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Characterization, Washing, Leaching, and Filtration of AZ-102 Sludge

K. P. Brooks
P. R. Bredt
S. K. Cooley
G. R. Golcar
L. K. Jagoda
K. G. Rappe
M. W. Urie

August 2000

Prepared for BNFL, Inc.
under Contract W375-LC-98-4168

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Summary

BNFL, Inc. developed flowsheets for the River Protection Project Waste Treatment Plant that use caustic leaching and/or water washing to pretreat Envelope D Hanford sludge before vitrifying the high-level waste (HLW). The pretreatment steps would remove a number of components to reduce the quantity of HLW generated. The flowsheets also use crossflow filtration to separate the leach and wash solutions from the solids between each step. The work reported here tested crossflow filtration and evaluated the washing and leaching characteristics of sludge from Tank AZ-102.

Battelle staff evaluated approximately 400 g of wet Hanford Tank AZ-102 Sludge by the pretreatment processes of ultrafiltration, dilute caustic washing, and elevated-temperature caustic leaching. The filterability of diluted AZ-102 sludge was measured with a 0.1- μm sintered metal Mott filter using a 24-in.-long, single-element, crossflow filtration system (cells unit filter [CUF]). Before washing and leaching, a 5.1-wt% solids^(a) slurry was filtered using a matrix of six 1-h conditions of varying transmembrane pressure (30 to 70 psid) and axial velocity (7 to 13 ft/s), with the filtrate being recirculated. The filtrate flux and backpulse efficiency were determined for each condition. The slurry was then concentrated to 13.7-wt% solids, a second matrix of six 1-h conditions performed at axial velocities ranging from 6 to 11.5 ft/s, and data analogous to that recorded in the first matrix obtained.

The low-solids-concentration matrix produced filtrate flux rates that ranged from 0.046 to 0.126 gpm/ft². The high-solids-concentration matrix produced filtrate flux rates that ranged from 0.020 to 0.051 gpm/ft². In the low solids matrix, the highest filtrate flux was at the first condition (50 psid, 9.5 ft/s). The initial filtrate flux declined significantly over the next four conditions, indicating filter fouling or more likely, particle deagglomeration. In the subsequent conditions, the initial filtrate flux remained steadier, and the highest filtrate flux was measured at the highest axial velocity. In the high solids matrix, the optimum condition was clearer and was found at the highest axial velocity.

For each matrix, the last test condition was a repeat of the first test condition. In both matrices, there was a significant decrease in filtrate flux between these two tests. This indicates some filter fouling occurred during each test matrix that could not be removed by backpulsing alone, although the backpulse number and duration were not optimized. The nitric acid cleaning performed at the end of the testing could partially recover the initial fluxes caused by this filter fouling.

Following testing of these two matrices, the material was washed in the CUF by continuously adding approximately 2.7 L of 0.01 M NaOH and then removing it as permeate by filtration. The purpose of these washing steps with 0.01 M NaOH was to remove water-soluble components. After washing the sludge with dilute caustic, it was combined with a concentrated caustic solution to produce a slurry containing ~3 M NaOH, which was then leached in a stirred, stainless steel vessel at 85°C for 8 h. This leaching was followed by two 0.01 M caustic washes, each conducted in a stainless steel vessel to dilute remaining soluble analytes from the interstitial liquids. Each rinse was performed at 85°C for 8 h. 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

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

for chemical and radiochemical constituents. The fraction of each component removed at each step was calculated. Key results are presented in Table S.1.

The primary components in the initial tank sludge were aluminum, iron, and sodium in roughly equal proportions. Following washing and caustic leaching, these three components remained the highest concentrations, but iron became the primary constituent, more than twice the concentration of sodium and aluminum. Based on the aluminum + iron + zirconium concentration limits in the HLW glass, the potential glass volume was reduced by ~23-30% as a result of washing and caustic leaching.

The rheological properties of the AZ-102 slurries were determined with a Haake viscometer. All three samples analyzed exhibited yield pseudoplastic and thixotropic behavior. This rheological behavior was expected, as the slurry initially added to the CUF was difficult to begin pumping, yet improved over time. The initial 5.1-wt% material had a viscosity between 4 and 16 cP. The concentrated slurry used in the second matrix had a measured solids concentration of 13.7 wt% and a viscosity between 82 and 100 cP (@ 33 s⁻¹) during the initial increase in shear^a. The viscosity steadily declined for both the decreasing shear curves and with subsequent runs. During the decreasing shear curve, the 13.7 wt% slurry had a measured viscosity at 33 s⁻¹ of between 25 and 47 cP. The final slurry samples with solids concentration of 9.2-wt % solids had viscosities at 33 s⁻¹, ranging from 167 to 209 cP. Foaming was observed in the final pretreated slurry sample (9.2 wt% solids), which may have contributed to the higher observed viscosities for this sludge.

Table S.1. Solubility of AZ-102 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	2.5	61.2	36.3
Cr	44.1	14.2	41.7
Fe	0.006	0.02	99.97
Na	80.2	-nd-	11.2
P	6.7	45.6	47.7
⁹⁰ Sr	0.003	0.007	99.99
¹³⁷ Cs	61.2	32.7	6.1

-nd- : Not determined due to difficulty in distinguishing leached sodium from added sodium. The fraction Na in the solids residue may be that added during the caustic leaching rather than that initially present in the sludge.

Particle-size distribution was measured on the initial, as-received sludge, on the 13.7 wt% concentrated slurry, and on the final caustic leached slurry. The initial sludge had a significantly larger particle-size distribution than subsequent material. Approximately 50% of the particles (on a volume weighted distribution) had a peak mode of 24 µm as compared to subsequent steps having >60% of the particles with a peak mode of approximately 1 µm. Sonication had a larger effect on the particles of the final caustic leached slurry when compared to the earlier steps. The significant reduction in particle size due to sonication may indicate that the washing and leaching steps weakened the agglomerate strength.

(a) BNFL selected 33 s⁻¹ as the reference point to compare all viscosity measurements.

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
DOE-RL	U.S. Department of Energy-Richland Operations
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
NIST	National Institute of Standards and Technology
PID	proportional-integral-derivative controller
PMG	precious metals group
PSD	particle size distribution
RPL	Radiochemical Processing Laboratory
RPP-WTP	River Protection Project Waste Treatment Plant
TMP	transmembrane pressure
TIC	total inorganic carbon
TOC	total organic carbon
TRU	transuranic
UPA	Ultrafine Particle Analyzer (Microtac)

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
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

British Nuclear Fuels Limited (BNFL) Inc. developed the River Protection Project Waste Treatment Plant (RPP-WTP) flowsheets, which plan to use caustic leaching and/or water washing to pretreat the Envelope D^(a) 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 filtration rate caused by the growth of a filter cake on the surface of the filter medium, in crossflow filtration, the majority of 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 (AZ-102) in a modified cells unit filter (CUF) filtration rig fabricated at Battelle. Similar to the studies done with AW-101, AN-107, and C-104, the permeability of the diluted AZ-102 sludge was evaluated for both high and low solids concentrations as a function of transmembrane pressure (TMP), axial velocity, and time through a single-element 0.1- μm Mott filter (Brooks et al. 1999; Hallen et al. 2000; Brooks et al. 2000). The radioactive tests with the single-element CUF unit will provide information for equipment-performance evaluation and a design basis for scaling up of the process.

The second objective of this work was to evaluate washing and leaching characteristics of the AZ-102 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 3 M 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 were 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 AZ-102 and filtrates generated during the washing and caustic-leaching steps.^(b) This report also provides a means of transmitting to BNFL the completed test instruction and raw filtration and analytical data.

(a) Envelope issues are explained in Specification 7 of Contract No. DE-RP06-96RL13308.

(b) The results presented in this report are based on work conducted under Test Plans TP-29953-069 and TP-29953-075, test instruction TP-29953-076, and Procedure TP-29953-020, Rev 1. Some data are recorded in Laboratory Record Book (LRB) #13745. Conditions for conducting these tests were given in the "AZ-102 Dewatering and Caustic Leach Test Specification," TSP-W375-99-014, Rev 0.

2.0 Test Conditions

Small-scale radioactive crossflow filtration, water-washing, and caustic-leaching tests using slurry samples from Tank AZ-102 were conducted from 1/10/2000 through 1/14/2000. The work was performed in the High Level Radiochemistry Facility (HLRF) hot cells located in the Radiochemical Processing Laboratory (RPL) facilities. Before testing, the material was homogenized and sub samples were pulled for analytical work with the as-received sample. A description of the material preparation and testing are described in the following sections.

2.1 Test Material Preparation

Battelle received samples from Tank AZ-102 from Hanford's 222-S laboratory on September 13, 1999. This material was received in four ~125-mL glass jars. Figure 2.1 lists the sample numbers along with the mass of material recovered from each jar. A total of 538.9 g of material in the jars was transferred to a 1-L glass bottle. This bottle was placed on a standard laboratory rolling mill, and mixed for 29 h and 25 min. The normal temperature in the hot-cell where the mixing process occurred is ~30–35°C.

Following mixing, the material was allowed to settle for slightly less than 4 days. After settling, the total amount of material was ~350-mL, consisting of 300 mL of settled solids and 50 mL of supernate. Approximately 58.32 g of supernate was decanted from the top of the slurry to a container labeled AZ-102 SUP. Three density measurements were made of the supernate and the average density was determined to be 1.14 g/mL. Other properties measured during this phase of testing and their values are shown in Table 2.1.

Table 2.1. AZ-102 Composite Initial Physical Properties Data

Property	Value
Settled Solids	85.7 vol%
Centrifuged Solids	Avg 75.5 vol%
Supernate Dissolved Solids	28 wt%
Supernate Density	Avg 1.14 g/mL
Approximate Undissolved Solids wt%	25 wt%
Approximate Composite wt% Solids	46 wt%

Figure 2.1. Flowsheet of Initial Compositing

The slurry remaining in AZ-102 COMP was stirred, and two ~8 mL samples were pulled with a wide-mouth pipet to verify physical homogeneity by comparing solid/liquid ratios. The two samples were put directly into centrifuge tubes and centrifuged for an hour at 1000X-G. The two subsamples were found to be 76.5 and 74.6 vol% centrifuged solids. Given that these results varied by less than 2 vol%, the sampling technique was determined to provide representative subsamples. The solids from the centrifuge tubes were recovered into a jar labeled AZ-102 Recovered Solids.

In the original test plan, two composite subsamples were needed for other tasks. The procedure for making composite subsamples involves transferring a target amount of solids material into sample jars and then adding a proportional amount of supernate back in based on the ratio of settled solids to supernatant in the original container. This procedure has been determined through previous testing to be the most consistent way to acquire repeatable representative slurry samples. The two composite subsamples were named AZ-102 MU and AZ-102 GL. Each had a target wt. of 50.0 g settled solids. The final compositions were 50.05 g of solids and 6.21 g supernate in AZ-102 MU and 49.66 g of solids and 6.05 g of supernate in AZ-102 GL. Additionally, 28.23 g of supernate was transferred from AZ-102 SUP to a bottle labeled AZ-102 SUP MU for supernatant analysis. After the supernate sample and two composites were prepared, the remaining supernate was added back to AZ-102 COMP, which was slated for CUF testing.

It was later determined that there were not enough solids to make the required amount of glass following CUF testing. Therefore, part of the solids from the composite subsamples would have to be used as CUF testing material. The work for which AZ-102 GL had been prepared was canceled and this material was used for CUF testing. AZ-102 MU was the sample intended for analytical testing. It was determined that the analytical work could be completed with less than 50.0 g of solids if extra supernate was provided. So a new bottle was labeled AZ-102 SUP-B, and 23.09 g of supernate from AZ-102 COMP and 7.18 g of supernate from AZ-102 SUP were transferred into it. Then AZ-102 MU was mixed with a steel scoop, and 27.3 g was transferred into a centrifuge tube labeled AZ-102 MU. From this point on in the report, these will be referred to as AZ-102 MU (CUF) and AZ-102 MU (analytical), respectively. AZ-102 MU (analytical) was analyzed to determine the starting composition. AZ-102 MU (CUF) was added to the CUF.

The original bottles and all tools were rinsed with 0.01 M NaOH into a bottle labeled AZ-102 Recovered Solids. AZ-102 Recovered Solids was added to the CUF during testing before the second testing matrix.

2.1.1 Observations

The AZ-102 material that was used for these tests is very cohesive/adhesive in nature. It was hard to transfer it into separate containers because the material would form a thick clinging layer on all the tools and sides of the vessels.

The material also forms strong agglomerations when it is allowed to sit for a few days. The first part of the composite work was finished toward the end of 1 week, and it was decided to pull some samples at the beginning of the next week. After the weekend when the material was being re-mixed, a couple of large lumps were observed. They broke apart without too much effort, and it was assumed that somehow they had been missed in the original mixing, even though the initial mixing had appeared quite thorough.

However, when AZ-102 MU was split after having sat for a couple of weeks, it was filled with hard grainy agglomerates that had not been present when the sample was pulled. These agglomerates varied in size, some as large as ~1 mm in diameter. These agglomerates were concentrated primarily in the bottom of the container. Attempts were made to break them apart during the re-mixing of the material, including vigorous stirring and compressing them between the jar wall and the steel scoop, with only moderate success.

2.2 Overview of Testing

For the AZ-102 crossflow filtration experiments, measurements of filtrate flux as a function of TMP and crossflow velocity were recorded for both a dilute and a concentrated slurry. The dilute feed was 5.1 wt% solids, and the concentrated feed was 13.7 wt% solids^a. Due to minimum volume constraints in the CUF and the small quantity of AZ-102 sludge available, the high solids concentration target of 20 wt% solids could not be achieved. 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 at 10-minute intervals. The system was back-pulsed at least twice between each condition, and not backpulsed at any time during the condition. The slurry temperature was maintained at $25 \pm 5^\circ\text{C}$ for all filtrate testing.

The filtration test target conditions presented later in Tables 2.2 and 2.3 were 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 tested the conditions of TMP (30 psid, 50 psid, 70 psid) and axial velocity (9.1 ft/s, 12.2 ft/s, 15.2 ft/s). However, the air pressure available and pump capability, even with the air booster, was insufficient to achieve the target velocities and pressures with the AZ-102 material. Therefore, some of the matrix points are at lower velocities.

It was hypothesized that the subsurface filter fouling (that cannot be removed by simple backpulsing) may have influenced the selection of optimum de-watering conditions as each test was 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.

The initial AZ-102 material exhibited some unexpected physical properties that affected the testing at several stages. The AZ-102 sludge composite was very cohesive and tended to form agglomerates when it settled. This may have caused problems with material transfer and with plugging and flow fluctuations in the CUF. It also could have affected the rheology measurements. After all CUF processing was complete, it was noted that the material entrained air in quantities large enough to increase its volume by almost a third. This entrained air may have been the result of pumping in the CUF. It is possible that part of the reason the target flowrates could not be reached during testing was due to these two unexpected physical properties, although entrained air was only observed at the conclusion of testing.

Following the filtration tests, the slurry was washed with 0.01 M NaOH at 25°C in the CUF system. This was accomplished through consecutive additions of the washing solution to the CUF slurry feed tank,

(a) Solids concentrations presented in this report are reported on an insoluble solids basis unless specifically delineated. Insoluble solids concentration is calculated by subtracting out the dissolved solids from the total solids.

followed by the removal of an equal amount of liquid as permeate 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 NaOH to produce a slurry consisting of ~3 M OH⁻, heated to 85°C, and held with steady mixing for 8 h. The leached slurry was then transferred back into the CUF, and de-watered. Following leaching, two 8-h, 0.01 M NaOH rinse cycles were performed at 85°C to reduce the dissolved solids contained in the interstitial liquid.

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 for the second matrix and the final slurry.

All wash solutions and slurry samples from selected process points were analyzed for chemical and radiochemical constituents. Additional samples were obtained to determine rheological properties and to verify the approximate solids loading at specified points. These samples were added back to the system after completion of the procedure when possible.

The final washed sludge was transferred into a storage container for melter feed rheological studies and HLW vitrification tests.

2.3 Testing Apparatus

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

- single tube filter module, 24-in. tube; 3/8-in. ID
- 0.1- μ m Mott liquid-service stainless steel filter
- re-circulation flow such that 5 m/s (15 ft/s) maximum linear crossflow velocity could be achieved with water; however, with the AZ-102 slurry, the target velocities (9.1–15.2 ft/s) could not be achieved.
- maximum TMP 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. An Oberdorfer progressive cavity pump (powered by an air motor) pumped the slurry from the slurry reservoir through the magnetic flow meter and the filter element. Three baffles were installed in the slurry reservoir to prevent vortex formations. The axial velocity and TMP were controlled by the pump speed (which was 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. During operation, the air booster created a pulsation at an approximate rate of 1 per second and a TMP of 5–10 psig. These pressure fluctuations may have impacted the filtration characteristics, although a test performed during the C-104 filtration compared this effect and indicated that it did not (Brooks et al. 2000).

Deionized (DI) water and dilute caustic (0.01 M NaOH) additions into the CUF were made by adding pre-weighed volumes in through a chemical addition tank located outside the hot cell. The chemical addition tank was hard piped into the cell where a long piece of flexible tubing was attached that could 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 back-pulse 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 rotometer. 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 back-pulsing was conducted by partially filling the back-pulse chamber with filtrate, pressurizing the back-pulse chamber with air, and forcing the filtrate in the back-pulse chamber back through the filter.

During the majority of the testing with 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 through a jacket surrounding 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 wash solutions at elevated temperatures (85°C), the chiller was turned off and heat tape surrounding the slurry reservoir and pump inlet tubing heated. The temperature was then maintained using a proportional-integral-derivative controller (PID) temperature controller.

The elevated-temperature caustic leaches/washes were performed in a large 2-L stainless steel beaker. The slurry in the stainless steel container was heated on a hotplate while being continuously stirred with a mixing blade. 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.

Figure 2.2. Crossflow Filtration Process Flow Diagram

The critical test measuring equipment included

- a magnetic flow meter to measure slurry re-circulation rate
- two flow meters to measure the filtrate flow rate 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 baseline filtrate flux was conducted with de-mineralized filtered water in the CUF at 20, 10, and 30 psid. The filtrate fluxes for these tests averaged 1.25, 0.55, and 1.54 gpm/ft², respectively. These fluxes were lower than the fluxes obtained with water before the start of previous testing with a C-104 sample, which averaged 1.59, 0.95, and 2.38 gpm/ft², respectively. This may indicate that a certain amount of irreversible fouling was occurring in the filter. The water testing showed very little filtrate flux reduction over the 60-min test time for the 20-psid test and 30-min test time for the 10-psid test. However, over the 30-min test at 30 psid, the flux dropped from 1.70 to 1.34. The majority of this drop was seen over the last 10-minute increment. It was decided that the CUF was sufficiently clean to begin testing with the AZ-102 sample since attempting to achieve better fluxes would be costly in both time and budget, and the water fluxes were above the required start values.

2.5 Experimental Approach

A flow sheet 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 **AZ-102 samples** were added to the CUF along with 0.01 M NaOH to create a slurry at 5.1-wt% insoluble solids concentration.

The AZ-102 material plugged the CUF for the first 20 minutes of operation and could not be pumped smoothly. For several hours after the start of testing, the system continued to show large variations in flow and pressure, indicating continued plugging. These problems could be either due to agglomerates in the slurry or just its high solids loading and cohesive nature. Once the flow and pressure readings had stabilized, it was noted that the target flowrate of 12.2 ft/s at 50 psid for the first condition could not be reached even with the help of an air booster. After several attempts to increase the flowrate, testing of the first matrix began with the highest flowrate achievable. During the first three hours of testing, occasional plugging of the CUF throttle valve occurred, resulting in pressure spikes. After that time, steady-state pressures and velocities were more easily achievable.

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 assess the effect of filter fouling over the course of testing (see Table 2.2). The system was back-pulsed two to four times between each condition. It was not backpulsed during the 1-hour test condition itself. The cognizant engineer determined the required amount of back-pulses required based on the drop in flux during the previous condition and on the amount of recovery achieved with the first two back-pulses. The filtrate flux results are found in Section 3.1.

Figure 2.3. AZ-102 Crossflow Filtration Test Experimental Steps

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

Condition #	Target Flowrate (ft/s)	Average Velocity (ft/s)	Target Pressure (psid)	Average Pressure (psid)
1	12.2	9.47	50	49.11
2	9.1	7.58	30	30.00
3	15.2	7.11	70	70.29
4	15.2	7.96	30	30.43
5	9.1	NA	70	NA
6	12.2	8.57	50	51.43
7	13.1 ^(b)	13.13	30 ^(b)	29.50

(a) Air Booster used to attain required pressure and flow.

(b) Determined to be the optimum condition.

An external air-booster was used originally for the test conditions, requiring both a high TMP and a high flow rate. However, since none of the targets could be reached with the AZ-102 even with the air-booster, it was used for all the conditions. Furthermore, Condition 5 was not performed during this testing matrix because it would have only been a replicate of Condition 3, since higher than 7.1 ft/s axial velocity was not possible at 70 psid transmembrane pressure. The test was moved directly to Condition 6.

After completion of the first test matrix, the slurry was de-watered, and the filtrate was collected in a bottle labeled **AZ-102 Filtrate #1**. Samples **AZ-102 GL**, **AZ-102 MU** and the Recovered Solids were then added to the CUF. The recovered solids material was approximately 10 vol% settled solids. The slurry was again dewatered, and the filtrate from the second de-watering processes was collected in **AZ-102 Filtrate #2**. Representative samples of these filtrates and the final combined slurry were taken for analysis. The combined slurry filtered during this second dewatering step contained agglomerates that plugged the system. The system had to be manually pulsed by repeated stop and starts of the pump for almost 45 min before steady consistent operation of the CUF was possible. The slurry was allowed to mix for an additional 15 min before proceeding with this second de-watering step.

This concentrated slurry had an insoluble solids concentration calculated at 13.7 wt%. It was run through a second test matrix to determine optimum de-watering conditions at these higher solids loading. Testing procedures for Matrix 2 were identical to the testing for Matrix 1. Since the target conditions could not be reached for Matrix 1, new achievable targets were decided upon for Matrix 2. The targeted and actual conditions are shown in Table 2.3. Once again, Condition 5 was not performed due to the difficulty in attaining high axial velocities at 70-psid transmembrane pressure.

Twenty minutes into Condition 6, the transmembrane pressure suddenly increased to >100 psid. Upon adjusting back to the original pressure setpoint, it was discovered that the maximum flowrate was up by about 20%. While this increase did not bring the flowrate up to the target, it was higher than had been achievable for any of the previous conditions. Therefore, after the completion of Condition 6, another condition was added with the pressure at 30 psid and the flowrate as high as possible. The sudden spike in pressure and corresponding jump in maximum attainable flowrate may indicate that a partial plug may have existed in the system, which was restricting flowrate throughout the previous testing. This plug may have broken loose 20 min into Condition 6, increasing the flow rate and the quantity of small particles to foul the filter. It is also possible that during Condition 6, air was suddenly entrained into the

sample, thus increasing the flowrate and blinding the filter with air bubbles. These hypotheses cannot be tested, but would be consistent with the previously noted nature of this material and observation of its performance in the CUF during the course of the test.

Table 2.3. Test Conditions for High-Solids-Loading Matrix^(a)

Condition #	Original Target Flowrate (ft/s)	Altered – Target Flowrate (ft/s)	Average Flowrate (ft/s)	Target Pressure (psid)	Average Pressure
1	12.2	7.55	7.87	50	51.07
2	9.1	6.39	6.59	30	29.00
3	15.2	5.81	5.96	70	67.74
4	15.2	As High As Possible	8.45	30	30.29
5	9.1	NA	NA	70	NA
6	12.2	As High As Possible	8.98	50	47.83
7	NA – Added Test	As High As Possible	11.53	30 ^(b)	29.36

(a) Air Booster used to attain required pressure and flow.

(b) Determined to be the optimum condition.

The slurry was washed three times with dilute caustic following the second test matrix. The total wash volume was divided into three parts and added batch-wise to the CUF. Each wash volume was approximately 900 mL. After each addition, the system was de-watered into a labeled bottle until a similar volume was removed. The wash water was added directly into the mixing tank.

After completion of the water-washing steps, the slurry was removed from the CUF and leached at 3 M NaOH in a separate vessel at 85°C for 8 h. To reach the 3 M NaOH target, 609.16 g of 7.37 M NaOH was added the slurry.

To rinse residual solids from the CUF, the slurry was pumped into the leaching beaker, and then the NaOH was added to the CUF and allowed to mix. This solution was then pumped into the leaching beaker to mix with the slurry. This resulted in a slurry with a calculated value of 5.9 wt% insoluble solids in the leaching beaker. The temperature of the slurry was raised to 85 ± 5°C and maintained there for 8 h. The slurry was continuously stirred with a mixer blade throughout the caustic leaching. Approximately 15 min before the end of the leaching period, the heat tape on the CUF was activated to pre-warm the system so that slurry could be maintained at 85 ± 5°C for the de-watering process. After 8 h, the slurry was weighed, and the amount of liquid lost through evaporation was determined. The slurry was then transferred back into the CUF for de-watering back to original solids concentration (~10 wt% solids). The amount of liquid lost through evaporation was used to rinse remaining solids out of the leach container into the CUF.

The slurry was pumped around in the CUF and heated to return its temperature to 85 ± 5°C before the de-watering process was started. Filtrate flux, TMP, temperature, and flow rate data were taken every 10 min during all de-watering steps when possible.

Following the caustic leaching steps, the solids were washed twice with 0.01 M NaOH. Each dilute caustic wash was conducted in the same manner as the caustic leach. Dilute Caustic Wash #1 consisted of 1390 g of 0.01 M NaOH added to the slurry. This produced a calculated solids concentration of ~4 wt% in the leaching beaker. The solution was heated to ~85°C and stirred for a period of 8 h, then de-watered to the pre-wash concentration. Dilute Caustic Wash #2 consisted of 1400.00 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 h, then de-watered at $85 \pm 5^\circ\text{C}$ to the pre-wash concentration. For both washes, the amount of liquid lost through evaporation was used to rinse remaining solids out of the leach container into the CUF. After the final wash, an additional ~130 mL of water was added to the system in an attempt to remove all the solids from the leaching vessel. Duplicate sub-samples of each permeate were taken and analyzed for soluble components removed.

Approximately 577 g of final washed and leached AZ-102 sludge was drained from the CUF and collected into a 1-L bottle. The CUF was rinsed once with 500 mL of DI water, and the solids were collected to be settled and recovered. The CUF was then rinsed four more times with 1000 mL of DI water, and the solids from these rinse steps were recovered to the extent possible.

It was noted in both the rheology testing and in the final collection of the material that this sludge has the capacity to retain large amounts of trapped gases (assumed to be air) when mixed in the CUF. The bottle that contained the "Final Washed Sludge" was visually full after transfer from the CUF. After settling overnight, the bottle was a little less than 2/3 full. This indicates that at the time of transfer, the material consisted of approximately 33-vol% entrained air, which was not easily removed in a short period of time.

The CUF was rinsed multiple times with water to remove all of the remaining solids and to recover the initial clean water fluxes. To assist in cleaning the filter, the water was added to the back-pulse chamber and forced backward through the filter. The wash water continued to come out of the CUF brown in color, indicating the presence of fine particles. 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 back-pulsing. 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 AZ-102 test, and it was determined that acid cleaning would be required.

One liter of 1-M HNO_3 was added to the CUF (about 600 mL of that was back-pulsed in) and pumped through the system. The nitric acid was then drained from the CUF and found to be very dark and full of solids. A second batch of ~600 mL of 1 M HNO_3 /2 M Citric acid was added to the CUF and allowed to re-circulate through the system. This second batch of nitric acid was lighter, but still brown. The system was then rinsed until a neutral pH was obtained. The external cartridge filter with a 0.05- μm rating was reattached and run before attempting to get clean water fluxes.

The clean water flux was measured again at 20, 10, and 30 psid. The resultant fluxes over 20-min tests showed a decrease in flux with time, indicating that the system was not yet free of fines. The fluxes averaged 0.3517, 0.2259, and 0.7352 gpm/ft², respectively. Comparing these values to the pre-AZ-102 water-test values of 1.25, 0.55, and 1.54 gpm/ft², respectively, it is obvious that pre-test conditions were

not achieved. Since this was the last test planned for the CUF, it was decided not to attempting further cleaning. However, it is expected that further cleaning would have eventually been able to achieve about an 80% recovery of pre-AZ-102 test flux capacity, based on the C-104 testing. Further cleaning in the full-scale 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 samples and associated analyses are shown in Table 2.4. 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 , ^{241}Am)
- strontium chemical separation followed by beta counting (^{90}Sr)
- alpha emission analysis (AEA) (for ^{241}Am , ^{239}Pu)
- total cyanide (CN^-).

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.

Table 2.4. Samples and Analyses Performed

Sampling Step	Sampling Number	Sample Type	Analysis
Initial Sludge Composite	AZ-102 MU	Sludge	PSD
	AZ-102 MU		Physical Properties
	AZ-102 MU		Chemical and Radiochemical
Initial Decanted Supernatant	AZ-102 MU SUP	Supernatant	Chemical and Radiochemical
First De-Watering After Matrix 1	CUF-AZ-102-001	Permeate	Chemical and Radiochemical
Second De-Watering After Matrix 1	CUF-AZ-102-003	Permeate	Chemical and Radiochemical
Dewatered Slurry (Before Matrix 2)	CUF-AZ-102-005	Slurry	Physical Properties
	CUF-AZ-102-006		PSD
Dewatered Slurry (After Matrix 2)	NA	Slurry	Rheology
Water Wash 1	CUF- AZ-102-015	Permeate	Chemical and Radiochemical
Water Wash 2	CUF- AZ-102-017	Permeate	Chemical and Radiochemical
Water Wash 3	CUF- AZ-102-019	Permeate	Chemical and Radiochemical
Washed Slurry	CUF- AZ-102-007	Slurry	Chemical and Radiochemical

Sampling Step	Sampling Number	Sample Type	Analysis
Caustic Leach	CUF- AZ-102-009	Permeate	Chemical and Radiochemical
After Caustic Leach	CUF- AZ-102-010	Slurry	Chemical and Radiochemical
	CUF- AZ-102-011		Physical Properties
	CUF- AZ-102-012		
Caustic Wash 1	CUF- AZ-102-013	Permeate	Chemical and Radiochemical
Caustic Wash 2	CUF- AZ-102-022	Permeate	Chemical and Radiochemical
Final Slurry	CUF- AZ-102-024	Slurry	PSD
	CUF- AZ-102-023		Chemical and Radiochemical
	CUF- AZ-102-026		
	CUF- AZ-102-025 and Final Rheology		Rheology & Physical Properties
Final Decanted Supernatant	CUF- AZ-102-027	Permeate	Acid Digest/ICP-AES (for Na)

In addition to the above analysis, several additional analyses were performed on the composited initial and final slurry. For these two slurries, a precious metals group (PMG) fusion was also performed to obtain the Pt concentration in the slurry. The additional analysis for these slurry samples included

- inductively coupled plasma-mass spectrometry (ICP-MS) (for ^{237}Np , ^{126}Sn , ^{129}I , ^{99}Tc , Pr, Rb, Ta, Pt)
- extraction and Beta Count for ^3H
- combustion release and beta count (for ^{14}C)
- cold vapor atomic adsorption spectroscopy (for Hg)
- ammonia concentration.

In addition to the analysis performed with the permeates, the original sample decanted supernatant analysis included the following:

- ICP-MS (for ^{237}Np , ^{129}I , ^{99}Tc)
- cold vapor atomic adsorption spectroscopy (for Hg)
- separations and beta counting for ^{99}Tc
- ammonia concentration.

The physical analyses of the slurries included density, weight percent dissolved and total 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 4.0. The rheological work measured shear stress as a function of shear rate. 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 further description of the experimental steps are provided in Section 5.0.

3.0 Ultrafiltration and Washing Results

This section is divided into two subsections: crossflow filtration results, and dilute caustic-washing and caustic-leaching results. The physical property and rheological results, and the particle size distribution results are presented in Sections 4.0 and 5.0, respectively

3.1 Crossflow Filtration Results

Two crossflow filtration matrices were conducted at low and high solids loading. During these matrices, filtrate fluxes were measured. Filtrate fluxes were also measured during the washing and leaching steps. These results are presented in the sections below.

3.1.1 Low Solids Loading Matrix Results

The low solids loading matrix consisted of six conditions. All were performed at 5.1-wt% insoluble solids concentration, and each was 1 hour in duration. The average filtrate fluxes from these conditions are shown in Table 3.1. As mentioned in Section 2.5, Condition 5 was not performed for this matrix. 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 was 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 and surface tension 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 (in °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 (~5.1 wt%)

Condition #	Average Velocity (ft/s)	Average Pressure (psid)	Average Filtrate Flux (gpm/ft ²)
1	9.5	49.1	0.126
2	7.6	30.0	0.082
3	7.1	70.3	0.046
4	8.0	30.4	0.055
5	NA	NA	NA
6	8.6	51.4	0.071
7	13.1	29.5	0.078

NA = Not performed

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

The initial condition (49.1 psid, 9.5 ft/s) had the highest average filtrate flux. As with previous crossflow filtration studies (Brooks et al. 1999; Brooks et al. 2000; Geeting and Reynolds 1997) on Hanford tank wastes, during each condition and between conditions, the filtrate flux steadily decreases over time. This was evidenced by the decrease in filtrate flux that was observed in Conditions #1 and #6 and Conditions #2 and #4. With similar pressure and slightly lower velocity, Condition #6 (51.4 psid, 8.6 ft/s) had a filtrate flux 44% lower than Condition #1. Condition #2 (30.0 psid, 7.6 ft/s) and Condition #4 (30.4 psid, 8.0 ft/s) were also nearly identical in pressure and velocity. In this case, there was a 32% decrease in filtrate flux from Condition #2 to Condition #4.

The initial, final, and average filtrate flux results are shown in Figure 3.2. The first four conditions show a linear decrease in initial filtrate flux in spite of backpulsing and varied TMPs. A similar linear decrease in average filtrate flux was seen with the first three conditions. This decrease was probably related more to filter fouling and the influence of particle break-up than from changes in TMP. Thus, no trend in TMP can be seen from the first three data points over the range of 30 to 70 psid. This result may indicate the need for higher backpulsing pressure and frequency to prevent this irreversible fouling.

In comparing the initial, average, and final flux values for all conditions, it can be concluded that after approximately 2-3 hours of slurry circulation in the CUF, the solids are eroded to a stable finer particle size distribution (See Section 5.1). The evidence for this assumption is determined by the rate at which equilibrium is established during one hour of circulation. During Conditions 1 & 2, the flux declines slowly over time. For Conditions 3-7, the equilibrium flux is reached more quickly. Thus in the later cases, the resistance of the deposited layer on the membrane surface is relatively unchanged during the one hour of circulation (Tarleton and Wakeman 1994).

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

Conditions #4, #6, and #7 better illustrate the influence of pressure and velocity. The lower pressure and velocity of Condition #4 (30.4 psid, 8.0 ft/s) yielded a lower filtrate flux than Conditions #6 and #7, with their higher pressure or axial velocity, respectively. The high pressure and low velocity of Condition #6 (51.4 psid, 8.6 ft/s) appeared to offset the high velocity and lower pressure of Condition #7 (29.5 psid, 13.1 ft/s) for nearly identical average filtrate fluxes. The increase seen between Condition #4 and #6 with higher transmembrane pressure would indicate a possible small, but measurable, influence of pressure on filtrate flux.

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 AZ-102 Comp (the initial feed), and again on the slurry after it contained all AZ-102 sludge material to be used, including AZ-102 MU, AZ-102 GL, and the recovery jar. The initial de-watering removed approximately 950 mL of supernatant at a filtrate flux of 0.094 gpm/ft². These filtrate flux measurements were performed at 48 psid and 10.2 ft/s. The results were approximately 30% higher than those seen for similar material during

Condition #6. The solids concentration varied from 5.1 wt% to approximately 9.2 wt% solids during the dewatering step. This increase in filtrate flux in spite of the higher solids loading may be the result of the higher axial velocity (10.2 ft/s vs. 8.6 ft/s). The second de-watering step removed an additional 730 mL of supernatant. The TMP was very similar to the first dewatering step, but the average axial velocity was much lower (5.7 vs. 10.2 ft/s) and decreased over time (from 7.3 to 4.3 ft/s). The filtrate flux for this step decreased from 0.067 to 0.024 gpm/ft². During this de-watering, the solids concentration increased from an estimated 8.7- to 13.7-wt% solids.

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 13.7 wt% solids. This matrix consisted of six conditions. As mentioned in Section 2.5, Condition 5 was not performed for this matrix. The first and sixth 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 3.3. As done previously, each condition was performed over the course of 1 h with 3 to 4 backpulses between each condition.

Table 3.2. Average Filtrate Flux for High-Solids Matrix (~13.7 wt%)

Condition #	Average Velocity (ft/s)	Average Pressure (psid)	Average Filtrate Flux (gpm/ft ²)
1	7.9	51.1	0.032
2	6.6	29.0	0.028
3	6.0	67.7	0.030
4	8.4	30.3	0.038
6	9.0	47.8	0.020
7	11.5	29.4	0.050

Unlike the previous matrix, the highest average flux did not occur at the first condition. Instead, it occurred at the condition of highest axial velocity (Condition 7). 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 increased velocity in Condition #6, there was a 38% decrease between the two conditions. Some of this decrease could be associated with a sudden change that occurred to the testing apparatus 23 min into the Condition. There was a spike in the pressure and then immediately the flow increased from 7.8 ft/s to 10.2 ft/s. The filtrate flux decreased by approximately 50%. It is postulated that build-up of particles somewhere in the system suddenly broke free, allowing an increase in flow and further coating the filter. This in turn resulted in a decrease in filtrate flux. A second postulate is that a sudden increase in entrained air in the slurry blinded the filter, resulting in a decrease in filtrate flux, although entrained air or its effects were noted only at the conclusion of testing.

Figure 3.3. Filtrate Flux as a Function of Time for the High Solids Matrix

The first three data points provide some indication as to the effect of TMP at constant axial velocity. The nearly constant filtrate flux seems to indicate that the filtrate flux was not impacted by TMP over the range from 30 to 70 psid.

A comparison between Conditions #2, #4, and #7 provides an indication of the effect of axial velocity at nearly constant TMP. With increased axial velocity, there was a significant increase in filtrate flux. This trend can best be seen in Figure 3.4 where filtrate flux was plotted as a function of axial velocity. The resulting graph is nearly linear. Overall, for the high solids matrix, the optimum condition was achieved at low pressures and high axial velocities.

Figure 3.4. Filtrate Flux as a Function of Axial Velocity for the High Solids Matrix at ~30 psid TMP

The initial, final, and average filtrate-flux results are shown in Figure 3.5. The initial filtrate flux for the first condition was significantly higher than all subsequent conditions. Condition 7 also has a slightly higher initial filtrate flux, possibly due to the significantly higher axial velocity, which removes more material during backpulsing. The remaining conditions were relatively constant in initial filtrate flux.

Figure 3.5. Initial, Average, and Final Filtrate Flux for Each Condition in the High-Solids Matrix

The filtrate fluxes for this high solids loading test matrix were lower than those seen in the previous matrix. With the 2.7-fold increase in solids concentration, the filtration flux decreased by an average of 2.3-fold (see Figure 3.6). In the case of Conditions #1, #2, and #6, the differences were larger.

3.1.4 Filtration Results During the Washing and Caustic Leaching Steps

Washing with dilute caustic was performed in three steps. In each step, approximately 900 mL was added to the CUF, and then an equal quantity of filtrate was removed. During each of the steps, the solids concentration should increase from approximately 7% up to 11%, assuming minimal solids dissolution during washing. The average filtrate fluxes for these washing steps are presented in Table 3.3.

Figure 3.6. Average Filtrate Flux Comparison between High and Low Solids Loading Matrices

Table 3.3. Average Filtrate Flux for Dewatering During Washing and Caustic Leach

Step	Average Velocity (ft/s)	Average Pressure (psid)	Average Filtrate Flux (gpm/ft ²)	High T Filtrate Flux Corrected for Temperature
Wash 1	7.4	53.2	0.038	(b)
Wash 2	7.3	49.8	0.028	(b)
Wash 3	4.0	48.2	0.014	(b)
Caustic Leach	8.8	31.0	0.17 ^(a)	0.042
Caustic Wash 1	8.4	29.5	0.17 ^(a)	0.042

(a) Performed at 85°C. Filtrate flux was not corrected for temperature using Equation 3.1.

(b) Performed near 25°C and corrected for temperature using Equation 3.1.

Each washing step required 20 to 40 min to remove the 900 mL of filtrate. With each washing step, the average filtrate flux decreased. In the case of Wash 3, this was the highest the pump would operate. The larger reduction in filtrate flux for this case was probably due to the significant reduction in average velocity.

Filtrate flux data were also taken during the caustic leach and first caustic wash de-watering steps. 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 Table 3.3. Overall, the insoluble solids concentration was increased from ~5.9 to ~8.3 wt% solids in the case of the caustic leach and from ~4.0 to ~8.8 wt% in the case of the first caustic wash. In both cases, the filtrate flux was much higher than matrices and de-watering done previously, indicating a possible significant increase in plant throughput as a result of higher operational temperatures. Elevated temperatures may, however, dissolve some solids that would reprecipitate in the filtrate after it is cooled. Using the correction in Equation 3.1, the filtrate flux was normalized to adjust the flux value on the same temperature basis. The resultant flux was reduced by approximately 75% and became more in line with the filtrate flux of Condition #1 in the low-solids test matrix.

3.1.5 Statistical Analysis

The goal of the statistical analysis is to estimate the error associated with these tests and to develop a model that adequately predicts the average flux for AZ-102 over the range of conditions studied.

The standard deviation estimate for the overall test was calculated by assuming that the change in flux with respect to time was constant after the first 10 min of each condition and calculating the standard deviation for each run. While this is not entirely true since the filtrate flux continues to decline slowly over time, it does provide a means of estimating the error associated with testing while the process is at steady-state operation. These individual condition standard deviations were then pooled over all 12 conditions using the formulas:

$$s_p = \sqrt{\frac{\sum_{j=1}^k \sum_{i=1}^{n_j} (y_{ij} - \bar{y}_i)^2}{\sum_{j=1}^k (n_j - 1)}} \quad \text{or} \quad s_p = \sqrt{\frac{\sum_{j=1}^k (n_j - 1) s_j^2}{\sum_{j=1}^k (n_j - 1)}} \quad (3.1)$$

where the y 's are the filtrate flux values, s 's are the standard deviations for an individual condition, the n 's are the number of observations in each run condition, and in our case, $k = 12$ since there were 12 sludge/run conditions. Using the above formula with all 77 data points taken, the pooled standard deviation was calculated to be 0.00786 gpm/ft².

