	70% = 1.076	0.495	82%	0.772
ma = 0.498	30% = 0.384	80% = 1.834			
cs = 12.04	40% = 0.483	90% = 7.137			
sd = 1.579	50% = 0.622	95% = 13.19			



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	92.25	1.48						
592.0	100.00	0.00	7.778	90.77	1.56						
497.8	100.00	0.00	6.541	89.21	1.56						
418.6	100.00	0.00	5.500	87.65	1.47						
352.0	100.00	0.00	4.625	86.18	1.33						
296.0	100.00	0.00	3.889	84.85	1.17						
248.9	100.00	0.00	3.270	83.68	1.06						
209.3	100.00	0.00	2.750	82.63	1.01						
176.0	100.00	0.00	2.312	81.62	1.15						
148.0	100.00	0.00	1.945	80.47	1.59						
124.5	100.00	0.00	1.635	78.88	2.57						
104.7	100.00	0.00	1.375	76.31	4.11						
88.00	100.00	0.00	1.156	72.20	5.62						
74.00	100.00	0.00	0.972	66.58	6.36						
62.23	100.00	0.00	0.818	60.22	6.46						
52.33	100.00	0.00	0.688	53.76	6.56						
44.00	100.00	0.00	0.578	47.20	6.94						
37.00	100.00	0.33	0.486	40.26	7.46						
31.11	99.67	0.55	0.409	32.80	7.74						
26.16	99.12	0.79	0.344	25.06	7.51						
22.00	98.33	1.00	0.289	17.55	6.39						
18.50	97.33	1.15	0.243	11.66	4.47						
15.56	96.18	1.24	0.204	6.69	2.89						
13.08	94.94	1.31	0.172	3.80	2.05						
11.00	93.63	1.38	0.145	1.75	1.75						