There were 10 filtrate-flux determinations made for Condition #7 at the 5.1% sludge concentration level. Of these, 2 were taken at the 41-min mark, and 4 were taken at the 60-min mark. In contrast to the previous data, these data points should provide a reasonable estimate of the standard deviation with a reduced effect of decline due to time. A pooled estimate for standard deviation was calculated using these points and was found to be 0.00681 gpm/ft².

A statistical model can be used to understand the important factors, predict filtrate-flux performance, and control for the 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. Since the Conditions for Matrix #1 and #2 were run in series, the value of "Run" ranges from 1 (Condition 1, Matrix 1) to 12 (Condition 7, Matrix 2).

A model was developed that incorporated both the low and high solids loading matrix. The regression analysis was conducted using the average flux of the 12 conditions studied in the high and low solids loading matrices. Multicollinearities were found among the regressor terms, so model selection followed an effort to eliminate redundant terms from the model and ultimately establish a model that fit the data well and provided reliable indicators of the effect of each term remaining in the model. The model with the best fit with only statistically significant terms took on the following form:

$$\text{Average Flux} = 0.0326 + 0.00734 \cdot \text{Velocity (ft/s)} - 0.00645 \cdot \text{Run} \quad (3.3)$$

For this model, the root mean square error was 0.01364 gpm/ft² with an R-square of 0.8079.

The model shows that axial velocity has a positive relationship, and run number has a negative relationship with filtrate flux. These results were not unexpected since higher velocities remove more solids from the surface of the filter and improve filtrate fluxes while continued operation tends to foul the filter and reduce the filtrate flux.

Transmembrane pressure was not included in the model because it was found to be collinear with velocity and did not contribute to the model. Transmembrane pressure may in fact be significant to the filtrate flux. Part of the reason for this may also be that no data were taken at high pressures and high velocities (due to equipment limitations); most of the data were taken either at high pressure and low velocity or low pressure and high velocity. Thus the two parameters were related in this data set.

Similarly, solids concentration was not included in the model because solids concentration and run number were also collinear. Since all low solids load data were taken during Runs 1–6 while all high solids loading data were taken during Runs 7–12, these two effects cannot be statistically separated from each other.

One of the purposes of these models was to determine the optimum conditions without the effect of run order for both solids concentrations. In the case of both the low and high solids loading matrix, the optimum filtration condition was the highest axial velocity. Higher solids loading, of course, reduces filtrate flux, although it was not included in the model.

3.2 Sludge-Washing and Caustic-Leaching Results

The chemical and radiochemical analyses obtained from the slurry-washing and caustic-leaching tests are presented in this section. Slurry samples were taken on the initial feed before the material was added to the CUF, following the three water washes, following the caustic leach, and at the conclusion of the tests following the two caustic washes. As discussed in Section 2, a sample of decanted supernatant was taken for analysis before the material was added to the CUF. Liquid samples were taken on all permeates removed throughout the course of the wash/leach steps. The results of these analyses for non-radioactive species are shown in Table 3.4. Slurry samples are presented here on a dry basis and permeate samples on a wet basis. When a sample is dried, it is done so by holding the sample at 105°C for 24 hours.

Table 3.4 shows that the primary metals in the initial slurry were, from highest to lowest concentration, iron, aluminum, sodium, uranium, cadmium, zirconium, nickel, and calcium. Significant soluble anions present in the slurry were nitrite, sulfate, and nitrate. Comparing the initial sample and intermediate Sample #1, the initial dewatering steps as well as dilute caustic washing appear to have removed a majority of the sodium, potassium, and the anions.

During the caustic leaching, aluminum, phosphorus, silicon and chromium were removed, as well as additional potassium. The concentration of metals in the final slurry remained roughly in the same order as in the initial slurry (for the top eight components). During the course of washing and leaching, most of the soluble anions measured by IC were washed from the solids, with small amounts of chloride, phosphate, sulfate, and fluoride remaining.

The radioactive component concentrations are shown in Table 3.5. 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. Non-Radioactive Component Concentrations

Analyte	Initial Sample ^(a) μg/g	Initial Sample Supernate μg/mL	Water Wash Permeate No. 1 μg/mL	Water Wash Permeate No. 2 μg/mL	Water Wash Permeate No. 3 μg/mL	Intermediate Sample No. 1 ^(a) μg/g	Caustic Leach Permeate μg/mL	Intermediate Sample No. 2 ^(a) μg/g	Caustic Wash Permeate No. 1 μg/mL	Caustic Wash Permeate No. 2 μg/mL	Final Sample ^(a) μg/g
Ag	352	<0.61	<0.25	<0.12	<0.1	390	<0.62	235	<0.25	<0.25	470
Al	1.25E+5	217	146	108	93.	1.85E+5	1.04E+4	1.01E+5	4.91E+3	2.18E+3	1.07E+5
As	<1E+3	<6.1	<2.5	<1.2	<1.	<1E+3	9.60	<2E+3	5.80	2.60	<1E+3
B	125	44.6	14.9	15.0	11.	230.0	23.9	250.0	10.00	10.5	225.0
Ba	550	0.260	<0.098	<0.05	<0.0	808	<0.25	521	<0.10	<0.10	958
Be	13.5	<0.24	<0.098	<0.05	<0.0	20.00	0.250	12.00	<0.10	<0.10	22.50
Bi	<596	<2.4	<0.98	<0.48	<0.5	<574	<2.5	<606	<0.99	<0.99	<592
Ca	7.20E+3	9.30	5.00	4.60	4.2	1.00E+4	14.0	8.15E+3	3.90	4.20	1.10E+4
Cd	2.07E+4	<0.37	<0.15	<0.07	<0.0	2.84E+4	15.0	1.79E+4	0.870	<0.15	3.30E+4
Ce	<1E+3	<4.9	<2.0	<0.96	<1.	1.30E+3	<4.9	<1E+3	<2.0	<2.0	1.55E+3
Co	69.0	<1.2	<0.49	<0.24	<0.2	100.0	<1.2	63.0	<0.50	<0.50	120.0
Cr	2.14E+3	826.5	36.3	20.6	11.	1.58E+3	43.8	969	17.4	8.82	1.64E+3
Cu	306	0.840	<0.25	<0.12	<0.1	488	<0.62	301	<0.25	<0.25	585
Dy	<298	<1.2	<0.49	<0.24	<0.2	<287	<1.2	<303	<0.50	<0.50	<296
Eu	<596	<2.4	<0.98	<0.48	<0.5	<574	<2.5	<606	<0.99	<0.99	<592
Fe	1.40E+5	0.685	0.320	0.200	0.35	1.87E+5	3.70	1.20E+5	1.000	0.810	2.21E+5
Hg	19.1	0.212	na	na	na	na	na	na	na	na	26.6
K	6.40E+3	3.74E+3	190	115	69.	<1E+4	190	<1E+4	61.0	27.0	<1E+4
La	4.19E+3	<1.2	<0.49	<0.24	<0.2	6.33E+3	<1.2	4.04E+3	<0.50	<0.50	7.41E+3
Li	<179	<0.73	<0.29	<0.14	<0.1	<172	<0.74	<182	<0.30	<0.30	<177
Mg	805	<2.4	<0.98	<0.48	<0.5	1.60E+3	<2.5	1.05E+3	<0.99	<0.99	1.95E+3
Mn	3.37E+3	<1.2	<0.49	<0.24	<0.2	4.50E+3	<1.2	2.90E+3	<0.50	<0.50	5.38E+3

Analyte	Initial Sample ^(a)	Initial Sample Supernate	Water Wash Permeate No. 1	Water Wash Permeate No. 2	Water Wash Permeate No. 3	Intermediate Sample No. 1 ^(a)	Caustic Leach Permeate	Intermediate Sample No. 2 ^(a)	Caustic Wash Permeate No. 1	Caustic Wash Permeate No. 2	Final Sample ^(a)
	µg/g	µg/mL	µg/mL	µg/mL	µg/mL	µg/g	µg/mL	µg/g	µg/mL	µg/mL	µg/g
Mo	90.0	61.4	3.00	1.70	0.970	<287	2.30	<303	1.30	2.50	<296
Na	1.17E+5	7.02E+4	3.71E+3	2.22E+3	1.43E+3	2.52E+4	5.41E+4	2.53E+5	2.06E+4	8.70E+3	8.33E+4
Nd	2.94E+3	<2.4	<0.98	<0.48	<0.50	4.46E+3	<2.5	2.83E+3	<0.99	<0.99	5.22E+3
Ni	9.31E+3	<0.73	<0.29	<0.14	<0.15	1.37E+4	<0.74	8.71E+3	<0.30	<0.30	1.62E+4
P	1.25E+3	144	6.60	6.52	6.63	2.50E+3	163	1.65E+3	69.9	28.4	2.80E+3
Pb	1.35E+3	<2.4	<0.98	<<0.48	<0.50	1.87E+3	6.10	1,155	2.40	<0.99	2.25E+3
Pd	<4E+3	<18	<7.4	<3.6	<3.8	<4E+3	<19	<5E+3	<7.4	<7.4	<4E+3
Rh	<2E+3	<7.3	<2.9	<1.4	<1.5	<2E+3	<7.4	<2E+3	<3.0	<3.0	<2E+3
Ru	<7E+3	<27	<11	<5.3	<5.5	<6E+3	<27	<7E+3	<11	<11	<7E+3
Sb	<3E+3	<12	<4.9	<2.4	<2.5	<3E+3	<12	<3E+3	<5.0	<5.0	<3E+3
Se	<1E+3	<6.1	<2.5	<1.2	<1.3	<1E+3	<6.2	<2E+3	<2.5	<2.5	<1E+3
Si	4.95E+3	302	33.0	33.7	28.7	5.50E+3	120	1.63E+4	56.9	45.0	7.10E+3
Sn	<9E+3	<37	<15	<7.2	<7.5	<9E+3	<37	<9E+3	<15	<15	<9E+3
Sr	265	<0.37	<0.15	<0.07	<0.08	393	<0.37	255	<0.15	<0.15	473
Te	<9E+3	<37	<15	<7.2	<7.5	<9E+3	<37	<9E+3	<15	<15	<9E+3
Th	720	<24	<9.8	<4.8	<5.0	<6E+3	<25	<6E+3	<9.9	<9.9	<6E+3
Ti	58.5	<0.61	<0.25	<0.12	<0.13	84.00	<0.62	54.50	<0.25	<0.25	185
Tl	<3E+3	<12	<4.9	<2.4	<2.5	<3E+3	<12	<3E+3	<5.0	<5.0	<3E+3
U	2.70E+4	<48.8	38.0	18.0	<10.00	3.68E+4	<49.4	2.32E+4	24.0	<19.8	4.24E+4
V	<298	<1.2	<0.49	<0.24	<0.25	<287	1.40	<303	0.700	<0.50	<296
W	<1E+4	<49	<20	<9.6	<10	<1E+4	<49	<1E+4	<20	<20	<1E+4
Y	<298	<1.2	<0.49	<0.24	<0.25	<287	<1.2	<303	0.50	<0.50	310
Zn	340	<1.2	<0.49	<0.24	0.330	500	3.40	475	3.70	0.980	970
Zr	1.59E+4	<1.2	<0.49	<0.24	<0.25	2.18E+4	<1.2	1.42E+4	0.50	<0.50	3.27E+4

Analyte	Initial Sample ^(a) µg/g	Initial Sample Supernate µg/mL	Water Wash Permeate No. 1 µg/mL	Water Wash Permeate No. 2 µg/mL	Water Wash Permeate No. 3 µg/mL	Intermediate Sample No. 1 ^(a) µg/g	Caustic Leach Permeate µg/mL	Intermediate Sample No. 2 ^(a) µg/g	Caustic Wash Permeate No. 1 µg/mL	Caustic Wash Permeate No. 2 µg/mL	Final Sample µg/g
F	1.70E+3	750	50	30	2	230	<10	590	<25	<25	
Cl	<250	<100	<10	<10	<1	400	890	4.15E+3	370	370	1.35
NO ₂	6.61E+4	4.17E+4	1.93E+3	1.12E+3	66	5.50E+3	420	1.75E+3	180	180	<
Br	<250	<100	<10	<10	<1	<27	<10	<250	<25	<25	<
NO ₃	2.14E+4	1.38E+4	740	380	22	1.60E+3	160	<490	100	100	<
PO ₄	569	435	<20	<20	3	960	210	2.05E+3	160	160	
SO ₄	2.93E+4	2.11E+4	1.13E+3	600	35	2.80E+3	220	995	110	110	
C ₂ O ₄	3.90E+3	3.29E+3	160	130	8	420	30	<490	<50	<50	<
CN	13.5	9.60	3.10	2.40	2.3	8.80	1.70	5.00	1.80	1.00	8
TIC	7.99E+3	8.62E+3	790	430	42	560	900	1.12E+4	3.13E+3	645	2.87
TOC	760	1.07E+3	50	40	<4	610	<40	1.79E+3	580	60	2.75
TC	8.75E+3	9.69E+3	840	470	42	1.17E+3	900	1.20E+4	3.71E+3	705	5.62

Note: na = No analysis performed.

(a) Dry weight basis.

Table 3.5. Radioactive Component Concentrations

	Initial Sample ^(a)	Initial Sample Supernate	Water Wash Permeate No. 1	Water Wash Permeate No. 2	Water Wash Permeate No. 3	Intermediate Sample No. 1 ^(a)	Caustic Leach Permeate	Intermediate Sample No. 2 ^(a)	Caustic Wash Permeate No. 1	Caustic Wash Permeate No. 2	Final Sample ^(a)
Analyte	μCi/g	μCi/mL	μCi/mL	μCi/mL	μCi/mL	μCi/g	μCi/mL	μCi/g	μCi/mL	μCi/mL	μCi/g
⁹⁰ Sr	1.64E+4	2.04	3.26E-2	1.30E-2	1.85E-2	2.17E+4	0.136	1.27E+4	3.42E-2	2.56E-2	2.49E+4
^{239, 240} Pu	6.33	3.02E-2	3.53E-5	1.75E-5	2.10E-5	7.63	9.77E-5	4.69	1.67E-5	1.69E-5	9.83
²³⁸ Pu	0.767	3.36E-3	5.15E-5	4.08E-5	5.61E-5	1.21	5.05E-5	0.712	4.45E-5	4.25E-5	1.57
²³⁶ Pu	<0.05	3.32E-5	<3E-7	<1E-6	<2E-7	<0.06	<1E-7	<0.05	<3E-7	<2E-7	<0.3
²⁴¹ Am (AEA)	113	2.09E-4	1.53E-4	3.41E-5	4.99E-5	135	3.24E-5	90.0	3.36E-5	1.32E-4	175
^{243, 244} Cm	0.300	2.82E-5	4.90E-5	4.81E-5	6.54E-5	0.25	4.33E-5	<0.3	5.30E-5	4.91E-5	0.281
²⁴² Cm	9.67E-2	<1.5E-5	<2E-7	<2E-7	<2E-7	7.95E-2	<1E-7	<0.07	<2E-7	<3E-7	<0.06
Total Alpha	143.3	na	na	na	na	na	na	na	na	na	
⁶⁰ Co	4.71	<6E-3	<4E-4	<5E-4	<4E-4	5.55	<5E-4	2.03	<5E-4	<6E-4	7.40
¹⁰⁶ RuRh	na	na	<1E-1	<7E-2	<6E-2	5.71	<9E-2	1.18	<6E-2	<4E-2	11.8
¹²⁵ Sb	24.1	<7E-1	<6E-2	<5E-2	<4E-2	19.6	<6E-2	11.0	<4E-2	<3E-2	40.3
¹³⁴ Cs	1.98	1.20	5.29E-2	3.05E-2	1.85E-2	<0.6	4.36E-2	<0.1	2.18E-2	9.95E-3	<7E-1
¹³⁷ Cs	1.80E+3	1.12E+3	48.0	29.1	17.8	546	42.6	160	20.8	9.09	169
¹⁵⁴ Eu	44.7	<3E-2	<3E-3	<2E-3	<2E-3	57.1	<2E-3	19.2	<2E-3	<2E-3	72.8
¹⁵⁵ Eu	83.4	<5E-1	<4E-2	<3E-2	<3E-2	105	<4E-2	35.6	<3E-2	<2E-2	134
²⁴¹ Am (GEA)	126	<5E-1	<4E-2	<3E-2	<3E-2	156	<4E-2	57.1	<3E-2	<2E-2	203
¹⁴ C	1.51E-3	na	na	na	na	na	na	na	na	na	2.36E-3

	Initial Sample ^(a)	Initial Sample Supernate	Water Wash Permeate No. 1	Water Wash Permeate No. 2	Water Wash Permeate No. 3	Intermediate Sample No. 1 ^(a)	Caustic Leach Permeate	Intermediate Sample No. 2 ^(a)	Caustic Wash Permeate No. 1	Caustic Wash Permeate No. 2	Final Sample ^(a)
	µg/g	ng/mL	ng/mL	ng/mL	ng/mL	ng/mL	ng/mL	ng/mL	ng/mL	ng/mL	µg/g
Cs	23.6	20.1	na	na	na	na	na	na	na	na	2.37
Pr	481	na	na	na	na	na	na	na	na	na	796
Ta	2.21	na	na	na	na	na	na	na	na	na	20.3
²³⁷ Np	103	0.140	na	na	na	na	na	na	na	na	175
Rb	232	na	na	na	na	na	na	na	na	na	228
⁹⁹ Tc	22.7	13.8	na	na	na	na	na	na	na	na	1.55
¹²⁶ Sn	<0.7	na	na	na	na	na	na	na	na	na	<0.9
¹²⁷ I	20.5	6.34E-2	na	na	na	na	na	na	na	na	19.4
¹²⁹ I	<3.1	1.53E-2	na	na	na	na	na	na	na	na	<2.5
^{135,137} Cs	0.443	0.505	na	na	na	na	na	na	na	na	0.423
Tritium	4.09E-2 ^(b)	na	na	na	na	na	na	na	na	na	1.61E-2 ^(b)

Note: na = No analysis performed.

(a) Dry weight basis. (b) Tritium is measured in µCi/g wet sample

Table 3.6. Distribution of Non-Radioactive Analytes in the Wash Steps

Analyte	Water Wash Efficiency^(b) (%)^(b)	Caustic Leach Efficiency (%)	Caustic Wash No. 1 Efficiency (%)	Caustic Wash No. 2 Efficiency (%)	Residue (%)
Ag	<0	<1	<1	<1	>96.9
Al	2.5	59.3	1.9	<0.003	36.3
As	<12	81.1	18.9	<19	0
B	62.5	20.8	<1	16.7	0
Ba	<0.1	<0.3	<0.1	<0.1	>99.4
Be	<3	13.9	<5	<5	86.0
Ca	60.4	39.6	<8	<8	0
Cd	<0.00	0.66	<0.01	<0.01	99.3
Ce	<1	<3	<2	<2	>92.4
Co	<3	<10	<5	<5	100.0
Cr	44.1	13.6	<0.1	0.54	41.7
Cu	<0.4	<1	<0.5	<0.6	>97.5
Fe	0.006	0.02	<0.001	<0.001	99.97
K	80.9	19.1	<2	<2	0
La	<0.1	<0.2	<0.1	<0.1	>99.6
Mg	<0	<1	<1	<1	>97.0
Mn	<0.1	<0.2	<0.1	<0.1	>99.5
Mo	65.9	11.6	2.9	19.6	0
Na	80.2	nd	nd	nd	11.2
Nd	<0.2	<0.5	<0.2	<0.3	>98.9
Ni	<0.02	<0.0	<0.02	<0.02	>99.9
P	6.7	44.5	<0.2	0.7	47.7
Pb	<0.4	3.8	<0.5	<0.6	<96.2, >94.7
Si	16.1	14.0	<1	4.5	<65.3, >64.7
Sr	<0.3	<1	<0.4	<0.4	>98.1
Ti	<1	<3	<2	<2	>92.0
U	1.8	<1	1.22	<1	<96.5, >95.8
V	<19	93.1	6.9	<30	0
Y	<1	<4	<2	<2	>90.5
Zn	0.6	4.3	4.5	<1	<90.6, >90.0
Zr	<0.01	<0.04	<0.02	<0.02	>99.9
F	77.5	<12	<44	<43	<22.5
Cl	<2	73.9	0	26.1	0
NO ₂	94.1	0	0	5.9	0
NO ₃	87.2	0	4.3	8.5	0
PO ₄	8.5	40.6	21.5	29.4	0
SO ₄	92.5	0	1.11	6.4	0
C ₂ O ₄	100	0	<30	<29	0
CN	75.5	0.19	20.3	4.2	0

Analyte	Water Wash Efficiency ^(b) (%) ^(b)	Caustic Leach Efficiency (%)	Caustic Wash No. 1 Efficiency (%)	Caustic Wash No. 2 Efficiency (%)	Residue (%)
TIC	20.0	9.9	70.1	0	0
TOC	4.3	<4	81.5	0	<14.2, >10.4
TC	18.0	8.5	73.4	0	0
(a) Accounts for carry-over of interstitial liquid.					
(b) Accounts for material removed in the initial dewatering steps.					
Note: nd = Not determined due to large amount of sodium added as wash steps.					

The removal efficiencies both for the initial dilute washing and the caustic leaching of the non-radioactive and radioactive components are shown in Table 3.6 and Table 3.7. The results indicate that 80.2% of the sodium was removed from the slurry during the water-washing steps. Nearly all of the soluble fluoride, nitrite, nitrate, sulfate, oxalate, and cyanide were removed during the first water washes. Chloride and phosphate were the only exceptions, with phosphate having only 8.5% removal, and no measurable chloride was removed during the water washes. Other non-radioactive components with significant removal efficiencies during the water wash were boron with 63% removal, calcium with 60% removal, chromium with 44% removal, potassium with 81% removal, and molybdenum with 66% removal.

The TIC and TOC concentrations and removal efficiencies are somewhat suspect. The highest concentrations of TIC and TOC were found in the first caustic-wash solution, where 70 and 82% of the total was removed. It would seem unlikely that both TIC and TOC would be removed in this particular step. Analytical error was possibly the culprit.

In terms of radioactive components, 61% of the ¹³⁷Cs was removed during the initial water-wash steps.

The caustic-leach and subsequent caustic-washing steps were performed at estimated 2.4, 0.9, and 0.38 M NaOH concentrations. The leaching efficiencies of these steps are also shown in Tables 3.6 and 3.7. While only 1.9% of the aluminum was removed during the dilute caustic washing, 59.3% was removed during the caustic-leaching step.

Table 3.7. Distribution of Radioactive Analytes in the Wash Steps

Analyte	Water Wash Efficiency (%) ^(b)	Caustic Leach Efficiency (%)	Caustic Wash No. 1 Efficiency (%)	Caustic Wash No. 2 Efficiency (%)	Residue (%)
⁹⁰ Sr	0.003	0.007	0	0.001	99.99
^{239, 240} Pu	0.008	0.01	0	0.002	99.98
²³⁸ Pu	0.11	0.01	0.03	0.033	99.81
²⁴¹ Am (AEA)	0.001	0	0.0002	0.001	99.997
^{243, 244} Cm	0.69	0	0.27	0.20	98.8
⁶⁰ Co	<0.2	<0.1	<0.2	<0.2	>99.4
¹⁰⁶ RuRh	<20	<11	<11	<7	>49.7
¹²⁵ Sb	<4	<2	<2	<2	>90.1
¹³⁴ Cs	65.2	30.1	3.8	0.81	0
¹³⁷ Cs	61.2	29.6	3.0	0.17	6.1
¹⁵⁴ Eu	<0.1	<0.04	<0.06	<0.06	>99.7
¹⁵⁵ Eu	<0.8	<0.4	<0.5	<0.3	>97.9
²⁴¹ Am (GEA)	<0.5	<0.3	<0.3	<0.2	>98.6
(a) Accounts for carry-over of interstitial liquid.					
(b) Accounts for material removed in the initial dewatering steps.					

The quantity of HLW glass produced from the as-received AZ-102 waste would have been limited by the high aluminum oxide concentration. With a DOE limit of 21% for the $\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 + \text{ZrO}_2$ and a mass of 207 g of dried initial sample going into the CUF, 450 g of HLW glass would be produced with the initial sludge. Even with almost 64% removal of Al from the sample after washing and leaching, $\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 + \text{ZrO}_2$ are still the limiting constituents in the glass. However, with the aluminum reduction, approximately 100-130 g of dried material would remain after caustic leaching, producing 315-345 g of HLW glass. Assuming no material was lost or removed during the process, this would be a glass mass reduction of 23-30%.

Other non-radioactive constituents significantly removed during the caustic-leaching process were arsenic, boron, calcium, potassium, and vanadium. Additionally, greater than 50% of the aluminum, chromium, phosphorus, and greater than 75% of the sodium, IC measured anions, TOC, and TIC were removed during the combined washing and leaching steps. Of the radioactive components, only ¹³⁴Cs, ¹³⁷Cs, and possibly tritium were removed in significant quantities during the caustic-leaching process.

Table 3.8 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

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

Table 3.8. Mass Recovery

Analyte	Recovery	Analyte	Recovery	Radionuclide	Recovery
Ag	81%	P	238%	⁹⁰ Sr	90%
Al	120%	Pb	103%	^{239, 240} Pu	92%
As	-	Si	134%	²³⁸ Pu	121%
B	532%	Sr	108%	²⁴¹ Am (AEA)	92%
Ba	106%	Th	0%	^{243, 244} Cm	54%
Be	106%	Ti	186%	²⁴² Cm	1%
Ca	97%	U	98%	⁶⁰ Co	91%
Cd	97%	V	-	¹²⁵ Sb	97%
Ce	-	Y	-	¹³⁴ Cs	79%
Co	105%	Zn	183%	¹³⁷ Cs	88%
Cr	102%	Zr	123%	¹⁵⁴ Eu	95%
Cu	116%	F	81%	¹⁵⁵ Eu	93%
Fe	96%	Cl	-	²⁴¹ Am (GEA)	94%
Hg	81%	NO ₂	78%	¹⁴ C	88%
K	88%	NO ₃	78%	Tritium	95%
La	107%	PO ₄	631%	Cs	6%
Mg	146%	SO ₄	86%	Pr	93%
Mn	97%	C ₂ O ₄	102%	Ta	517%
Mo	115%	CN	674%	²³⁷ Np	95%
Na	114%	TIC	269%	Rb	55%
Nd	108%	TOC	455%	⁹⁹ Tc	4%
Ni	105%	TC	285%	¹²⁷ I	53%
				^{135, 137} Cs	54%

This value provides a means of evaluating the closure of the mass balance (i.e., how much of each component was not accounted for). In general, the recoveries were reasonably close to 100%. There were some cases where the recoveries were very much larger or much smaller than 100%. These generally occur for analytes at very low concentrations throughout the process. These deviations also occur when there is significant error in one or more of the analyses (e.g., TOC, TIC). The major constituent recoveries indicate that very little of the total slurry mass was unaccounted for during the process.

Recoveries for the radionuclides also appear reasonably close to 100%, especially with the major radionuclides. In some cases, analysis was performed on the starting and final material, but not on the permeate samples removed throughout the washing and leaching process. In this case, the recovery provides an estimate of the removal efficiency during the washing and leaching. For example, the recovery indicates that >96% of the ⁹⁹Tc, 47% of the ¹²⁷I, and only 5% of the tritium were removed during processing. Without the full data set, it is not possible, however, to distinguish poor mass recoveries from high removal efficiencies.

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. The ^{241}Am water-wash DFs were between 100,000 and 450,000, and the caustic-leach DFs were 400,000. These high DFs indicate good solid/liquid separations using the Mott 0.1- μm sintered metal filter.

4.0 Physical-Properties and Rheological-Properties Testing

This section describes physical and rheological analyses conducted on samples of AZ-102 slurry. The physical and rheological analyses included density, weight percent (wt%) and volume percent (vol%) solids, and shear stress as a function of shear rate.

Physical measurement samples were taken three times during the course of testing: 1) before the second filtration test matrix but after the material had been dewatered to approximately 13.7 wt% solids (concentrated slurry—CUF-AZ102-006); 2) after the caustic leaching step and the caustic permeate had been removed from the slurry to approximately 9 wt% solids (caustic leached slurry—CUF-AZ102-012); and 3) at the end of testing, after washing and caustic leaching the slurry at approximately 9.2 wt% solids (final slurry—CUF-AZ102-025).

Rheological measurements were taken three times during the course of testing: 1) after the first filtration test matrix at approximately 5.1-wt% solids (initial slurry); 2) after the second filtration test matrix at approximately 13.7 wt% solids (concentrated slurry); and 3) at the end of testing, after washing and caustic leaching the slurry at approximately 9.2 wt% solids (final slurry). Each of these subsamples were taken directly from the ultrafiltration loop and immediately characterized by shear stress versus shear rate. Following rheological measurements, the material was returned to the CUF for continued testing, thus no sample number applies for these measurements.

4.1 Physical-Properties Analysis

The above-described AZ-102 physical property samples were analyzed for density of the bulk slurries, settled solids, centrifuged solids, and centrifuged supernatant. The density results are listed in Table 4.1. The weight percent (wt%) and volume percent (vol%) settled solids (on a wet basis), wt% and vol% centrifuged solids (on a wet basis), and wt% total solids (on a dry basis) were measured for these samples as well. The wt% and vol% solids results are listed in Table 4.2.

For this testing, a known mass of each slurry was placed in duplicate in volume-graduated centrifuge cones. The duplicates were then allowed to settle for 3 days. The total mass (M_B) and volume (V_B) of the slurry after it was allowed to settle were recorded and the density of the bulk slurry was calculated ($D_B = M_B/V_B$). These results appeared to be biased low and had a high relative percent difference, probably because of entrained gas as well as an inability to clearly measure the total sample volume due to material smeared on the sides of the centrifuge tubes. Therefore, the bulk slurry densities were recalculated later in the work using volumes recorded following centrifugation. Following settling, the volume of the settled solids (V_{ss}) and volume of clarified supernatant (V_{sl}) were recorded. The vol% settled solids were then calculated ($Vol\%_{ss} = V_{ss}/V_B \times 100\%$). For previous samples in the BNFL project, a portion of the clarified supernatant was then transferred to a graduated cylinder to determine supernatant density. For these samples of AZ-102, there was insufficient liquid to make this determination.

Table 4.1. Density Measurements for Samples of AZ-102

Average Values	Bulk Density, g/mL			
	Centrifuged Slurry	Settled Solids	Centrifuged Solids	Centrifuged Supernatant
Concentrated Slurry CUF-AZ102-006	1.09	1.07	1.36	1.016
Relative % Difference	2%	6%	2%	0.1%
Caustic Leached Slurry CUF-AZ102-012	1.16	1.15	1.35	1.108
Relative % Difference	1%	2%	1%	4%
Final Slurry CUF-AZ102-025	1.05	1.07	1.20	1.040
Relative % Difference	0%	NA	6%	0%

(a) The relative % difference is between the sample and its duplicate and provides a means for showing sample variability.

Table 4.2. Wt% and Vol% Solids Measurements for Samples of AZ-102

Average Values	Wt% Settled (wet basis)	Wt% Centrifuged (wet basis)	Vol% Settled (wet basis)	Vol% Centrifuged (wet basis)	Wt% Total (dry basis)
Concentrated Slurry CUF-AZ102-006	95	33	96	26	14.8
Relative % Difference	2%	0%	5%	4%	0%
Caustic Leached Slurry CUF-AZ102-012	96	32	96	28	16.0
Relative % Difference	1%	3%	3%	4%	39%
Final Slurry CUF-AZ102-025	94	30	93	26	9.8
Relative % Difference	1%	3%	NA	8%	5%

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

Before centrifugation, the supernatant following solids settling could not be removed from the centrifuge cone without disturbing the settled solids; therefore, the mass of the settled solids (M_{ss}) could not be measured directly. The mass of the settled solids was calculated. This was done by first calculating the mass of the settled supernatant in the centrifuge cone using the measured centrifuged supernatant following solids settling density and volume ($M_{sl}=D_{cl} \times V_{sl}$), then subtracting this mass for 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 ($D_{ss}=M_{ss}/V_{ss}$) as well as the wt% settled solids ($Wt\%_{ss}=M_{ss}/M_B \times 100\%$).

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 that all mass lost during the drying process was water and not another volatile component, the weight percent total solids in the bulk slurry was calculated ($Wt\% \text{ total solids} = [M_{dcs}+M_{dcl}]/[M_{cs}+M_{cl}] \times 100\%$).

The density of the centrifuged supernatant for the concentrated slurry was 1.016 g/mL. As would be expected, this value increased to 1.108 g/mL following addition of the 3 M NaOH leach solution. Following the final water wash, the supernatant density was down to 1.040 g/mL.

The wt% settled solids were between 94 and 96% and the vol% settled solids were between 93 and 96% for all three samples analyzed. Similarly, the wt% and vol% centrifuged solids for all three samples were between 30 and 33% and 26 and 28%, respectively. Based on the similarity between the wt% and vol% settled and centrifuged solids over the course of testing, it would seem that the CUF test matrix and leaching did not significantly effect the solids packing characteristics of this sludge.

An additional calculation was performed to determine the wt% solids in the samples, excluding all interstitial liquid (wt% undissolved solids). This 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:

$$Wt\% \text{ undissolved solids} = \left(\frac{1 - \frac{M_{dsc}}{M_{cs}}}{1 - \frac{M_{dcl}}{M_{cl}}} \right) \times \frac{M_{cs}}{M_B} \times 100\% \quad (4.1)$$

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 was water loss from interstitial liquid. The results of this calculation are listed in Table 4.3 along with the wt% dried residue from the centrifuged solids (Solids Residue= $M_{dcs}/M_{cs} \times 100\%$), and dried centrifuged supernatant (Supernatant Residue= $M_{dcl}/M_{cl} \times 100\%$).

Table 4.3. Results of Wt% Residual Solids and Undissolved Solids Calculation Following Drying at 105°C for 24h for Samples of AZ-102

Sample	Wt% Residual Centrifuged Solids	Wt% Residual Centrifuged Supernatant	Wt% Undissolved Solids
Concentrated Slurry CUF-AZ102-006 Average	41.65	1.55	13.6
Relative % Difference	0.2	6	1
Caustic Leached Slurry CUF-AZ102-012 Average	37.4	10.45	9.5
Relative % Difference	1	10	2
Final Slurry CUF-AZ102-025 Average	32.6	1.7	9.2
Relative % Difference	NA	NA	NA

NA – Not applicable, the centrifuged liquid from CUF-AZ102-025 was spilled so no duplicate was available. A relative percent difference requires two values.

4.2 Rheological and Flow Properties

The AZ-102 initial diluted feed, dewatered slurry (similar to CUF-AZ102-006), and final slurry (same as CUF-AZ102-025) were analyzed for shear stress as a function of shear rate from approximately 0.1 to 500 s⁻¹ or 1000 s⁻¹. The slurries were analyzed at 25°C using a Haake M5 measuring head modified for hot cell operations. An MVI measuring geometry was used on the Haake. A 49.9 cP standard, Brookfield lot 102298, was used to check the calibration of the instrument before samples were analyzed.

Samples of the initial diluted slurry and the dewatered slurry were collected directly from the ultrafiltration test loop and immediately analyzed. The final slurry sample was collected in a 125-mL wide-mouth jar and analyzed approximately 1 day later. Before analyzing the final slurry sample, the sample in the jar was shaken to combine the separated liquid and solid layers. Shear stress as a function of shear rate data was obtained by measuring the shear stress produced at a specific shear rate.

As requested by the client, samples were allowed to sit in the system for 5 minutes before analysis. The shear rate was then gradually increased from 0 to either 500 s⁻¹ or 1000 s⁻¹, generating the increasing shear rate curve, and then back down to 0 s⁻¹ generating the decreasing curve. For analysis conducted to 1000 s⁻¹, the shear rate was increased to 1000 s⁻¹ over 5 min. The shear rate was then held at 1000 s⁻¹ for 5 min, and then decreased to 0 s⁻¹ over 5 min. In general, analyses of this duration can exhibit solids settling problems. Shorter duration runs from 0 to 500 s⁻¹ over 2 min followed by a decreasing curve from 500 to 0 s⁻¹ over 2 min were used to look for settling behavior. As will be discussed, no evidence of settling in the slurries was observed during these analyses.

Rheograms for the standards, samples, and duplicates are presented in Figures 1 through 15 in Appendix F. The measured viscosity at 33 s⁻¹ for both the increasing and decreasing curve are presented in Table 4.4. The first sample of the initial diluted feed was analyzed from 0 to 1000 s⁻¹ two times without removing the sample from the instrument. The viscosity of the sample was near the lower limit for the instrument, roughly 2 cP. This sample displayed a low viscosity, between 12 and 2.5 cP, between 33 and 430 s⁻¹. A yield point was detected, but below the quantification limit of 1 Pa. A decrease in viscosity with increasing shear rate above a yield point is referred to as yield pseudoplastic behavior. All AZ-102 samples displayed yield pseudoplastic behavior. The initial slurry samples showed a decrease in viscosity between the increasing shear rate curve and the decreasing shear-rate curve as well as between runs. This decrease in viscosity with increasing shear history is referred to as thixotropic behavior.

Above approximately 430 s⁻¹, a sharp rise in shear stress with shear rate was observed for the initial slurry. This represents the onset of Taylor Vortices. Taylor Vortices are the result of a secondary flow that occurs as the inner cylinder of the concentric-cylinder instrument rotates. Taylor Vortices result from analyzing a material at too high a shear rate. All data collected above the onset of Taylor vortices is invalid. Taylor vortices are expected when the following two equations are satisfied:

$$Re > 41.3[R_2/(R_2-R_1)]^{1/2}$$

$$Re = \gamma(R_2-R_1)^2\rho/\mu$$

Where R_1 = Radius of Inner Cylinder (0.02004 m for MVI)

R_2 = Radius of Outer Cylinder (0.02100 m for MVI)

γ = Shear Rate (s⁻¹)

ρ = density (g/mL)

μ = Apparent viscosity (Pa•s)

For the initial slurry, the density was 1.09 g/mL and the apparent viscosity of the slurry at the onset of Taylor Vortices was approximately 2.5 cP (0.0025 Pa•s). Applying the above equations, Taylor Vortices would be expected at 480 s⁻¹. This value of 480 s⁻¹ is within 12% of the observed value of 430 s⁻¹. Several analyses of the initial slurry were conducted only to 500 s⁻¹ to provide better resolution of the onset. These analyses show an onset of 460 s⁻¹, which is within 4% of the predicted value.

Table 4.4. Results for Yield Stress and Viscosity at 33 sec⁻¹ for Samples of AZ-102 Slurry Along with Observed Onset of Taylor Vortices

Sample	Analysis	Yield Stress (Pa)	Upper Shear Rate (s ⁻¹)	Viscosity (cP)		Taylor Vortices Onset (s ⁻¹)
				Increasing Curve At 33 s ⁻¹	Decreasing Curve At 33 s ⁻¹	
Initial Slurry	Sample 1 Run 1	<1	1000	12	BD	430
	Sample 1 Run 2	<1	1000	5	BD	430
	Sample 2 Run 1	<1	500	16	6	460
	Sample 2 Run 2	<1	500	13	4	460
	Sample 2 Run 3	<1	1000	11	5	430
Concentrated Slurry	Sample 1 Run 1	3	1000	100	29	ND
	Sample 1 Run 2	1.2	1000	39	25	940
	Sample 2 Run 1	2.6	500	82	47	ND
	Sample 2 Run 2	1.8	500	58	40	ND
	Sample 2 Run 3	1.5	1000	49	35	940
	Sample 2 Run 4	1.3	1000	43	32	890
Final Slurry	Sample 1 Run 1	8	1000	172	171	NA
	Sample 2 Run 1	6.5	1000	167	189	NA
	Sample 2 Run 2	8	1000	205	209	NA

ND – Not detected, analysis only conducted to 500s⁻¹.

NA – Not applicable, onset of Taylor Vortices above limit of viscometer.

BD – Below detection, detection limit for Haake with MVI is approximately 2cP.

The concentrated slurry displayed yield pseudoplastic and thixotropic behavior. This sample displayed a yield stress of approximately 2.5-3.5 Pa. The initial sample displayed high viscosity on the increasing shear-rate curve along with a bump between 700 and 900 s^{-1} . This behavior was not seen during the decreasing shear rate curve of this sample or any of the duplicate analyses. This behavior was probably the result of large solids or agglomerates that were broken up at the higher shear rates. The onset of Taylor vortices occurred at 940 s^{-1} , and 890 s^{-1} for this sample. Using a density of 1.16 g/mL and a viscosity of approximately 0.045 Pa•s, the expected onset was 815 s^{-1} . The expected value of 815 s^{-1} was within approximately 15% of the observed values. As with the initial slurry sample, all data collected above the onset of Taylor vortices is invalid.

The final slurry displayed yield pseudoplastic behavior, but no thixotropy was observed. This sample displayed a yield stress of approximately 6 – 8 Pa. This sample was removed from the ultrafiltration unit and stored in a jar for approximately 1 day before the shear-stress versus shear-rate analysis was conducted. This sample was gently agitated in the jar and loaded into the rheometer. The level of material in the rheometer was monitored during the analysis. The level of material was observed to drop during the course of each analysis, ~20 min in duration. This level drop appeared to be the result of gas entrained in the slurry. As the material was sheared, these entrained bubbles were released, decreasing the level of material in the instrument. The level did not drop sufficiently to cause analytical errors in the analyses.

The apparent viscosity of the final slurry decreased from approximately 200 to 12 cP over the range of 33 to 1000 s^{-1} . This appears unexpectedly high compared to the viscosity of the dewatered slurry which decreased from approximately 50 to 6 cP over the range of 33 to 940 s^{-1} . The concentrated slurry had slightly a higher solids content than the final slurry, 9.5 versus 9.2 wt% undissolved solids, and 32 versus 30 wt% centrifuged solids, respectively. Given the slightly higher solids content, one may anticipate that the viscosity of the two slurries would be similar or that the concentrated slurry would be higher. The higher viscosity of the final slurry must be the result of a change in the solids properties beyond just solids content and could be the result of entrained gas bubbles mentioned previously. Entrained gas bubbles generally increase the viscosity of a slurry. As discussed in Section 5.0, the final slurry also contained significantly more fine particles. These finer particles could also be responsible for the increase in viscosity of this sample over the concentrated slurry sample.

5.0 Particle Size Distribution

The PSD of the initial sludge composite (Sample CUF-AZ-102 MU), the de-watered slurry before Matrix 2 (Sample CUF-AZ-102-005), and the final slurry (Sample CUF-AZ-102-024) is described below. The first sample was the initial composite AZ-102 sludge before it was diluted and fed into the CUF (initial sludge). The second sample was the slurry that was taken after Matrix 1 testing was completed and the slurry was de-watered (concentrated slurry). The third sample was the final slurry, which was caustic leached and was caustic washed twice (final slurry). The sample number associated with the process flowsheet step of crossflow filtration testing is presented in Table 2.3, and the experimental process is described in detail in Section 2.6.

5.1 Experimental

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 the Microtrac X-100 and Microtrac UPA analyzers was checked against National Institute of Standards and Technology (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 PSD of these three samples and their duplicates was 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 sec, and 4) circulation at 60 mL/s with 40 W sonication for 90 sec for the second time. 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 PSD of these three samples and their duplicates was then repeated using the Microtrac UPA under conditions of Brownian motion. The results on a volume-weighted basis and on a number-weighted basis are reported.

The suspending medium for these analyses was surrogate supernatants based on the ICP-AES and IC data obtained for the applicable AZ-102 supernatant. The composition of these three supernatants is reported in Table 5.1.

Table 5.1. Surrogate Supernatant Composition

	Initial Sludge	Concentrated Slurry	Final Slurry
Component	Concentration (M)	Concentration (M)	Concentration (M)
SiO ₂	1.08 E-02	1.32 E-03	1.61 E-03
NaNO ₃	1.27 E-01	8.18 E-03	1.61 E-03
NaOH	3.00 E-01	2.60 E-02	1.78 E-01
KNO ₃	9.59 E-02	8.44 E-03	6.92 E-03
Al(OH) ₃	8.00 E-03	6.04 E-03	8.07 E-02
Na ₂ C ₂ O ₄	3.74 E-02	3.41 E-03	5.68 E-04
Na ₂ SO ₄	2.19 E-01	1.65 E-02	1.15 E-03
Na ₂ HPO ₄ - 7 H ₂ O	4.58 E-03	5.26 E-04	1.68 E-03
NaF	3.95 E-02	4.74 E-03	1.32 E-03
NaNO ₂	9.05 E-02	8.07 E-02	5.43 E-04
Na ₂ CO ₃	7.18 E-01	9.25 E-02	5.38 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.

5.2 Particle Size Distribution Results

In Figure 5.1, the averaged PSDs for the initial sludge, concentrated slurry, and final slurry in cumulative under-size-percentage form are presented for the Microtrac X-100 system. In Figure 5.2, these results are illustrated in a histogram on the volume-weighted PSD. The reproducibility of the two replicate PSD plots for the initial sludge and concentrated slurry samples suggest that the slurry was thoroughly homogenized, and each extracted sample was a representative specimen. A close examination of the two final slurry replicate plots (Figures 5.1 and 5.2) shows a well-defined peak at 6.5 μm in the final slurry duplicate sample occupying 23% of the volume or mass particles. The final slurry sample, on the other hand, exhibits a less-defined peak at 6.5 μm that occupied 9% of the volume-weighted distribution. Despite this slight difference, the overall distribution shape and the location of the peaks for both final slurry replicates compare very well and suggest that the replicates are reasonably reproducible.

Figure 5.1. Cumulative Under-Size Percentage Distribution for AZ-102 Slurries Using the Microtrac X-100

Figure 5.2. Volume-Weighted Distribution for AZ-102 Slurries Using the Microtrac X-100

The cumulative under-sized-percentage plots using the UPA system (see Figure 5.3) show the samples and their duplicates. Once again the replicates are reasonably reproducible within experimental error. The PSD analysis (Figures 5.1 and 5.3 combined) of the concentrated slurry and the final slurry indicate that the large majority (>90%) of the volume and number of the particles have diameters greater than 0.03 and less than 10 μm . The PSD of the initial slurry, on the other hand, indicates that 45 % of the volume of particles are greater than 0.1 and less than 10 μm , and 55% of the volume of particles have diameters greater than 10 and less than 75 μm .

The volume-weighted distribution plots (see Figure 5.2) of the initial sludge show a bimodal distribution formed from overlapping two Gaussian distribution peaks while the concentrated slurry and the final slurry distribution plots show a trimodal distribution. The major particle-size peaks along with the relative volume or number percentage that each peak represents are summarized in Tables 5.2 and 5.3.

Table 5.2. Peak Mode Location for Volume-Weighted Particle Distribution of AZ-102 Samples

Sample	<u>X-100 (40 mL/sec)</u>		<u>UPA (Brownian Motion)</u>	
	Mode Diameter (μm)	Vol %	Mode Diameter (μm)	Vol %
Initial Sludge (CUF-AZ102 MU)	24.280	51	1.174	100
	4.647	49		
Initial Sludge Duplicate (CUF-AZ102 MU DUP)	24.590	49	5.537	2
	4.604	51	1.412	98
Concentrated Slurry (CUF-AZ-102-005)	1.561	92	1.794	100
	0.287	8		
Concentrated Slurry Duplicate (CUF-AZ-102-005 DUP)	6.236	27	1.683	96
	1.271	66	0.346	4
	0.286	7		
Final Slurry (CUF-C104-024)	6.525	9	2.826	83
	1.112	69	0.411	17
	0.319	22		
Final Slurry Duplicate (CUF-C104-024 DUP)	6.505	23	1.614	89
	1.103	58	0.163	3
	0.319	19	0.078	4
			0.034	4

Figure 5.3. Cumulative Under-Size Percentage Distribution for AZ-102 Slurries Using the Microtrac UPA

Table 5.3. Peak Mode Location for Number-Weighted Particle Distribution of AZ-102 Samples

Sample	<u>X-100 (40 mL/sec)</u>		<u>UPA (Brownian Motion)</u>	
	Mode Diameter (μm)	Num %	Mode Diameter (μm)	Num %
Initial Sludge (CUF-AZ102 MU)	0.798	100	1.035	100
Initial Sludge Duplicate (CUF-AZ102 MU DUP)	0.793	100	1.245	100
Concentrated Slurry (CUF-AZ-102-005)	0.979	7	1.598	100
	0.241	93		
Concentrated Slurry Duplicate (CUF-AZ-102-005 DUP)	0.284	100	1.310	20
			0.311	77
Final Slurry (CUF-C104-024)	0.261	100	0.169	100
Final Slurry Duplicate (CUF-C104-024 DUP)	0.260	100	0.029	100

The significant decrease in the volume-weighted distribution histogram from the initial sludge to the concentrated slurry shown in Figure 5.2 represents the extent of solids de-agglomeration after the solids were re-circulated in the CUF line for 6 hours at various crossflow-velocity and TMP conditions described in Table 3.1. The production of finer particles exhibited in the concentrated slurry PSD confirms that particle break-up is a significant factor in reducing filtration flux from Conditions 1 to 7 (see Figure 3.1). A possible explanation for reduction in flux can be described in terms of forming higher resistance “cakes” (deposited layer on the membrane surface) as the solids deposited at or near the membrane surface are enriched progressively with finer particles. In crossflow filtration, a slight decrease in the filtrate flux is caused by the formation of a porous filter cake as the particles are deposited on the membrane surface. However, as small fine particles begin to plug the filter cake, the filtrate flux could decrease very rapidly and necessitate back flushing to regenerate the membrane. Upon each backpulsing and progressive de-agglomeration, the dispersed fine particles are capable of penetrating the pores of the membrane and reducing the filtration flux further.

Figures 5.4 and 5.5 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. Figure 5.6 and 5.7 compare the volume-weighted and number-weighted distribution of these samples before and after sonication. It can be seen from these plots (Figures 5.4, 5.5, and 5.6) that after the slurry was caustic leached and caustic washed, the nature of agglomerate compaction was changed. The final slurry solids are more shear sensitive and are easier to de-agglomerated compared to the concentrated slurry (same agglomeration compaction nature as initial slurry) under identical variation in the circulation flowrate and sonication treatments as the concentrated slurry.

Furthermore, the comparison of the volume-weighted distribution plots (see Figure 5.6 and 5.7) of the concentrated slurry and the final slurry before sonication show that the overall distribution shape and the location of the peaks in both samples (concentrated slurry and final slurry) are almost the same and similar, respectively.

Figure 5.4. Histogram of the Volume- Weighted AZ-102 Slurries Using the Microtrac X-100 at 40 and 60 mL/s Circulation Flow Rate

Figure 5.5. Histogram of the Number-Weighted AZ-102 Slurries Using the Microtrac X-100 at 40 and 60 mL/s Circulation Flow Rate

Figure 5.6. Histogram of the Volume Weighted AZ-102 Slurries before and after Sonication Using the Microtrac X-100

Figure 5.7. Histogram of the Number Weighted AZ-102 Slurries before and after Sonication Using the Microtrac X-100

6.0 Conclusions

Based on the testing and analysis performed on AZ-102 sludge described in this document, the following conclusions have been obtained. They have been divided into categories for clarity.

AZ-102 Crossflow Filtration

- For a slurry at 5.1 wt% insoluble solids, the average filtrate flux over 50 min of operation ranged from 0.046 to 0.126 gpm/ft². The dominant variable affecting the filtrate flux data during the first three conditions appears to be operating time. During these conditions, there was a steady decline in filtrate flux. Changes in the TMP appeared to have little effect. For the three subsequent runs, the axial velocity and TMP may have slightly increased the flux rate.
- For a slurry at 13.7 wt% insoluble solids, the average filtrate fluxes ranged from 0.020 to 0.051 gpm/ft² over 50 min of operation. This filtrate flux is ~2.3 times less than the low solids matrix, and ~2.7 times higher in solids concentration. Over the range of conditions studied, higher axial velocities improve filtrate flux. Once again, TMPs appear to have little effect.
- The filtrate DFs for ²⁴¹Am (ratio of concentrations in the slurry to the concentration in the filtrate) were between 100,000 and 450,000 for the filtrates collected, indicating excellent solid-liquid separations.
- A statistical model of filtrate flux was developed and found to be a function of axial velocity and run order. Based on this modeling effort, the optimum condition for both low and high solids loading is to maximize the axial velocity. Due to collinearities between pressure and velocity, and run order and solids concentration in the current data set, from a statistical standpoint, it is not possible to determine the effect of TMP and solids concentration on filtrate flux.
- Even after two cleanings with (1 M) nitric acid, the CUF was not capable of recovering the clean water filtrate flux. In full-scale operation, higher temperatures and longer cleaning times may be required. Acid cleaning at temperatures greater than ambient was not possible in the CUF due to the potential for corrosion of the CUF material.

AZ-102 Wash and Caustic Leach Testing

- Dilute caustic washing removed 80.4% of the sodium and >90% of the soluble anions, including nitrate, nitrite, and sulfate. Significant quantities of fluoride and cyanide were also removed. The only radioactive isotopes significantly removed by washing were ¹³⁷Cs and possibly ⁹⁹Tc, and ¹²⁷I.
- The first caustic leach removed 60% of the aluminum, but only 1.2% was removed during the subsequent caustic wash steps. Caustic leaching also increased the removal of P as well as the water-soluble components, such as K and ¹³⁷Cs.
- Fe, Al, and Na were the analytes of highest concentration, respectively, in the final composition of the final leached/washed sludge.
- The total mass of solids was reduced by approximately 44% during the course of water washing and caustic leaching, and the mass of HLW glass (assuming Al + Fe + Zr limiting the glass) was reduced by potentially ~27%.

AZ-102 Physical, Rheological, and Particle Size Properties

- The initial slurry exhibited significant plugging in the CUF during the start of operations. After several hours of operations, the plugging effects were reduced. The final slurry was found to entrain significant quantities of air that remained in the material for hours. This entrained air may have impacted the pumpability and filterability of the material, although entrained air and its effects were only observed on the final slurry.
- The initial dilute slurry, concentrated slurry, and final slurry were found to be yield pseudoplastic and thixotropic.
- There was a significant decrease in particle size from the initial as-received sludge to the concentrated slurry. This decrease is attributed to particle deagglomeration during pumping in the CUF. In contrast, there is less difference in particle size between the concentrated slurry and the final washed and leached material.
- The slurry exhibited increased shear sensitivity after washing and leaching as evidenced by its large reduction in number distribution after sonication.

7.0 References

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

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

PNNL Test Instruction		Document No.: BNFL-TP-29953-076 Rev. No.: 0
Title: HLW Filtration and Caustic Leaching of AZ-102		
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 <small>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.</small>		
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		1/8/00
Technical Reviewer		Jan 8, 2000

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 381 grams of AZ-102 slurry, which contains approximately 127.6 grams of undissolved solids.

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-29953-069, 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 – Mac Zumhoff, Ralph Lettau, Don Rinehart
- Radiological Control Technician

4.0 Materials, Equipment, Supplies and Reagents Needed

4.1 Materials Required

- AZ-102 Samples “A,” “MU,” and “GL.” AZ-102 Recovery Jar.
- One 250 mL glass jar labelled “AZ-102 Initial Feed”
- Twenty-seven 20-mL glass scintillation vials for filtrate and slurry samples, pre-labeled as follows:
 - CUF-AZ102-001 through CUF-AZ102-027.
- Two 1-L bottles, labeled “AZ-102 Filtrate#1” and “AZ-102 Filtrate#2” with mark(s) indicating 970 milliliters.
- Two 1-L polyethylene bottle labeled “CUF AZ-102 First Wash.” It should be marked with a line for 1-L volume.
- Three 1-L polyethylene bottles labeled “AZ-102 WW permeate#1” through “AZ-102 WW permeate#3”. Each bottle should have a mark indicating a volume of 900-mL.
- One 1-L polyethylene bottle labeled “AZ-102 CL permeate.”
- Two 1-L polyethylene bottles labeled “AZ-102 CW permeate#1” and “AZ-102 CW permeate#2.”
- Two 1-L plastic-coated glass bottles labeled “AZ-102 final sample” and “AZ-102 decant.”
- Three graduated centrifuge tubes.
- Tubing for de-watering mode.
- 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.
- One 2-L bottle labeled “tank overflow container.”

- Tubing to go from valve V-10 to the caustic leaching/washing vessel.
- 10 liters of 0.2-micron filtered DI water for washing the CUF upon completion of AZ-102 processing.

4.2 Equipment

- 4000 gram balance
- Video tape
- pH paper
- Hand held camera. Used for making difficult readings and volume estimations.
- Stopwatch
- Calculator
- CUF Ultrafiltration system with 100 mL plug in place
- 1000 watt Chiller

4.3 Reagents Needed

All containers that will be put into the cell should be labeled with "AZ-102" and "CUF testing."

- 1-L of filtered, distilled water labeled "start-up water."
- 1.62 liters of 0.01-M NaOH_{aq} labeled "5 wt % dilution 1."
- TBD mL (837 g – ~~rinse bottle liquid~~ ^{recovery jar liquid}) of 0.01-M NaOH_{aq} labeled "5 wt % dilution 2."
- 2.7 liters of 0.01-M NaOH_{aq} labeled "water wash solution" split up into three separate 1-L containers containing 900-mL of solution in each.
- NaOH_{aq} labeled "caustic leach solution." Amount and molarity to be determined. Will be approximately 600 mL.
- 100 mL bottle of 3 M NaOH_{aq} labeled "3M caustic rinse." Determine mass of caustic in bottle.
- Two containers of 0.01-M NaOH_{aq} labeled "caustic wash solution#1" and "caustic wash solution#2." Amount to be determined. Will be approximately 1600 mL of solution in *each* container.
- 1 liter of filtered, distilled water labeled "AZ-102 CUF rinse."
- 1 liter of 1-M HNO_{3aq} labeled "AZ-102 CUF wash acid."
- 1 liter 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. Due to the shortage of test sample, *do not* remove any additional slurry other than what is specifically requested for in this test instruction.

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 this document.

5.1.3 Weight AZ-102 Sample A and tare weigh the bottle labeled "AZ-102 Initial Feed"

AZ-102 Sample A

Total 788.56 g
w/ cap & w/o metal clamp

Tare AZ-102 Initial Feed

Total 352.33 g (w/ cap)
327.89 g (w/o cap)

5.1.4 Transfer 284 g of AZ-102 from AZ-102 Sample A to AZ-102 Initial Feed.

Reweigh both containers. Any solids remaining on the tools used for the transfer should be saved and will be rinsed into the CUF.

Not performed. See LRB.

AZ-102 Sample A

Total 466.57 g

AZ-102 Initial Feed

Total 328.54 g

5.1.5 Conduct the "0.0 Pre-Start" operations in BNFL-TP-29953-020. Replace the overflow container with the bottle labeled "tank overflow container."

** Steps 5.1.6 through 5.1.12 have already been completed. They will remain in the instruction for reference purposes only. Operations should begin with step 5.1.13.

5.1.6 Perform "1.0 Start-Up" operations in BNFL-TP-29953-020 with lay-up solution currently in CUF with one variation: V4 the filtrate control valve should be closed. Run CUF for 5 minutes between 4 and 6 gallons per minute (gpm).

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

- 5.1.8 Perform "1.0 Start-Up" operations in BNFL-TP-29953-020 with 1.0 liter of filtered, distilled water.
- 5.1.9 Perform "6.0 Back-pulsing" operations in BNFL-TP-29953-020.
- 5.1.10 Shut off the system and conduct the "10.0 Draining the system" operation in BNFL-TP-29953-020.
- 5.1.11 Perform "1.0 Start-Up" operations in BNFL-TP-29953-020 using the liquid in the bottle labeled "start-up water".
- 5.1.12 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 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	30	
2	4.20	55	
3	4.20	70	

- 5.1.13 Shut off the system and 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: (Small)
 Calib ID 326-06-01-061 Calib Exp Date 8/2000
 Location A-cell South

✓ Balance 2: (large)
 Calib ID 326-06-01-054 Calib Exp Date 8/2000
 Location A-cell North

TC 1 Calib ID/Date 3003/8/00 P-201 Calib ID/Date 325-30-2/1/2000 ^{Exp.}

P-202 Calib ID/Date 325-30-3 ^{Exp.} P-203 Calib ID/Date 3054/Jan. 2001
Exp. 1/2000

- 5.2.2 Weigh the jars containing the AZ-102 samples and the "5 wt% dilution 1." Record their weights below.

AZ-102 Sample MU

Total 175.320 g

AZ-102 Sample GL

Total 180,833 g

"5 wt% dilution 1"

Total See LRB g

5.2.3 Conduct the "1.0 Start-Up" operations in BNFL-TP-29953-020 starting with approximately one-half of the material in "5wt% dilution 1." Once that is pumping, add the contents of AZ-102 Initial Feed. Shake the jar containing the AZ-102 sample thoroughly before adding it to the slurry reservoir. Use the remaining "5wt% dilution" material to recover any solids left in the sample jar that cannot be transferred by shaking. Pay close attention to the level of material in the CUF.

5.2.4 Record the weights of the empty jars and lids in the spaces provided below. Calculate the amount of material remaining in the sample jar. If the difference is greater than ~ 2 g or significant solids are visible, further solids recovery is warranted. Record the method of recovery in the LRB.

<u>AZ-102 Sample A</u>	AZ-102 Initial Feed	"5wt% dilution"
466.57 g	Jar + Lid <u>328.54</u> g	Jar + Lid <u>See LRB</u> g
788.56	Tare <u>327.89</u> g	Tare <u>See LRB</u> g
<u>321.99 g</u>	Total <u>0.65</u> g	Total <u>1849.86</u> g

5.2.5 Record the level in the slurry reservoir.

Height _____ inches 22.2 cm 6" = 15 cm

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	<u>LRB</u> <u>1/10/00</u>

2	3.13	30	FR 1/10/00
3	5.23	70	KGR 1/10/00
4	5.23	30	KGR 1/10/00
5	3.13	70	—
6	4.20	50	KGR 1/10/00
7	Optimum from 1 - 6	Optimum from 1 - 6	2109 1/10/00

~ 4.5 (best achieved) 30

KGR 5.3.2 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. Perform rheological measurement per Table 4 in BNFL-TP-29953-069.

Viscometer

Make HAAKE

Model M5 RVRC 20

Geometry mv1

24.7°C

5.3.3 Tare-weigh the 1-L bottle labeled "AZ-102 Filtrate#1" *1A*

18:29 pm Weight of empty "AZ-102 Filtrate#1" with lid 102.626 g

5.3.4 Pre-weigh two of the labeled 20-mL glass scintillation vials and record its weights. Mid-way through the following step (5.3.5), 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. *# -001 & -002 19:00 (pm)*

18:45 - 2 back pulses - *target 4.2 ppm, 50 psig*
5.3.5 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 "AZ-102 Filtrate#1" to the 970 mL mark, or as high as possible if this mark is unattainable. *actual ~ 3.3 ppm / 50 psig 19:16 pm*

5.3.6 Replace the lid on the bottle and weigh.

Weight of "AZ-102 Filtrate#1" with lid 1060.60 g

Total weight of material removed in 957.97 g

AZ-102 Filtrate#1

+ 2 samples
= total

5.3.7 Record the level in the slurry reservoir.

Height ~1-2" inches

19:12 video

5.3.8 Weigh the bottle labeled "5 wt% dilution 2" and the AZ-102 recovery jar. Calculate the weight of liquid/slurry in the recovery jar.

Tare Weight of "5 wt% dilution 2" with lid NA g

Weight of AZ-102 recovery jar 735.69 g

Tare Weight of AZ-102 recovery jar 351.87 g

Weight of liquid/slurry in recovery jar 383.82 g

visually settle
~ 1050 Solids
~ 9050 Supernate
see vial

5.3.9 Calculate and add the proper weight of 0.01M NaOH to the bottle labeled "5 wt% dilution 2."

Weight required to be added to CUF 837 g

Weight of liquid/slurry in recovery jar 383.82 g

Weight needed in "5 wt% dilution 2" 453.18 g

Actual weight of "5 wt% dilution 2" added 453.19 g

307.31
145.88

Pre - $M_u = 175.320$
GL - $GL = 180.83$
5.3.10 Add the remaining slurry material from "AZ-102 Sample A", "AZ-102 Sample MU," and "AZ-102 Sample GL" to the CUF. *Final Composite*

5.3.11 Use the AZ-102 recovery jar and 5 wt% dilution 2 liquids to rinse out the three AZ-102 sample jars. Pour this material into the CUF. *finished @ ~ 2030*

5.3.12 Weigh the empty containers.

When looked in CUF no slurry movement - Plugs - had to put se manually to break - allowing 150 min to mix & break-up any flocs/aggrates

Weight of "AZ-102 Sample A" 465.10 g

Weight of "AZ-102 Sample MU" 134.485 g

Weight of "AZ-102 Sample GL" 134.169 g

Weight of recovery jar 351.87 g

Weight added to CUF

Weight of "AZ-102 Sample A" 465.10 g

Weight of "AZ-102 Sample MU" 134.485 g

Weight of "AZ-102 Sample GL" 134.169 g

Weight of recovery jar 735.84 g

5.3.13 Tare-weigh the 1-L bottle labeled "AZ-102 Filtrate#2."

Weight of empty "AZ-102 Filtrate#2" with lid 100.361 g

5.3.14 Pre-weigh two of the labeled 20-mL glass scintillation vials and record its weights. Mid-way through the following step (5.3.15), 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.

Height before de-watering 4-4.5"
(close to 4)

* Having lots of plugging problems.

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 5.3.1. Fill "AZ-102 Filtrate#2" to the 970 mL mark, or as high as possible if this mark is unattainable.

~10:15 started

5.3.16 Replace the lid on the bottle and weigh.

Weight of "AZ-102 Filtrate#2" with lid 828.18 g
Total weight of material removed 727.819 g

5.3.17 Record the level in the slurry reservoir.

Height ~1" inches

very thick - not mixing well in tank

added back ~200 ml of permeate from filtrate #2

New Height ~2-2.5"

Second Test Matrix

5.3.18 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, and check the level in the slurry reservoir (video-taping periodically, as necessary).

Condition	Flowrate (gpm)	Transmembrane Pressure (psig)	Initial and date when complete
1	4.20 2.6	50	JTB 1/11/00
2	3.13 2.2	30	JTB 1/11/00
3	5.23 2.0	70	JTB 1/11/00
4	5.23	30	JTB 1/11/00
5	3.13	70	Not performed
6	4.20	50	JTB 1/11/00

5.3.19 Obtain two slurry samples of 20 and 10 grams each, respectively, 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. Record the weights and sample numbers in Data Sheet 3.

~11:45 pm

5.3.20 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. See Table 4 in BNFL-TP-29953-069 for a description of this process.

JTB 1/10/00

5.3.21 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. Perform rheological measurement per Table 4 in BNFL-TP-29953-069. K7B 4/19/00

5.3.22 Increase the flow and pressure to its optimal value and repeat step 5.3.20 and 5.3.21 for a second 40-mL slurry sample.

Three Water Washes

5.3.23 Weigh all three one-liter bottles labeled "water wash solution."

Bottle with solution (solution only)	#1 <u>896.53</u> g	#2 <u>905.42</u> g	#3 <u>895.80</u> g
--	-----------------------	-----------------------	-----------------------

5.3.24 Obtain the tare weight of the three empty two-liter bottles labeled "AZ-102 WW permeate#1" through "AZ-102 WW permeate#3."

	permeate#1	permeate#2	permeate#3
Empty bottle	<u>100.00</u> g	<u>100.29</u> g	<u>100.58</u> g

5.3.25 Add the first bottle of "water wash solution" to the slurry reservoir.

5.3.26 Conduct the "4.0 Operation during Ultrafilter De-watering Mode" operations in BNFL-TP-29953-020 using the optimum conditions from step 5.3.12, K7B 4/19/00 acquiring flux measurements periodically. Fill "AZ-102 WW permeate#1" to the 900-mL mark. Add the second bottle of "water wash solution", and begin filling "AZ-102 WW permeate#2." Add the third bottle of "water wash solution" once the 900-mL mark is reached in the second permeate bottle. Continue dewatering into "AZ-102 water wash permeate#3," filling it to its 900-mL mark, or as high as possible if the 900-mL mark is unattainable. Following each dewatering (after each 900-mL mark is reached), check the slurry height. Video tape if necessary.

5.3.27 Weigh the three empty one-liter bottles labeled "water wash solution."

Bottle without solution	#1 <u>N/A</u> g	#2 <u>N/A</u> g	#3 <u>N/A</u> g
Total added	<u>896.53</u> g	<u>905.42</u> g	<u>895.80</u> g

5.3.28 Weight the three one-liter bottles labeled "AZ-102 WW permeate#1" through "AZ-102 WW permeate#3."

	permeate#1	permeate#2	permeate#3
with permeate	<u>978.57 g</u>	<u>981.14 g</u>	<u>1054.65 g</u>
Total permeate	<u>978.57 g</u>	<u>980.85 g</u>	<u>954.07 g</u>

5.3.29 Obtain the tare weight of a graduated centrifuge tube.

Tare weight of centrifuge tube = 6.4899 g

5.3.30 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 5 minutes, remove, and determine volume of centrifuged solids (V_{solids}).

Weight (tube and sample) = 15.91 g

$W_{t_{\text{sample}}} =$

Weight (tube and sample) - weight of centrifuge tube = 9.4201 g
5:40 pm Wt of empty tube after return of sample to CUF 6.817 g

$V_{\text{sample}} =$ 8.6 mL

$V_{\text{solids}} =$ 1.6 mL

$V_{\text{slurry/tank}} =$ 0.186 mL

slurry wt = 0.321 g

1/11/00

5.3.31 Calculate the volume of required leaching solution, ~~3M~~ NaOH_{aq} and place in "caustic leach solution." Place a mark indicating this volume on the bottle labeled "AZ-102 CL permeate#1". V_{slurry} represents the volume of the slurry.

Volume of ~~3M~~ NaOH_{aq} to be used =

$3 * V_{\text{slurry/tank}} * (V_{\text{solids}} / V_{\text{sample}}) =$ 483 mL

see LRB pg 98

5.3.32 Record the level in the slurry reservoir.

Height ~1" / 1.5 inches

5.3.33 Obtain one slurry sample of at least 20 grams following "7.0 Slurry Sampling" in BNFL-TP-29953-020 and using the pre-labeled sample vials. This will be used for physical analyses. Record the weight and sample number in Data Sheet 3.

6:25 pm 1/11/00

Caustic Leach

5.3.34 Measure the Tare of the empty caustic leach container.

-No lid / no baffles etc.

Tare container 1124.41 g

5.3.35 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. - Rinsed sample port into caustic leach & approx 10ml added (Dr. Hro)

- 5.3.36 Prepare the NaOH_{aq} solution. The volume of this solution was determined above. The concentration is to be sufficient to create a 3M NaOH solution above the centrifuged solids layer.

Volume NaOH solution required = $\frac{483 \text{ ml}}{7.37} \text{ g}$
 Molarity of NaOH solution = $\frac{7.37}{676.49} \text{ M}$
 Bottle with 3-M NaOH_{aq} = 676.49 g - Empty 67.331 g
 Both solution Both tare 66.93 g

- 5.3.37 Add the above volume of solution to the slurry reservoir and pump through the system for five minutes. Drain as described above (5.3.35) into the caustic-leaching vessel. Determine the weight of 3-M NaOH_{aq} added.

Leach 2114 pm start 5m
 Container with 3-M NaOH_{aq} = 2800.66
 Level in leaching container $2'' = \sim 1600 \text{ ml}$ Slurry 609.159 g added
 0.4018 g in bottle
 2154

- 5.3.38 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 $10:15 \text{ AM/PM}$ 1/11/00
 Estimated Shutdown time (above time plus 8 hours) $6:05 \text{ AM/PM}$ 1/12/00
 Shut off CUF - $6:33 \text{ AM}$ 1/12/00

- 5.3.39 Obtain the tare weight of the empty one-liter bottle labeled "AZ-102 CL permeate."

AZ-102 CL permeate
 Empty bottle 67.000 g
 Rinsed Container w/

- 5.3.40 Begin heating the CUF at the end of the caustic leach. When leaching is complete, stop the heating/mixing the caustic leach. Measure weight and level of full container.

Container w/slurry 2721.54 g
 Level $\frac{34}{32}$

- 5.3.41 Add the slurry to the slurry reservoir. Pump until the temperature reaches $85 \pm 5^\circ\text{C}$.

- 5.3.42 Calculate water lost during leaching.

Full container pre-leach 2800.66 g

Full container post-leach	<u>2721.54</u> g
Amount of water lost	<u>79.12</u> g
	+ 28.53 g already added

5.3.43 Add amount of water equal to that lost into the container, swirl and transfer into the slurry reservoir.

Actual Water Added to CUF 81.46 g

Note if too many solids remain in the container, rinse the caustic leach vessel with an additional 100-mL of 3-M NaOH_{aq} to remove any residual solids on the bottom or sides and add to the reservoir. Determine the weight of 3-M NaOH_{aq} added.

Container with 3-M NaOH _{aq} =	<u>Not performed</u> g
Container without 3-M NaOH _{aq} =	<u>Not performed</u> g

5.3.44 Pre-weigh two of the labeled 20-mL glass scintillation vials and record their weights. Mid-way through the following step (5.3.45), obtain two filtrate samples.

5.3.45 Conduct the "4.0 Operation during Ultrafilter De-watering Mode" operations in BNFL-TP-29953-020 using the optimum conditions from step 5.3.18. 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. Fill "AZ-102 CL permeate" to the mark made in step 5.3.31, or as high as possible if mark is unattainable.

5.3.46 Weight the full one-liter bottle labeled "AZ-102 ^{Caustic Add} ~~CL permeate~~."

	AZ-102 CL permeate
With permeate	<u>596.58</u> g

5.3.47 Record the level in the slurry reservoir.

Height not done inches

5.3.48 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. These will be used for chemical and physical analyses. Record the weights and sample numbers in Data Sheet 3.

First Caustic Wash

5.3.49 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.

Level with slurry 13 1/16 - inch
 Volume of slurry 702.3 mL

5.3.50 Add sufficient volume of 0.01-M NaOH_{aq} to the slurry reservoir from "caustic wash solution#1" to make 2400 mL total volume (slurry and and pump through the system for five minutes. Drain as described above (5.3.34) into the caustic-leaching vessel. Determine the weight of 0.01-M NaOH_{aq} added. Determine the weight of full leaching container. 1/10/00

labeled "AZ-102 CW permeate#1" ←
 Volume 0.01-M NaOH_{aq} required (2400-slurry volume) 1400 mL
 Volume 0.01-M NaOH_{aq} added 1400 mL
 Weight of Full Container with 0.01-M NaOH_{aq} 1659.86 g tare
 Level in leaching container 2 - 7/8 - inches 2.288 - liters
 Mass in leaching container 3520.60 g

5.3.51 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 1 2:54 AM/PM
 Shutdown time (above time plus 8 hours) 0 8:54 AM/PM

5.3.52 Obtain the tare weight of the empty one-liter bottle labeled "AZ-102 CW permeate#1."

permeate#1
 Empty bottle 267.63 g

5.3.53 Begin heating the CUF at the end of the caustic wash. When washing is complete, stop the heating/mixing the caustic wash. Measure weight and level of full container. 21 00

Container w/slurry 3415.80 g
 Level 2 - 7/8 "

5.3.54 Add the slurry to the slurry reservoir. Pump until the temperature reaches 80°C.

5.3.55 Calculate water lost during leaching.

Full container pre-leach 3520.60 g
 Full container post-leach 3415.80 g
 Amount of water lost 104.80 g

5.3.56 Add an amount of water equal to that lost into the container, swirl, and transfer into the slurry reservoir.

Actual water added to CUF 105.25 g

Note if too many solids remain in the container, rinse with an addition 100 mL of 0.01 M NaOH solution. Pour into the CUF. Determine the amount added.

Additional Water added to the CUF Not performed g

✓ 5.3.57 Pre-weigh two of the labeled 20-mL glass scintillation vials and record their weights. Mid-way through the following step, obtain two filtrate samples.

✓ 5.3.58 Conduct the "4.0 Operation during Ultrafilter De-watering Mode" operations in BNFL-TP-29953-020 using the optimum conditions from step 5.3.18, recording flux measurements periodically. 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. Fill "AZ-102 CW permeate#1" to the amount added in step 5.3.48, or as high as possible if mark in unattainable.

50 1/10/00

5.3.59 Record the level in the slurry reservoir.

Height Not Performed inches (video taped)

Second Caustic Wash

5.3.60 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.

Tare container 1174.41 g

Level with slurry Not performed - taken from weight & volume below

Volume of slurry Not performed - taken from weight & volume below

5.3.61 Add sufficient volume of 0.01-M NaOH_{aq} to the slurry reservoir from "caustic wash solution#2" to make 2400 mL total volume (slurry and and pump through the system for five minutes. Drain as described above (5.3.34) into the caustic-leaching vessel. Determine the weight of 0.01-M NaOH_{aq} added. Determine the weight of the full leach container.

35 1/10/00

Volume 0.01-M NaOH_{aq} required (2400-slurry volume) 1400 mL

Volume 0.01-M NaOH_{aq} added 1400 mL

Weight of Full Container with 0.01-M NaOH_{aq} 1661.98 g

Level in leaching container 2 11/16" (2144 mL)

Weight of Full Leach Container 3259.25

5.3.62 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 4:13 AM/PM → 12:13
Shutdown time (above time plus 8 hours) 12:13 AM/PM

5.3.63 Obtain the tare weight of the empty one-liter bottle labeled "AZ-102 CW permeate#2."

permeate#2
Empty bottle 261.982 g

5.3.64 Begin heating the CUF at the end of the caustic wash. When washing is complete, stop the heating/mixing the caustic leach.

Container w/slurry 3237.10 - 100.068g
Level not taken

5.3.65 Add the slurry to the slurry reservoir. Pump until the temperature reaches 80°C.

5.3.66 Calculate the water lost during heating.

Full container pre-leach 3259.25 g
Full container post-leach 3137.032 g
Amount of water lost 122.218 g

5.3.67 Add an amount of water equal to that lost into the container, swirl, and transfer into the slurry reservoir.

Actual water added to CUF 100.068 g

Note if too many solids remain in the container, rinse with an addition 100 mL of 0.01 M NaOH solution. Pour into the CUF. Determine the amount added.

Additional Water added to the CUF 150.092 g

5.3.68 Pre-weigh two of the labeled 20-mL glass scintillation vials and record their weights. Mid-way through the following step, obtain two filtrate samples.

Measure filtrate flux initial, middle, end (over course of 10 minutes).

5.3.69 Conduct the "4.0 Operation during Ultrafilter De-watering Mode" operations in BNFL-TP-29953-020 using the optimum conditions from step 5.3.18, recording flux measurements periodically. 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. Fill "AZ-102 CW permeate#2" to the amount added in step 5.3.61, or as high as possible if mark in unattainable.

5.3.70 Record the level in the slurry reservoir.

Height not done inches

two #18 1/13/00
~~one~~ 2

5.3.71 Weight the ~~two~~ full ~~one~~-liter bottles labeled "AZ-102 CW permeate#1" and "~~AZ-102 CW permeate#2.~~"

(start. #18 1/13/00

	permeate#1	permeate#2
With permeate	<u>1759.69g</u>	<u>1801.32g</u>
Tare	<u>267.63 g</u>	<u>261.982g</u>
	<u>1492.06</u>	<u>1539.338</u>

Testing Finale

(Call Paul - if he is available do this. If not, collect a 80g sample for him.)

Acquiring
some of sample
for testing
later.

5.3.72 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.

5.3.73 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. The 40-mL of material will be recovered at a later time. Determine weight of material used.

5.3.74 Increase the flow and pressure to its optimal value and repeat step 5.3.62 and 5.3.63 for a second 40-mL slurry sample.

73 1/10/00

5.3.75 Obtain four slurry samples of at least 10 (1) & 20 (3) grams each, respectively, following "7.0 Slurry Sampling" in BNFL-TP-29953-020 and using the pre-labeled sample vials. These will be used for chemical and physical analyses, and particle size distribution. Record the weights and sample numbers in Data Sheet 3.

OK
6:33 PM

Draining and Rinsing the System

5.4.1 Tare-weigh the 2-L bottle labeled "AZ-102 final sample."

1L glass (w/ plastic coating)

Weight of empty "AZ-102 final sample" with lid 520.37 g

5.4.2 Drain the system of slurry following "10.0 Draining the System" into "AZ-102 final sample."

Weight of full "AZ-102 final sample" with lid 1097.35 g

576.98g
in Final
Sample

Tare AZ-102 washed solids 354.756 g
Full AZ-102 washed solids 673.04 g

318.284g
removed

- 5.4.3 Add the 1-L of filtered, distilled water in "AZ-102 CUF rinse" to the reservoir.
Open V4 and pump through the entire system for 5 minutes.

Weight of full "AZ-102 CUF rinse" _____ g
Weight of empty "AZ-102 CUF rinse" _____ g See LRB
Weight of "AZ-102 CUF rinse" _____ g for details

- 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."

Weight of full "CUF rinse" See LRB _____ g

- 5.4.6 Conduct the "9.0 Rinsing the system" operation in BNFL-TP-29953-020 for the second and third rinses. The second rinse should be done with 2-L of filtered, distilled water, and the third 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.

- Q Back pulse during 2nd Rinse / None for 1st Rinse.
5.4.7 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	30	KGB 1/14/00
2	4.20	55 40	KGB 1/14/00
3	4.20	70	

ops should have been 10, 20, 30 psig 1/14/00 7:45 p.m.

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

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

"AZ-102 Final Sample"

- 5.4.10 Allow the slurry to settle in the 2-L bottle. Analysis of the permeate from the second caustic wash, "AZ-102 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 "AZ-102 decant" until 1 to 2 inches of liquid remain. Weigh the amount of liquid removed.

Weight of empty "AZ-102 decant" with lid _____ g
Weight of full "AZ-102 decant" with lid _____ g

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 Brett. 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 for physical testing (except for particle size distribution analysis samples, which will go to Lab 312) will remain in the HLRF. 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.

Liquid Samples

- One sample from the first de-watering step.
- One sample from the second de-watering step.
- One sample taken from the bottle labeled "AZ-102 water wash permeate#1"
- One sample taken from the bottle labeled "AZ-102 water wash permeate#2"
- One sample taken from the bottle labeled "AZ-102 water wash permeate#3"
- One sample taken during the AZ-102 caustic leach
- One sample taken during the first AZ-102 caustic wash #1
- One sample taken during the second AZ-102 caustic wash #2
- One sample taken from the bottle labeled "AZ-102 decant"

Slurry Samples

- One sample after three water washes
- Two samples after the caustic leach
- Two samples after the final dilute caustic wash

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. See BNFL-TP-29953-069 for further details.

AZ-102 Test

1/10/00

The CUF has been rinsed w/ $1M HNO_3$ / $0.2M$ citric acid to finish cleaning it after the AN-107 run.

It has been rinsed to neutral pH and the clean water flux measured. A new pressure gauge on permeate side was needed & the leak on the chilled water is fixed. We are concerned about whether the water we insert in the tank outside the cell all ends up in the CUF

Weight of water ^(0.01M NaOH) outside	=	243.51g
Tare Weight of Container inside	=	102.32g
Weight of full container inside	=	<u>323.84g</u>
Weight of 0.01M NaOH inside		<u>221.52g</u>
Delta		21g

Weight of water outside _(0.01M) = 688.07g (#1)

Weight of (0.01M) NaOH outside 711.95g (#2)

We attempted to transfer the solids to a different container. In the process, material was being lost, dried on and getting spread everywhere. Instead we will send all of the material both in AZ-102 A and the other new container into the CUF.

We may have lost 2-3g dried solid on the cloths where we set the spatulas. We may have lost an additional ~~2-3~~ 3-4g on the spatulas.