Particle Size Analysis

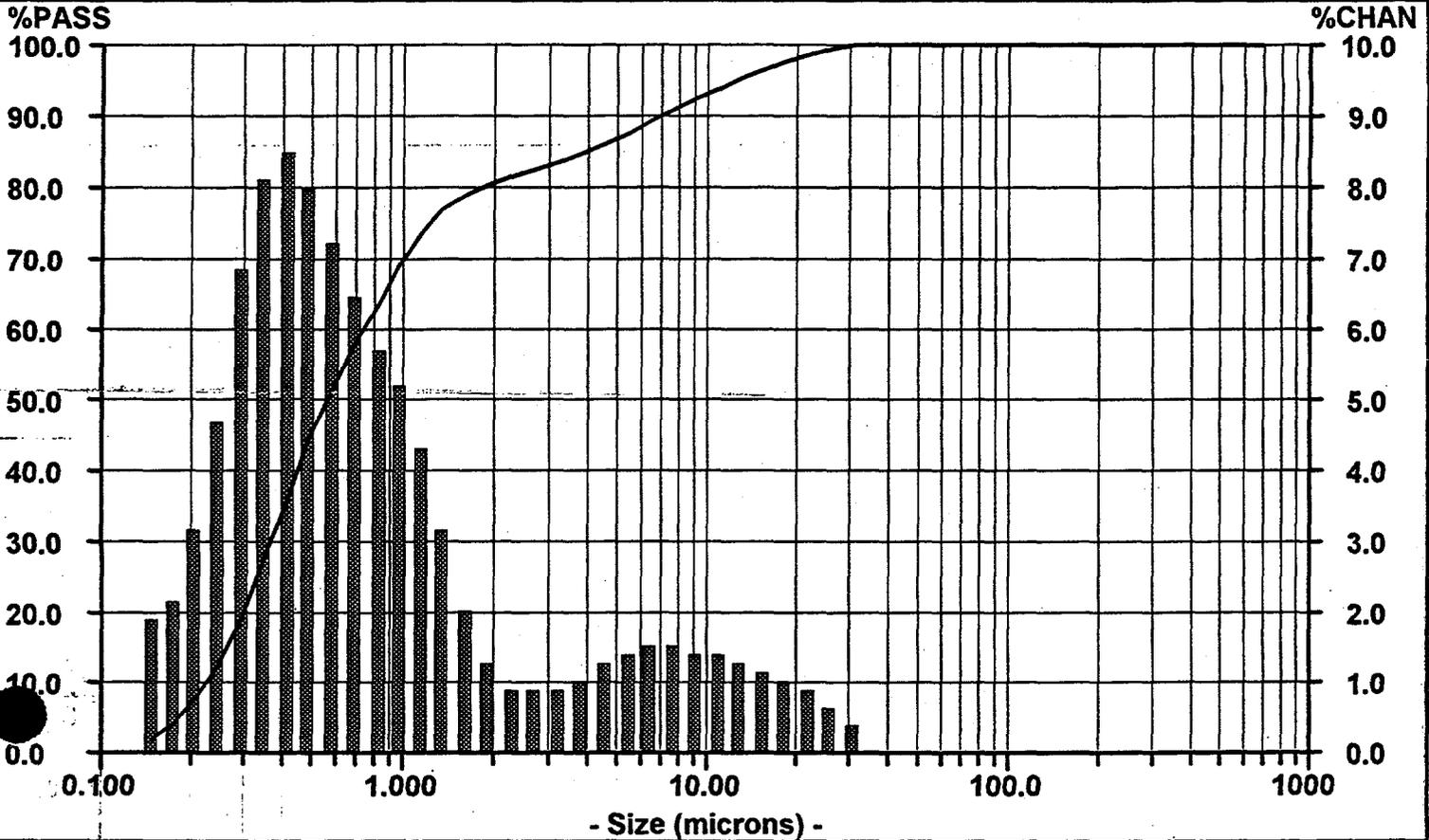
C104 Slurry DUP
Sample CUP-C104-012

Date: 02/08/89 Meas #: 00092
Time: 16:14 Pres #: 01

C104 Slurry DUP; Sample CUP-C104-012
In #12 Supernatant Simulant
Sonicated at 40W for 90 sec; 60 ml/sec
2nd Sonication—Duplicate Sample

Summary	Percentiles		Dia	Vol%	Width
mv = 2.268	10% = 0.226	60% = 0.726	7.650	18%	12.99
mn = 0.223	20% = 0.294	70% = 0.996	0.453	82%	0.698
ma = 0.472	30% = 0.362	80% = 1.803			
cs = 12.72	40% = 0.444	90% = 7.017			
sd = 1.694	50% = 0.556	95% = 12.61			

Volume Distribution



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	92.50	1.54						
592.0	100.00	0.00	7.778	90.96	1.62						
497.8	100.00	0.00	6.541	89.34	1.62						
418.6	100.00	0.00	5.500	87.72	1.53						
352.0	100.00	0.00	4.625	86.19	1.36						
296.0	100.00	0.00	3.889	84.83	1.19						
248.9	100.00	0.00	3.270	83.64	1.05						
209.3	100.00	0.00	2.750	82.59	0.98						
176.0	100.00	0.00	2.312	81.61	1.06						
148.0	100.00	0.00	1.945	80.55	1.39						
124.5	100.00	0.00	1.635	79.16	2.11						
104.7	100.00	0.00	1.375	77.05	3.25						
88.0	100.00	0.00	1.156	73.80	4.47						
74.00	100.00	0.00	0.972	69.33	5.33						
62.23	100.00	0.00	0.818	64.00	5.89						
52.33	100.00	0.00	0.688	58.11	6.51						
44.00	100.00	0.00	0.578	51.60	7.36						
37.00	100.00	0.00	0.486	44.24	8.18						
31.11	100.00	0.51	0.409	36.06	8.55						
26.16	99.49	0.77	0.344	27.51	8.24						
22.00	98.72	1.00	0.289	19.27	6.96						
18.50	97.72	1.17	0.243	12.31	4.88						
15.55	96.55	1.27	0.204	7.43	3.20						
13.00	95.28	1.35	0.172	4.23	2.29						
11.00	93.93	1.43	0.145	1.94	1.94						