Flow Mx+B settings:

M = 1.9901

B = -1.9987

Project No.	29953	Date of Work	1/10/00
Entered By	RP Brooks	Date	1/10/00
Disclosed To and Understood By			
Signed 1.		Date	
2.		Date	

AZ-102

93

2:10 am

Attached new pressure gauge.
Drained CUF
Turned on DAS

Added 106.09 g of 0.01 M NaOH to
the AZ-102 Initial feed container (maybe 10g went
to AZ-102 Sample A) - (from existing squirt bottle)

Added 221.52g of 0.01 M NaOH to AZ-102 sample A

Weight of 788.58g
Container + 0.01 M NaOH. $\rightarrow$ 686.26 g

Poured AZ-102 Initial Feed into CUF (w/ 106.09g water)

Weight of 815.20 g
Container + 0.01 M NaOH $\rightarrow$ 712.88g

Poured into AZ-102 Sample A to clean bottle.

4:02 Repeated as needed. Cleaned up spatulas as well as could.

AZ-102 Sample A has a lid that says
"AZ-102 Recovered Solids Rin"
Side says "AZ-102"

4:35 Started CUF
- Initially no flow & pressure built up in the inlet pressure gauge
but not the outlet. Lots of flow variation/pressure variation -
Added
173.11 g of 0.01 M NaOH additional to get it to 5wt%

4:52 Opened V4 and began flux measurements.
Needed air stepper to get to 50 psi & still can't get
to 4.2 gpm.

Project No. 29953 Date of Work 1/10/00
Entered By KP Brooks Date 1/10/00
Disclosed To and Understood By
Signed 1. Date
2. Date

A2-102

5:20 Continues to creep up in pressure over time. It also appears that the flow rate is increasing over time. The flux is decreasing over time.

6:06 Started Condition #2

6:16 Adjusted velocity down. Since 4.2 gpm wasn't achieved, we need to run the matrix at lower axial velocities.

7:23 System plugs at condition #3 such that pressure zooms up to 100 psi & then you have to back way down to get it to equilibrate. All the way down to 30 psi. Readjust & the pressure jumps to 70 psi again.

7:28 Finally holding more or less steady at ~70 psi

7:37 Still bouncing around & plugging.

~12:00pm - V3 toggle switch came off.
This had to be repaired before Condition 7 could be ~~done~~ started.

~2:00 Repaired V3 - running 7 / Decided to do highest gpm @ 30 psi for 7

~3:30 Rheology taken - ~~Fluxing 7~~

Because optimum was 4.2 gpm @ 50 psi - but had already been done 2 times

* When rinsing Cup added ~30 extra ml DI to CUF

7:00 pm sampled - 001 & 002 @ Sw. 5.3.4 in test plan.

6:45 pm back pulsed 2 times / started De watering.
target 4.2 gpm / 50 psi - Actual 3.5 gpm / 50 psi.

7:16 pm Finished De watering

Project No.

Date of Work

Entered By

Date

Disclosed To and Understood By

Signed 1.

Date

2.

Date

AZ-102 HOT CUF

8:30pm Finished adding all additional material to the CUF.

Recovery solids visually ~1050 settled solids
~9050 Supernate

Ralph used Supernate to rinse other samples into CUF.

When addition was complete noted no movement in the CUF and no flow rate -

The system was plugged.

~9:30 ~45 minutes of manual start and stop (bump the pump) action was required before the system would run continuously. An additional 45 min of running and bumping where needed before de-watering could be started.

* See notes from Composite & Sampling from this date.

10:50 De-watering Complete - Slurry is too thick and stagnant in zones - See video -

Added ~50 ml permeate back - still stagnant
then added ~100-110 ml permeate back - mostly flowing but not uniform
then added ~50 more back - smooth flow (uniform)

~~Started~~ Took Slurry samples
~11:40-11:45

~~Started~~

11:51pm Started 2nd test matrix. Cannot reach target cross flow velocity.

1/11/00 1:15 am

Started condition 2

2:25 am

The slurry appears to not be mixing - added 102.3g DI water to improve mixing. Volume may be lower than before - hard to tell. (correction)

Project No. _____ Date of Work 1/10/00
Entered By [Signature] Date 1/10/00
Disclosed To and Understood By _____
Signed 1. _____ Date _____

AZ-102

2:50

The flow appears to be a constant. Higher pressures (up to 30-40) down to 0 psi all produce ~2.5 gpm. Increased air flow doesn't appear to help.

At 60 psi air feed & 30 psi ΔP , we achieved 2.4 gpm w/ Air booster at ~80 (70-90 psi pulsing), with 30 psi ΔP we achieved 2.4 gpm.

Tare weights of sample ~~bottles~~ ^{flasks} Vials.

7 - 16.926g

8 - 16.893g

9 - 17.004g

3:30

Took filter off back end of CUF air motor to increase flow. Maybe 0.3 gpm increase.

4:14

Started Condition 4

5:22

Skipped Condition 5

5:24

Started Condition 6

5:47

Something happened. Pressure jumped to ~100 psi momentarily we then reduced the pressure and the flowrate jumped to 1 gpm more. The filtrate flux decreased by more than half. The pressure drop across the filter went from 2-4 to ~7-8 psi. The flow rate is 3.3 gpm as compared to 2.5-2.7 gpm before.

6:43

Running ~~test~~ ^{flask} test at 30 psi & 3.7 gpm since suddenly we can get there.

AZ-102 WW Permeate #1
" " " #2
" " " #3

Tare Wt.

100.00g

100.29g

100.58g

Project No. _____ Date of Work _____

Entered By _____ Date _____

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K6R

AZ - 102.

10:40 Washing Paul's apparatus.

11:45a finished washing viscometer. Added 1st water wash solution, beginning permeate removal of wash. Lost ~10 ml total.

11:55a Added oil to oiler for pump.

12:00p going to 50 psi & max flow.
Backpulsing. Twice.

12:35p beginning ww2. Backpulsing twice.
Add water. Initiate permeate removal. Backpulse. Take flow rate.

1:35p done w/ ww2.
Added ww3. Backpulsing.

3:15p done water

3:30p centrifuge tube. 6.4899g

Empty water wash bottles.

#1 102.8602 g

#2 102.9535 g

#3 102.9225 g

Centrifuged sample 8.6 ml

Centrifuged solids 1.6 ml

~ Volume of slurry calculated to be 865 ml @ this point (Ken Rep calculation)

See next page for NaOH calculations

* Large vessel Calibration (KPB Brooks)

< 750 - not linear

7/8" = 750 ml

1 3/4" = 1400 ml

2 1/2" = 2000 ml

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AZ-102

Caustic Leach Calculations

$$3 \times \frac{V_{\text{solid}}}{V_{\text{total}}} \times V_{\text{slurry}} = X \text{ volume NaOH}$$

$$3 \times \frac{1.6}{8.6} \times 865 = 483 \text{ ml}$$

$$V_{\text{slurry}} - \frac{V_{\text{solid}}}{V_{\text{total}}} \times V_{\text{slurry}} = Y \text{ volume liquid target @ } 3M \text{ NaOH}$$

$$865 - 160.9 = 704 \text{ ml}$$

$$\frac{3 \text{ M NaOH}}{1 \text{ L}} \left(\frac{X+Y}{1187} \right) \text{ L} = \text{moles NaOH needed}$$

$$= 3.561 \text{ moles NaOH}$$

∴ 483 ml of 7.37 M NaOH needed to add
as Caustic Leach
NaOH make-up (KP Brooks)

142.50g NaOH powder
483 ml H₂O

Tare bottle & lid = 66.93g
Bottle, lid & Soln = 676.49g

609.56 g of 7.37 M NaOH were prepared
for Caustic Leach in CUF.

6:25 pm
1825-

Slurry Sample CUF-AZ-102-007 taken

The Sample Port. was removed from the CUF to
attach the hose for transfer of the Slurry to the
Leaching Vessel. It was rinsed with ~10 ml DI into
the leaching vessel.

*
*

Problem arose with vessel lid - it is too small
and baffles are too short. Another was fabricated
before Slurry transfer to vessel.

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AZ-102

2152 All is set and starting of leach/heat cycle
1/11/00 T=0 Temp=24°C begin S₁
2153 pm

Time = 2157 Temp = 34°C

1/11/00	Time	Temp C	Time	Temp C
	2159	39	0305	88
	2201	47	0330	85
	2202	50	0405	87
	2206	60	0431	86
	2209	70	0500	84
	2214	83	0530	82
	2215	85	0600	83
	2220	88	0628	86
	2223	88		
	2224	87		
	2230	85		
	2234	82		
	2254	85		
	2302	82		
	2317	86		
	2321	84		
	2346	85		
1/12/00	0005	88		
	0031	85		
	0100	88		
	0132	84		
	0201	85		
	0232	81		

Controller Calibration Expires 8/2001
S/N 2013

Pot TC TC-325-426 - 8/00 Expires

Rinsed out "AZ-102 Caustic Addl" it will replace

"AZ-102 CL permeate"

387.321g DI water container initial

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AZ-102

361.782 g DI water after rinsing off the impeller
25.539 g DI water added

6:55 Weighed & measured level on container
7:07 Added slurry to CNF. Turned on heat tape
7:27 Rinsed ~~CNF~~ ^{leach container} with DI water
7:30 $T_{CNF} = 58^{\circ}\text{C}$
7:35 $T_{CNF} = 67^{\circ}\text{C}$
7:42 $T_{CNF} = 75^{\circ}\text{C}$
7:46 $T_{CNF} = 79^{\circ}\text{C}$
7:50 $T_{CNF} = 83^{\circ}\text{C}$
8:00 $T_{CNF} = 88^{\circ}\text{C}$

KGR
1/13/00

8:22 Dewatered caustic leach.
Acquired -008 & -009 after dewatering.

8:09:17 Tare wt's

CNF-AZ-102-010	16.988g
-011	17.008g
-012	17.023g

10:27a 596.58g - wt. of caustic leach permeate removed. Total. Bottle & solution,

wt. of 1st c.w. solution w/ bottle.
1659.86g
bottle 267.63g

Project No. _____ Date of Work _____
Entered By Cunneen, R. [Signature] Date 1/13/00
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11/6 P
1/12/00

AZ 102

Drained slurry. 10:55a. level in washing vessel. $13/16$ -inch 101

Tare wts.

CuF-A2702-013	16.861 g
-014	16.969 g
-017	16.913 g
-016	16.803 g

11:40p Drained caustic wash solution into vessel. Draining pump housing & blowing out upper loop. Draining pump housing again.

Rinsing out drain container. Determining how much rinse we add.

tare wt. 145.000g w/wash 154.500g
added 9.500g wash

Level $2-7/8$ -inches in leaching container.

3520.6g slurry in leaching vessel.

12:30p Heating caustic wash vessel. Now @ 40°C .

<u>Time</u>	<u>Temp ($^{\circ}\text{C}$)</u>
12:30p	43
12:40p	62
12:50p	82
12:54p	(85)
13:25	86
13:56	87
14:26	88
19:42	87
21:00	84

9:00 pm Heat time complete -

* Manipulator hand/band snapped - working with one manipulator
Cannot pull samples from "middle" of dewatering.

Project No. _____ Date of Work 1/12/00
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AZ-102

Slurry successfully transferred into CUF

Started @ $\sim 65^{\circ}\text{C}$ 2153 Temp up to 74.3 2225 - Temp @ 88 - Working on manipulator repairClimbed to 92 - turned on chiller for $<1\text{ min}$
dropped back to 85 2328 $T = 79.7$ Still working on manipulator repair

0054 Started dewatering

1:03 Stopped dewatering & pulled samples.

Transferred the slurry to the Pot

Made up some more 0.01 M NaOH - needed 300 mL Added 0.12 g of NaOH pellets to container to
make 1400.7 g total weight of liquid. (already had
 1100 mL of 0.01 M NaOH from the rest of the test)**0.010 M NaOH**10X dilution of standardized 0.1 M NaOH

7.0 Liters prepared

Expiration date 1/1/2001

RE: Chemres-37 - rgs

LAB 13745 p.102

This label was for all of the other 0.01 M NaOH used during the
washes, ~~etc.~~

2:35 Add water to CUF & mixed around

6:52 Transferred liquid into pot

Project No. _____ Date of Work _____

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Discovered that the heat tape was running continuously during the last run - last ones to start it hook it directly to power.

Port has been uncovered for the last 30 minutes lagging ~0.01g/sec to evaporation

3:16

Put on lid

4:02

Started stirring

4:13

Reached 85°C

del

4:30	84°C
5:00	83°C
5:35	83°C
6:02	83°C
6:30	82°C
7:01	85°C
7:30	84°C
8:00	83°C
08:30	86°C
09:00	87°C
09:30 09:30	88°C
10:00	87°C
10:30	84°C
11:00	82°C
11:30	82°C
12:00	83°C
12:12	shut down

K62

12:13p

mixing & heating stopped. Disassembling cut wash vessel.

1/13/00

* Significant solids stuck to mixing baffles.
Attempting to scrap off.

Project No. _____ Date of Work _____
 Entered By Lennell R. Rapp Date 1/13/00
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K6R
1/13/00

** significant solids also stuck to mixer.
Will attempt to rinse off.

Squirt bottle before rinse : 432.51 g
after rinse : 332.442 g
100.068

Rinsing appeared successful aside from some
crusted-on material on stem of impeller.

Weighing pot. 3237.10g

Rinsing pot. Twice. Large chunks did not come
out after initial pot dump.

Additional water added: bottle 182.35g
 $332.442 - 182.35g = 150.092g$

01:30p everything in CUF, heating.

Time	Temp
1:30p	61°C
2:06p	76°C
3:00p	83°C (not increasing)

wt. of empty
leach vessel.

1136.78g

12.37g slurry & water lost

Need weights of: Empty caustic wash permeate #2 bottle
Tare wt. of CUF-AZ-102-015

Need samples of Water Wash Permeates #1, #2, & #3. 2 each.

Tare wt of CW permeate #2 bottle: 261.982g

Tare wts of:	CUF-AZ-102-015	16.714
	-018	16.951
	-019	16.846
	-020	16.924

Project No. _____ Date of Work _____

Entered By Kenneth J. [Signature] Date 1/13/00

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KGR

1/13/00

Tare wts. CUF-AZ-102-021 16.8810
 022 16.5307
 023 16.9559
 024 17.0717
 025 17.0804
 026 17.1539 41

75

3:05p Setting CUF to 30 psi & max cross-flow.

Flowrate initial

3:12p 1/13/00 4.59 sec. 20 ml 1.9 gpm 28 - 30 psi

3:30p 85°C reached. Beginning de-watering.
 Half complete

3:45p Acquired samples CUF-AZ-102-021 & -022.

3:50p Resume de-watering. Done. filling ~ 100 ml extra due to additional water added for rinsing wash vessel.

Sample weights.

CUF-AZ-102-021 35.402g
 -022 37.734g

** Filtrate appears quite bubbly, like possibly getting a lot of foaming
 Rheology vid for Paul's final sample. 5.3.72.

tare: 133.3506g
 filling with ~ 80 ml of slurry.

** Heat tape controller has been going screwy. Realized slurry temp. had dropped to < 70°C. Heating back up.
 Saw T.C. line was getting slightly charred by the heat tape.

4:30p Pump shut down - no flow.
 Looked in slurry tank, no mixing, way too thick.
 Got ~~about 40 ml~~ ~ 40 ml of sample for Paul's rheology.

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Entered By Therese Rapp Date 1/13/00

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106

K68
1/13/00AZ-102

Tried restarting pump. No flow. Shut back down.

Tare wt. of Caustic W. P. #1 bottle. 102.8196g
303.6576g

Adding

200.8374 g ΔW.

AG
1/13/00

5:20pm

Slurry is pumping again but visual show a thick coating of solids all along the walls of the CUF Tank.

Attempting to scrape the walls down before proceeding.

Temp ~~of~~ of slurry was ~47°C. Turned on chiller.

5:35 T = 20.6°C

Sent in 3 bottles - 1. Large plastic coated (n/quant)
"AZ-102 Final Sample" → Tare 20.37
N300ml plastic coated → 354.756g
"AZ-102 Washed Solids 1st Rinse"
1 L plastic polyethylene
AZ-102 CUF 1st Rinse → 105.575g

5:55 Scraping sides did not return any solids to slurry but recover some to be record in Rinse.

Taking Rheology Sample
Final wt. 192.540 g

lost ~ 5g from spout -

4 Slurry samples taken

Project No.

Date of Work

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1/13/00
1/13/00

AZ-102

1

Transferred Washed Sludge from CUF to
AZ-102 Final Sample - 1576.98g transferred

6:50 First Rinse
Weight of DI Bottle @ start 542.93g

Washing down tank sides

DI bottle after washing 63.179g

DI 479.7502g added

7:10 - CUF on allowing to mix.

7:40 - Drained into ~500 ml Glass - AZ-102 Washed Solids 1st Rinse
318.284g removed

Put in 1L DI H_2O from Chem add tank.

AZ-102-CUF Rinse #1 1040.26g = Full
tare 105.575g

934.685g Slurry in
bottle
removed from
CUF

Sent in ~~three~~ ^{5 added} more 1L Poly bottles

	Tare	Full
AZ 102 CUF Rinse #1b	<u>102.89g</u>	<u>405.16g</u>

AZ 102 CUF Rinse #2	<u>102.97g</u>	<u>1065.97g</u>
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AZ-102 CUF Rinse #2b	<u>102.94g</u>	<u>714.54g</u>
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9:50 AZ-102 CUF Rinse #3	<u>100.016g</u>	<u>1069.41g</u>
--------------------------	-----------------	-----------------

9:50 AZ-102 CUF Rinse #4	<u>100.551g</u>	<u>1037.09g</u>
--------------------------	-----------------	-----------------

9:00pm - Ralph noted V4 maybe failing

11:30 - Hooking up cartridge -

Project No.

Date of Work

1/13/00

Entered By

Date

1/13/00

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Date

1/14/00

ATB

1:03 am started the 0.05 μ m filter cleaning the CUF
(external filter)

2:21 Backpulsed twice — very slow!
Backpulsed again

2:30 Drained system — still kind of brown

2:40 Re-filled w/ water (1 l) attached filter & ran

Checked water exiting filter — very clear
Checked water out of V9 — still very yellow
Filtrate flow rate is very slow — clean water fluxes not recovered.
Pulled off supernate from A2-102 final material. Picked
up some solids — will have to filter.

4:45 Backpulsed ~ 600 ml of HNO_3 thru the top

5:06 Turned on CUF w/ 1 l of 1 M HNO_3

6:00 Drain 1 M HNO_3 — muddy brown colored

Put ~ 600 ml of 1 M HNO_3 / 0.2 M Citric
into CUF

6:30 Removed acid & replaced w/ 1.5 l of DI
water

The acid is lighter this time, but still fairly brown

7:00 Backpulsed several times and drained liquid. Still
fairly brown.

7:12 Backpulsed 3 thru the top & drained the liquid

7:20 Added second batch of water — running thru 0.05 μ m filter

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1/14/00
KGR

109

12:00p rinsing rheology equipment.
Everything to go into 'AZ-102 Final Sample' jar.

DI water bottle weight.

Before rinsing: 509.62g

After rinsing: 399.22g

Rinsed with 110.40g DI water.

Good recovery of sample !!

Final Sample jar weight:

'AZ-102 Final Sample' = 1254.17g

Checking ptt of second rinse. Done. have no standards, so leaving in cell.

Checking water filtrate flux measurement.

^{30 psig}
** Max pressure attainable, just < 40 psig.
Therefore, just running @ 30 psig & 40 psig.

2:20p Backpulsed once upon completion of water tests.
Opened V2 & V10 to flow through external filter cartridge. Ran pressure up to ~ 25 psig for ~ 15 minutes.

2:35p Backpulsed twice. Drained.

* Looks clear.

1/14/00 * Note Clean water fluxes not achieved ~~At~~ 1/14/00

* Note Visually Solids have settled well in final Sample & washed Solids rinse #1 - but in CUF Rinse 1 - 4 not yet settled enough to recover. ∴ leaving All till Monday. Additionally - based on the settling in the high Solids jar, a lot of air was entrained in the slurry when removed from CUF.

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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
10-Jan-00	Sample A		197	315.64	1.60		131.8	41.8%
	5 wt% dilution #1		1850	1849.86	1.00		0.0	0.0%
	Test Sample #1		2047	2165.5	1.06		110.4	5.1%
	First Test Matrix & Dewatering							
	Rheology equipment rinse		30	30.0	1.00	0	0	0.0%
	Dewatering #1		951	957.97	1.008		0	0.0%
	Filtrate Sample -001		17	17.18	1.008	0	0	0.0%
	Filtrate Sample -002		17	16.90	1.008	0	0	0.0%
	Test Sample #1		1092.3	1203.4	1.102		110.4	9.2%
	Sample MU		25	40.835	1.60		17.1	41.8%
	Sample GL		29	46.664	1.60		19.5	41.8%
	Recovery Jar		384	383.82	1.00		38.4	10.0%
	5 wt% dilution #2		453	453.19	1.00	0.010	0	0%
	Test Sample #2		1983.9	2128.0	1.073		185.4	8.7%
	Dewatering #2		723	727.82	1.006		0	0%
	Filtrate Sample -003		23	22.89	1.006	0	0	0%
	Filtrate Sample -004		19	19.03	1.006	0	0	0%
	Test Sample #2		1246	1358.2	1.090		185.4	13.7%
	Slurry Sample -005		13	14.133	1.090	0	1.9	13.7%
	Slurry Sample -006		17	18.806	1.090	0	2.6	13.7%
	Water for mixing assistance		102	102.3	1.00	0	0	0%
11-Jan-00	Second Test Matrix & Water Washes							
	Water Wash #1 Solution		887	886.53	1.00	0.01	0	0%
	Water Wash #2 Solution		905	905.42	1.00	0.01	0	0%
	Water Wash #3 Solution		896	895.80	1.00	0.01	0	0%
	Water Wash #1 Permeate		871	875.71	1.006		0	0%
	Water Wash #2 Permeate		877	878.19	1.001		0	0%
	Water Wash #3 Permeate		956	951.73	0.995		0	0%
	Test Sample		1287	1409.71	1.10		159.77	11.3%
	Centrifuge Sample		0.3	0.327	1.10	0.00	0.04	11.3%
	Slurry Sample -007		18	19.378	1.10	0.00	2.2	11.3%
	Sample Port Rinse		10	10	1.00	0	0	0%
11-Jan-00	Caustic Leach							
	Caustic Leach Solution		483	609.16	1.26	7.37	0	0%
12-Jan-00	DI impeller rinse		26	25.54	1.00	0.00	0	0%
	Evaporation		105	104.66	1.00	0.00	0	0.0%
	DI impeller rinse		81	81.46	1.00	0.00	0	0%
	Caustic Leach Permeate		485	529.58	1.092		0	0%
	Filtrate Sample -008		17	18.708	1.092	0	0	0%
	Filtrate Sample -009		20	21.285	1.092	0	0	0%
	Test Sample		1238	1441.9	1.165		136.98	9.5%
	Slurry Sample -010		15	17.011	1.165	0	1.62	9.5%
	Slurry Sample -011		13	14.846	1.165	0	1.41	9.5%

Date/Time	Description		Volume (mL)	Mass (g)	Density (g/mL)	Caustic Molarity (M)	Undissolved Solids (g)	Weight Percent Undissolved Solids
	Slurry Sample -012		13	15.377	1.165	0	1.46	9.5%
	Drain container rinse		10	9.50	1.00	0.00	0	0%
12-Jan-00	Caustic Washes							
	Caustic Wash #1 Solution		1392	1392.23	1.00	0.01	0	0%
	Test Sample		2598.89	2796.43	1.08		132.5	4.7%
	Evaporation		105	104.80	1.00	0.00	0	4.7%
	Water added		105	105.25	1.00	0.00	0	0%
13-Jan-00	Caustic Wash #1 Permeate		1435	1492.06	1.040		0	0%
	Filtrate Sample -013		19	19.898	1.040	0	0	0%
	Filtrate Sample -014		15	15.208	1.040	0	0	0%
	Test Sample		1130.6	1269.7	1.123		116.8	9.2%
	Caustic Wash #2 Solution		1400	1400.00	1.00	0.01	0	0%
	Evaporation		122	122.22	1.00	0.00	0	0.0%
	Drain container rinse		100	100.07	1.00	0.00	0	0%
	Water added		150	150.09	1.00	0.00	0	0%
	Caustic Wash #2 Permeate		1508	1539.34	1.021		0	0%
	Filtrate Sample -021		18	18.521	1.021	0	0	0%
	Filtrate Sample -022		21	21.203	1.021	0	0	0%
	Water added		201	200.84	1.00	0.00	0	0%
	Test Sample		1048	1100.0	1.050		101.2	9.2%
	Slurry Sample -023		15	16.274	1.050	0	1.50	9.2%
	Slurry Sample -024		10	10.823	1.050	0	1.00	9.2%
	Slurry Sample -025		15	15.609	1.050	0	1.44	9.2%
	Slurry Sample -026		14	14.472	1.050	0	1.33	9.2%
13-Jan-00	Test Finale							
	Test Sample		1352	1419.4	1.050		1259.2	88.7%

Appendix C: Analytical Results

Appendix C: Analytical Results

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

Project: 29953
Client: K. Brooks

ACL Number(s): 00-0902 through 00-0917

Client ID: "AZ-102 MU" through "CUF-AZ-102-027"

ASR Number: 5667, 5668

Total Samples: 16

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

Analyst: D.R. Sanders

Analysis Date (Filename): 02-02-00 (A0579) [ALO-115 KOH/Ni fusion]
02-08-00 (A0582) [ALO-128 Acid Digestion - liquids]
02-24-00 (A0586) [ALO-129 Acid Leach - solids]

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

4-7-00
Reviewed by
4-7-00
Concur

4/7/00

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

ASR-5667

Sample **AZ-102 MU** (ACL# 00-0902) was prepared in duplicate by the Shielded Analytic Lab (SAL) using ALO-115 KOH/Ni fusion procedure. About 0.19 grams dried slurry sample was fused and diluted to a final volume of 100ml. Samples dissolved leaving a slightly cloudy mixture prior to adding 2% hydrochloric acid during dilution. An additional dilution (11.24-fold) was prepared by SAL prior to ICPAES analysis because of ALARA concerns. Measurement results are reported in $\mu\text{g/g}$ dry weight and corrected for all preparation and analytical dilution.

Sample **AZ-102 MU** (ACL# 00-0902) was also prepared in duplicate by SAL using ALO-129 Acid Leach procedure. About 0.48g of dried slurry sample was processed and diluted to a final volume of about 25ml. The prepared samples did not completely dissolve leaving a 'light-gray' residue in the bottom of the digestion vessel. The samples were also diluted 11.20-fold because of ALARA concerns. ICPAES measurement results are reported as $\mu\text{g/g}$ dry weight. Comparing results of the fusion prepared samples with the acid leached samples, it appears that aluminum, iron and zirconium were incompletely dissolve by acid leach procedure. Analysis results should be based upon the fusion prepared sample data.

Sample **AZ-102 M SUP** (ACL#00-0903) was prepared in duplicate, plus matrix-spike and blank-spike by SAL using ALO-128 Acid Digestion procedures for liquids. About 5.1 ml of supernatant was processed and diluted to a final volume of about 25ml. No ALARA dilution was performed prior to ICPAES analysis. Measurement results are reported in $\mu\text{g/ml}$.

Silver recovery was low in the spiked blank (about 19%) and sample ACL# 00-0903 (about 13%) because hydrochloric acid was used during sample preparation causing silver to precipitate.

Barium recovery was acceptable in the spiked blank (about 105%) but very low in the matrix spiked sample (about 5%). The low recovery of barium in the matrix-spiked sample is mainly due to high concentration of sulfate in the sample (ACL# 00-0903).

Silver recovery is expected to be low for all samples since hydrochloric acid was used to prepare all samples. Barium concentration will be low in all samples that have high sulfate concentration.

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ASR-5668

Four dry-slurry samples plus one duplicate and one wet-slurry sample, CUF-AZ-102-007 to CUF-AZ-102-026 (ACL# 00-0909 to 00-0916) were prepared by SAL using ALO-115 KOH/Ni fusion procedure. About 0.19g of dried slurry sample (except sample ACL# 00-0912) was fused and diluted to a final volume of 100ml. Samples dissolved leaving only a slightly cloudy mixture prior to adding 2% hydrochloric acid during dilution. Sample ACL# 00-0912 was prepared using about 0.95g of wet slurry (18.5% solids) and processed similarly using ALO-115 fusion procedure. Sample weight for ACL# 00-0912 was adjusted for percent solids. All samples were diluted 11.24-fold by SAL prior to ICPAES analysis because of ALARA concerns. Measurement results are reported in $\mu\text{g/g}$ dry weight corrected for all preparation and analytical dilution.

Five wet-slurry samples (same ACL#'s as above) were prepared by SAL using ALO-129 Acid Leach procedure. About 1.1 to 1.5g of wet-slurry material (0.48g dry-slurry) was processed and diluted to a final volume of about 25ml. The prepared samples did not completely dissolve leaving a 'dark-brown' residue in the bottom of the digestion vessel. One ml of concentrated hydrochloric acid added to the leached samples did not appear to dissolve any of the residues. Samples were further diluted 11.20-fold because of ALARA concerns. ICPAES measurement results are reported as $\mu\text{g/g}$ dry weight (wet-slurry sample weights adjusted for percent solids). Comparing results of the fusion prepared samples with the acid leached samples, it appears that aluminum, iron and zirconium were incompletely dissolve by the acid leach procedure. Also, sodium concentration appears higher (up to about 25% higher) in the wet-slurry leached samples prepared using ALO-129 procedure than for the same dry-slurry fusion prepared samples except for sample ACL# 00-0912. This sample was prepared by both ALO-129 and ALO-115 procedure using wet-slurry material. Except for potassium and nickel, all results should be based upon the fusion prepared sample data. Potassium and nickel concentration may need to be adjusted based upon sodium concentration between the acid leach and fusion results.

Nine supernatant samples, CUF-AZ-102-001 to CUF-AZ-102-027 (ACL# 00-0904 to 00-0917) were prepared by SAL using ALO-128 Acid Digestion procedure for liquids. About 5.1ml of supernatant sample was processed and diluted to a final volume of about 25ml. No ALARA dilution was performed prior to ICPAES analysis. ICPAES measurement results are reported as $\mu\text{g/ml}$. Supernatant aliquot weights were recorded during sample preparation and later used to convert to volume based upon original sample density information. Measurement results are reported in $\mu\text{g/ml}$ corrected for all preparation and analytical dilution.

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Volumes and weights have been recorded on bench sheets and included with this report. Values reported earlier have been changed from $\mu\text{g/g}$ to $\mu\text{g/ml}$ in this report for supernatant samples only. Values reported earlier for acid leached slurries have been changed to reflect percent solids content of the samples.

For ICPAES analytes of interest (22 analytes) see attached table 3A in ASR-5667/5668.

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

Five fold serial dilution:

(Solid & liquid samples) Results were within tolerance limit of $\leq 10\%$ for all analytes tested after correcting for dilution except as follows. Potassium was slightly low in 5-fold serial dilution in sample ACL# 00-0903. Chromium was very high in the 5-fold dilution of sample ACL# 00-0910. Two other analytes (not analytes of interest) were similarly high. The discrepancy is most likely due to pipetting error during preparation of the 5-fold dilution. Measurements reported for these analytes did not involve the serial dilution used for this test.

Duplicate RPD (Relative Percent Difference):

(Solid & liquid samples) All analytes tested were recovered within tolerance limit of $\leq 20\%$ relative percent difference (RPD) except as follows. RPD for aluminum and, although not an analyte of interest, zirconium were very different in sample ACL# 00-0902 and 00-0902-D. Aluminum RPD for the duplicate samples measured 56% (Zr RPD was 26%). The original sample had higher concentration for both analytes than the duplicate sample. The duplicate samples were prepared using ALO-129 acid leach procedure. Both aluminum and zirconium are more resistive to acid attack. Aluminum and zirconium in the fusion prepared sample ACL# 00-0902 was about twice as high in concentration as the acid leach prepared sample. Less resistant analytes were similar in concentration for both fusion and acid leach prepared samples.

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Post-Spiked Samples (Group A):

(Solid & liquid samples) All analytes of interest were recovered within tolerance of 75% to 125% except potassium in the acid digestion (ALO-128) prepared sample ACL# 00-0903. Potassium recovery (134%) was above the tolerance limit. All other analytes above EQL were within tolerance. The reason for the discrepancy is not known.

Post-Spiked Samples (Group B):

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

Blank Spike:

(Solid & liquid samples) A blank spike is not require for fusion prepared samples. All analytes of interest in solid and liquid prepared samples tested were recovered within tolerance limit of 80% to 120% recovery except as follows. Silver in the blank spike prepared using ALO-129 and ALO-128 procedure produced low recovery (27% and 19% respectively) because hydrochloric acid was used during sample preparation, causing silver to precipitate from solution.

Matrix Spiked Sample:

(Solid & liquid samples) A matrix spike is not require for fusion prepared samples. All analytes of interest in solid and liquid prepared samples tested were recovered within tolerance limit of 75% to 125% recovery, except as follows. Arsenic in the matrix-spiked sample ACL# 00-0909, prepared using ALO-129 procedure, produced low recovery of 22%. The reason for the low arsenic recovery is unclear but may be related to a high sulfate concentration in the sample. Silver and barium in the matrix-spiked sample ACL# 00-0903 prepared using ALO-128 procedure were both low (13% and 5% respectively). Silver recovery is low because hydrochloric acid was used to prepare the sample. Barium recovery is low most likely because of high concentration of sulfate in the sample, which will precipitate barium from solution.

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Quality Control Check Standards (solid and liquid samples):

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

High Calibration Standard Check (solid and liquid samples):

Verification of the high-end calibration for all analytes is within tolerance limits of $\pm 5\%$ accuracy except for potassium. Potassium was slightly high (6% to 8%) during analysis of solids prepared using ALO-129 acid leach procedure.

Process Blank:

(solid and liquid samples) All analytes of interest were within tolerance limit of $\leq$ EQL or $< 5\%$ of sample concentration in the prepared samples.

Laboratory Control Standard (LCS):

(Fusion prepared samples) All analytes measured at a concentration equal to or greater than EQL were recovered within tolerance limit of 75% to 125%. SRM-2710 Montana Soil was used for the LCS in PNL-ALO-115 fusion preparations. Calcium was the only analyte that exceeded tolerance limits. Without blank correction calcium recovery was about 140%. With blank correction calcium recovery was too low, about 28%. Typically a blank correction is applied for calcium determination in the SRM-2710 using ALO-115 KOH/Ni fusion procedure since there calcium is known to be present in the KOH reagent. If a sample contaminated the fusion blank it would lead to low recovery, which may have occurred in this case.

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 HNO₃ 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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Multiplier= ALO#=		5871.0 00-0902-PB-Ni	6039.8 00-0902-Ni	5887.9 00-0902-DUP-Ni	5737.6 00-00909-Ni	5755.2 00-00911-Ni
Client ID= Run Date= (Analyte)		<u>Process</u> <u>Blank</u> 2/2/00 ug/g dry	<u>AZ102 MU</u> 2/2/00 ug/g dry	<u>AZ102 MU</u> 2/2/00 ug/g dry	<u>CUF-AZ102-007</u> 2/2/00 ug/g dry	<u>CUF-AZ102-010</u> 2/2/00 ug/g dry
Det. Limit (ug/mL)						
0.025	Ag	—	[340]	[350]	[350]	[210]
0.060	Al	[510]	130,000	119,000	185,000	94,200
0.250	As	—	—	—	—	—
0.050	B	—	—	—	—	[2,800]
0.010	Ba	—	[560]	[540]	724	[430]
0.010	Be	—	—	—	—	—
0.100	Bi	—	—	—	—	—
0.250	Ca	[14,000]	[7,300]	[7,100]	[10,000]	[7,600]
0.015	Cd	—	21,100	20,300	25,300	14,600
0.200	Ce	—	—	—	[1,300]	—
0.050	Co	—	—	—	—	—
0.020	Cr	—	2,200	2,070	1,520	[910]
0.025	Cu	—	[270]	[280]	[440]	[250]
0.050	Dy	—	—	—	—	—
0.100	Eu	—	—	—	—	—
0.025	Fe	[350]	142,000	137,000	170,000	103,000
0.050	La	—	4,180	4,080	5,250	3,080
0.030	Li	—	—	—	—	—
0.100	Mg	[1,600]	[950]	[660]	[1,600]	[1,000]
0.050	Mn	—	3,400	3,330	4,090	[2,600]
0.050	Mo	—	—	—	—	—
0.150	Na	[2,400]	105,000	96,100	21,300	228,000
0.100	Nd	—	[2,900]	[2,900]	[3,700]	[2,200]
0.100	P	—	[1,200]	[1,300]	[2,500]	[1,900]
0.100	Pb	—	[1,400]	[1,300]	[1,800]	[640]
0.750	Pd	—	—	—	—	—
0.300	Rh	—	—	—	—	—
1.100	Ru	—	—	—	—	—
0.500	Sb	—	—	—	—	—
0.250	Se	—	—	—	—	—
0.500	Si	—	[5,000]	[4,900]	[5,500]	[28,000]
1.500	Sn	—	—	—	—	—
0.015	Sr	—	[270]	[260]	[360]	[220]
1.500	Te	—	—	—	—	—
1.000	Th	—	—	—	—	—
0.025	Ti	—	—	—	—	—
0.500	Tl	—	—	—	—	—
2.000	U	—	[27,000]	[27,000]	[32,000]	[19,000]
0.050	V	—	—	—	—	—
2.000	W	—	—	—	—	—
0.050	Y	—	—	—	—	—
0.050	Zn	[450]	[370]	[310]	[500]	[440]
0.050	Zr	—	15,900	15,800	21,800	14,300

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).

		Multiplier= ALO#=	6372.2 00-00912-Ni	5711.4 00-00915-Ni	6118.7 00-00916-Ni			
		Client ID=	CUF-AZ102- 011 (wet slurry)	CUF-AZ102- 023	CUF-AZ102- 026			
Det. Limit (ug/mL)	Run Date= (Analyte)	2/2/00 ug/g dry	2/2/00 ug/g dry	2/2/00 ug/g dry				
0.025	Ag	[230]	[460]	[450]	--		--	
0.060	Al	108,000	108,000	106,000	--		--	
0.250	As	--	--	--	--		--	
0.050	B	--	--	--	--		--	
0.010	Ba	[490]	936	920	--		--	
0.010	Be	--	--	--	--		--	
0.100	Bi	--	--	--	--		--	
0.250	Ca	[8,700]	[11,000]	[11,000]	--		--	
0.015	Cd	16,600	31,600	31,000	--		--	
0.200	Ce	--	[1,600]	[1,500]	--		--	
0.050	Co	--	--	--	--		--	
0.020	Cr	[1,000]	1,650	1,620	--		--	
0.025	Cu	[300]	[620]	[550]	--		--	
0.050	Dy	--	--	--	--		--	
0.100	Eu	--	--	--	--		--	
0.025	Fe	117,000	223,000	219,000	--		--	
0.050	La	3,440	6,870	6,670	--		--	
0.030	Li	--	--	--	--		--	
0.100	Mg	[1,100]	[2,000]	[1,900]	--		--	
0.050	Mn	[3,000]	5,410	5,350	--		--	
0.050	Mo	--	--	--	--		--	
0.150	Na	240,000	67,500	66,400	--		--	
0.100	Nd	[2,400]	[4,800]	[4,700]	--		--	
0.100	P	[1,400]	[3,500]	[2,100]	--		--	
0.100	Pb	[1,300]	[2,300]	[2,200]	--		--	
0.750	Pd	--	--	--	--		--	
0.300	Rh	--	--	--	--		--	
1.100	Ru	--	--	--	--		--	
0.500	Sb	--	--	--	--		--	
0.250	Se	--	--	--	--		--	
0.500	Si	[4,500]	[7,000]	[7,200]	--		--	
1.500	Sn	--	--	--	--		--	
0.015	Sr	[240]	[470]	[460]	--		--	
1.500	Te	--	--	--	--		--	
1.000	Th	--	--	--	--		--	
0.025	Ti	--	[180]	[190]	--		--	
0.500	Tl	--	--	--	--		--	
2.000	U	[21,000]	[39,000]	[38,000]	--		--	
0.050	V	--	--	--	--		--	
2.000	W	--	--	--	--		--	
0.050	Y	--	[310]	--	--		--	
0.050	Zn	[510]	[980]	[960]	--		--	
0.050	Zr	14,000	34,800	30,500	--		--	

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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Multiplier= ALO#=		585.6 00-0902-PB1	579.8 00-0902-PB2	576.9 00-0902	583.9 00-0902-DUP
Client ID= Run Date=		Process Blank #1 (dry solids equiv.) 2/8/00	Process Blank #2 (dry solids equiv.) 2/8/00	AZ102 MU (dry solids) 2/8/00	AZ102 MU (dry solids) 2/8/00
Det. Limit (ug/mL)	(Analyte)	ug/g dry	ug/g dry	ug/g dry	ug/g dry
0.025	Ag	--	--	376	327
0.060	Al	[44]	[39]	68,200	38,600
0.250	As	--	--	--	--
0.050	B	[53]	[70]	[120]	[130]
0.010	Ba	--	--	540	514
0.010	Be	--	--	[14]	[13]
0.100	Bi	--	--	--	--
0.250	Ca	[710]	[450]	4,600	4,350
0.015	Cd	--	--	20,200	19,900
1.500	Ce	--	--	--	--
0.050	Co	--	--	[71]	[67]
0.020	Cr	--	--	2,170	2,110
0.025	Cu	[19]	--	318	293
0.050	Dy	--	--	--	--
0.100	Eu	--	--	--	--
0.025	Fe	[18]	--	135,000	127,000
2.000	K	--	--	[6,400]	[6,400]
0.050	La	--	--	4,330	4,040
0.030	Li	--	--	--	--
1.500	Mg	--	--	--	--
0.050	Mn	--	--	2,970	2,750
0.150	Mo	--	--	[90]	--
0.150	Na	[120]	[120]	109,000	124,000
0.100	Nd	--	--	3,050	2,820
0.030	Ni	[22]	[19]	9,550	9,060
0.100	P	--	--	--	[140]
0.100	Pb	--	--	1,290	1,190
1.500	Pd	--	--	--	--
0.300	Rh	--	--	--	--
1.250	Ru	--	--	--	--
0.500	Sb	--	--	--	--
0.250	Se	--	--	--	--
0.500	Si	--	--	[1,200]	[2,900]
4.000	Sn	--	--	--	--
0.015	Sr	--	--	248	233
1.500	Te	--	--	--	--
1.250	Th	--	--	[720]	--
0.025	Ti	--	--	[61]	[56]
0.500	Tl	--	--	--	--
2.000	U	--	--	27,400	26,100
0.075	V	--	--	--	--
2.000	W	--	--	--	--
0.400	Y	--	--	--	--
0.050	Zn	[31]	--	[180]	[160]
0.050	Zr	--	--	8,630	6,620

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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Multiplier=		1558.3	1542.8	1650.3	1097.7	1064.4
ALO#=		00-0909-PB1	00-0909-PB2	00-0909	00-0911	00-0912
Client ID=		<u>Process Blank #1</u> <u>(wet slurry equiv.)</u>	<u>Process Blank #2</u> <u>(wet slurry equiv.)</u>	<u>CUF-AZ102-007</u> <u>(wet slurry)</u>	<u>CUF-AZ102-010</u> <u>(wet slurry)</u>	<u>CUF-AZ102-011</u> <u>(wet slurry)</u>
Det. Limit	Run Date=	2/8/00	2/8/00	2/8/00	2/8/00	2/8/00
(ug/mL)	(Analyte)	ug/g dry	ug/g dry	ug/g dry	ug/g dry	ug/g dry
0.025	Ag	--	--	[390]	[230]	[240]
0.060	Al	[120]	[100]	142,000	65,700	67,200
0.250	As	--	--	--	--	--
0.050	B	[140]	[190]	[230]	[260]	[240]
0.010	Ba	--	--	808	511	531
0.010	Be	--	--	[20]	[12]	[12]
0.100	Bi	--	--	--	--	--
0.250	Ca	[1,900]	[1,200]	7,420	4,910	4,790
0.015	Cd	--	--	28,400	17,700	18,100
1.500	Ce	--	--	--	--	--
0.050	Co	--	--	[100]	[61]	[65]
0.020	Cr	--	--	1,580	948	990
0.025	Cu	[51]	--	488	297	304
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	[49]	--	187,000	118,000	121,000
2.000	K	--	--	--	--	--
0.050	La	--	--	6,330	3,980	4,090
0.030	Li	--	--	--	--	--
1.500	Mg	--	--	--	--	--
0.050	Mn	--	--	4,500	2,860	2,940
0.150	Mo	--	--	--	--	--
0.150	Na	[330]	[310]	25,200	264,000	241,000
0.100	Nd	--	--	4,460	2,790	2,870
0.030	Ni	[60]	[51]	13,700	8,580	8,830
0.100	P	--	--	--	--	--
0.100	Pb	--	--	1,870	1,140	1,170
1.500	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.250	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	--	--	[4,900]	[3,000]	[1,600]
4.000	Sn	--	--	--	--	--
0.015	Sr	--	--	393	252	258
1.500	Te	--	--	--	--	--
1.250	Th	--	--	--	--	--
0.025	Ti	--	--	[84]	[53]	[56]
0.500	Tl	--	--	--	--	--
2.000	U	--	--	36,800	22,900	23,500
0.075	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.400	Y	--	--	--	--	--
0.050	Zn	[81]	--	[330]	[290]	[300]
0.050	Zr	--	--	7,900	7,170	8,540

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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Multiplier=		2012.6	1963.1		
ALO#=		00-0915	00-0916		
Client ID=		<u>CUF-AZ102-023 (wet slurry)</u>	<u>CUF-AZ102-026 (set slurry)</u>		
Det. Limit	Run Date=	2/8/00	2/8/00		
(ug/mL)	(Analyte)	ug/g dry	ug/g dry		
0.025	Ag	[460]	[480]	--	--
0.060	Al	44,600	44,600	--	--
0.250	As	--	--	--	--
0.050	B	[260]	[190]	--	--
0.010	Ba	937	979	--	--
0.010	Be	[22]	[23]	--	--
0.100	Bi	--	--	--	--
0.250	Ca	8,970	9,000	--	--
0.015	Cd	32,200	33,700	--	--
1.500	Ce	--	--	--	--
0.050	Co	[120]	[120]	--	--
0.020	Cr	1,500	1,570	--	--
0.025	Cu	562	586	--	--
0.050	Dy	--	--	--	--
0.100	Eu	--	--	--	--
0.025	Fe	215,000	225,000	--	--
2.000	K	--	--	--	--
0.050	La	7,240	7,580	--	--
0.030	Li	--	--	--	--
1.500	Mg	--	--	--	--
0.050	Mn	5,240	5,480	--	--
0.150	Mo	--	--	--	--
0.150	Na	84,500	82,000	--	--
0.100	Nd	5,090	5,340	--	--
0.030	Ni	15,800	16,500	--	--
0.100	P	--	--	--	--
0.100	Pb	2,080	2,170	--	--
1.500	Pd	--	--	--	--
0.300	Rh	--	--	--	--
1.250	Ru	--	--	--	--
0.500	Sb	--	--	--	--
0.250	Se	--	--	--	--
0.500	Si	[3,700]	[3,500]	--	--
4.000	Sn	--	--	--	--
0.015	Sr	462	483	--	--
1.500	Te	--	--	--	--
1.250	Th	--	--	--	--
0.025	Ti	[100]	[110]	--	--
0.500	Tl	--	--	--	--
2.000	U	41,400	43,400	--	--
0.075	V	--	--	--	--
2.000	W	--	--	--	--
0.400	Y	--	--	--	--
0.050	Zn	[870]	[880]	--	--
0.050	Zr	18,100	18,600	--	--

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).

Det. Limit (ug/mL)	Run Date= (Analyte)	Multiplier= ACL#= Client ID= 2/24/00 ug/mL	4.8 00-0903-PB Process Blank 2/24/00 ug/mL	24.5 00-0903 @5 AZ102 M SUP 2/24/00 ug/mL	24.3 00-0903-DUP @5 AZ102 M SUP 2/24/00 ug/mL	9.8 00-0904 @2 CUF-AZ102- 001 2/24/00 ug/mL	9.8 00-0905 @2 CUF-AZ102- 003 2/24/00 ug/mL
0.025	Ag	--	--	--	--	--	--
0.060	Al	4.50	214	219	184	163	
0.250	As	--	--	--	--	--	--
0.050	B	7.44	43.3	45.8	17.6	13.5	
0.010	Ba	--	[0.26]	--	--	--	--
0.010	Be	--	--	--	--	--	--
0.100	Bi	--	--	--	--	--	--
0.250	Ca	[4.8]	[8.8]	[9.8]	[5.8]	[6.1]	
0.015	Cd	--	--	--	--	--	--
0.200	Ce	--	--	--	--	--	--
0.050	Co	--	--	--	--	--	--
0.020	Cr	--	822	831	66.2	66.9	
0.025	Cu	--	[0.84]	--	--	--	--
0.050	Dy	--	--	--	--	--	--
0.100	Eu	--	--	--	--	--	--
0.025	Fe	[0.18]	[0.70]	[0.67]	[0.25]	[0.25]	
2.000	K	--	3,650	3,830	325	329	
0.050	La	--	--	--	--	--	--
0.030	Li	--	--	--	--	--	--
0.100	Mg	--	--	--	--	--	--
0.050	Mn	--	--	--	--	--	--
150	Mo	--	61.0	61.7	5.10	5.12	
150	Na	15.7	67,600	72,700	5,980	6,020	
0.100	Nd	--	--	--	--	--	--
0.030	Ni	[0.35]	--	--	--	--	--
0.100	P	--	143	145	[8.8]	[7.2]	
0.100	Pb	--	--	--	--	--	--
0.750	Pd	--	--	--	--	--	--
0.300	Rh	--	--	--	--	--	--
1.100	Ru	--	--	--	--	--	--
0.500	Sb	--	--	--	--	--	--
0.250	Se	--	--	--	--	--	--
0.500	Si	[21]	299	304	[43]	[37]	
1.500	Sn	--	--	--	--	--	--
0.015	Sr	--	--	--	--	--	--
1.500	Te	--	--	--	--	--	--
1.000	Th	--	--	--	--	--	--
0.025	Ti	--	--	--	--	--	--
0.500	Tl	--	--	--	--	--	--
2.000	U	--	--	--	[23]	[29]	
0.050	V	--	--	--	--	--	--
2.000	W	--	--	--	--	--	--
0.050	Y	--	--	--	--	--	--
0.050	Zn	--	--	--	--	--	--
0.050	Zr	--	--	--	--	--	--

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

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

Multiplier=		9.8	4.8	5.0	24.7	9.9
ACL#=		00-0906 @2	00-0907	00-0908	00-0910 @5	00-0913 @2
Client ID=		CUF-AZ102-015	CUF-AZ102-017	CUF-AZ102-019	CUF-AZ102-009	CUF-AZ102-013
Det. Limit	Run Date=	2/24/00	2/24/00	2/24/00	2/24/00	2/24/00
(ug/mL)	(Analyte)	ug/mL	ug/mL	ug/mL	ug/mL	ug/mL
0.025	Ag	—	—	—	—	—
0.060	Al	146	108	93.8	10,400	4,910
0.250	As	—	—	—	[9.6]	[5.8]
0.050	B	14.9	15.0	11.3	23.9	10.0
0.010	Ba	—	—	—	—	—
0.010	Be	—	—	—	[0.25]	—
0.100	Bi	—	—	—	—	—
0.250	Ca	[5.0]	[4.6]	[4.2]	[14]	[3.9]
0.015	Cd	—	—	—	15.0	[0.87]
0.200	Ce	—	—	—	—	—
0.050	Co	—	—	—	—	—
0.020	Cr	36.3	20.6	11.8	43.8	17.4
0.025	Cu	—	—	—	—	—
0.050	Dy	—	—	—	—	—
0.100	Eu	—	—	—	—	—
0.025	Fe	[0.32]	[0.20]	[0.35]	[3.7]	[1.0]
2.000	K	[190]	115	[69]	[190]	[61]
0.050	La	—	—	—	—	—
0.030	Li	—	—	—	—	—
0.100	Mg	—	—	—	—	—
0.050	Mn	—	—	—	—	—
0.050	Mo	[3.0]	[1.7]	[0.97]	[2.3]	[1.3]
0.150	Na	3,710	2,220	1,430	54,100	20,600
0.100	Nd	—	—	—	—	—
0.030	Ni	—	—	—	—	—
0.100	P	[6.6]	6.52	6.63	163	69.9
0.100	Pb	—	—	—	[6.1]	[2.4]
0.750	Pd	—	—	—	—	—
0.300	Rh	—	—	—	—	—
1.100	Ru	—	—	—	—	—
0.500	Sb	—	—	—	—	—
0.250	Se	—	—	—	—	—
0.500	Si	[33]	33.7	28.7	[120]	56.9
1.500	Sn	—	—	—	—	—
0.015	Sr	—	—	—	—	—
1.500	Te	—	—	—	—	—
1.000	Th	—	—	—	—	—
0.025	Ti	—	—	—	—	—
0.500	Tl	—	—	—	—	—
2.000	U	[38]	[18]	—	—	[24]
0.050	V	—	—	—	[1.4]	[0.70]
2.000	W	—	—	—	—	—
0.050	Y	—	—	—	—	—
0.050	Zn	—	—	[0.33]	[3.4]	[3.7]
0.050	Zr	—	—	—	—	—

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

2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.

3) "—" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

		Multiplier=	9.9	9.6				
		ACL#	00-0914 @2	00-0917 @2				
			CUF-AZ102-	CUF-AZ102-				
		Client ID	022	027				
Det. Limit	Run Date		2/24/00	2/24/00				
(ug/mL)	(Analyte)		ug/mL	ug/mL				
0.025	Ag		--	--	--	--	--	--
0.060	Al		2,180	1,790	--	--	--	--
0.250	As		[2.6]	--	--	--	--	--
0.050	B		10.5	16.0	--	--	--	--
0.010	Ba		--	--	--	--	--	--
0.010	Be		--	--	--	--	--	--
0.100	Bi		--	--	--	--	--	--
0.250	Ca		[4.2]	[3.9]	--	--	--	--
0.015	Cd		--	--	--	--	--	--
0.200	Ce		--	--	--	--	--	--
0.050	Co		--	--	--	--	--	--
0.020	Cr		8.82	8.05	--	--	--	--
0.025	Cu		--	--	--	--	--	--
0.050	Dy		--	--	--	--	--	--
0.100	Eu		--	--	--	--	--	--
0.025	Fe		[0.81]	--	--	--	--	--
2.000	K		[27]	[20]	--	--	--	--
0.050	La		--	--	--	--	--	--
0.030	Li		--	--	--	--	--	--
0.100	Mg		--	--	--	--	--	--
0.050	Mn		--	--	--	--	--	--
0.150	Mo		[2.5]	[2.1]	--	--	--	--
0.150	Na		8,700	7,110	--	--	--	--
0.100	Nd		--	--	--	--	--	--
0.030	Ni		--	--	--	--	--	--
0.100	P		28.4	38.5	--	--	--	--
0.100	Pb		--	--	--	--	--	--
0.750	Pd		--	--	--	--	--	--
0.300	Rh		--	--	--	--	--	--
1.100	Ru		--	--	--	--	--	--
0.500	Sb		--	--	--	--	--	--
0.250	Se		--	--	--	--	--	--
0.500	Si		[45]	[47]	--	--	--	--
1.500	Sn		--	--	--	--	--	--
0.015	Sr		--	--	--	--	--	--
1.500	Te		--	--	--	--	--	--
1.000	Th		--	--	--	--	--	--
0.025	Ti		--	--	--	--	--	--
0.500	Tl		--	--	--	--	--	--
2.000	U		--	--	--	--	--	--
0.050	V		--	--	--	--	--	--
2.000	W		--	--	--	--	--	--
0.050	Y		--	--	--	--	--	--
0.050	Zn		[0.98]	[0.57]	--	--	--	--
0.050	Zr		--	--	--	--	--	--

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).

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

Client: K Brooks
ACL Numbers: 00-0902 to 00-0916
Analyst: MJ Steele

Charge Code/Project: W45577/29953
ASR Number: 5667 and 5668
Analysis Date: 2/11/00 and 3/15/00

Procedure: PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography"
M&TE: IC system (WD25214); Balance (360-06-01-031) --- See Chemical Measurement Center 98620 RIDS IC File for Calibration, Standards Preparations, and Maintenance Records.

Final Results:

The AZ-102 as received samples (ASR 5667) and the AZ-102 CUF samples (ASR 5668) were analyzed by ion chromatography (IC) for inorganic anions as specified in the governing ASR. The final results are presented in Tables 1 through 3 below. Table 1 presents the slurry anion results in µg/g on a dry weight basis (i.e., corrected for weight percent solids) and Table 2 presents the liquid samples (supernatant, filtrates, washes, and leaches) in µg/ml. Although not requested by the ASR, Table 3 presents the slurry concentration in µg/g of wet slurry. All sample were diluted at the IC workstation to appropriate levels to ensure that all anions reported were measured within the calibration range. The diluted slurry samples were analyzed (after filtering) on February 11 and the liquids on March 15.

Table 1: Slurry Results on a Dry Weight Basis		Solids	Wt%	F	Cl	NO ₂	Br	NO ₃	PO ₄	SO ₄	C ₂ O ₄
Lab ID	Sample ID	Dil Fctr	Solids	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g
00-00902 PB	Process Blank (equiv Dil)	103.3	41.8	< 25	41	< 50	< 25	< 50	< 50	< 50	< 50
00-00902	AZ-102 MU As Rec Slurry	103.0	41.8	1,400	< 250	63,300	< 250	20,400	660	27,400	3,500
00-00902 Dup	AZ-102 MU As Rec Slurry dup	103.6	41.8	2,000	< 250	68,800	< 250	22,300	470	31,100	4,300
	RPD			35%	n/a	8%	n/a	9%	34%	13%	21%
	Matrix Spike True @10x			0.4	0.4	1.2	0.4	1.2	1.2	1.2	1.2
00-00902 MS	AZ-102 As Rec Slurry MS Rec			121%	205%	48%	87%	6%	97%	85%	93%
	Matrix Spike True			4.0	4.0	12.0	4.0	12.0	12.0	12.0	12.0
00-00902 PS	AZ-102 As Rec Slurry PS Rec			107%	104%	114%	95%	110%	102%	98%	106%
00-00902 BS	AZ-102 As Rec Slurry BS Rec			103%	195%	95%	96%	105%	82%	104%	98%
00-00902 BS	AZ-102 As Rec Slurry BS Rec			106%	197%	98%	94%	105%	101%	109%	106%
00-00909VB	Dil Blank --(equiv. Dil/Wt%)	13.9	13.3	< 30	< 30	< 60	< 30	< 60	< 60	< 60	< 60
00-00909	CUF-AZ-102-007 Slurry Wash #3	12.6	11.3	230	400	5,500	< 30	1,600	960	2,800	420
00-00911	CUF-AZ-102-010 Slurry Caustic Leach	17.9	18.0	590	4,200	1,500	< 250	< 500	2,200	1,000	< 500
00-00912	CUF-AZ-102-011 Slurry Caustic Leach	18.0	18.5	< 250	4,100	2,000	< 250	< 500	1,900	990	< 500
00-00915	CUF-AZ-102-023 Final Slurry	11.0	10.4	300	1,500	< 550	< 300	< 550	850	690	< 550
00-00916	CUF-AZ-102-026 Final Slurry	11.5	10.7	< 300	1,200	< 550	< 300	< 550	870	< 550	< 550
00-00916 Rep	CUF-AZ-102-026 Final Slurry Rep	11.5	10.7	< 300	1,200	< 550	< 300	< 550	970	< 550	< 550
	RPD			n/a	0%	n/a	n/a	n/a	11%	n/a	n/a

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

MS Rec = Matrix Spike Standard % Recovery, PS Rec = Post Spike % Recovery, BS Rec = Blank Spike Standard % Recovery

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

Table 2: Liquid Samples

Lab ID	Sample ID	F ug/ml	Cl ug/ml	NO ₂ ug/ml	Br ug/ml	NO ₃ ug/ml	PO ₄ ug/ml	SO ₄ ug/ml	C ₂ O ₄ ug/ml
00-00903	AZ-102 (M Sup) As Received	720	< 100	41,600	< 100	14,000	440	21,300	2,890
00-00903 D	AS-102 (M Sup) As Received Dup	780	< 100	41,700	< 100	13,600	430	20,800	3,690
	RPD	8%	n/a	0%	n/a	3%	2%	2%	24%
00-00903 MS	AZ-102 (M Sup) As Received MS Rec	98%	94%	99%	120%	118%	106%	101%	106%
00-00903 MS	AZ-102 (M Sup) As Received MS Rec	108%	109%	105%	107%	106%	105%	106%	105%
Woring Spike	Recovery	98%	94%	98%	118%	119%	106%	101%	106%
Woring Spike	Recovery	109%	109%	105%	107%	106%	106%	106%	105%
00-00904	CUF-AZ-102-001 Filtrate 1	90	< 25	3,820	< 25	1,080	< 50	1,630	250
00-00905	CUF-AZ-102-003 Filtrate 2	90	< 25	3,710	< 25	1,030	< 50	1,580	300
00-00906	CUF-AZ-102-015 Wash 1	50	< 10	1,930	< 10	740	< 20	1,130	160
00-00907	CUF-AZ-102-017 Wash 2	30	< 10	1,120	< 10	380	< 20	600	130
00-00908	CUF-AZ-102-019 Wash 3	20	< 10	660	< 10	220	30	350	80
00-00910	CUF-AZ-102-009 Caustic Leach	< 10	890	420	< 10	160	210	220	30
00-00913	CUF-AZ-102-013 Caustic Wash 1	< 25	370	180	< 25	100	160	110	< 50
00-00914	CUF-AZ-102-022 Caustic Wash 2	< 25	370	180	< 25	100	160	110	< 50

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

MS Rec = Matrix Spike Standard % Recovery, PS Rec = Post Spike % Recovery, BS Rec = Blank Spike Standard % Recovery

Table 3: Slurry Results on a Wet Weight Basis

Lab ID	Sample ID	Solids Dil Fctr	F ug/g	Cl ug/g	NO ₂ ug/g	Br ug/g	NO ₃ ug/g	PO ₄ ug/g	SO ₄ ug/g	C ₂ O ₄ ug/g
00-00902 PB	Process Blank (equiv Dil as slurries)	103.3	< 10	17	< 20	< 10	< 20	< 20	< 20	< 20
00-00902	AZ-102 MU As Rec Slurry	103.0	600	< 100	26,400	< 100	8,500	280	11,400	1,500
00-00902 Dup	AZ-102 MU As Rec Slurry dup	103.6	820	< 100	28,700	< 100	9,300	200	13,000	1,800
00-00909 PB	Process Blank (equiv. Dil as slurries)	13.9	< 4.0	< 4.0	< 7.0	< 4.0	< 7.0	< 7.0	< 7.0	< 7.0
00-00909	CUF-AZ-102-007 Slurry Wash #3	12.6	30	50	620	< 3.0	180	110	320	50
00-00911	CUF-AZ-102-010 Slurry Caustic Leach	17.9	110	750	280	< 50	< 90	400	190	< 90
00-00912	CUF-AZ-102-011 Slurry Caustic Leach	18.0	< 50	750	360	< 50	< 90	350	180	< 90
00-00915	CUF-AZ-102-023 Final Slurry	11.0	30	160	< 60	< 30	< 60	90	70	< 60
00-00916	CUF-AZ-102-026 Final Slurry	11.5	< 30	120	< 60	< 30	< 60	90	< 60	< 60
00-00916 Rep	CUF-AZ-102-026 Final Slurry Rep	11.5	< 30	120	< 60	< 39	< 60	100	< 60	< 60

Q.C. Comments for Slurry Analyses (February 11, 2000) :

Duplicates: Duplicate samples were extracted from the AZ-102 as received slurry sample (00-0902) and diluted in the hot cells. These dilution were than transferred to the IC Workstation and treated as duplicate for the IC analysis. At the IC Workstation, the CUF-AZ-102-026 final slurry sample (00-0916) which was diluted in the hot cell was split and analyzed in replicate. For anions above the estimated quantitation level (EQL; i.e., the lowest calibration standard), the Relative Percent Difference (RPD) is within the acceptance criteria of 20%, except for fluoride, phosphate and oxalate on the duplicate processed in the hot cell. Oxalate is not an analyte of

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interest stated on the ASR and the phosphate results are just marginally above the EQL. Interferences from suspected organic anion significantly affect the ability to accurately quantify the fluoride peak and the fluoride results should be considered qualitative.

Matrix Spike: A matrix spike of the AZ-102 as received sample (00-0902) were prepared in the hot cells. Unfortunately the spiking level was insufficient to recover the spike after the required analytical dilutions at the IC Workstation. Recoveries are reported for this MS; however, the spike level is right at the EQL following dilutions. Since no Matrix Spike results could be obtained, the sample was post-spike at the IC Workstation. All post-spike recoveries were within the spiking acceptance criteria of 75% to 125%.

Blank Spike: A blank spike was prepared in the hot cells. Except for chloride which has a recovery about double (i.e., 195%) that anticipated and phosphate which recovered low (i.e., 82%), the blank spike recoveries were within the acceptance criteria of 90% to 110%. The reason for the phosphate low recovery is unknown. Since all verification standard phosphate recoveries are acceptable, it is unlikely that the low phosphate recovery is due to column degradation. The high chloride results are suspected to be from a chloride contamination introduced during processing in the hot cell. Reanalysis of the blank spike produced good recovery for phosphate (i.e., 101%), but produced the same high chloride recovery (i.e., 197%). Since the source of the blank spike contamination can not be determined, the reported chloride results may be higher than the actual chloride sample concentration.

System Blank/Processing Blanks: Four system blanks were process during the analysis of the slurry samples. No anions were detected in the system blanks above the estimate quantitation level.

Quality Control Calibration Verification Check Standards: Four mid-range verification standards were analyzed throughout the analysis runs. Except for two chloride, one nitrite, and one nitrate result, the reported results for all anions of interest were recovered within the acceptance criteria of 90% to 110% for the verification standard. For those result outside the acceptance criteria, no results had recovery less than 88%, and therefore these results should have little, if any, effect on the reported slurry sample results.

Q.C. Comments for Liquids Analyses (March 15, 2000):

Duplicates: Although there is only one AZ-102 as received supernatant sample (00-0902), two samples were extracted from the supernatant sample in the hot cells and transferred to the IC workstation for analysis. The samples were treated as duplicates. The RPDs for the measured anion are less than 10% and are well within the acceptance criteria of 20%.

Matrix Spike: Two matrix spikes were prepared from the AZ-102 as received sample (00-0902) after the sample was delivered to the IC Workstation. No attempt to spike the supernatant in the

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hot cells was made. All anion recoveries for all anions were within the 75% to 125% recovery acceptance criteria.

Blank Spike: No blank spikes were prepared in the hot cells. However, the working spike (i.e., the spike solution used to prepare the matrix spike samples) was measured at the same time as the Matrix Spike samples. One working spike analysis produced higher than expected recoveries for bromide and nitrate. Repeating the working spike analysis produced recoveries within the acceptance criteria of 90% to 110% recovery.

System Blank/Processing Blanks: Thirteen system blanks were processed during the analysis of the liquid samples. Other than three oxalate system blanks, no anions were detected in the system blanks above the estimate quantitation level. The low level of oxalate detected in the system blank has not affect on the reported oxalate values.

Quality Control Calibration Verification Check Standards: Eleven mid-range verification standards were analyzed throughout the analysis runs. For most of the anions recoveries were within the acceptance criteria from 90% to 110% for the verification standard. Two nitrite results, two phosphate results and one fluoride result recovered below the 90% lower limit; however these recoveries were all greater than 85%. A single fluoride result recovered at about 60%, well below the acceptance criteria. In general the reported fluoride results should be considered qualitative. Not only do the samples appear to adversely affect the column, there appears to be significant interferences in the fluoride region of the chromatograms (possibly from organic anions).

General Comments:

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- 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.
- Routine precision and bias are typically $\pm 15\%$ or better for non-complex aqueous samples that are free of interference and have similar concentrations as the measured anions.

Analyst:

Date

Approval:

Date

Archive Information:

Files: ASR 5667-8 Brooks.doc

ASR 5667-8 Final.xls

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

Client: K. Brooks
ACL Numbers: 00-0902 to 00-0916
Analyst: MJ Steele

Charge Code/Project: W45577 / 29953
ASR Number: 5667 and 5668
Analysis Date: April 5 and 7, 2000

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:

Slurry Samples								
Lab Number	Sample ID	Wt g	TIC µg C/g	TIC RPD %	TOC µg C/g	TOC RPD %	TC µg C/g	TC RPD %
00-0902 Dup	AZ-102 MU (As Received) Dup	0.1254	7,240		760		8,000	
00-0902	AZ-102 MU (As Received)	0.2004	8,740	19	760	1	9,500	17
00-0909	CUF-AZ102-007	0.1875	560		610		1,170	
00-0911	CUF-AZ102-010	0.1041	11,200		780		11,980	
00-0912	CUF-AZ102-011	0.1368	lost		2,790		n/a	
00-0915	CUF-AZ102-023	0.1441	2,910		2,710		5,620	
00-0915 MS	CUF-AZ102-023 MS	0.1150	94.4%		96.2%		95.1%	
00-0916	CUF-AZ102-026	0.1663	2,820		2,790		5,610	

Liquid Samples								
Lab Number	Sample ID	Vol ml	TIC µg C/ml	TIC RPD %	TOC µg C/ml	TOC RPD %	TC µg C/ml	TC RPD %
00-0903	AZ-102 M Sup (As Received)	0.10	8,670		1,200		9,870	
00-0903 Dup	AZ-102 M Sup (As Received) Dup	0.20	8,570	1	930	25	9,500	4
00-0903 MS	AZ-102 M Sup (As Received) MS	0.10	99.4%		87.6%		93.7%	
00-0904	CUF-AZ102-001 Filtrate 1	0.50	1,070		120		1,190	
00-0904 Dup	CUF-AZ102-001 Filtrate 1 Dup	0.30	1,070	0	< 60	n/a	1,070	11
00-0905	CUF-AZ102-003 Filtrate 2	0.50	1,100		140		1,240	
00-0905 Dup	CUF-AZ102-003 Filtrate 2 Dup	0.30	1,120	2	60	n/a	1,180	5
00-0906	CUF-AZ102-015 Wash 1	0.50	790		50		840	
00-0906 Dup	CUF-AZ102-015 Wash 1 Dup	0.20	510	42	< 90	n/a	510	48
00-0907	CUF-AZ102-017 Wash 2	0.50	430		40		470	
00-0907 Dup	CUF-AZ102-017 Wash 2 Dup	0.30	430	1	< 60	n/a	430	9
00-0908	CUF-AZ102-019 Wash 3	0.50	420		< 40		420	
00-0908 Dup	CUF-AZ102-019 Wash 3 Dup	0.18	430	2	< 100	n/a	430	2
00-0910	CUF-AZ102-009 Caustic Leach	0.50	890		< 40		890	
00-0910 Dup	CUF-AZ102-009 Caustic Leach Dup	0.20	910	1	< 90	n/a	910	1
00-0913	CUF-AZ102-013 Caustic Wash 1	0.50	3,090		570		3,660	
00-0913 Dup	CUF-AZ102-013 Caustic Wash 1 Dup	0.10	3,160	2	590	n/a	3,750	2
00-0914	CUF-AZ102-022 Caustic Wash 2	0.50	640		50		690	
00-0914 Dup	CUF-AZ102-022 Caustic Wash 2 Dup	0.30	650	3	70	n/a	720	5

RPD = Relative Percent Difference (between sample and duplicate/replicate); calculated from unrounded data.

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

The analyses of the samples submitted under ASRs 5667 and 5668 were 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.

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

April 5, 2000 QC *****

Calibration Standards: The QC system calibration standards were all within acceptance criteria of 90% to 110%, with the average recovery being about 95% for TIC and 95% for TOC.

Calibration Blanks: The four calibration blanks run at the beginning, middle, and end of the analysis run were acceptable, averaging about 11 μgC TIC and 33 μgC TOC. These calibration/system blanks are considered good and the standard deviations for the TIC and TOC blanks is within the historical pooled standard deviation used to establish the method detection limits.

Duplicates: The precision between the duplicates, as demonstrated by the Relative Percent Difference (RPD), is acceptable. The RPDs for sample 00-0902 (AZ-102 as received) are within the acceptance criteria of 20%. However, the relatively high RPDs (i.e., 19% for TIC and 17% for TOC) most likely indicate difficulties in obtaining representative "slurry" samples.

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. A matrix spike was prepared from sample 00-0915 (CUF-AZ102-023). The matrix spike demonstrated recoveries of about 94% for TIC and 96% for TOC; well within the acceptance criteria of 75% to 125% recovery.

April 7, 2000 QC *****

Calibration Standards: The QC system calibration standards were all within acceptance criteria, with the average recovery being about 99% for TIC and 97% for TOC. This is well within the acceptance criteria of 90% to 110%.

Calibration Blanks: The four calibration blanks run at the beginning, middle, and end of the analysis run were acceptable, averaging about 18 μgC TIC and 58 μgC TOC. These

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

calibration/system blanks are considered good and the standard deviations for the TIC and TOC blanks is within the historical pooled standard deviation used to establish the method detection limits.

Duplicates: The precision between the duplicates, as demonstrated by the Relative Percent Difference (RPD), is very good except for sample 00-0906 (CUF-AZ102-015). The RPDs for most samples are within the acceptance criteria of 20%. Sample 00-0906 demonstrated an RPD of 42% to 48%. Other than sample heterogeneity or undetected analysis error, no explanation can be offered for the poor precision.

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. A matrix spike was prepared from sample 00-0902 (AZ-102 as received). The matrix spike demonstrated recoveries of about 99% for TIC and 88% for TOC; well within the acceptance criteria of 75% to 125% recovery.

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 Thie 4/11/00

Review/Approval by: DLT Goldw.

Date 4/12/00

Archive Information:

Files: ASR 5667 5668 Brooks.doc

ASR 5644 5667 5668 5744 Liq+Solids.xls

Date March 17, 2000

To Kriston Brooks

From Michael Urie

 Subject Cyanide Results for As Received AZ-102
Samples and CUF-AZ102 Samples

Table 1: Slurry Samples

Lab #	Client ID	(µg CN / g Slurry)		RPD Rec (%)	Rec. (%)	ug CN / g dry solids	
		Sample	Dup			Sample	Dup
00-0902 CB	Hot Cell Blank	<0.2	--	--	--	--	--
00-0902	AZ102 As Rec Slurry	5.6	5.7	2.6	--	13.4	13.6
00-0909	CUF-AZ-102-007	1.0	--	--	--	8.8	--
00-0911	CUF-AZ-102-010	0.90	--	--	--	5.0	--
00-0912	CUF-AZ-102-011	0.92	--	--	--	5.0	--
00-0915	CUF-AZ-102-023	0.87	--	--	--	8.3	--
00-0916	CUF-AZ-102-026	0.89	--	--	--	8.3	--
00-0916 Spike	CUF-AZ-102-026 Spike	--	--	--	68	--	--

Table 2: Supernate, Filtrate and Wash Samples

Lab #	Client ID	(µg CN / mL)		RPD Rec (%)	Rec. (%)
		Sample	Dup		
00-0903 CB	Hot Cell Blank	< 0.03	--	--	--
00-0903	AZ102 As Rec Supernate	lost	9.6	n/a	--
00-0903 Spike	AZ102 As Rec Supernate Spike	--	--	--	94
00-0904	CUF-AZ-102-001	3.3	--	--	--
00-0905	CUF-AZ-102-003	3.2	--	--	--
00-0906	CUF-AZ-102-015	3.1	--	--	--
00-0907	CUF-AZ-102-017	2.4	--	--	--
00-0908	CUF-AZ-102-019	2.3	--	--	--
00-0910	CUF-AZ-102-009	1.7	--	--	--
00-0913	CUF-AZ-102-013	1.8	--	--	--
00-0914	CUF-AZ-102-022	1.0	--	--	--

The CN results for AZ-102 As Received samples and the CUF AZ-102 samples analyzed on March 9-10, 2000 per ASR 5667 (as received) and ASR 5668 (CUF) are reported in the Tables 1 and 2 above. The samples were weighed in the Shielded Analytical Laboratory and delivered, ready for distillation, to Laboratory 400 in the Radiochemical Processing Laboratory. The samples were distilled with the addition of sulfamic acid to ensure there would be no interference if nitrates were present in the sample. The samples were analyzed using a Lachat QuickChem AE Autoanalyzer (WC36517). The reporting limits are estimated to be approximately 0.2 µg CN/g (for solids/slurries) or 0.03 µg CN/mL (for liquids).

An independent mid-range calibration check solution run at the beginning, middle, and end of the analysis batches gave an average recovery of 99% over the two days of analysis. These calibration check standards ranged from 92% recovery to 106% recovery which is within the 85% to 115% acceptance criteria. A solid laboratory control standard (ERA-LSC) was analyzed at 178 µg/g, well within the certified advisory range of 77 to 301 µg/g.

A liquid sample (00-0903 AZ-102 As Received) and a slurry sample (00-0916 CUF-AZ-102-026) were spiked with CN. The spike recovery for the liquid sample was within the acceptance criteria of 75% to 125%; however, the spike recovery for the slurry sample did not meet this criterion. Since the CN concentration of the slurry is reasonably low, the poor recovery is not due to sampling heterogeneity. The slurry concentration is less than 10% of the expected concentration of the spiked slurry sample and sampling heterogeneity should have little effect on the measured spike concentration. A blank spike at the same concentration as the slurry matrix spike measured 92%. Other than potential matrix interferences or reactions, no explanation is offered for the poor slurry spike recovery.

One liquid sample and one slurry sample per batch were analyzed in duplicate. The Relative Percent Difference (RPD, precision) could not be calculated for the liquid sample since one of the samples was inadvertently lost. The RPD for the slurry samples was 2.6%, well within the acceptance criteria of 20%.

All sample preparation sheets, standard preparation information, and analytical data are included with this report.

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

page 1 of 3

WO/Project: W45577/29953
Client: K. Brooks

ACL Numbers: 00-00902, 00-00903, 00-00915 & 00-00916
ASR Number 5667, 5668

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

Analyst: J. J. Wagner

Digestion Date: March 14, 2000 **Analysis Date:** March 15, 2000

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: Jeremy Wagner 4-17-00
Approval: MW **Date** 4-17-00

Final Results:

The samples were analyzed by cold vapor atomic absorption spectrophotometry for inorganic mercury as specified in ASR 5667,5668. Approximately 0.09 to 0.19g weight of wet slurry and approximately 0.1g weight of supernatant liquid samples were processed and diluted to a final volume of 25ml per procedure ALO-131. Additional analytical dilution of 3 to 10-fold was necessary for some samples. The mercury concentration results are presented in the table below.

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

page 2 of 3

Lab ID	Solid Sample ID	Sample Weight (g wet)	Digestion Factor	Analytic Dilution Factor	Hg ug/g (wet wt)	Percent Solids (%)	Hg ug/g (dry wt)	RPD (%)
00-00902CB	Cell Blank	0.1650	151.5	1	<0.02			
00-00902	AZ102 MU	0.1947	128.4	10	7.56	41.76	18.1	
00-00902D	AZ102 MU	0.1299	192.5	10	8.37	41.76	20.0	10
00-00915*	CUF-AZ102-023	0.0905	276.2	3	2.78	10.44	26.6	
00-00916*	CUF-AZ102-026	0.1101	227.1	5	2.83	10.69	26.5	0.4

Lab ID	Liquid Sample ID	Sample Weight (g)	Digestion Factor	Analytic Dilution Factor	Hg ug/g	RPD (%)
00-00903CB	Cell Blank	0.0974	256.7	1	0.08	
00-00903	AZ102 M SUP	0.1120	223.2	1	0.21	
00-00903D	AZ102 M SUP	0.1089	229.6	1	0.16	N/A

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

N/A = RPD is not calculated when results are less than 5 x IDL

“*” Field duplicate 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 detection limit (IDL) 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 96% and 95%, well within the acceptance criteria of 80% to 120%.

Matrix Spiked Sample: A matrix spike was prepared for the slurry and supernatant samples submitted under ASR-5667 and ASR-5668. However, recovery of the matrix spike (45% & 55%) fell below the acceptance criteria of 75% to 125%. Failure of the matrix spike recover may have been caused by excess organic material present which would requiring more oxidizing reagent than specified in the digestion procedure.

Duplicate: Relative percent difference for duplicate wet slurry samples is within acceptance criteria of $\leq 20\%$ RPD for RPL# 00-00902, 00-00902D (10% RPD) and RPL# 00-00915 and 00-00916 field duplicate samples (0.4%RPD).

Laboratory Control Sample (solids): Sample recovery of mercury in SRM-2709 San Joaquin Valley Soil certified by NIST to contain $1.40 \pm 0.08 \mu\text{g/g}$ was recovered within acceptance criteria of 75% to 125%.

Laboratory Control Sample (liquids): Sample recovery of mercury in SRM-1641c "Mercury in H_2O " certified by NIST to contain $1.47 \mu\text{g/ml}$ was recovered (35% and 49%) below acceptance criteria of 75% to 125%. NIST originally specified an expiration date of 1 year from date of delivery (Dec. 1, 1997) but had extended the expiration date until May 4, 2000. Perhaps the standard was too old resulting in poor recovery. However, two blank spikes were processed similarly to the samples at the same time using NIST traceable certified standards from an alternate vendor, different than the calibration standards. The blank spike recoveries (96% and 95%) were well within acceptance tolerance of 80% to 100% recovery indicating that sample preparation and analysis were acceptable.

Cell Blanks/Processing Blanks: A cell blank, consisting of an aliquot of water used in the hot cell, was processed at the same time as the samples. The concentration measured was less than two times detection limit. Some samples were about the same concentration as the process blank and other samples were many times this level.

Quality Control Calibration Verification Check Standards: Six 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.

Date March 17, 2000
To K.P.Books
From Tom Farmer *Quell Thomas Jarama*
Subject ICP/MS Analysis of Submitted Samples
(ALO#00-00902, 00-00915, 00-00916)

329/4 File
LSO Project File
Mike Urie

Pursuant to your request, the 6 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.

Values for tin were obtained using the response of a natural tin standard. Interference corrections for ^{126}Xe and ^{126}Te were calculated. 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.

No known cesium 135, 137 standard was available. Results reported are direct cesium ratios using natural barium standards for mass bias corrections.

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

Brooks Analysis

March 17, 2000

J. Brooks
3/17/00

Results are reported in μg analyte/ g (ppm) of solid sample.

Sample ID	Client ID	ICP/MS Number	Rb $\mu\text{g/g} \pm 1\text{SD}$	Tc-99 $\mu\text{g/g} \pm 1\text{SD}$	•Sn-126 $\mu\text{g/g}$	†I-127 $\mu\text{g/g} \pm 1\text{SD}$	†I-129 $\mu\text{g/g} \pm 1\text{SD}$
00-00902-PB-Ni	PROCESS BLANK	00208b16	182 $\pm$ 3	1.0 $\pm$ 0.1	<0.9	25 $\pm$ 5	<7.7
00-00902-Ni	AZ102 MU	00208b17	244 $\pm$ 3	24.5 $\pm$ 0.6	<0.9	23 $\pm$ 4	<3.9
00-00902-DUP-Ni	AZ102 MU	00208b18	219 $\pm$ 4	20.9 $\pm$ 1.0	<0.7	18 $\pm$ 1	<3.1
00-00902 + spike	AZ102 MU	00210a16				41 $\pm$ 10	<4.0
Spike Recovery						88%	
00-00915-Ni	CUF-AZ102-023	00208b19	230 $\pm$ 6	1.7 $\pm$ 0.7	<0.9	14.7 $\pm$ 0.1	<2.5
00-00915-Ni Dup	CUF-AZ102-023	00208b20	222 $\pm$ 6	1.7 $\pm$ 0.2	<0.9		
00-00916-Ni	CUF-AZ102-026	00208b21	229 $\pm$ 2	1.4 $\pm$ 0.1	<0.9	24 $\pm$ 8	<6.0
00-00916-Ni + spike	CUF-AZ102-026	00208b22	254 $\pm$ 1	4.8 $\pm$ 0.6			
Spike Recovery			102%	93%			
SRM 2710	LCS/00-00902/Ni	00208b24	334 $\pm$ 9	0.6 $\pm$ 0.2	<0.9	11 $\pm$ 1	<2.4
CCV and Blank results are reported in ng/g (ppb)							
1%HNO3		00208b1	<0.03	<0.02	<0.06	0.04 $\pm$ 0.004	<0.2
1%HNO3		00208b15	<0.02	<0.02	<0.06	0.04 $\pm$ 0.02	<0.1
5ppb Rb, Pr, Cs		00208b12	5.09 $\pm$ 0.13				
5ppb Rb, Pr, Cs		00208b27	5.12 $\pm$ 0.12				
0.5ppb Tc-99		00208b14		0.515 $\pm$ 0.049			
0.5ppb Tc-99		00208b26		0.496 $\pm$ 0.014			
1.0ppb I-127		00210a15				0.87 $\pm$ 0.02	
0.1ppb I-129		00210a11					0.11 $\pm$ 0.02
1ppb Ta/2ppb Np-237		00209a3					
1ppb Ta/2ppb Np-237		00209a17					

*Results are from procedure 00209a.