Particle Size Analysis

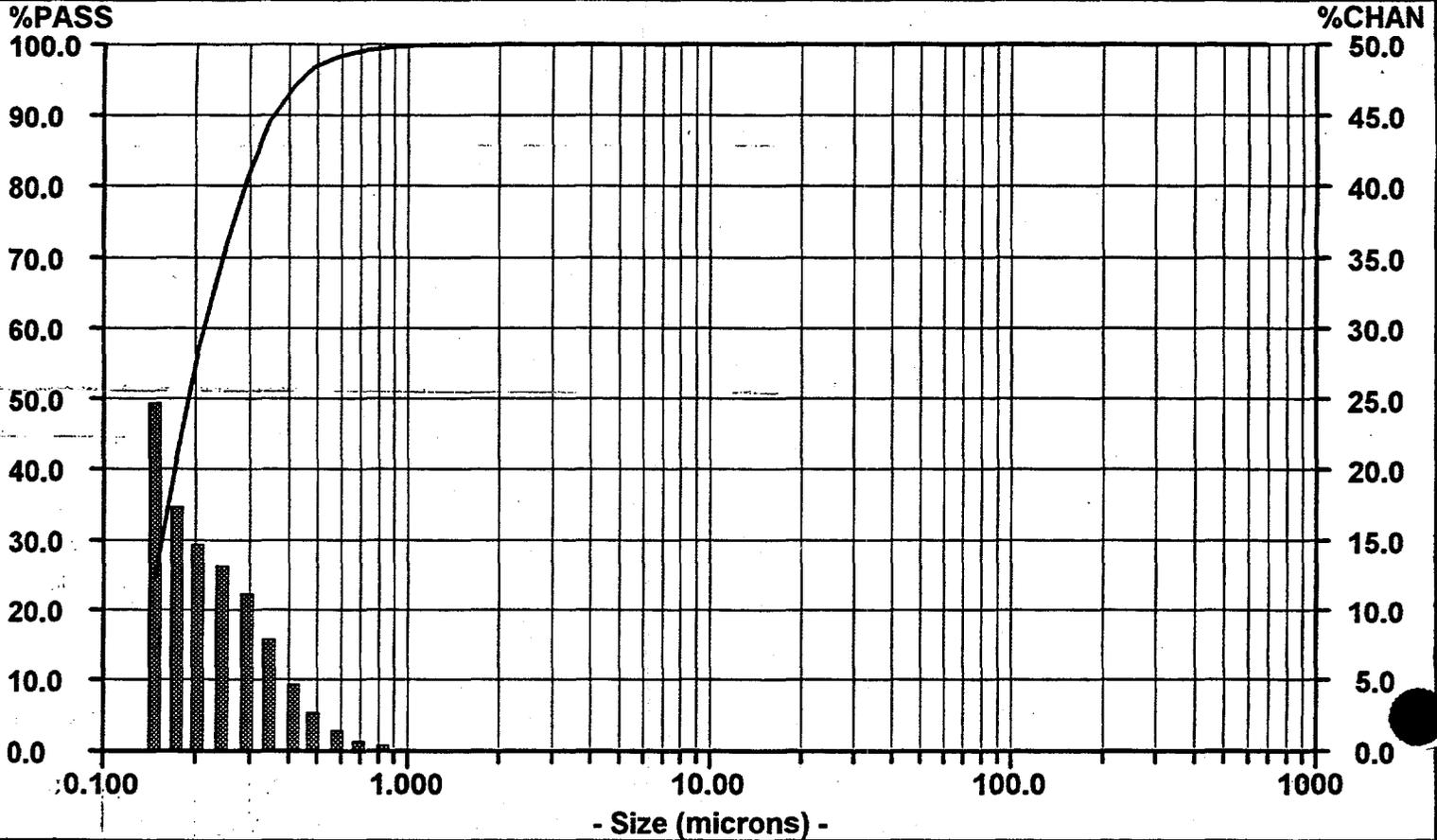
C104 Slurry DUP
Sample CUP-C104-012

Date: 02/08/89 Meas #: 00092
Time: 16:14 Pres #: 01

C104 Slurry DUP; Sample CUP-C104-012
in #12 Supernatant Simulant
Sonicated at 40W for 90 sec; 60 ml/sec
2nd Sonication—Duplicate Sample

Summary	Percentiles	Dia	Vol%	Width	
mv = 2.268	10% = 0.131	60% = 0.212	0.188	100%	0.168
mn = 0.223	20% = 0.140	70% = 0.243			
ma = 0.472	30% = 0.151	80% = 0.283			
cs = 12.72	40% = 0.168	90% = 0.352			
sd = 0.084	50% = 0.188	95% = 0.428			

Number Distribution



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	100.00	0.00						
692.0	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	0.00						
296.0	100.00	0.00	3.889	100.00	0.00						
248.9	100.00	0.00	3.270	100.00	0.00						
209.3	100.00	0.00	2.750	100.00	0.00						
176.0	100.00	0.00	2.312	100.00	0.00						
148.0	100.00	0.00	1.945	100.00	0.01						
124.5	100.00	0.00	1.635	99.99	0.02						
104.7	100.00	0.00	1.375	99.97	0.05						
88.00	100.00	0.00	1.156	99.92	0.11						
74.00	100.00	0.00	0.972	99.81	0.23						
62.23	100.00	0.00	0.818	99.58	0.42						
52.33	100.00	0.00	0.688	99.16	0.78						
44.00	100.00	0.00	0.578	98.38	1.48						
37.00	100.00	0.00	0.486	96.90	2.77						
31.11	100.00	0.00	0.409	94.13	4.87						
26.16	100.00	0.00	0.344	89.26	7.92						
22.00	100.00	0.00	0.289	81.34	11.23						
18.50	100.00	0.00	0.243	70.11	13.21						
15.56	100.00	0.00	0.204	56.90	14.70						
13.08	100.00	0.00	0.172	42.20	17.43						
11.00	100.00	0.00	0.145	24.77	24.77						

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