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

†Results are from procedure 00210a.

**Calculated value was greater than the detection limit, but less than the reporting limit.

Rev: O.T. James

Date: 17 Mar 00 10/3

Brooks Analysis

March 17, 2000

J. Brown
3/17/00

Results are reported in µg analyte/ g (ppm) of solid sample

Sample ID	Client ID	ICP/MS Number	Cs µg/g ± 1SD	Pr µg/g ± 1SD	*Ta µg/g ± 1SD	*Np-237 µg/g ± 1SD
00-00902-PB-Ni	PROCESS BLANK	00208b16	<0.12	0.11 ± 0.04	1.9 ± 0.2	<0.3
00-00902-Ni	AZ102 MU	00208b17	24.4 ± 0.5	487 ± 7	1.7 ± 0.2	103 ± 4
00-00902-DUP-Ni	AZ102 MU	00208b18	22.7 ± 1.2	475 ± 4	2.72 ± 0.2	103 ± 1
00-00902 + spike	AZ102 MU	00210a16				
Spike Recovery						
00-00915-Ni	CUF-AZ102-023	00208b19	2.4 ± 0.3	814 ± 13	4.93 ± 0.3	177 ± 1
00-00915-Ni Dup	CUF-AZ102-023	00208b20	2.29 ± 0.16	800 ± 10	7.23 ± 0.4	178 ± 3
00-00916-Ni	CUF-AZ102-026	00208b21	2.4 ± 0.3	784 ± 7	34.6 ± 1.3	172 ± 2
00-00916-Ni + spike	CUF-AZ102-026	00208b22	30.5 ± 1.6	809 ± 9	54.2 ± 1.4	214 ± 2
Spike Recovery			115%	102%	107%	114%
SRM 2710	LCS/00-00902/Ni	00208b24	79.7 ± 2.0	8.24 ± 0.42	14.3 ± 0.5	<0.3
CCV and Blank results are reported in ng/g (ppb)						
1%HNO3		00208b1	<0.01	<0.02	<0.04	<0.02
1%HNO3		00208b15	<0.01	<0.02	<0.02	<0.02
5ppb Rb, Pr, Cs		00208b12	5.18 ± 0.13	5.25 ± 0.07		
5ppb Rb, Pr, Cs		00208b27	5.12 ± 0.07	4.96 ± 0.18		
0.5ppb Tc-99		00208b14				
0.5ppb Tc-99		00208b26				
1.0ppb I-127		00210a15				
0.1ppb I-129		00210a11				
1ppb Ta/2ppb Np-237		00209a3			0.911 ± 0.045	1.85 ± 0.03
1ppb Ta/2ppb Np-237		00209a17			0.955 ± 0.020	1.87 ± 0.07

*Results are from procedure 00209a.

•Obtained using response of natural tin. Should be consider

†Results are from procedure 00210a.

**Calculated value was greater than the detection limit, but

DATA REVIEW

Reviewed O. T. Farmer

Date: 17 Mar 00 Page: 20/3

Brooks Analysis

March 17, 2000

Sample ID	Client ID	ICP/MS Number	avg	Cs(135/137) std	%rsd
0.5M HNO3		000316a1	-4.525	11.339	-250.59
00-00902-PB	PROCESS BLANK	000316a0	-2.096	9.181	-438.05
00-00902-Ni	AZ102 MU	000316a2	0.446	0.024	5.37
00-00902-DUP-Ni	AZ102 MU	000316a4	0.440	0.015	3.40
00-00915-Ni	CUF-AZ102-023	000316a3	0.416	0.063	15.26
00-00916-Ni	CUF-AZ102-)26	000316a5	0.430	0.036	8.37
SRM 2710	LCS/00-00902/Ni	000316a	-0.383	2.552	-665.85

DATA REVIEW

Reviewed: O.J. James

Date: 12 March 2000 Page 3 of 3

AB
3/17/00

Date March 31, 2000
To K.P.Books
From Tom Farmer *T.J. Farmer 3/1/2000*
Subject ICP/MS Analysis of Submitted Samples
(ALO#00-00903)

329/4 File
LSO Project File
Mike Urie

Pursuant to your request, the 5 acid digested samples that you submitted for analysis were analyzed by ICPMS for selected elements. The results of this analysis are reported on the attached page.

An Alfa Aesar cesium standard, Amersham ^{99}Tc standard, Isotope Products ^{129}I and ^{237}Np standards and an ^{127}I standard, prepared from Fisher potassium iodide (LOT# 35260) were used to generate the calibration curves. Independent standards of each element were used as the continuing calibration verification (CCV) standards.

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.

No known cesium 135, 137 standard was available. Results reported are direct cesium ratios using natural barium standards for mass bias corrections.

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

March 30, 2000

J.P. Brooks
3/31/00

Results are reported in µg analyte/g (ppm) of original sample.

Sample ID	Client ID	ICP/MS Number	Tc-99 µg/g ± 1 Stdev	†I-127 µg/g ± 1 Stdev	†I-129 µg/g ± 1 Stdev	Cs µg/g ± 1 Stdev
00-00903-PB	Process Blank	00323a7	<0.06	0.0234 ± 0.0010	<0.003	<0.2
00-00903-BS	Blank Spike	00323a8	<0.06	0.0189 ± 0.0010	<0.003	<0.2
00-00903	AZ102 M SUP	00323a9	12.2 ± 0.2	0.0656 ± 0.0023	0.011 ± 0.007	17.9 ± 0.5
00-00903-DUP	AZ102 M SUP	00323a10	11.8 ± 0.5	0.0550 ± 0.0005	0.019 ± 0.002	17.6 ± 1.1
00-00903-MS	AZ102 M SUP	00323a11	12.1 ± 0.4	0.0456 ± 0.0010	0.010 ± 0.001	17.2 ± 0.2
00-00903 + spike	AZ102 M SUP	00323a12	22.0 ± 1.1	0.0880 ± 0.0010	0.019 ± 0.002	44.9 ± 0.9
Spike Recovery						
			96%	81%	97%	94%
CCV and blank results are reported in ng/g (ppb)						
1%HNO3		00323a1	<0.03	1.2 ± 0.01	<0.08	<0.1
1%HNO3		00323a6	<0.03	0.5 ± 0.03	<0.08	<0.1
1%HNO3		00323a18	0.08 ± 0.01	<0.0001	<0.08	0.23 ± 0.01
5ppb Tc-99		00323a4	4.96 ± 0.03			
5ppb Tc-99		00323a19	5.13 ± 0.18			
20ppb Co		00323a20	0.06 ± 0.02			
0.5ppb I-127		00327a4		0.644 ± 0.013		
1ppb I-127		00327a17		0.910 ± 0.079		
0.1ppb I-129		00327a2			0.105 ± 0.012	
0.5ppb I-129		00327a13			0.461 ± 0.056	
14ppb Cs		00323a4				13.6 ± 0.4
14ppb Cs		00323a19				13.5 ± 0.1
5ppb Np-237		00324a4				
5ppb Np-237		00324a14				

*Results are from procedure 00324a.

†Results are from procedure 00327a.

DATA REVIEW

Reviewed by: *O.J. James*

Date: *3/31/00* Pages: *1 of 2*

K.P. Brooks Analysis

March 30, 2000

J.P. Brooks
3/31/00

Results are reported in μg analyte/g (ppm) of original sample.

Sample ID	Client ID	ICP/MS Number	Cs 135/137 $\pm$ 1 Stdev	%RSD	*Np-237 $\mu\text{g/g}$ $\pm$ 1 Stdev
00-00903-PB	Process Blank	00323a7	not detected		0.007 $\pm$ 0.002
00-00903-BS	Blank Spike	00323a8	not detected		0.011 $\pm$ 0.009
00-00903	AZ102 M SUP	00323a9	0.443 $\pm$ 0.01	1.1%	0.117 $\pm$ 0.004
00-00903-DUP	AZ102 M SUP	00323a10	0.438 $\pm$ 0.01	2.2%	0.118 $\pm$ 0.005
00-00903-MS	AZ102 M SUP	00323a11	0.442 $\pm$ 0	0.5%	0.133 $\pm$ 0.008
00-00903 + spike	AZ102 M SUP	00323a12			1.21 $\pm$ 0.03
Spike Recovery					102%
CCV and blank results are reported in ng/g (ppb)					
1%HNO3		00323a1			<0.02
1%HNO3		00323a6			0.08 $\pm$ 0.02
1%HNO3		00323a18			0.05 $\pm$ 0.02
5ppb Tc-99		00323a4			
5ppb Tc-99		00323a19			
20ppb Co		00323a20			
0.5ppb I-127		00327a4			
1ppb I-127		00327a17			
0.1ppb I-129		00327a2			
0.5ppb I-129		00327a13			
14ppb Cs		00323a4			
14ppb Cs		00323a19			
5ppb Np-237		00324a4			5.25 $\pm$ 0.14
5ppb Np-237		00324a14			5.18 $\pm$ 0.05

*Results are from procedure 00324a.

†Results are from procedure 00327a.

DATA REVIEW

Reviewed by: *D.J. Jarman*

Date: *3/14/00* Pages: *2 of 2*

Date March 31, 2000

To K.P.Books

From Tom Farmer *T.J. Farmer III 31mar00*Subject ICP/MS Analysis of Submitted Samples
(ALO#00-00902 PMG Fusions)329/4 File
LSO Project File
Mike Urie

Pursuant to your request, the 6 fusion samples that you submitted for analysis were analyzed by ICPMS for platinum. The results of this analysis are reported on the attached page.

An Alfa Aesar platinum standard was used to generate the calibration curve and an Independent CPI platinum standard was used as the continuing calibration verification (CCV) standard.

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

March 31, 2000

J. Brannon
3/31/00

Results are reported in μg analyte/ g of original sample.

Sample ID	Client ID	ICP/MS Number	Pt $\mu\text{g/g}$	± 1 Stdev
00-00902-PB-Zr	Process Blank	00329a7	<0.41	
00-00902-Zr	AZ102 MU	00329a9	<0.41	
00-00902-DUP-Zr	AZ102 MU	00329a10	<0.39	
00-00915-Zr	CUF-AZ102-023	00329a11	<0.40	
00-00916-Zr	CUF-AZ102-026	00329a12	<0.38	
00-00916 + spike	CUF-AZ102-026	00329a13	0.971 $\pm$	0.036
Spike Recovery			116%	

SRM 2710-Zr	LCS/00-00902/Zr	00329a8	<0.34
-------------	-----------------	---------	-------

CCV and blank results are reported in ng/g (ppb)

1%HNO3	00329a1	<0.034	
1%HNO3	00329a6	<0.030	
1%HNO3	00329a14	<0.030	
0.05ppb Pt	00329a4	0.0473	0.006
0.1ppb Pt	00329a15	0.101	0.005

DATA REVIEW

Reviewed by: *O.J. Janner*

Date: *31 Mar 00* Page: *1 of 1*

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

00-0902

3/7/2000

Client : Brooks

Cognizant Scientist:

JR Greenwood

Date : 3-7-00

Concur :

T Trang-le

Date : 3/8/00

Procedure: PNL-ALO-450

Equipment: Gamma detectors G

Measured Activities (uCi/g) with 1-sigma error

ALO ID Client ID	Co-60 Error %	Sb-125 Error %	Cs-134 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
00-0902 PB Process Blank	<3.E-2	<7.E-2	<3.E-2	6.10E-2 22%	<7.E-2	<7.E-2	<7.E-2
00-0902 AZ102MU	4.73E+0 4%	2.06E+1 8%	2.13E+0 11%	1.86E+3 2%	4.45E+1 2%	8.30E+1 3%	1.22E+2 5%
00-0902 DUP AZ102MU	4.68E+0 3%	2.76E+1 5%	1.82E+0 8%	1.73E+3 2%	4.48E+1 2%	8.37E+1 3%	1.30E+2 4%
RPD	1%	29%	16%	7%	1%	1%	6%
00-0903 PB Process Blank	<3.E-5	<7.E-5	<3.E-5	9.67E-4 3%	<7.E-5	<6.E-5	<6.E-5
00-0903 AZ102 M SUP	<6.E-3	<8.E-1	1.03E+0 2%	9.71E+2 2%	<3.E-2	<6.E-1	<5.E-1
00-0903 DUP AZ102 M SUP	<5.E-3	<6.E-1	1.06E+0 2%	9.83E+2 2%	<3.E-2	<4.E-1	<4.E-1
RPD			3%	1%			

3/10/00

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

Client : Brooks

Cognizant Scientist:

L R Greenwald

Date :

3/10/00

Concur :

T Trang-le

Date :

3/10/00

Procedure: PNL-ALO-450
Equipment: Gamma detectors G

Measured Activities (uCi/g) with 1-sigma error

ALO ID Client ID	Co-60 Error %	RuRh-106 Error %	Sb-125 Error %	Cs-134 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
00-0904 CUF-AZ-102-001	<5.E-4	<2.E-1	<8.E-2	8.77E-2 3%	8.35E+1 2%	<3.E-3	<5.E-2	<5.E-2
00-0905 CUF-AZ-102-003	<6.E-4	<2.E-1	<8.E-2	9.04E-2 3%	8.58E+1 2%	<4.E-3	<6.E-2	<5.E-2
00-0906 CUF-AZ102-015	<4.E-4	<1.E-1	<6.E-2	5.29E-2 3%	4.80E+1 2%	<3.E-3	<4.E-2	<4.E-2
00-0907 CUF-AZ-102-017	<5.E-4	<7.E-2	<5.E-2	3.05E-2 4%	2.91E+1 2%	<2.E-3	<3.E-2	<3.E-2
00-0908 CUF-AZ-102-019	<4.E-4	<6.E-2	<4.E-2	1.85E-2 4%	1.78E+1 2%	<2.E-3	<3.E-2	<3.E-2
00-0909 CUF-AZ-102-007	5.55E+0 4%	5.71E+0 25%	1.96E+1 6%	<6.E-1	5.46E+2 2%	5.71E+1 2%	1.05E+2 3%	1.56E+2 4%

Measured Activities (uCi/g) with 1-sigma error

ALO ID Client ID	Co-60 Error %	RuRh-106 Error %	Sb-125 Error %	Cs-134 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
00-0910 CUF-AZ-102-009	<5.E-4	<9.E-2	<6.E-2	4.36E-2 3%	4.26E+1 2%	<2.E-3	<4.E-2	<4.E-2
00-0911 CUF-AZ-102-010	3.34E+0 4%	<5.E0	1.67E+1 6%	<5.E-1	2.64E+2 2%	3.16E+1 2%	5.88E+1 3%	9.49E+1 5%
00-0912 CUF-AZ-102-011	3.86E+0 4%	6.40E+0 22%	2.86E+1 4%	<5.E-1	2.98E+2 2%	3.70E+1 2%	6.72E+1 3%	1.04E+2 5%
00-0913 CUF-AZ-102-013	<5.E-4	<6.E-2	<4.E-2	2.18E-2 4%	2.08E+1 2%	<2.E-3	<3.E-2	<3.E-2
00-0914 CUF-AZ-102-022	<6.E-4	<4.E-2	<3.E-2	9.95E-3 6%	9.09E+0 2%	<2.E-3	<2.E-2	<2.E-2
00-0915 CUF-AZ-102-023	7.38E+0 3%	<7.E0	5.33E+1 3%	<7.E-1	1.71E+2 2%	7.37E+1 2%	1.38E+2 3%	2.01E+2 4%
00-0916 CUF-AZ-102-026	7.41E+0 3%	1.18E+1 15%	2.73E+1 4%	<7.E-1	1.66E+2 2%	7.19E+1 2%	1.30E+2 3%	2.04E+2 4%

Client : Brooks

Cognizant Scientist:

LR Heenwood

Date :

3/23/00

Concur :

T Trang-le

Date :

3/23/00

Procedure: PNL-ALO-496, 417, 476

Measured Activities (uCi/g) with 1-sigma error

ALO ID Client ID	Sr-90 Error +/-	Pu-239+ Pu-240 Error +/-	Pu-238 Error +/-	Pu-236 Error +/-	Am-241 Error +/-	Cm-243+ Cm-244 Error +/-	Cm-242 Error +/-
00-0902 PB Process Blank	4.09E-1 29%	1.76E-3 40%	<2.E-3	<5.E-4	<3.E-3	<7.E-4	<5.E-4
00-0902 AZ102MU	1.66E+4 3%	6.60E+0 7%	6.40E-1 38%	<2.E-1	1.11E+2 5%	3.00E-1 28%	<4.E-2
00-0902 DUP AZ102MU	1.61E+4 3%	6.06E+0 7%	8.94E-1 21%	<5.E-2	1.15E+2 5%	<2.E-1	9.67E-2 50%
RPD	3%	9%	33%		4%		
00-0903 PB Process Blank	3.24E-3 4%	1.04E-5 15%	3.37E-5 9%	<5.E-7	2.64E-5 11%	3.46E-5 9%	<4.E-7
00-0903 AZ102 M SUP	1.83E+0 6%	2.65E-2 4%	2.90E-3 5%	2.90E-5 47%	2.01E-4 16%	2.46E-5 48%	<9.E-6
00-0903 DUP AZ102 M SUP	1.74E+0 6%	2.62E-2 4%	2.97E-3 5%	<2.E-5	1.64E-4 25%	<5.E-5	<4.E-5
RPD	5%	1%	2%		20%		
Matrix Spike	96%	104%			90%		
Spike	100%	107%			97%		
Blank	<3.E-3	<8.E-2	<3.E-1	<2.E-1	<1.E-1	<6.E-2	<6.E-2

Measured Activities (uCi/g) with 1-sigma error

ALO ID Client ID	Sr-90 Error +/-	Pu-239+ Pu-240 Error +/-	Pu-238 Error +/-	Pu-236 Error +/-	Am-241 Error +/-	Cm-243+ Cm-244 Error +/-	Cm-242 Error +/-
00-0909 CUF-AZ-102-007	2.17E+4 3%	7.63E+0 5%	1.21E+0 9%	<6.E-2	1.35E+2 5%	2.50E-1 23%	7.95E-2 41%
00-0910 CUF-AZ-102-009	1.36E-1 3%	9.77E-5 4%	5.05E-5 5%	<1.E-7	3.24E-5 6%	4.33E-5 6%	<1.E-7
00-0911 CUF-AZ-102-010	1.15E-4 3%	4.39E+0 7%	7.12E-1 15%	<5.E-2	9.00E+1 5%	<3.E-1	<7.E-2
00-0912 CUF-AZ-102-011	1.38E+4 3%	4.98E+0 6%	5.01E-1 19%	<3.E-2	9.32E+1 5%	2.42E-1 27%	<7.E-2
00-0913 CUF-AZ-102-013	3.42E-2 5%	1.67E-5 7%	4.45E-5 5%	<3.E-7	3.36E-5 6%	5.30E-5 6%	<2.E-7
00-0914 CUF-AZ-102-022	2.56E-2 6%	1.69E-5 7%	4.25E-5 5%	<2.E-7	1.32E-4 5%	4.91E-5 6%	<3.E-7
00-0915 CUF-AZ-102-023	2.53E+4 3%	9.80E+0 6%	1.03E+0 26%	<3.E-1	1.71E+2 5%	2.69E-1 38%	<6.E-2
00-0916 CUF-AZ-102-026	2.44E+4 3%	9.85E+0 6%	2.11E+0 13%	<3.E-1	1.79E+2 5%	2.93E-1 40%	<1.E-1
Matrix Spike	96%	104%			90%		
Spike	100%	107%			97%		
Blank	<3.E-3	<3.E-7	<7.E-7	<6.E-7	<3.E-7	<2.E-7	<2.E-7

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

3/23/00

Client : Brooks

Cognizant Scientist:

LR Greenwood

Date :

3/24/00

Concur :

T Trang-le

Date :

3/23/00

Procedure: PNL-ALO-476, 417, 496

Measured Activities (uCi/g) with 1-sigma error

ALO ID Client ID	Sr-90 Error +/-	Pu-239+ Pu-240 Error +/-	Pu-238 Error +/-	Pu-236 Error +/-	Am-241 Error +/-	Cm-243+ Cm-244 Error +/-	Cm-242 Error +/-
00-0904 CUF-AZ-102-001	1.22E-2 39%	6.31E-5 4%	4.25E-5 4%	<2.E-7	4.14E-5 6%	4.42E-5 6%	<2.E-7
00-0904 REP CUF-AZ-102-001	1.26E-2 38%	6.20E-5 5%	4.66E-5 5%	<4.E-7	4.08E-5 6%	4.74E-5 6%	<2.E-7
RPD	3%	2%	9%		1%	7%	
00-0905 CUF-AZ-102-003	3.65E-2 6%	4.68E-5 4%	4.95E-5 4%	<3.E-7	3.28E-5 7%	5.28E-5 6%	<2.E-7
00-0906 CUF-AZ102-015	3.26E-2 7%	3.53E-5 6%	5.15E-5 5%	<3.E-7	1.53E-4 5%	4.90E-5 6%	<2.E-7
00-0907 CUF-AZ-102-017	1.30E-2 8%	1.75E-5 8%	4.08E-5 6%	<1.E-6	3.41E-5 6%	4.81E-5 5%	<2.E-7
00-0908 CUF-AZ-102-019	1.85E-2 4%	2.10E-5 5%	5.61E-5 4%	<2.E-7	4.99E-5 6%	6.54E-5 6%	<2.E-7

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

00-0902

5/2/2000

Client : Brooks

Cognizant Scientist:

Dandra K. Fokun

Date :

5/2/00

Concur :

L.R. Greenwood

Date :

5/2/00

Procedure: PNL-ALO-432

Measured Activities (uCi/g) with 1-sigma error

<u>ALO ID</u> <u>Client ID</u>	<u>Tc-99</u> <u>Error +/-</u>
00-0903 PB Process Blank	<3.E-6
00-0903 AZ102 M SUP	1.25E-1 4%
00-0903 DUP AZ102 M SUP	1.33E-1 4%
RPD	6.2%
Standard	99%
Matrix Spike	73%
Blank	<3.E-6

Note: all QC parameters met specification per the QA plan in the PNNL standards-based management system except the matrix spike recovery. The Tc-99 spike was added as pertechnetate in sufficient quantity to be measured above the sample activity (two times the sample activity). The sample matrix may have been reducing causing some pertechnetate spike reduction. The procedure calls for the use of sodium dichromate to oxidize Tc species to Tc+7. However, for this matrix, this may not have been oxidizing enough. For a worst-case scenario, the sample Tc concentration may be biased low by about 25%.

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

00-0904

3/21/00

Client : Brooks

Cognizant Scientist:

JRGreenwood

Date :

3/21/00

Concur :

T Teang-le

Date :

3/21/00

Procedure: PNL-ALO-4014

Equipment: Chemcheck KPA11R

Measured Concentration (ug/g) with 1- σ error

ALO ID Client ID	Uranium Error %	Standards	Yield,%
00-0904	2.48E+1	Pre 1	104%
CUF-AZ-102-001	4%	Pre 2	101%
00-0904 Rep	2.36E+1	Post 1	98%
CUF-AZ-102-001	4%	Post 2	100%
00-0905	2.96E+1	Blank	<2.E-5
CUF-AZ-102-003	4%		
00-0906	4.01E+1		
CUF-AZ102-015	4%		
00-0907	1.92E+1		
CUF-AZ-102-017	4%		
00-0908	8.44E+0		
CUF-AZ-102-019	4%		
00-0909	2.87E+4		
CUF-AZ-102-007	4%		
00-0910	2.12E+1		
CUF-AZ-102-009	4%		
00-0911	1.74E+4		
CUF-AZ-102-010	4%		
00-0912	1.91E+4		
CUF-AZ-102-011	4%		
00-0913	1.81E+1		
CUF-AZ-102-013	4%		
00-0914	1.29E+1		
CUF-AZ-102-022	4%		
00-0915	3.63E+4		
CUF-AZ-102-023	4%		
00-0916	3.52E+4		
CUF-AZ-102-026	4%		

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

00-0902

3/21/00

Client : Brooks

Cognizant Scientist:

LR Green

Date :

3/21/00

Concur :

T Trang-le

Date :

3/21/00

Procedure: PNL-ALO-4014

Equipment: Chemcheck KPA11R

Measured Concentration (ug/g) with 1- σ error

<u>ALO ID</u> <u>Client ID</u>	<u>Uranium</u> <u>Error +/-</u>
00-0902 PB Process Blank	9.28E-1 3%
00-0902 AZ102MU	2.26E+4 4%
00-0902 DUP AZ102MU	2.31E+4 4%
RPD	2%
00-0903 PB Process Blank	1.65E-2 3%
00-0903 AZ102 M SUP	1.69E+1 3%
00-0903 DUP AZ102 M SUP	1.69E+1 3%
RPD	0%
Pre Standard 1	104%
Pre Standard 2	101%
Post Standard 1	98%
Post Standard 2	100%
Blank	<2.E-5

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

00-0902 H3
Rev. 1
7/10/2000

Client : Brooks

Cognizant Scientist:

Date : 7/10/00

Concur :

Date : 7/10/00

Measured Activity (uCi/g) with 1- σ error

<u>ALO ID</u> <u>Client ID</u>	<u>Tritium*</u> <u>Error +/-</u>
00-0902 PB Process Blank	1.87E-3 24%
00-0902 AZ102MU	3.92E-2 3%
00-0902 DUP AZ102MU	4.26E-2 3%
RPD	8%

*Tritium values are reported per gram of wet slurry.

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

00-0915 H3
4/13/2000

Client : Brooks

Cognizant Scientist: LR Greenwood Date: 4/13/00
Concur: T Trang-le Date: 4/13/00

Measured Activities (uCi/g) with 1-sigma error

<u>ALO ID</u> <u>Client ID</u>	<u>Tritium*</u> <u>Error %</u>
00-0915	9.04E-3
CUF-AZ-102-023	4%
00-0915 MS**	8.44E-3
CUF-AZ-102-023	4%
RPD	7%
00-0916	2.35E-2
CUF-AZ-102-026	3%
Blank	<4.E-4

*Tritium is reported per gram of wet slurry.

**The MS refers to an inorganic matrix spike; hence, this sample is a duplicate for tritium.

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

00-0902 C14

5/11/2000

Client : Brooks
Procedure PNL-ALO-482

Cognizant Scientist:

S. K. Fisk

Date :

5/11/00

Concur :

J. R. Greenwood

Date :

5/11/00

Measured Activity (uCi/g) with 1- σ error

<u>ALO ID</u> <u>Client ID</u>	<u>C-14</u> <u>Error +/-</u>
00-0902 AZ102MU	1.39E-3 10%
00-0902 DUP AZ102MU	1.62E-3 10%
RPD	15%
Pre Run System Blank	<3.E-4
Post Run System Blank	<3E-4
Blank Spike	106%
Sample Spike	106%

C-14 was measured on direct aliquots of the wet slurry. The weight percent solids data were used to correct the results for dried weight, although it should be noted that some of the C-14 would of course be lost if the samples had actually been dried prior to analysis. The C-14 activity in the sample is slightly below the requested MRQ limit of 1.8E-3 uCi/g.

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

00-0915 C14
5/11/2000

Client : Brooks

Cognizant Scientist:

S.K. Fisk

Date :

5/11/00

Concur :

J. Green

Date :

5/11/00

Measured Activities (uCi/g) with 1-sigma error

PNL-ALO-482

ALO ID
Client ID

C-14
Error %

00-0915
CUF-AZ-102-023

<1.2E-3

00-0916
CUF-AZ-102-026

2.36E-3
24%

Pre Run System Blank

<1.2E-3

Post Run System Blank

<1.2E-3

Blank Spike

106%

Sample Spike

106%

C-14 was measured on direct aliquots of the wet slurry. The weight percent solids data were used to correct the results for dried weight, although it should be noted that some of the C-14 would of course be lost if the samples had actually been dried prior to analysis. The C-14 activity in the sample is slightly below the requested MRQ limit of 1.8E-3 uCi/g.

Shielded Analytical Laboratory Bench Sheet

Client: KP BROOKS

WP Number: W45577

TI#/ASR: ASRs 5667/5668

Procedure: Bench Instruction

AZ102 DENSITIES

SAMPLE IDENTIFICATION

LAB NUMBER	SAMPLE IDENT	FLASK TARE WT (g)	FLASK GROSS WEIGHT (g)	FLASK SIZE (mL)	DENSITY (g/mL)
00-00903	AZ102 M SUP	9.3700	20.5161	10	1.1446
00-00904	CUF-AZ-102-001	9.3822	19.4575		1.0075
00-00905	CUF-AZ-102-003	9.4674	19.5298		1.0062
00-00906	CUF-AZ-102-015	9.3944	19.4528		1.0058
00-00907	CUF-AZ-102-017	9.3495	19.3605		1.0011
00-00908	CUF-AZ-102-019	9.4822	19.4344		0.9952
00-00910	CUF-AZ-102-009	9.4853	20.4000		1.0915
00-00913	CUF-AZ-102-013	9.4258	19.8241		1.0398
00-00914	CUF-AZ-102-022	9.3758	19.5814		1.0206
00-00917	CUF-AZ-102-027	10.6546	15.6959	15ml	1.0073

$$\text{DENSITY} = \frac{\text{FLASK GROSS (g)} - \text{FLASK TARE (g)}}{\text{FLASK SIZE (mL)}}$$

M&TE: ☒ Cell 2 (360-06-01-016) Mettler AE160 Balance Other _____

_____ Cell 5 (360-06-01-019) Sartorius LP4200S Balance

_____ Bench (510-06-01-014) Mettler AT201 Balance

_____ Bench (360-06-01-040) Denver A160 Balance

Analyst:

Date:

Reviewer:

Date:

[Signature] 2-18-00 [Signature] 2/18/00

SW

WT% SOLIDS DATA SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

CLIENT: Kriston Brooks WORK PACKAGE: W45577 ASR/ARF/LOI/TI: ASRs 5667/5668
QA PLAN: SBMS IMPACT LEVEL: PROCEDURE NUMBER: PNL-ALO-504

AZ102 MU AND CUF-AZ102-XXX SLURRIES
SAMPLE IDENTIFICATION

ACL NUMBER	CLIENT IDENTIFICATION	TARE WEIGHT (G)	(A) SAMPLE WET WEIGHT PLUS TARE	(B) SAMPLE DRY WEIGHT PLUS TARE	WEIGHT % SOLIDS
00-00902	AZ102 MU	16.5411	19.2798	17.6915	42.01
00-00902-DUP	AZ102 MU	16.3881	19.3956	17.6479	41.89
00-00902-TRI	AZ102 MU	16.3954	19.4756	17.6700	41.38
00-00909	CUF-AZ102-007	16.4055	17.5118	16.5316	11.40
00-00909-DUP	CUF-AZ102-007	16.3833	17.4581	16.5049	11.31
00-00909-TRI	CUF-AZ102-007	16.4583	17.5339	16.5797	11.29
00-00911	CUF-AZ102-010	16.3408	17.5356	16.5548	17.91
00-00911-DUP	CUF-AZ102-010	16.4402	17.6017	16.6479	17.88

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

DATE/TIME IN: 1-26-00 1500 OVEN TEMPERATURE: 107 °C

DATE/TIME OUT: 1-27-00 0815 OVEN TEMPERATURE: 106 °C

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

THERMOCOUPLE: 2116

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

Analyst:

Date:

Reviewer:

Date:

[Signature]

1-27-00

[Signature]

1-27-00

WT% SOLIDS DATA SHEET (325 SHIELDED ANALYTICAL LABORATORY)

CLIENT: Kriston Brooks WORK PACKAGE: W45577 ASR/ARF/LOI/TT: ASRs 5667/5668
QA PLAN: SBMS IMPACT LEVEL: PROCEDURE NUMBER: PNL-ALO-504

AZ102 MU AND CUF-AZ102-XXX SLURRIES SAMPLE IDENTIFICATION

ACL NUMBER	CLIENT IDENTIFICATION	TARE WEIGHT (G)	(A) SAMPLE WET WEIGHT PLUS TARE	(B) SAMPLE DRY WEIGHT PLUS TARE	WEIGHT % SOLIDS
00-00911-TRI	CUF-AZ102-010	16.4343	17.5938	16.6441	18.09
00-00912	CUF-AZ102-011 16.2588	16.5183	17.4627	16.4834	18.66
00-00912-DUP	CUF-AZ102-011	16.5183	17.6822	16.7292	18.12
00-00912-TRI	CUF-AZ102-011	16.3896	17.5627	16.6084	18.65
00-00915	CUF-AZ102-023	16.3902	17.5855	16.5154	10.47
00-00915-DUP	CUF-AZ102-023	16.3409	17.4874	16.4607	10.45
00-00915-TRI	CUF-AZ102-023	16.3474	17.4767	16.4650	10.41
00-00916	CUF-AZ102-026	16.4523	18.0280	16.6303	11.30

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

DATE/TIME IN: 1-26-00 1500 OVEN TEMPERATURE: 107 °C

DATE/TIME OUT: 1-27-00 0815 OVEN TEMPERATURE: 106 °C

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

THERMOCOUPLE: 2116

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

Analyst:

Date:

Reviewer:

Date:

[Signature]

1-27-00

[Signature]

1-27-00

02/11/00 FRI 15:09 FAX

003

WT% SOLIDS DATA SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

CLIENT: Kriston Brooks

WORK PACKAGE: W45577

ASR/ARF/LOI/TI: ASRs 5667/5668

QA PLAN: SBMS

IMPACT LEVEL: _____

PROCEDURE NUMBER: PNL-ALO-504

AZ102 MU AND CUF-AZ102-XXX SLURRIES
SAMPLE IDENTIFICATION

ACL NUMBER	CLIENT IDENTIFICATION	TARE WEIGHT (G)	(A) SAMPLE WET WEIGHT PLUS TARE	(B) SAMPLE DRY WEIGHT PLUS TARE	WEIGHT % SOLIDS
00-00916-DUP	CUF-AZ102-026	16.4017	17.8831	16.5596	10.66
00-00916-TRI	CUF-AZ102-026	16.2676	17.8006	16.4224	10.10

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

DATE/TIME IN: 1-26-00 1500 OVEN TEMPERATURE: 107 °C

DATE/TIME OUT: 1-27-00 0815 OVEN TEMPERATURE: 106 °C

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

THERMOCOUPLE: 2116

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

Analyst:

Date:

Reviewer:

Date:

[Signature]

1-27-00

[Signature]

1-27-00

Appendix D: Crossflow Filtration Raw Data

Appendix D: Crossflow Filtration Raw Data

AZ-102 Sludge Flux vs. Time
at 49.2 psig and 3.3 gpm
(Matrix 1, Condition 1)

Time (hr:min)	Filtrate Flux (gpm/ft ²)
0:00	0.180000
0:14	0.140000
0:28	0.130000
0:43	0.110000
0:57	0.110000
1:12	0.110000

AZ-102 Sludge Permeability vs. Time at
49.2 psig and 3.3 gpm
(Matrix 1, Condition 1)

Time (hr:min)	Permeability (gpm/ft ² /psi)
0:00	0.003800
0:14	0.003200
0:28	0.002800
0:43	0.002400
0:57	0.002400
1:12	0.002400

Condition Number	Time	Total Time Elapsed (Min)	Slurry	Filter	Filter Inlet Pressue (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)	Filtrate Flux (gpm/ft2)	Permeability (gpm/ft2/psi)
			Loop Flow Rate (gpm)	Outlet Pressure (psig)										
2	6:06	0:00	3	30	31	1	40	30.12	1.328	21.4	6.970	3.314	0.118815	0.003893
2	6:17	0:11	2.4	30	31	1	40	36.35	1.100	22.7	5.564	2.646	0.094843	0.003107
2	6:27	0:21	2.4	30	31	1	40	34.88	1.147	27.3	5.094	2.422	0.086834	0.002845
2	6:42	0:36	2.5	29	31	2	40	46.2	0.866	20.9	4.610	2.229	0.078588	0.002618
2	6:52	0:46	2.57	29	31	2	40	36.5	1.096	25	5.191	2.509	0.088487	0.002947
2	7:02	0:56	2.67	28	30	2	40	45.75	0.874	26	4.027	2.014	0.068643	0.002365
2	7:06	1:00	2.7	29	30	1	40	49.19	0.813	21.7	4.231	2.080	0.072126	0.002443
RAW			Average Pressure =			30	Average Flow =		1.032	Average Flux =		4.786		
	Average Slurry Flow =		2.61				Filtrate			With First Point Removed

RAW	Average Slurry Flow =	2.45	Average Pressure =	70.28571	Average Flow =	0.665	Average Flux =	2.696
					Filtrate		With First Point Removed

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)	Filtrate Flux (gpm/ft2)	Permeability (gpm/ft2/psi)
4	8:45	0:00	2.8	30	32	2	20	28.35	0.705	24.7	3.370	1.577	0.057446	0.001852
4	8:55	0:10	2.8	28	30	2	20	31.1	0.643	26.7	2.904	1.453	0.049512	0.001706
4	9:05	0:20	2.8	29	30	1	20	31.91	0.627	21.6	3.270	1.608	0.055752	0.001889
4	9:15	0:30	2.7	28	31	3	20	29.38	0.681	26.6	3.083	1.516	0.052557	0.001780
4	9:25	0:40	2.75	30	31	1	20	27.5	0.727	26.3	3.321	1.579	0.056622	0.001855
4	9:35	0:50	2.75	30	31	1	20	32.03	0.624	21.6	3.258	1.549	0.055544	0.001820
4	9:45	1:00	2.6	32	34	2	20	25.06	0.798	26.2	3.655	1.606	0.062309	0.001887

RAW Average Slurry Flow = 2.74 Average Pressure = 30.42857 Average Flow = 0.687 Average Flux = 3.249
 Filtrate Filtrate With First Point Removed

AZ-102 Sludge Flux vs. Time
at 30.4 psig and 2.7 gpm
(Matrix 1, Condition 4)

AZ-102 Sludge Permeability vs. Time at
30.4 psig and 2.7 gpm
(Matrix 1, Condition 4)

RAW	Average Slurry Flow =	Average Pressure =	51.42857	Average Flow =	0.922	Average Flux =	0.072
		2.95		Filtrate		With First Point Removed

RAW	Average Slurry Flow =	4.52	Average Pressure =	29.5	Average Flow =	0.919	Average Flux =	4.580
					Filtrate		With First Point Removed

RAW	Average Slurry Flow =	2.71	Average Pressure =	51.07143	Average Flow =	0.500	Average Flux =	1.857
					Filtrate		First point removed

Condition Number	Time	Total Time Elapsed (Min)	Slurry	Filter	Filter Inlet Pressue (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)	Filtrate Flux (gpm/ft2)	Permeability (gpm/ft2/psi)
			Loop Flow Rate (gpm)	Outlet Pressure (psig)										
2	1:15	0:00	2.2	30	31	1	30	54.49	0.551	26.6	2.493	1.186	0.042507	0.001393
2	1:26	0:11	2.18	28	30	2	30	84.17	0.356	22.8	1.797	0.899	0.030632	0.001056
2	1:36	0:21	2.5	27	30	3	20	57.28	0.349	22.6	1.770	0.901	0.030180	0.001058
2	1:45	0:30	2.1	28	30	2	20	55.35	0.361	26.9	1.623	0.812	0.027666	0.000953
2	1:56	0:41	2.18	28	30	2	20	64.28	0.311	22.1	1.600	0.800	0.027282	0.000940
2	2:06	0:51	2.3	27	30	3	20	62.45	0.320	22.6	1.624	0.826	0.027682	0.000971
2	2:16	1:01	2.4	27	30	3	20	57.41	0.348	27.2	1.552	0.790	0.026452	0.000927
RAW	Average Slurry Flow =		Average Pressure =		29	Average Flow =		0.371	Average Flux =		1.661	First point removed		
			2.27			Filtrate

RAW	Average Slurry Flow =	2.05	Average Pressure =	67.78571	Average Flow =	0.392	Average Flux =	1.733
					Filtrate		First point removed

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)	Filtrate Flux (gpm/ft2)	Permeability (gpm/ft2/psi)
4	4:14	0:00	2.7	30	30		0	20	32.66	27.4	2.712	1.311	0.046240	0.001540
4	4:25	0:11	2.9	28	30	2	20	20	46.8	23.5	2.112	1.056	0.036002	0.001241
4	4:35	0:21	2.9	29	32	3	20	20	42.18	25	2.246	1.068	0.038286	0.001254
4	4:46	0:32	3	30	32	2	20	20	40.88	27	2.191	1.025	0.037354	0.001204
4	4:58	0:44	3	28	32	4	20	20	48.06	22.4	2.122	1.026	0.036176	0.001205
4	5:11	0:57	3	28	32	4	20	20	40.38	28.5	2.128	1.029	0.036281	0.001209
4	5:15	1:01	2.9	30	33	3	20	20	35.37	26.2	2.590	1.192	0.044146	0.001400
RAW			Average Slurry Flow = 2.91			Average Pressure = 30.28571			Average Flow = 0.497		Average Flux = 2.231		First point removed

RAW	Average Slurry Flow =	3.09	Average Pressure =	47.83333	Average Flow =	0.300	Average Flux =	1.166
					Filtrate		First point removed

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)	Filtrate Flux (gpm/ft2)	Permeability (gpm/ft2/psi)
7	6:43	0:00	3.8	28	30		2	20	29.77	22	3.465	1.733	0.059076	0.002036
7	6:53	0:10	4	27	30		3	20	32.59	24.3	2.965	1.509	0.050540	0.001772
7	7:03	0:20	4.05	27	30		3	20	31.59	26.8	2.851	1.451	0.048609	0.001704
7	7:14	0:31	3.95	28	31		3	20	33.84	21.8	3.066	1.508	0.052271	0.001771
7	7:23	0:40	4	28	32		4	30	42.5	27.1	3.153	1.524	0.053746	0.001790
7	7:33	0:50	4.05	28	32		4	30	49.74	24.7	2.881	1.393	0.049113	0.001636
7	7:43	1:00	3.95	28	32		4	30	50.48	22.5	3.022	1.461	0.051515	0.001716

RAW Average Slurry Flow = 3.97 Average Pressure = 29.35714 Average Flow = 0.630 Average Flux = 2.990
 Filtrate First point removed

Appendix E: Physical Properties Test Instruction

Appendix E: Physical Properties Test Instruction

NP# w45559

PNNL Test Instruction		Document No.: BNFL-TP-29953-078 Rev. No.: 0
Title: BNFL Ultrafiltration Physical Testing: AZ-102		
Work Location: Radiochemical Processing Laboratory	Page 1 of 12	
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		1/17/00
Technical Reviewer		1/17/00

BNFL Ultrafiltration Physical Testing: AZ-102

Scope:

This test instruction defines work to be conducted on slurry samples collected during the ultrafiltration of materials from tanks 241-AZ-102. Under this test instruction, samples of the concentrated slurry (CUF-AZ102-006), caustic leached slurry (CUF-AZ102-012), and final slurry (CUF-AZ102-025) will be analyzed for select 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.

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:

rcy
rgz Balance 1:
Calib ID 388-06-01-020 Calib Exp Date 8/2000
Location 325/601/C cell 1-18-2000 100.0001 g = 100.001 g ± .002 g

Balance 2:
Calib ID _____ Calib Exp Date _____
Location _____

only one balance used
PAB
6/9/00

Signature Date 1-18-2000

Work Instructions

- 1) Thoroughly agitate samples CUF-AZ102-006, CUF-AZ102-012, and CUF-AZ102-025 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.

6A
Total 28.688 g
Tare 20.335 g
Slurry 8.553 g

6B
Total 28.439 g
Tare 20.118 g
Slurry 8.321 g

12A
Total 26.958 g
Tare 19.770 g
Slurry 7.188 g

12B
Total 26.952 g
Tare 19.834 g
Slurry 7.118 g

25A
Total 27.985 g
Tare 19.992 g
Slurry 7.993 g

25B
Total 25.901 g
Tare 19.156 g
Slurry 6.745 g

- 2) Agitate the cones and record the time and date.

Not really necessary (ry3)

Time 1:30 PM Date 1-19-2000

* Wash A
Total 27.530 g
14.2737 ⇒ TARE 19.269 g
Slurry 8.261 g

WASH - B
Total 27.263 g
TARE 19.015 g ← 19.0235
Slurry 8.188 g

* Slurry rinses with Associated Solids were transferred (on 1-17-2000 by D. Ruchant) to a 500mL Bottle labeled AZ102 CUF Rinse. ^{@ ~ 3pm}
Additive 1-18-2000 - excess liquid was discarded
1-19-2000 The Liquid layer was removed temporarily & Solids were Agitated prior to taking 2 ~ 8mL ^{duplicate} Aliquots Above (See Attached Addendum to this test plan)

Signature [Signature] Date 1-19-2000

ry3
1/19/2000

- 3) One hour after agitation, record the volume of the total sample and the volume of settled solids.

Date _____ Time _____

Initial Fill

8.1	6A	Total _____ ml	6B	Total _____ ml	8.15
		Solids _____ ml		Solids _____ ml	
		Liquid _____ ml		Liquid _____ ml	
8.6	12A	Total _____ ml	12B	Total _____ ml	6.4
		Solids _____ ml		Solids _____ ml	
		Liquid _____ ml		Liquid _____ ml	
7.7	25A	Total _____ ml	25B	Total _____ ml	7.5
		Solids _____ ml		Solids _____ ml	
		Liquid _____ ml		Liquid _____ ml	
8.1	WASH A	Total _____ ml	WASH B	Total _____ ml	8.05
		Solids _____ ml		Solids _____ ml	
		Liquids _____ ml		Liquid _____ ml	

Skip this in lite of the short Time diff
from here to end of shift (see next page)
rgs

Signature _____

Date _____

1/19/00

Note:

The following steps (4-9) are used to record data from the settling study. It is not necessary to record data in each step or at the specific time as stated, but data should be record as often as possible to document the stability of the final volume measurements in step 9.

- 4) Before the end of the shift (day 1), record the volume of the total sample and the volume of settled solids.

Date 1-19-2000

Time 15:30 hr.

6A
Total 8.1 ml
Solids 7.8 ml
Liquid 0.3 ml

6B
Total 8.1 ml
Solids 7.9 ml
Liquid 0.2 ml

12A
Total 6.4 ml
Solids 6.3 ml
Liquid 0.1 ml

12B
Total 6.4 ml
Solids 6.3 ml
Liquid 0.1 ml

25A
Total 7.9 ml
Solids 7.7 ml
Liquid 0.2 ml

25B
Total 7.5 ml
Solids 7.4 ml
Liquid 0.1 ml

WASH A
Total 8.0 ml
Solids 7.8 ml
Liquid 0.2 ml

WASH B
Total 7.8 ml
Solids 7.65 ml
Liquid 0.15 ml

Signature Date 1/19/00

- 5) At the start of the next day (day 2), record the volume of the total sample and the volume of settled solids.

Date 1-20-00

Time 8:20-12

6A
Total 8.1 ml
Solids 7.4 ml
Liquid 0.7 ml

6B
Total 8.0 ml
Solids 7.6 ml
Liquid 0.4 ml

	W-A	W-B
Total	<u>7.7</u>	<u>8.6</u>
Solids	<u>7.4</u>	<u>7.3</u>
Liquid	<u>0.3</u>	<u>0.3</u>

12A
Total 6.3 ml
Solids 6.1 ml
Liquid 0.2 ml

12B
Total 6.3 ml
Solids 6.1 ml
Liquid 0.2 ml

25A
Total 7.45 ml
Solids 7.25 ml
Liquid 0.25 ml

25B
Total 7.00 ml
Solids 6.80 ml
Liquid 0.20 ml

- 6) At the end of the day (day 2), record the volume of the total sample and the volume of settled solids.

Date 1-20-00

Time 15:00 hr

6A
Total 7.80 ml
Solids 7.35 ml
Liquid 0.45 ml

6B
Total 8.0 ml
Solids 7.6 ml
Liquid 0.4 ml

	W-A	W-B
Total	<u>7.70</u>	<u>7.60</u>
Solids	<u>7.25</u>	<u>7.10</u>
Liquid	<u>0.45</u>	<u>0.40</u>

12A
Total 6.3 ml
Solids 6.0 ml
Liquid 0.3 ml

12B
Total 6.3 ml
Solids 6.1 ml
Liquid 0.2 ml

25A
Total 7.45 ml
Solids 7.2 ml
Liquid 0.25 ml

25B
Total 7.0 ml
Solids 6.8 ml
Liquid 0.2 ml

Signature R. J. Bredt Date 1-20-00

- 7) At the start of the next day (day 3), record the volume of the total sample and the volume of settled solids.

Date 1-21-2000 Time 0800 hr
1500 hr

6A		6B	
Total	<u>7.7</u> ml	Total	<u>8.0</u> ml
Solids	<u>7.3</u> ml	Solids	<u>7.6</u> ml
Liquid	<u>0.4</u> ml	Liquid	<u>0.4</u> ml

12A		12B	
Total	<u>6.2</u> ml	Total	<u>6.2</u> ml
Solids	<u>6.0</u> ml	Solids	<u>6.0</u> ml
Liquid	<u>0.2</u> ml	Liquid	<u>0.2</u> ml

25A		25B	
Total	<u>7.50</u> ml	Total	<u>7.00</u> ml
Solids	<u>7.10</u> ml	Solids	<u>6.60</u> ml
Liquid	<u>0.4</u> ml	Liquid	<u>0.4</u> ml

- 8) At the end of the day (day 3), record the volume of the total sample and the volume of settled solids.

Date 1-21-2000 Time 1500

6A		6B		WA		WB	
Total	<u>7.7</u> ml	Total	<u>8.0</u> ml	<u>7.7</u>	Total	<u>7.6</u> ml	
Solids	<u>7.3</u> ml	Solids	<u>7.6</u> ml	<u>7.0</u>	Solids	<u>6.85</u> ml	
Liquid	<u>0.4</u> ml	Liquid	<u>0.4</u> ml	<u>0.7</u>	Liquids	<u>0.75</u> ml	

12A		12B	
Total	<u>6.2</u> ml	Total	<u>6.2</u> ml
Solids	<u>6.0</u> ml	Solids	<u>6.0</u> ml
Liquid	<u>0.2</u> ml	Liquid	<u>0.2</u> ml

25A		25B	
Total	<u>7.5</u> ml	Total	<u>7.0</u> ml
Solids	<u>7.1</u> ml	Solids	<u>6.6</u> ml
Liquid	<u>0.4</u> ml	Liquid	<u>0.4</u> ml

Signature Date 1-21-2000

PR Bredt
January 17, 2000

After weekend
Settling

Test Instruction: TI-29953-078
Page 8 of 12

- 9) At the beginning of the next day (day 4), record the volume of the total sample and the volume of settled solids.

Date 1/24/00

Time 10 AM

6A
Total 7.8 ml
Solids 7.3 ml
Liquid 0.5 ml

6B
Total 8.0 ml
Solids 7.65 ml
Liquid 0.35 ml

W-A
7.80 ml
6.85
.95

W-B
7.65
6.8
.85

12A
Total 6.2 ml
Solids 5.9 ml
Liquid 0.3 ml

12B
Total 6.15 ml
Solids 6.00 ml
Liquid 0.25 ml

25A
Total 7.50 ml
Solids 7.10 ml
Liquid 0.40 ml

25B
Total 7.0 ml
Solids 6.6 ml
Liquid 0.4 ml

- 10) 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.

6A
Total _____ g
Tare 25.488 g
Liquid _____ g
Liquid _____ ml

6B
Total _____ g
Tare 25.175 g
Liquid _____ g
Liquid _____ ml

W-A
Total _____ g
Tare 25.217 g
Liquid _____ g
Liquid _____ ml

12A
Total _____ g
Tare 25.625 g
Liquid _____ g
Liquid _____ ml

12B
Total _____ g
Tare 25.420 g
Liquid _____ g
Liquid _____ ml

25A
Total _____ g
Tare 25.931 g
Liquid _____ g
Liquid _____ ml

25B
Total _____ g
Tare 25.339 g
Liquid _____ g
Liquid _____ ml

Not done since
Less than 1 ml of
Visible Liquid is present in
all the sample < 0.5 ml
on 1st 6 samples

rg
1/24/00

Signature rg Bredt Date 1/24/00

Balance Check 200g = 199.995g
 100g = 99.997g

MT#E# 388-06-A-020

Loc. 325/60/C001

PR Bredt
 January 17, 2000

1/24/00

Test Instruction: TI-29953-078
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- 11) 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 1/24/00 @ 11:25
 Stopped @ 12:45

6A
 Total 28.839 g
 Tare 20.335 g
 Sample 8.504 g

Total 7.8 ml
 Solids 2.1 ml
 Liquid 5.7 ml

6B
 Total 28.432 g
 Tare 20.118 g
 Sample 8.314 g

Total 7.7 ml
 Solids 2.0 ml
 Liquid 5.7 ml

12A
 Total 26.944 g
 Tare 19.770 g
 Sample 7.174 g

Total 6.20 ml
 Solids 1.65 ml
 Liquid 4.55 ml

12B
 Total 26.916 g
 Tare 19.834 g
 Sample 7.082 g

Total 6.10 ml
 Solids 1.70 ml
 Liquid 4.40 ml

25A
 Total 27.973 g
 Tare 19.992 g
 Sample 7.981 g

Total 7.60 ml
 Solids 1.90 ml
 Liquid 5.70 ml

25B
 Total 25.892 g
 Tare 19.156 g
 Sample 6.736 g

Total 6.40 ml
 Solids 1.70 ml
 Liquid 4.70 ml

Start centrifuge @ 13:45 hr
 Stopped @ 14:50

W-A
 Total 27.519 g
 Tare 19.269 g
 Sample 8.250 g
 Total 7.75 ml
 Solids 2.1 ml
 Liquid 5.65 ml

W-B
 Total 27.188 g
 Tare 19.015 g
 Sample 8.173 g
 Total 7.65 ml
 Solids 2.1 ml
 Liquid 5.55 ml

Signature [Signature] Date 1/24/2000

- 12) 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.

6A
Liquid+Grad 31.128 g
Grad 25.488 g
Liquid 5.64 g
Liquid 5.55 ml

$$\rho = 1.0162 \text{ g/ml}$$

Solids+cone 23.171 g
Cone 20.335 g
Solids 2.836 g

6B
Liquid+Grad 30.705 g
Grad 25.175 g
Liquid 5.53 g
Liquid 5.46 ml

$$\rho = 1.0147 \text{ g/ml}$$

Solids+cone 22.815 g
Cone 20.118 g
Solids 2.757 g

12A
Liquid+Grad 30.536 g
Grad 25.625 g
Liquid 4.911 g
Liquid 4.35 ml

$$\rho = 1.129$$

Solids+cone 22.017 g
Cone 19.770 g
Solids 2.247 g

12B
Liquid+Grad 30.205 g
Grad 25.420 g
Liquid 4.785 g
Liquid 4.40 ml

$$\rho = 1.088$$

Solids+cone 22.108 g
Cone 19.834 g
Solids 2.274 g

25A
Liquid+Grad 28.271 g
Grad 25.931 g
Liquid 2.340 g
Liquid 2.250 ml

$$\rho = 1.040$$

Solids+cone 22.343 g
Cone 19.992 g
Solids 2.351 g

25B
Liquid+Grad 30.144 g
Grad 25.339 g
Liquid 4.805 g
Liquid 4.62 ml

$$\rho = 1.04$$

Solids+cone 21.151 g
Cone 19.156 g
Solids 1.995 g

W-A
Liquid+Grad 30.828 g
Grad 25.217 g
Liquid 5.611 g
Liquid 5.70 ml

$$\rho = 1.984$$

Solids+cone 21.894 g
Cone 19.269 g
Solids 2.625 g

W-B
Liquid+Grad 31.196 g
Grad 25.587 g
Liquid 5.609 g
Liquid 5.55 ml

$$\rho = 1.011$$

Solids+cone 21.568 g
Cone 19.015 g
Solids 2.553 g

Signature [Signature] Date 1-25-2000

Spilled on balance
when removing the
cover.
Lost entire
sample at
this point.

I double
checked
this value
173

BALANCE Check
MET # 388-06-01-020

100g = 99.998g

PR Bredt
January 17, 2000

LOC.. 325/601/Cell

Test Instruction: TI-29953-078
Page 11 of 12

- 13) Transfer the decanted supernatant to preweighed 20 ml vials with lids rated to 105°C. Weigh the loaded beakers.

6AL
Total 22.364 g
Tare 16.827 g
Supernatant 5.537 g

6BL
Total 22.267 g
Tare 16.882 g
Supernatant 5.385 g

W-A L
Total 22.483 g
Tare 16.982 g
Supernatant 5.501 g

12AL
Total 17.742 g
Tare 17.012 g
Supernatant 0.730 g

12BL
Total 21.678 g
Tare 17.031 g
Supernatant 4.647 g

W-B L
Total 22.522 g
Tare 17.015 g
Supernatant 5.507 g

25AL
Total 19.080 g
Tare 17.018 g
Supernatant 2.062 g

25BL
Total 16.872 g
Tare 16.872 g
Supernatant 0.000 g

LOST sample

- 14) 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.
- 15) Transfer the solids and liquids to an oven at 105°C for 24 hours.
- 16) Remove the solids and liquids from the oven and cap the vials. Allow the vial to cool ~1 hour and reweigh.

6AL
Total 16.908 g
Tare 16.827 g
sample .081 g

6BL
Total 16.966 g
Tare 16.882 g
sample .084 g

W-A L
Total 16.992 g
Tare 16.982 g
Sample .010 g

W-B L
Total 17.022 g
Tare 17.015 g
Sample .007 g

6A
Total 21.519 g
Tare 20.335 g
sample 1.184 g

6B
Total 21.266 g
Tare 20.118 g
sample 1.148 g

12AL
Total 17.084 g
Tare 17.012 g
sample .072 g

12BL
Total 17.544 g
Tare 17.031 g
sample .513 g

Signature Don E. Kuehn Date 2/2/00

PR Bredt
January 17, 2000

Test Instruction: TI-29953-078
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12A
Total 20.615 g
Tare 19.770 g
sample 0.845 g

12B
Total 20.680 g
Tare 19.834 g
sample 0.846 g

25AL
Total 17.053 g
Tare 17.018 g
sample 0.035 g

~~25BL~~
~~Total _____ g~~
~~Tare _____ g~~
~~sample Lost g~~
~~sample Sample g~~

25A
Total 20.758 g
Tare 19.992 g
sample 0.766 g

25B
Total 19.799 g
Tare 19.156 g
sample 0.643 g

W-A
total 20.134 g
tare 19.269 g
Sample 0.865 g

W-B
total 19.867 g
tare 19.015 g
Sample 0.852 g

Signature La E. Bredt Date 2/2/00

PNNL Test Plan Addendum

Document No.: BNFL-TP-29953-46
Rev. No.: 0
Document Control: Only the original
signed copy is controlled

Title: BNFL Ultrafiltration Physical Testing: AZ-102

Work Location: Radiochemical Processing
Laboratory

Page 1 of 3

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
- ☒ Project Manager
- ☐ Building Manager
- ☐ RPL Manager
- ☐ Radiological Control
- ☐ SFO Manager
- ☐ ES&H
- ☐ Quality Engineer

Are One-Time Modifications Allowed to this Procedure? ☒ 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

1/18/00

Technical Reviewer

1/18/00

BNFL Ultrafiltration Physical Testing: AZ-102

Scope

This addendum define the preparation of sample **AZ-102 CUF Rinse** for physical properties testing under test instruction 29953-078. Sample **AZ-102 CUF Rinse** was generated by rinsing of the CUF unit after the AZ-102 run. **AZ-102 CUF Rinse**, in a 500 ml glass jar, contains roughly 200 ml of solids. These solids need to be characterized prior to use in the HLW Vitrification task.

The liquid in **AZ-102 CUF Rinse** will be decanted. The density of this liquid will then be measured. The settled solids will then be mixed using a spatula and a 20 ml subsample of the settled solids will then be collected. The liquid will then be added back to the settled solids in the 500 ml jar. The 20 ml of settled solids will then be analyzed for solids content under Test Instruction 29953-078 in parallel with samples CUF-AZ102-006, CUF-AZ102-012, and CUF-AZ102-025.

Balance m# TE# 362-06-01-049

True wt 1000.01

meas wt 1000.00

OK 1/19/2000

Work Instructions

- 1) Label a clean 500 ml jar "**AZ-102 WASH SUP**". Record the mass of the empty jar.

AZ-102 WASH SUP

Tare 105.70 g

105.48

- 2) Record the mass of **AZ-102 CUF RINSE**.

● **AZ-102 CUF RINSE**

Bottle labeled)

(Slurry Rinse (First) 1-13-2000)

Total 811.20 g

Tare 354.756 g

Slurry 456.444 g

← Lorette has this #

from Kristen via
e-mail on 2/3/00
PRB

- 3) Decant as much supernatant as possible from **AZ-102 CUF Rinse** and transfer to **AZ-102 WASH SUP**. Record the new mass of **AZ-102 WASH SUP** and **AZ-102 CUF RINSE**.

AZ-102 WASH SUP

Total 323.119 g

Tare 105.70 g

Sup 217.419 g

AZ-102 CUF RINSE

Total 543.70 g

Tare 354.756 g

Solids 238.94 g

Signature

Date

1-19-2000

- 4) Transfer ~9ml of liquid from **AZ-102 WASH SUP** to a preweighed 10 ml graduated cylinder (or a 5 ml graduated cylinder may be used with ~4 ml of liquid). Record the mass and volume of liquid in the graduated cylinder. Discard this liquid after the mass and volume have been recorded.

Grad (25ml)

Total	<u>76.08</u>	g
Tare	<u>55.38</u>	g
Sup	<u>20.70</u>	g
Sup	<u>20.0</u>	ml

Density = 1.035 g/ml

- 5) Label a clean 20 ml vial **AZ-102 RIN**. Record the mass of the empty vial. *I labeled this as W-A*

^{WA}
AZ-102 RIN (WA)

Tare 17.033 g

- 6) Mix the solids in **AZ-102 CUF Rinse** with a spatula and transfer ~20 ml to **AZ-102 RIN**. Record the new mass of **AZ-102 RIN**. ^{WA} *WA and AZ-102 CUF Rinse*

After 20ml
17.920

^{WA}
AZ-102 RIN

Total	<u>34.414</u>	g
Tare	<u>17.033</u>	g
Solids	<u>17.385</u>	g

AZ-102 CUF Rinse

total	<u>575.53</u>	g
tare	<u>354.756</u>	g
solids	<u>220.774</u>	g

- 7) Transfer the liquid in **AZ-102 WAS SUP** back into **AZ-102 CUF RINSE**. Record the new mass of **AZ-102 CUF RINSE**.

AZ-102 CUF RINSE

Total	<u>791.56</u>	g
Tare	<u>354.756</u>	g
Slurry	<u>436.804</u>	g

- 8) Perform solids analysis on **AZ-102 CUF RINSE** as defined under Test Instruction 29953-078.

Signature *[Signature]*

Date 1-19-2000

BNFL Ultrafiltration Physical Testing: Feed Characterization

BNFL-TP-29953-078

AZ-102 - Filtered Slurry

01/18/2000 start 2/2/2000 finish

R.G. Swoboda

Step 1

Step 9

Step 10

Target initial volume graduation mark (8 or 9 mL)

Slurry, in 10 ml centrifuge cone														Vol% Settled Solid in cone after 5 days				Settled Superantant transferred to grad after 5 days	
Sample ID	desc.	Abbrev. ID	Mass, g	Volume, ml	Density	Average	RPD	volume, ml	Vol %	Average	RPD	Mass, g	Volume, ml						
CUF-AZ102-006-A	sam.	6-A	8.553	7.80	1.10	1.09	1.5%	7.30	93.6%	96.5%	6.0%	minimal liquid visible							
CUF-AZ102-006-B	dup.	6-B	8.321	7.70	1.08			7.65	99.4%			minimal liquid visible							
CUF-AZ102-012-A	sam.	12-A	7.188	6.20	1.16	1.16	0.6%	5.90	95.2%	96.8%	3.3%	minimal liquid visible							
CUF-AZ102-012-B	dup.	12-B	7.118	6.10	1.17			6.00	98.4%			minimal liquid visible							
CUF-AZ102-025-A	sam.	25-A	7.993	7.60	1.05	1.05	0.2%	7.10	93.4%	98.3%	9.9%	minimal liquid visible							
CUF-AZ102-025-B	dup.	25-B	6.745	6.40	1.05			6.60	103.1%			minimal liquid visible							
AZ102-Wash-A	sam.	W-A	8.261	7.75	1.07	1.07	0.4%	6.85	88.4%	88.6%	0.6%	minimal liquid visible							
AZ102-Wash-B	dup.	W-B	8.188	7.65	1.07			6.80	88.9%			minimal liquid visible							

after 105 deg C and 1hr cool										
step 12										
Sample	Tare	Wet Total	Sample	Dry Total	Sample	Wt Fraction Dry Solids	Water	Superanta	Wt% Solids Undissolved	Total
6-A	20.335	23.171	2.836	21.519	1.184	0.417	0.583	0.591	13.56%	14.8%
6-AL	16.827	22.364	5.537	16.908	0.081	0.015	0.985			
6-B	20.118	22.875	2.757	21.266	1.148	0.416	0.584	0.593	13.49%	14.8%
6-BL	16.882	22.267	5.385	16.966	0.084	0.016	0.984			
12-A	19.770	22.017	2.247	20.615	0.845	0.376	0.624	0.692	9.62%	12.8%
12-AL	17.012	17.742	0.730	17.084	0.072	0.099	0.901			
12-B	19.834	22.108	2.274	20.680	0.846	0.372	0.628	0.706	9.40%	19.1%
12-BL	17.031	21.678	4.647	17.544	0.513	0.110	0.890			
25-A	19.992	22.343	2.351	20.758	0.766	0.326	0.674	0.686	9.24%	10.0%
25AL	17.018	19.080	2.062	17.053	0.035	0.017	0.983			
25-B	16.188	21.181	1.993	16.799	0.623	0.322	0.678	0.670	10.43%	10.6%
25-BL	16.972	19.081	2.109	16.799	0.623	0.322	0.678	0.670	10.43%	10.6%
W-A	19.269	21.894	2.625	20.134	0.865	0.330	0.670	0.672	10.43%	10.6%
W-AL	16.982	22.483	5.501	16.992	0.010	0.002	0.998			
W-B	19.015	21.568	2.553	19.867	0.852	0.334	0.666	0.667	10.38%	10.5%
W-BL	17.015	22.522	5.507	17.022	0.007	0.001	0.999			

Table for Reporting

Sample ID	desc.	Abbrev. ID	Density, g/ml			Vol% Solids		Wt% Solids			Undissolved
			Slurry	Supernatant	Centrifuged Solids	Settled	Centrifuged	Settled	Centrifuged	Total	
CUF-AZ102-006-A	sam.	6-A	1.10	1.016	1.35	93.6%	26.9%	94.1%	33.2%	14.8%	13.6%
CUF-AZ102-006-B	dup.	6-B	1.08	1.015	1.38	99.4%	26.0%	95.7%	33.1%	14.8%	13.5%
CUF-AZ102-012-A	sam.	12-A	1.16	1.129	1.36	95.2%	26.6%	95.3%	31.3%	12.8%	9.6%
CUF-AZ102-012-B	dup.	12-B	1.17	1.088	1.34	98.4%	27.9%	96.2%	31.9%	19.1%	9.4%
CUF-AZ102-025-A	sam.	25-A	1.05	1.040	1.24	93.4%	25.0%	94.8%	29.4%	10.0%	9.2%
CUF-AZ102-025-B	dup.	25-B	1.05	1.040	1.17	103.1%	26.6%	93.8%	29.6%	9.5%	10.5%
AZ102-Wash-A	sam.	W-A	1.07	0.984	1.25	88.4%	27.1%	88.7%	31.8%	10.6%	10.4%
AZ102-Wash-B	dup.	W-B	1.07	1.011	1.22	88.9%	27.5%	89.5%	31.2%	10.5%	10.38%

Step 12

Step 9

Sample ID	Centrifuged Supernatant after 1 hour at 1000xg				Settled Solids after 5 days					
	Mass, g	Volume, ml	Density	Average RPD	Liquid, ml	Solids, g	wt% settle	Average RPD	Density	Average RPD
CUF-AZ102-006-A	5.640	5.55	1.016	1.015	0.2%	0.5	0.508108	8.044892	94.1%	1.07
CUF-AZ102-006-B	5.530	5.45	1.015	1.015	0.2%	0.35	0.355138	7.965862	95.7%	1.04
CUF-AZ102-012-A	4.911	4.35	1.129	1.108	3.7%	0.3	0.33869	6.84931	95.3%	1.15
CUF-AZ102-012-B	4.785	4.40	1.088	1.088	0.0%	0.25	0.271875	6.846125	96.2%	1.14
CUF-AZ102-025-A	2.340	2.25	1.040	1.040	0.0%	0.4	0.416	7.577	94.8%	1.01
CUF-AZ102-025-B	4.805	4.62	1.040	1.040	0.0%	0.4	0.416017	6.328983	93.8%	0.96
AZ102-Wash-A	5.811	5.70	0.984	0.998	2.6%	0.95	0.935167	7.325833	88.7%	1.07
AZ102-Wash-B	5.609	5.55	1.011	1.011	2.6%	0.85	0.859036	7.328964	89.5%	1.08

settled liquid volume in cone after 72 hours

Step 12

Step 11

Sample ID	Centrifuged Solids, after 1000xg for 1 hr				Settled Solids after 5 days					
	Mass, g	Volume, ml	Density	Average RPD	Liquid, ml	Solids, g	wt% settle	Average RPD	Density	Average RPD
CUF-AZ102-006-A	2.836	2.1	26.9%	26.4%	3.6%	0.1%	0.1%	1.35	1.36	2.1%
CUF-AZ102-006-B	2.757	2.0	26.0%	26.0%	3.1%	0.1%	0.1%	1.38	1.38	1.8%
CUF-AZ102-012-A	2.247	1.65	26.6%	27.2%	4.6%	0.1%	0.1%	1.36	1.35	1.8%
CUF-AZ102-012-B	2.274	1.7	27.9%	27.9%	31.9%	0.6%	0.6%	1.34	1.24	5.3%
CUF-AZ102-025-A	2.351	1.9	25.0%	25.8%	6.1%	0.1%	0.1%	1.24	1.21	5.3%
CUF-AZ102-025-B	1.995	1.7	26.6%	27.3%	1.3%	0.1%	0.1%	1.17	1.25	2.8%
AZ102-Wash-A	2.625	2.1	27.1%	27.3%	1.3%	0.1%	0.1%	1.25	1.23	2.8%
AZ102-Wash-B	2.553	2.1	27.5%	27.5%	31.2%	0.1%	0.1%	1.22	1.22	2.8%

Appendix F: Rheograms for AZ-102 and Standards

Appendix F: Rheograms for AZ-102 and Standards

Figure 1. 50 cP Brookfield Standard Lot 102298 Analyzed on 1/7/00 Using the In-Cell Remoted Haake M5

Figure 2. AZ-102 Initial Slurry (5.1 wt% Solids) Sample 1 Run 1. Suspect data shaded.

Figure 3. AZ-102 Initial Slurry (5.1 wt% Solids) Sample 1 Run 2

Figure 4. AZ-102 Initial Slurry (5.1 wt% solids) Sample 2 Run 1

Figure 5. AZ-102 Initial Slurry (5.1 wt% solids) Sample 2 Run 2

Figure 6. AZ-102 Initial Slurry (5.1 wt% Solids) Sample 2 Run 3

Figure 7. AZ-102 Concentrated Slurry (13.6 wt% Solids) Sample 1 Run 1

Figure 8. AZ-102 Concentrated Slurry (13.6 wt% Solids) Sample 1 Run 2

Figure 9. AZ-102 Concentrated Slurry (13.6 wt% Solids) Sample 2 Run 1

Figure 10. AZ-102 Concentrated Slurry (13.6 wt% Solids) Sample 2 Run 2

Figure 11. AZ-102 Concentrated Slurry (13.6 wt% Solids) Sample 2 Run 3

Figure 12. AZ-102 Concentrated Slurry (13.6 wt% Solids) Sample 2 Run 4

Figure 13. AZ-102 Final Slurry (9.2 wt% Solids) Sample 1 Run 1

Figure 14. AZ-102 Final Slurry (9.2 wt% Solids) Sample 2 Run 1

Figure 15. AZ-102 Final Slurry (9.2 wt% Solids) Sample 2 Run 2

Appendix G: Key Personnel Affiliated with AZ-102 Testing

Appendix G: Key Personnel Affiliated with AZ-102 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 Brecht	Rheology and Physical Properties Measurement	(509) 376-3777 paul.brecht@pnl.gov
Scott Cooley	Statistical Analysis	(509) 375-3604 scott.cooley@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 gr.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 Analysis

Duke Standards
301um #19136

Date: 05/18/89 Meas #: 00004
Time: 10:02 Pres #: 01

NIST Duke Standard
301um, Lot #19136

Summary

mv = 302.7
mn = 287.2
ma = 297.2
cs = 0.020
sd = 41.12

Percentiles

10% = 253.4 60% = 309.4
20% = 266.0 70% = 321.8
30% = 276.9 80% = 336.3
40% = 287.4 90% = 357.5
50% = 298.1 95% = 378.4

Dia Vol% Width
298.1 100% 82.25

Volume Distribution ; X-100 System

%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	1.11	6.541	0.00	0.00						
418.6	98.89	10.55	5.500	0.00	0.00						
352.0	88.34	40.22	4.625	0.00	0.00						
296.0	48.12	41.31	3.889	0.00	0.00						
248.9	6.81	6.81	3.270	0.00	0.00						
209.3	0.00	0.00	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						
12.08	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 Standards *616*

Date: 05/18/89

Meas #: 00004

~~20.0 um lot #19444~~ *301um*

Time: 10:02

Pres #: 01

NIST Duke Standard
20.0 um, Lot #19444
in 1.0 uM KCl solution

301um Lot # 19136

Summary

mv = 302.7
mn = 287.2
ma = 297.2
cs = 0.020
sd = 36.21

Percentiles

10% = 241.9 60% = 290.7
20% = 254.7 70% = 301.9
30% = 264.1 80% = 316.4
40% = 272.7 90% = 336.4
50% = 281.3 95% = 351.7

Dia Vol% Width

281.3 100% 72.41

Number Distribution; X-100 System

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	0.29	6.541	0.00	0.00						
418.6	99.71	4.62	5.500	0.00	0.00						
352.0	95.09	29.65	4.625	0.00	0.00						
296.0	65.44	51.23	3.889	0.00	0.00						
248.9	14.21	14.21	3.270	0.00	0.00						
209.3	0.00	0.00	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 Standards
20.0 um lot#19411

Date: 05/18/89 Meas #: 00008
Time: 10:23 Pres #: 01

NIST Duke Standard
20.0 um, Lot #19411
In 1.0 uM KCl solution

Summary

mv = 21.20
mn = 19.57
ma = 20.59
cs = 0.291
sd = 3.454

Percentiles

10% = 16.95 60% = 21.45
20% = 18.10 70% = 22.47
30% = 19.01 80% = 23.85
40% = 19.80 90% = 26.02
50% = 20.59 95% = 28.24

Dia Vol% Width
20.59 100% 6.907

Volume Distribution; X-100 System

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	0.47	0.578	0.00	0.00						
37.00	99.53	1.74	0.486	0.00	0.00						
31.11	97.79	7.25	0.409	0.00	0.00						
26.16	90.54	24.48	0.344	0.00	0.00						
22.00	66.06	42.16	0.289	0.00	0.00						
18.50	23.90	21.71	0.243	0.00	0.00						
15.56	2.19	2.19	0.204	0.00	0.00						
12.50	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 Standards
20.0 um lot#19411

Date: 05/18/89- Meas #: 00008
Time: 10:23 Pres #: 01

00 6125

NIST Duke Standard
20.0 um, Lot #19411
In 1.0 uM KCl solution

Summary

mv = 21.20
mn = 19.57
ma = 20.59
cs = 0.291
sd = 2.827

Percentiles

10% = 15.98 60% = 19.86
20% = 16.87 70% = 20.69
30% = 17.65 80% = 21.69
40% = 18.38 90% = 23.34
50% = 19.11 95% = 24.97

Dia	Vol%	Width
19.11	100%	5.654

Number Distribution - X-100 System

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	0.06	0.578	0.00	0.00						
37.00	99.94	0.36	0.486	0.00	0.00						
31.11	99.58	2.50	0.409	0.00	0.00						
26.16	97.08	14.21	0.344	0.00	0.00						
22.00	82.87	41.17	0.289	0.00	0.00						
18.50	41.70	35.66	0.243	0.00	0.00						
15.56	6.04	6.04	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 Standards
2.013 um lot#15992

Date: 05/18/89 Meas #: 00012
Time: 10:39 Pres #: 01

NIST Duke Standard
2.013 um, Lot #15992
In 1.0 uM KCl solution

Summary

mv = 1.793
mn = 1.662
ma = 1.745
cs = 3.438
sd = 0.290

Percentiles

10% = 1.440 60% = 1.826
20% = 1.533 70% = 1.908
30% = 1.612 80% = 2.017
40% = 1.684 90% = 2.199
50% = 1.753 95% = 2.368

Dia Vol% Width
1.753 100% 0.580

Volume Distribution ; X-100 System

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	1.07						
209.3	100.00	0.00	2.750	98.93	5.09						
176.0	100.00	0.00	2.312	93.84	19.71						
148.0	100.00	0.00	1.945	74.13	41.14						
124.5	100.00	0.00	1.635	32.99	28.33						
104.7	100.00	0.00	1.375	4.66	4.66						
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	0.00	0.486	0.00	0.00						
31.11	100.00	0.00	0.409	0.00	0.00						
26.16	100.00	0.00	0.344	0.00	0.00						
22.00	100.00	0.00	0.289	0.00	0.00						
18.50	100.00	0.00	0.243	0.00	0.00						
15.56	100.00	0.00	0.204	0.00	0.00						
13.00	100.00	0.00	0.172	0.00	0.00						
10.00	100.00	0.00	0.145	0.00	0.00						

Particle Size Analysis

Duke Standards
2.013 um lot#15992

Date: 05/18/89 Meas #: 00012
Time: 10:39 Pres #: 01

NIST Duke Standard
2.013 um, Lot #15992
In 1.0 uM KCl solution

Summary

mv = 1.793
mn = 1.662
ma = 1.745
cs = 3.438
sd = 0.243

Percentiles

10% = 1.359 60% = 1.686
20% = 1.438 70% = 1.761
30% = 1.499 80% = 1.850
40% = 1.558 90% = 1.982
50% = 1.619 95% = 2.112

Dia Vol% Width
1.619 100% 0.486

Number Distribution; X-100 System

- Size (microns) -

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	0.19						
209.3	100.00	0.00	2.750	99.81	1.55						
176.0	100.00	0.00	2.312	98.26	10.12						
148.0	100.00	0.00	1.945	88.14	35.55						
124.5	100.00	0.00	1.635	52.59	41.21						
104.7	100.00	0.00	1.375	11.38	11.38						
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	0.00	0.486	0.00	0.00						
31.11	100.00	0.00	0.409	0.00	0.00						
26.16	100.00	0.00	0.344	0.00	0.00						
22.00	100.00	0.00	0.289	0.00	0.00						
18.50	100.00	0.00	0.243	0.00	0.00						
15.56	100.00	0.00	0.204	0.00	0.00						
13.08	100.00	0.00	0.172	0.00	0.00						
11.00	100.00	0.00	0.145	0.00	0.00						

Particle Size Analysis

NIST Duke Standard
304 nm Lot#15928

Date: 05/19/89 Meas #: 00001
Time: 18:26 Pres #: 01

NIST Traceable Duke Standard
304 nm Lot #:15928
In 1.0 mM KCl Solution

Summary

Percentiles

Dia	Vol%	Width
-----	------	-------

```
mv = .3354
mn = .2445
ma = .3123
cs = 19.22
sd = .0717
```

10% = .2341	60% = .3591
20% = .2782	70% = .3773
30% = .3033	80% = .3983
40% = .3232	90% = .4279
50% = .3416	95% = .4540

.3416 100% .1435

PA-System
volume Distribution

[illegible]

Particle Size Analysis

NIST Duke Standard
96 nm Lot#16339

Date: 05/19/89 Meas #: 00002
Time: 18:33 Pres #: 01

NIST Traceable Duke Standard

196 nm Lot #:16339

In 1.0 mM KCl Solution

Volume Distribution; UPA System

Summary

$$m_V = .1015$$

mn = .0966

ma = .0999

CS = 60.06

sd = .0133

Percentiles

10% = .0853 60% = .1049

20% = .0902 70% = .1087

30% = .0940 80% = .1130

40% = .0977 90% = .1185

50% = .1012 95% = .1223

Dia	Vol%	Width
-----	------	-------

.1012	100%	.0266
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[illegible]

Particle Size Distribution Plots
For Initial Sludge: Sample CUF-AZ-102 MU R1D1
Replicate No: 1

40.

14.0
62.23

15.58	99.96	0.02	0.172	0.00	0.00				
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Particle Size Analysis

AZ-102 Waste
CUF-AZ102-MU-R2D1

Date: 05/19/89 Meas #: 00116
Time: 15:18 Pres #: 01

Blank - CUF-AZ102
Sample CUF-AZ102-MU-R2D1
In AZ-102-MU Simulated Supernatant
60ml/s

Summary

mv = 14.95
mn = 0.955
ma = 4.844
cs = 1.239
sd = 12.80

Percentiles

10% = 1.886 60% = 14.65
20% = 3.555 70% = 19.89
30% = 5.290 80% = 25.67
40% = 7.213 90% = 33.96
50% = 10.04 95% = 41.64

Dia

23.35
4.628

Vol%

48%
52%

Width

22.33
6.535

ume Distribution; X-100 System

%PASS

%CHAN

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	47.74	5.27						
592.0	100.00	0.00	7.778	42.47	5.71						
497.8	100.00	0.00	6.541	36.76	5.58						
418.6	100.00	0.00	5.500	31.18	4.97						
352.0	100.00	0.00	4.625	26.21	4.26						
296.0	100.00	0.00	3.889	21.95	3.62						
248.9	100.00	0.00	3.270	18.33	3.08						
209.3	100.00	0.00	2.750	15.25	2.61						
176.0	100.00	0.00	2.312	12.64	2.26						
148.0	100.00	0.00	1.945	10.38	2.07						
124.5	100.00	0.00	1.635	8.31	1.98						
104.7	100.00	0.00	1.375	6.33	1.84						
88.00	100.00	0.22	1.156	4.49	1.55						
74.00	99.78	0.57	0.972	2.94	1.14						
62.23	99.21	1.11	0.818	1.80	0.78						
52.33	98.10	2.12	0.688	1.02	0.53						
44.00	95.98	3.59	0.578	0.49	0.39						
37.00	92.39	5.21	0.486	0.10	0.10						
31.11	87.18	6.43	0.409	0.00	0.00						
26.16	80.75	6.89	0.344	0.00	0.00						
22.00	73.86	6.49	0.289	0.00	0.00						
18.50	67.37	5.62	0.243	0.00	0.00						
15.56	61.75	4.81	0.204	0.00	0.00						
	56.94	4.49	0.172	0.00	0.00						
	52.45	4.71	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-MU-R2D1

Date: 05/19/89 Meas #: 00116
Time: 15:18 Pres #: 01

005129

Blank CUF-AZ102
Sample CUF-AZ102-MU-R2D1
In AZ-102-MU Simulated Supernatant
60ml/s

Summary

mv = 14.95
mn = 0.955
ma = 4.844
cs = 1.239
sd = 0.369

Percentiles

10% = 0.494 60% = 0.838
20% = 0.540 70% = 0.966
30% = 0.588 80% = 1.151
40% = 0.654 90% = 1.524
50% = 0.736 95% = 2.059

Dia	Vol%	Width
0.736	100%	0.739

Number Distribution; X-100 System

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	99.92	0.07						
592.0	100.00	0.00	7.778	99.85	0.12						
497.8	100.00	0.00	6.541	99.73	0.20						
418.6	100.00	0.00	5.500	99.53	0.29						
352.0	100.00	0.00	4.625	99.24	0.42						
296.0	100.00	0.00	3.889	98.82	0.60						
248.9	100.00	0.00	3.270	98.22	0.86						
209.3	100.00	0.00	2.750	97.36	1.23						
176.0	100.00	0.00	2.312	96.13	1.79						
148.0	100.00	0.00	1.945	94.34	2.76						
124.5	100.00	0.00	1.635	91.58	4.44						
104.7	100.00	0.00	1.375	87.14	6.93						
88.00	100.00	0.00	1.156	80.21	9.83						
74.00	100.00	0.00	0.972	70.38	12.13						
62.23	100.00	0.00	0.818	58.25	13.96						
52.33	100.00	0.00	0.688	44.29	16.00						
44.00	100.00	0.00	0.578	28.29	19.78						
37.00	100.00	0.00	0.486	8.51	8.51						
31.11	100.00	0.00	0.409	0.00	0.00						
26.16	100.00	0.00	0.344	0.00	0.00						
22.00	100.00	0.01	0.289	0.00	0.00						
18.50	99.99	0.01	0.243	0.00	0.00						
15.56	99.98	0.01	0.204	0.00	0.00						
13.08	99.97	0.02	0.172	0.00	0.00						
11.00	99.95	0.03	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-MU-R2D1

Date: 05/19/89 Meas #: 00120
Time: 15:32 Pres #: 01

CUF-AZ102
Sample CUF-AZ102-MU-R2D1
In AZ-102-MU Simulated Supernatant
60ml/s Sonicate 40 watts 90 seconds

Summary

mv = 12.95
mn = 0.736
ma = 4.285
cs = 1.400
sd = 11.07

Percentiles

10% = 1.696 60% = 12.60
20% = 3.238 70% = 17.26
30% = 4.857 80% = 22.36
40% = 6.599 90% = 29.37
50% = 8.903 95% = 35.61

Dia Vol% Width
21.32 44% 18.57
4.545 56% 6.592

ume Distribution ; X-100 System

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	51.18	5.53						
592.0	100.00	0.00	7.778	45.65	5.95						
497.8	100.00	0.00	6.541	39.70	5.85						
418.6	100.00	0.00	5.500	33.85	5.26						
352.0	100.00	0.00	4.625	28.59	4.53						
296.0	100.00	0.00	3.889	24.06	3.86						
248.9	100.00	0.00	3.270	20.20	3.28						
209.3	100.00	0.00	2.750	16.92	2.77						
176.0	100.00	0.00	2.312	14.15	2.40						
148.0	100.00	0.00	1.945	11.75	2.20						
124.5	100.00	0.00	1.635	9.55	2.11						
104.7	100.00	0.00	1.375	7.44	1.99						
88.00	100.00	0.00	1.156	5.45	1.68						
74.00	100.00	0.00	0.972	3.77	1.24						
62.23	100.00	0.58	0.818	2.53	0.84						
52.33	99.42	1.25	0.688	1.69	0.58						
44.00	98.17	2.42	0.578	1.11	0.43						
37.00	95.75	4.02	0.486	0.68	0.36						
31.11	91.73	5.64	0.409	0.32	0.32						
26.16	86.09	6.73	0.344	0.00	0.00						
22.00	79.36	6.79	0.289	0.00	0.00						
18.50	72.57	6.16	0.243	0.00	0.00						
15.56	66.41	5.32	0.204	0.00	0.00						
13.00	61.09	4.89	0.172	0.00	0.00						
10.00	56.20	5.02	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-MU-R2D1

Date: 05/19/89 Meas #: 00120
Time: 15:32 Pres #: 01

CUF-AZ102
Sample CUF-AZ102-MU-R2D1
In AZ-102-MU Simulated Supernatant
60ml/s Sonicare 40 watts 90 seconds

Summary

mv = 12.95
mn = 0.736
ma = 4.285
cs = 1.400
sd = 0.321

Percentiles

10% = 0.371 60% = 0.619
20% = 0.393 70% = 0.746
30% = 0.422 80% = 0.928
40% = 0.467 90% = 1.246
50% = 0.529 95% = 1.648

Dia Vol% Width

0.529 100% 0.643

Number Distribution; X-100 System

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	99.95	0.04						
592.0	100.00	0.00	7.778	99.91	0.07						
497.8	100.00	0.00	6.541	99.84	0.12						
418.6	100.00	0.00	5.500	99.72	0.18						
352.0	100.00	0.00	4.625	99.54	0.26						
296.0	100.00	0.00	3.889	99.28	0.37						
248.9	100.00	0.00	3.270	98.91	0.52						
209.3	100.00	0.00	2.750	98.39	0.74						
176.0	100.00	0.00	2.312	97.65	1.08						
148.0	100.00	0.00	1.945	96.57	1.67						
124.5	100.00	0.00	1.635	94.90	2.69						
104.7	100.00	0.00	1.375	92.21	4.26						
88.00	100.00	0.00	1.156	87.95	6.06						
74.00	100.00	0.00	0.972	81.89	7.50						
62.23	100.00	0.00	0.818	74.39	8.55						
52.33	100.00	0.00	0.688	65.84	9.96						
44.00	100.00	0.00	0.578	55.88	12.40						
37.00	100.00	0.00	0.486	43.48	17.43						
31.11	100.00	0.00	0.409	26.05	26.05						
26.16	100.00	0.00	0.344	0.00	0.00						
22.00	100.00	0.00	0.289	0.00	0.00						
18.50	100.00	0.01	0.243	0.00	0.00						
15.56	99.99	0.01	0.204	0.00	0.00						
13.08	99.98	0.01	0.172	0.00	0.00						
11.00	99.97	0.02	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-MU-R2D1

Date: 05/19/89 Meas #: 00124
Time: 15:43 Pres #: 01

CUF-AZ102
Sample CUF-AZ102-MU-R2D1
In AZ-102-MU Simulated Supernatant
60ml/s 2nd Sonication 40 watts 90 seconds

Summary

mv = 12.37
mn = 0.598
ma = 3.944
cs = 1.521
sd = 10.68

Percentiles

10% = 1.574 60% = 11.87
20% = 3.032 70% = 16.47
30% = 4.590 80% = 21.46
40% = 6.261 90% = 28.20
50% = 8.416 95% = 34.19

Dia

Vol%

Width

20.89 42% 17.73
4.423 58% 6.578

Volume Distribution - X-100 System

%PASS

%CHAN

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	52.92	5.47						
592.0	100.00	0.00	7.778	47.45	5.94						
497.8	100.00	0.00	6.541	41.51	5.91						
418.6	100.00	0.00	5.500	35.60	5.38						
352.0	100.00	0.00	4.625	30.22	4.67						
296.0	100.00	0.00	3.889	25.55	4.00						
248.9	100.00	0.00	3.270	21.55	3.39						
209.3	100.00	0.00	2.750	18.16	2.87						
176.0	100.00	0.00	2.312	15.29	2.49						
148.0	100.00	0.00	1.945	12.80	2.30						
124.5	100.00	0.00	1.635	10.50	2.23						
104.7	100.00	0.00	1.375	8.27	2.11						
88.00	100.00	0.00	1.156	6.16	1.79						
74.00	100.00	0.00	0.972	4.37	1.32						
62.23	100.00	0.46	0.818	3.05	0.90						
52.33	99.54	1.02	0.688	2.15	0.61						
44.00	98.52	2.07	0.578	1.54	0.45						
37.00	96.45	3.62	0.486	1.09	0.37						
31.11	92.83	5.30	0.409	0.72	0.36						
26.16	87.53	6.54	0.344	0.36	0.36						
22.00	80.99	6.75	0.289	0.00	0.00						
18.50	74.24	6.17	0.243	0.00	0.00						
15.56	68.07	5.32	0.204	0.00	0.00						
8	62.75	4.86	0.172	0.00	0.00						
50	57.89	4.97	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-MU-R2D1

Date: 05/19/89 Meas #: 00124
Time: 15:43 Pres #: 01

CUF-AZ102
Sample CUF-AZ102-MU-R2D1
In AZ-102-MU Simulated Supernatant
60ml/s 2nd Sonication 40 watts 90 seconds

Summary

mv = 12.37
mn = 0.598
ma = 3.944
cs = 1.521
sd = 0.268

Percentiles

10% = 0.308 60% = 0.471
20% = 0.324 70% = 0.576
30% = 0.341 80% = 0.753
40% = 0.367 90% = 1.060
50% = 0.407 95% = 1.396

Dia Vol% Width
0.407 100% 0.535

Number Distribution ; X-100 System

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	99.97	0.02						
692.0	100.00	0.00	7.778	99.95	0.04						
497.8	100.00	0.00	6.541	99.91	0.08						
418.6	100.00	0.00	5.500	99.83	0.12						
352.0	100.00	0.00	4.625	99.71	0.17						
296.0	100.00	0.00	3.889	99.54	0.24						
248.9	100.00	0.00	3.270	99.30	0.35						
209.3	100.00	0.00	2.750	98.95	0.49						
176.0	100.00	0.00	2.312	98.46	0.72						
148.0	100.00	0.00	1.945	97.74	1.11						
124.5	100.00	0.00	1.635	96.63	1.82						
104.7	100.00	0.00	1.375	94.81	2.89						
88.00	100.00	0.00	1.156	91.92	4.13						
74.00	100.00	0.00	0.972	87.79	5.11						
62.23	100.00	0.00	0.818	82.68	5.86						
52.33	100.00	0.00	0.688	76.82	6.70						
44.00	100.00	0.00	0.578	70.12	8.30						
37.00	100.00	0.00	0.486	61.82	11.45						
31.11	100.00	0.00	0.409	50.37	18.75						
26.16	100.00	0.00	0.344	31.62	31.62						
22.00	100.00	0.00	0.289	0.00	0.00						
18.50	100.00	0.00	0.243	0.00	0.00						
15.56	100.00	0.01	0.204	0.00	0.00						
13.08	99.99	0.01	0.172	0.00	0.00						
11.00	99.98	0.01	0.145	0.00	0.00						

Particle Size Analysis		CUF AZ-102 Waste CUF-AZ102-MU-R1D#		Date: 05/20/89 Time: 11:11		Meas #: 00019 Pres #: 01	
CUF AZ-102 Waste UPA Sample: CUF-AZ102-MU-R1D# <i>Volume Distribution; UPA System</i>		Summary mv = 1.164 mn = 1.058 ma = 1.127 cs = 5.323 sd = .2054		Percentiles 10% = .9065 20% = .9846 30% = 1.042 40% = 1.095 50% = 1.147 60% = 1.202 70% = 1.262 80% = 1.334 90% = 1.440 95% = 1.539		Dia Vol% Width 1.147 100% .4109	

Sample: CUF-AZ102-MU-R1D

Volume Distribution; UPA System

CUF AZ-102 Waste
CUF-AZ102-MU-R1D

Date: 05/20/89 Meas #: 00019
Time: 11:11 Pres #: 01

Summary

Percentiles

Dia	Vol%	Width
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mv = 1.164	10% = .9065	60% = 1.202
mn = 1.058	20% = .9846	70% = 1.262
ma = 1.127	30% = 1.042	80% = 1.334
cs = 5.323	40% = 1.095	90% = 1.440
sd = .2054	50% = 1.147	95% = 1.539

10% = .9065	60% = 1.202
20% = .9846	70% = 1.262
30% = 1.042	80% = 1.334
40% = 1.095	90% = 1.440
50% = 1.147	95% = 1.539

1.147 100% .4109

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100

[illegible]

Particle Size Analysis		CUF AZ-102 Waste CUF-AZ102-MU-R1D1 SON		Date: 05/20/89 Time: 11:39	Meas #: 00027 Pres #: 01
CUF AZ-102 Waste UPA Sample: CUF-AZ102-MU-R1D1 Sonicated		Summary		Percentiles	
Volume Distribution; UPA System		mv = 2.219		10% = .4593	60% = 2.552
		mn = .2523		20% = 1.033	70% = 2.807
		ma = 1.156		30% = 1.717	80% = 3.119
		cs = 5.189		40% = 2.055	90% = 3.610
		sd = 1.278		50% = 2.314	95% = 4.094
		Dia	Vol%	Width	
		2.521	83%	1.668	
		.4167	17%	.4251	

Sample: CUF-AZ102-MU-R1D1 Sonicated

Volume Distribution; UPA System

Date: 05/20/89 Meas #: 00027
Time: 11:39 Pres #: 01

Dia	Vol%	Width
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mv = 2.219	10% = .4593	60% = 2.552
mn = .2523	20% = 1.033	70% = 2.807
ma = 1.156	30% = 1.717	80% = 3.119
cs = 5.189	40% = 2.055	90% = 3.610
sd = 1.278	50% = 2.314	95% = 4.094

2.521	83%	1.668
.4167	17%	.4251

[illegible]

00 67K55
100- Mass #

50% = .1920 95% = .4960

[illegible]

Particle Size Distribution Plots
For Initial Sludge: Sample CUF-AZ-102 MU R2D1
Replicate No: 2

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-MU-R2D1

Date: 05/19/89 Meas #: 00112
Time: 15:04 Pres #: 01

Blank - CUF-AZ102
Sample CUF-AZ102-MU-R2D1
In AZ-102-MU Simulated Supernatant
40ml/s

Summary	Percentiles	Dia	Vol%	Width
mv = 15.92	10% = 1.940	24.59	49%	24.88
mn = 1.019	20% = 3.614	4.604	51%	6.489
ma = 5.013	30% = 5.380			
cs = 1.197	40% = 7.383			
sd = 13.86	50% = 10.44			
	60% = 15.46			
	70% = 21.14			
	80% = 27.53			
	90% = 36.75			
	95% = 45.21			

ume Distribution; X-100 System

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	46.78	5.13						
592.0	100.00	0.00	7.778	41.65	5.55						
497.8	100.00	0.00	6.541	36.10	5.44						
418.6	100.00	0.00	5.500	30.66	4.87						
352.0	100.00	0.00	4.625	25.79	4.20						
296.0	100.00	0.00	3.889	21.59	3.60						
248.9	100.00	0.00	3.270	17.99	3.07						
209.3	100.00	0.00	2.750	14.92	2.62						
176.0	100.00	0.00	2.312	12.30	2.27						
148.0	100.00	0.00	1.945	10.03	2.08						
124.5	100.00	0.00	1.635	7.95	1.97						
104.7	100.00	0.00	1.375	5.98	1.82						
88.00	100.00	0.43	1.156	4.16	1.51						
74.00	99.57	0.80	0.972	2.65	1.09						
62.23	98.77	1.53	0.818	1.56	0.73						
52.33	97.24	2.75	0.688	0.83	0.48						
44.00	94.49	4.28	0.578	0.35	0.35						
37.00	90.21	5.67	0.486	0.00	0.00						
31.11	84.54	6.49	0.409	0.00	0.00						
26.16	78.05	6.59	0.344	0.00	0.00						
22.00	71.46	6.05	0.289	0.00	0.00						
18.50	65.41	5.23	0.243	0.00	0.00						
15.56	60.18	4.54	0.204	0.00	0.00						
13.00	55.64	4.30	0.172	0.00	0.00						
11.00	51.34	4.56	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-MU-R2D1

Date: 05/19/89 Meas #: 00112
Time: 15:04 Pres #: 01

Blank - CUF-AZ102
Sample CUF-AZ102-MU-R2D1
In AZ-102-MU Simulated Supernatant
40ml/s

Summary

mv = 15.92
mn = 1.019
ma = 5.013
cs = 1.197
sd = 0.389

Percentiles

10% = 0.534 60% = 0.897
20% = 0.575 70% = 1.029
30% = 0.633 80% = 1.222
40% = 0.708 90% = 1.620
50% = 0.793 95% = 2.197

Dia Vol% Width
0.793 100% 0.778

Number Distribution; X-100 System

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	99.91	0.07						
592.0	100.00	0.00	7.778	99.84	0.13						
497.8	100.00	0.00	6.541	99.71	0.22						
418.6	100.00	0.00	5.500	99.49	0.33						
352.0	100.00	0.00	4.625	99.16	0.48						
296.0	100.00	0.00	3.889	98.68	0.70						
248.9	100.00	0.00	3.270	97.98	1.00						
209.3	100.00	0.00	2.750	96.98	1.43						
176.0	100.00	0.00	2.312	95.55	2.08						
148.0	100.00	0.00	1.945	93.47	3.22						
124.5	100.00	0.00	1.635	90.25	5.13						
104.7	100.00	0.00	1.375	85.12	7.96						
88.00	100.00	0.00	1.156	77.17	11.11						
74.00	100.00	0.00	0.972	66.06	13.46						
62.23	100.00	0.00	0.818	52.60	15.16						
52.33	100.00	0.00	0.688	37.44	16.82						
44.00	100.00	0.00	0.578	20.62	20.62						
37.00	100.00	0.00	0.486	0.00	0.00						
31.11	100.00	0.00	0.409	0.00	0.00						
26.16	100.00	0.00	0.344	0.00	0.00						
22.00	100.00	0.01	0.289	0.00	0.00						
18.50	99.99	0.01	0.243	0.00	0.00						
15.56	99.98	0.01	0.204	0.00	0.00						
13.08	99.97	0.02	0.172	0.00	0.00						
11.00	99.95	0.04	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-MU-R2D1

Date: 05/19/89 Meas #: 00116
Time: 15:18 Pres #: 01

Blank CUF-AZ102
Sample CUF-AZ102-MU-R2D1
In AZ-102-MU Simulated Supernatant
60ml/s

Summary

mv = 14.95
mn = 0.955
ma = 4.844
cs = 1.239
sd = 12.80

Percentiles

10% = 1.886 60% = 14.65
20% = 3.555 70% = 19.89
30% = 5.290 80% = 25.67
40% = 7.213 90% = 33.96
50% = 10.04 95% = 41.64

Dia Vol% Width

23.35 48% 22.33
4.628 52% 6.535

Volume Distribution; X-100 System

%PASS

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	47.74	5.27						
592.0	100.00	0.00	7.778	42.47	5.71						
497.8	100.00	0.00	6.541	36.76	5.58						
418.6	100.00	0.00	5.500	31.18	4.97						
352.0	100.00	0.00	4.625	26.21	4.26						
296.0	100.00	0.00	3.889	21.95	3.62						
248.9	100.00	0.00	3.270	18.33	3.08						
209.3	100.00	0.00	2.750	15.25	2.61						
176.0	100.00	0.00	2.312	12.64	2.26						
148.0	100.00	0.00	1.945	10.38	2.07						
124.5	100.00	0.00	1.635	8.31	1.98						
104.7	100.00	0.00	1.375	6.33	1.84						
88.00	100.00	0.22	1.156	4.49	1.55						
74.00	99.78	0.57	0.972	2.94	1.14						
62.23	99.21	1.11	0.818	1.80	0.78						
52.33	98.10	2.12	0.688	1.02	0.53						
44.00	95.98	3.59	0.578	0.49	0.39						
37.00	92.39	5.21	0.486	0.10	0.10						
31.11	87.18	6.43	0.409	0.00	0.00						
26.16	80.75	6.89	0.344	0.00	0.00						
22.00	73.86	6.49	0.289	0.00	0.00						
18.50	67.37	5.62	0.243	0.00	0.00						
15.56	61.75	4.81	0.204	0.00	0.00						
13.00	56.94	4.49	0.172	0.00	0.00						
10.00	52.45	4.71	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-MU-R2D1

Date: 05/19/89 Meas #: 00116
Time: 15:18 Pres #: 01

Blank CUF-AZ102
Sample CUF-AZ102-MU-R2D1
In AZ-102-MU Simulated Supernatant
60ml/s

Summary

mv = 14.95
mn = 0.955
ma = 4.844
cs = 1.239
sd = 0.369

Percentiles

10% = 0.494 60% = 0.838
20% = 0.540 70% = 0.966
30% = 0.588 80% = 1.151
40% = 0.654 90% = 1.524
50% = 0.736 95% = 2.059

Dia Vol% Width
0.736 100% 0.739

Number Distribution; X-100 System

%PASS

100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0

%CHAN

20.0
18.0
16.0
14.0
12.0
10.0
8.0
6.0
4.0
2.0
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	99.92	0.07						
592.0	100.00	0.00	7.778	99.85	0.12						
497.8	100.00	0.00	6.541	99.73	0.20						
418.6	100.00	0.00	5.500	99.53	0.29						
352.0	100.00	0.00	4.625	99.24	0.42						
296.0	100.00	0.00	3.889	98.82	0.60						
248.9	100.00	0.00	3.270	98.22	0.86						
209.3	100.00	0.00	2.750	97.36	1.23						
176.0	100.00	0.00	2.312	96.13	1.79						
148.0	100.00	0.00	1.945	94.34	2.76						
124.5	100.00	0.00	1.635	91.58	4.44						
104.7	100.00	0.00	1.375	87.14	6.93						
88.00	100.00	0.00	1.156	80.21	9.83						
74.00	100.00	0.00	0.972	70.38	12.13						
62.23	100.00	0.00	0.818	58.25	13.96						
52.33	100.00	0.00	0.688	44.29	16.00						
44.00	100.00	0.00	0.578	28.29	19.78						
37.00	100.00	0.00	0.486	8.51	8.51						
31.11	100.00	0.00	0.409	0.00	0.00						
26.16	100.00	0.00	0.344	0.00	0.00						
22.00	100.00	0.01	0.289	0.00	0.00						
18.50	99.99	0.01	0.243	0.00	0.00						
15.56	99.98	0.01	0.204	0.00	0.00						
13.08	99.97	0.02	0.172	0.00	0.00						
11.00	99.95	0.03	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-MU-R2D1

Date: 05/19/89 Meas #: 00120
Time: 15:32 Pres #: 01

CUF-AZ102
Sample CUF-AZ102-MU-R2D1
In AZ-102-MU Simulated Supernatant
60ml/s Sonicate 40 watts 90 seconds

Summary	Percentiles		Dia	Vol%	Width
mv = 12.95	10% = 1.696	60% = 12.60	21.32	44%	18.57
mn = 0.736	20% = 3.238	70% = 17.26	4.545	56%	6.592
ma = 4.285	30% = 4.857	80% = 22.36			
cs = 1.400	40% = 6.599	90% = 29.37			
sd = 11.07	50% = 8.903	95% = 35.61			

ume Distribution; X-100 System

%PASS

- Size (microns) -

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	51.18	5.63						
592.0	100.00	0.00	7.778	45.65	5.95						
497.8	100.00	0.00	6.541	39.70	5.85						
418.6	100.00	0.00	5.500	33.85	5.26						
352.0	100.00	0.00	4.625	28.59	4.53						
296.0	100.00	0.00	3.889	24.06	3.86						
248.9	100.00	0.00	3.270	20.20	3.28						
209.3	100.00	0.00	2.750	16.92	2.77						
176.0	100.00	0.00	2.312	14.15	2.40						
148.0	100.00	0.00	1.945	11.75	2.20						
124.5	100.00	0.00	1.635	9.55	2.11						
104.7	100.00	0.00	1.375	7.44	1.99						
88.00	100.00	0.00	1.156	5.45	1.68						
74.00	100.00	0.00	0.972	3.77	1.24						
62.23	100.00	0.58	0.818	2.53	0.84						
52.33	99.42	1.25	0.688	1.69	0.58						
44.00	98.17	2.42	0.578	1.11	0.43						
37.00	95.75	4.02	0.486	0.68	0.36						
31.11	91.73	5.64	0.409	0.32	0.32						
26.16	86.09	6.73	0.344	0.00	0.00						
22.00	79.36	6.79	0.289	0.00	0.00						
18.50	72.57	6.16	0.243	0.00	0.00						
15.56	66.41	5.32	0.204	0.00	0.00						
13.00	61.09	4.89	0.172	0.00	0.00						
10.00	56.20	5.02	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-MU-R2D1

Date: 05/19/89 Meas #: 00120
Time: 15:32 Pres #: 01

CUF-AZ102
Sample CUF-AZ102-MU-R2D1
In AZ-102-MU Simulated Supernatant
60ml/s Sonicare 40 watts 90 seconds

Number Distribution; X-100 System

Summary

mv = 12.95
mn = 0.736
ma = 4.285
cs = 1.400
sd = 0.321

Percentiles

10% = 0.371 60% = 0.619
20% = 0.393 70% = 0.746
30% = 0.422 80% = 0.928
40% = 0.467 90% = 1.246
50% = 0.529 95% = 1.648

Dia	Vol%	Width
0.529	100%	0.643

%PASS

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	99.95	0.04						
592.0	100.00	0.00	7.778	99.91	0.07						
497.8	100.00	0.00	6.541	99.84	0.12						
418.6	100.00	0.00	5.600	99.72	0.18						
352.0	100.00	0.00	4.625	99.54	0.26						
296.0	100.00	0.00	3.889	99.28	0.37						
248.9	100.00	0.00	3.270	98.91	0.52						
209.3	100.00	0.00	2.750	98.39	0.74						
176.0	100.00	0.00	2.312	97.65	1.08						
148.0	100.00	0.00	1.945	96.57	1.67						
124.5	100.00	0.00	1.635	94.90	2.69						
104.7	100.00	0.00	1.375	92.21	4.26						
88.00	100.00	0.00	1.156	87.95	6.06						
74.00	100.00	0.00	0.972	81.89	7.50						
62.23	100.00	0.00	0.818	74.39	8.55						
52.33	100.00	0.00	0.688	65.84	9.96						
44.00	100.00	0.00	0.578	55.88	12.40						
37.00	100.00	0.00	0.486	43.48	17.43						
31.11	100.00	0.00	0.409	26.05	26.05						
26.16	100.00	0.00	0.344	0.00	0.00						
22.00	100.00	0.00	0.289	0.00	0.00						
18.50	100.00	0.01	0.243	0.00	0.00						
15.56	99.99	0.01	0.204	0.00	0.00						
13.08	99.98	0.01	0.172	0.00	0.00						
11.00	99.97	0.02	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-MU-R2D1

Date: 05/19/89 Meas #: 00124
Time: 15:43 Pres #: 01

CUF-AZ102
Sample CUF-AZ102-MU-R2D1
In AZ-102-MU Simulated Supernatant
60ml/s 2nd Sonication 40 watts 90 seconds

Summary	Percentiles	Dia	Vol%	Width
mv = 12.37	10% = 1.574	20.89	42%	17.73
mn = 0.598	20% = 3.032	4.423	58%	6.578
ma = 3.944	30% = 4.590			
cs = 1.521	40% = 6.261			
sd = 10.68	50% = 8.416			
	60% = 11.87			
	70% = 16.47			
	80% = 21.46			
	90% = 28.20			
	95% = 34.19			

Volume Distribution; X-100 System

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	52.92	5.47						
592.0	100.00	0.00	7.778	47.45	5.94						
497.8	100.00	0.00	6.541	41.51	5.91						
418.6	100.00	0.00	5.500	35.60	5.38						
352.0	100.00	0.00	4.625	30.22	4.67						
296.0	100.00	0.00	3.889	25.55	4.00						
248.9	100.00	0.00	3.270	21.55	3.39						
209.3	100.00	0.00	2.750	18.16	2.87						
176.0	100.00	0.00	2.312	15.29	2.49						
148.0	100.00	0.00	1.945	12.80	2.30						
124.5	100.00	0.00	1.635	10.50	2.23						
104.7	100.00	0.00	1.375	8.27	2.11						
88.00	100.00	0.00	1.156	6.16	1.79						
74.00	100.00	0.00	0.972	4.37	1.32						
62.23	100.00	0.46	0.818	3.05	0.90						
52.33	99.54	1.02	0.688	2.15	0.61						
44.00	98.52	2.07	0.578	1.54	0.45						
37.00	96.45	3.62	0.486	1.09	0.37						
31.11	92.83	5.30	0.409	0.72	0.36						
26.16	87.53	6.54	0.344	0.36	0.36						
22.00	80.99	6.75	0.289	0.00	0.00						
18.50	74.24	6.17	0.243	0.00	0.00						
15.56	68.07	5.32	0.204	0.00	0.00						
13.00	62.75	4.86	0.172	0.00	0.00						
11.00	57.89	4.97	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-MU-R2D1

Date: 05/19/99 Meas #: 00124
Time: 15:43 Pres #: 01

CUF-AZ102
Sample CUF-AZ102-MU-R2D1
In AZ-102-MU Simulated Supernatant
60ml/s 2nd Sonication 40 watts 90 seconds

Summary

mv = 12.37
mn = 0.598
ma = 3.944
cs = 1.521
sd = 0.268

Percentiles

10% = 0.308 60% = 0.471
20% = 0.324 70% = 0.576
30% = 0.341 80% = 0.753
40% = 0.367 90% = 1.060
50% = 0.407 95% = 1.396

Dia Vol% Width
0.407 100% 0.535

Number Distribution ; X-100 System

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	99.97	0.02						
592.0	100.00	0.00	7.778	99.95	0.04						
497.8	100.00	0.00	6.541	99.91	0.08						
418.6	100.00	0.00	5.500	99.83	0.12						
352.0	100.00	0.00	4.625	99.71	0.17						
296.0	100.00	0.00	3.889	99.54	0.24						
248.9	100.00	0.00	3.270	99.30	0.35						
209.3	100.00	0.00	2.750	98.95	0.49						
176.0	100.00	0.00	2.312	98.46	0.72						
148.0	100.00	0.00	1.945	97.74	1.11						
124.5	100.00	0.00	1.635	96.63	1.82						
104.7	100.00	0.00	1.375	94.81	2.89						
88.00	100.00	0.00	1.156	91.92	4.13						
74.00	100.00	0.00	0.972	87.79	5.11						
62.23	100.00	0.00	0.818	82.68	5.86						
52.33	100.00	0.00	0.688	76.82	6.70						
44.00	100.00	0.00	0.578	70.12	8.30						
37.00	100.00	0.00	0.486	61.82	11.45						
31.11	100.00	0.00	0.409	50.37	18.75						
26.16	100.00	0.00	0.344	31.62	31.62						
22.00	100.00	0.00	0.289	0.00	0.00						
18.50	100.00	0.00	0.243	0.00	0.00						
15.56	100.00	0.01	0.204	0.00	0.00						
13.08	99.99	0.01	0.172	0.00	0.00						
11.00	99.98	0.01	0.145	0.00	0.00						

Particle Size Analysis

CUF AZ-102 Waste
CUF-AZ102-MU-R2D1

Date: 05/20/89 Meas #: 00021
Time: 11:18 Pres #: 01

CUF AZ-102 Waste

UPA

Sample: CUF-AZ102-MU-R2D1

Volume Distribution; UPA System

Summary

Percentiles

```
mv = 1.532
mn = 1.281
ma = 1.409
cs = 4.259
sd = .3006
```

10% = 1.093	60% = 1.496
20% = 1.195	70% = 1.583
30% = 1.274	80% = 1.697
40% = 1.347	90% = 1.895
50% = 1.420	95% = 2.119

<u>Dia</u>	<u>Vol%</u>	<u>Width</u>
5.537	2%	1.133
1.412	98%	.5745

[illegible]

Particle Size Analysis

CUF AZ-102 Waste
CUF-AZ102-MU-R2D1SON

Date: 05/20/89 Meas #: 00027
Time: 11:39 Pres #: 01

CUF AZ-102 Waste

UPA

Sample: CUF-AZ102-MU-R1D2 Sonicated

R2D1

lume Distribution; ZPA System

Summary

mv = 2.219
mn = .2523
ma = 1.156
cs = 5.189
sd = 1.278

Percentiles

10% = .4593	60% = 2.552
20% = 1.033	70% = 2.807
30% = 1.717	80% = 3.119
40% = 2.055	90% = 3.610
50% = 2.314	95% = 4.094

[illegible]

2.521	83%	1.668
.4167	17%	.4251

[illegible]

Particle Size Distribution Plots
For Concentrated Slurry: Sample CUF-AZ-102-005
Replicate No: 1

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-05

Date: 05/19/89 Meas #: 00152
Time: 17:28 Pres #: 01

CUF AZ-102 Waste
Sample: CUF- AZ102-05
In AZ-102-05 Simulated Supernatant
40ml/s,

Summary

mv = 2.820
mn = 0.326
ma = 1.076
cs = 5.575
sd = 2.053

Percentiles

10% = 0.476 60% = 1.789
20% = 0.855 70% = 2.556
30% = 1.059 80% = 3.984
40% = 1.235 90% = 6.826
50% = 1.444 95% = 10.23

Dia	Vol%	Width
1.561	92%	4.267
0.287	8%	0.147

Volume Distribution; X-100 System

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	93.96	2.10						
592.0	100.00	0.00	7.778	91.86	2.52						
497.8	100.00	0.00	6.541	89.34	2.94						
418.6	100.00	0.00	5.500	86.40	3.30						
352.0	100.00	0.00	4.625	83.10	3.61						
296.0	100.00	0.00	3.889	79.49	3.83						
248.9	100.00	0.00	3.270	75.66	3.95						
209.3	100.00	0.00	2.750	71.71	4.13						
176.0	100.00	0.00	2.312	67.58	4.77						
148.0	100.00	0.00	1.945	62.81	6.44						
124.5	100.00	0.00	1.635	56.37	9.27						
104.7	100.00	0.00	1.375	47.10	11.52						
88.00	100.00	0.00	1.156	35.58	10.31						
74.00	100.00	0.00	0.972	25.27	6.69						
62.23	100.00	0.00	0.818	18.58	3.86						
52.33	100.00	0.00	0.688	14.72	2.49						
44.00	100.00	0.00	0.578	12.23	2.00						
37.00	100.00	0.00	0.486	10.23	1.91						
31.11	100.00	0.00	0.409	8.32	1.99						
26.16	100.00	0.38	0.344	6.33	2.08						
22.00	99.62	0.59	0.289	4.25	1.94						
18.50	99.03	0.84	0.243	2.31	1.43						
15.56	98.19	1.11	0.204	0.88	0.88						
13.08	97.08	1.40	0.172	0.00	0.00						
11.00	95.68	1.72	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-05

Date: 05/19/89 Meas #: 00152
Time: 17:28 Pres #: 01

CUF AZ-102 Waste
Sample: CUF-AZ102-05
In AZ-102-05 Simulated Supernatant
100-1/s,

Summary

mv = 2.820
mn = 0.326
ma = 1.076
cs = 5.575
sd = 0.100

Percentiles

10% = 0.187 60% = 0.271
20% = 0.199 70% = 0.302
30% = 0.212 80% = 0.356
40% = 0.229 90% = 0.520
50% = 0.248 95% = 0.834

Dia Vol% Width
0.979 7% 0.547
0.241 93% 0.151

Number Distribution; X-100 System

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	100.00	0.00						
592.0	100.00	0.00	7.778	100.00	0.00						
497.8	100.00	0.00	6.541	100.00	0.00						
418.6	100.00	0.00	5.500	100.00	0.00						
352.0	100.00	0.00	4.625	100.00	0.01						
296.0	100.00	0.00	3.889	99.99	0.02						
248.9	100.00	0.00	3.270	99.97	0.03						
209.3	100.00	0.00	2.750	99.94	0.05						
176.0	100.00	0.00	2.312	99.89	0.09						
148.0	100.00	0.00	1.945	99.80	0.20						
124.5	100.00	0.00	1.635	99.60	0.50						
104.7	100.00	0.00	1.375	99.10	1.04						
88.00	100.00	0.00	1.156	98.06	1.56						
74.00	100.00	0.00	0.972	96.50	1.70						
62.23	100.00	0.00	0.818	94.80	1.65						
52.33	100.00	0.00	0.688	93.15	1.79						
44.00	100.00	0.00	0.578	91.36	2.42						
37.00	100.00	0.00	0.486	88.94	3.88						
31.11	100.00	0.00	0.409	85.06	6.80						
26.16	100.00	0.00	0.344	78.26	11.99						
22.00	100.00	0.00	0.289	66.27	18.79						
18.50	100.00	0.00	0.243	47.48	23.24						
16	100.00	0.00	0.204	24.24	24.24						
14	100.00	0.00	0.172	0.00	0.00						
12	100.00	0.00	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-05

Date: 05/19/89 Meas #: 00156
Time: 17:39 Pres #: 01

20 G/L

CUF AZ-102 Waste
Sample: CUF- AZ102-05
In AZ-102-05 Simulated Supernatant
60ml/s,

Summary

mv = 2.845
mn = 0.330
ma = 1.002
cs = 5.989
sd = 2.285

Percentiles

10% = 0.434 60% = 1.676
20% = 0.736 70% = 2.681
30% = 0.935 80% = 4.310
40% = 1.105 90% = 7.200
50% = 1.310 95% = 10.58

Dia	Vol%	Width
5.101	33%	7.302
0.996	67%	1.050

Volume Distribution; X-100 System

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	93.50	2.31						
592.0	100.00	0.00	7.778	91.19	2.79						
497.8	100.00	0.00	6.541	88.40	3.25						
418.6	100.00	0.00	5.500	85.15	3.61						
352.0	100.00	0.00	4.625	81.54	3.80						
296.0	100.00	0.00	3.889	77.74	3.75						
248.9	100.00	0.00	3.270	73.99	3.50						
209.3	100.00	0.00	2.750	70.49	3.27						
176.0	100.00	0.00	2.312	67.22	3.46						
148.0	100.00	0.00	1.945	63.76	4.49						
124.5	100.00	0.00	1.635	59.27	6.78						
104.7	100.00	0.00	1.375	52.49	9.68						
88.00	100.00	0.00	1.156	42.81	10.65						
74.00	100.00	0.00	0.972	32.16	8.49						
62.23	100.00	0.00	0.818	23.67	5.60						
52.33	100.00	0.00	0.688	18.07	3.70						
44.00	100.00	0.00	0.578	14.37	2.78						
37.00	100.00	0.00	0.486	11.59	2.41						
31.11	100.00	0.00	0.409	9.18	2.33						
26.16	100.00	0.38	0.344	6.85	2.33						
22.00	99.62	0.61	0.289	4.52	2.10						
18.50	99.01	0.89	0.243	2.42	1.51						
15.56	98.12	1.20	0.204	0.91	0.91						
13.08	96.92	1.53	0.172	0.00	0.00						
11.00	95.39	1.89	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-05

Date: 05/19/89 Meas #: 00156
Time: 17:39 Pres #: 01

CUF AZ-102 Waste
Sample: CUF-AZ102-05
In AZ-102-05 Simulated Supernatant
60ml/s,

Summary

mv = 2.845
mn = 0.330
ma = 1.002
cs = 5.989
sd = 0.113

Percentiles

10% = 0.188 60% = 0.278
20% = 0.201 70% = 0.313
30% = 0.215 80% = 0.376
40% = 0.233 90% = 0.558
50% = 0.254 95% = 0.806

Dia

0.254

Vol%

100%

Width

0.226

Number Distribution; X-100 System

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	0.03						
176.0	100.00	0.00	2.312	99.93	0.06						
148.0	100.00	0.00	1.945	99.87	0.13						
124.5	100.00	0.00	1.635	99.74	0.33						
104.7	100.00	0.00	1.375	99.41	0.79						
88.00	100.00	0.00	1.156	98.62	1.47						
74.00	100.00	0.00	0.972	97.15	1.96						
62.23	100.00	0.00	0.818	95.19	2.18						
52.33	100.00	0.00	0.688	93.01	2.42						
44.00	100.00	0.00	0.578	90.59	3.06						
37.00	100.00	0.00	0.486	87.53	4.45						
31.11	100.00	0.00	0.409	83.08	7.24						
26.16	100.00	0.00	0.344	75.84	12.22						
22.00	100.00	0.00	0.289	63.62	18.49						
18.50	100.00	0.00	0.243	45.13	22.31						
15.56	100.00	0.00	0.204	22.82	22.82						
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

AZ-102 Waste
CUF-AZ102-05

Date: 05/19/89 Meas #: 00160
Time: 17:54 Pres #: 01

00626

CUF AZ-102 Waste

Sample: CUF- AZ102-05

In AZ-102-05 Simulated Supernatant

60ml/s, Sonicate 40 watts 90 seconds (1st sonication)

Number Distribution; X-100 System

Summary

mv = 2.925
mn = 0.323
ma = 0.956
cs = 6.275
sd = 0.112

Percentiles

10% = 0.187 60% = 0.280
20% = 0.201 70% = 0.316
30% = 0.215 80% = 0.378
40% = 0.234 90% = 0.529
50% = 0.255 95% = 0.731

Dia

0.255 100% 0.224

%CHAN

%PASS

- Size (microns) -

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	0.03						
176.0	100.00	0.00	2.312	99.93	0.05						
148.0	100.00	0.00	1.945	99.88	0.10						
124.5	100.00	0.00	1.635	99.78	0.24						
104.7	100.00	0.00	1.375	99.54	0.56						
88.00	100.00	0.00	1.156	98.98	1.07						
74.00	100.00	0.00	0.972	97.91	1.62						
62.23	100.00	0.00	0.818	96.29	2.06						
52.33	100.00	0.00	0.688	94.23	2.58						
44.00	100.00	0.00	0.578	91.65	3.46						
37.00	100.00	0.00	0.486	88.19	5.03						
31.11	100.00	0.00	0.409	83.16	7.80						
26.16	100.00	0.00	0.344	75.36	12.43						
22.00	100.00	0.00	0.289	62.93	18.14						
18.50	100.00	0.00	0.243	44.79	21.95						
15.56	100.00	0.00	0.204	22.84	22.84						
13.08	100.00	0.00	0.172	0.00	0.00						
11.00	100.00	0.00	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-05

Date: 05/19/89 Meas #: 00164
Time: 18:04 Pres #: 01

CUF AZ-102 Waste
Sample: CUF- AZ102-05
In AZ-102-05 Simulated Supernatant
50ml/s, 2nd Sonication 40 watts 90 seconds

Summary

mv = 2.971
mn = 0.320
ma = 0.951
cs = 6.312
sd = 2.509

Percentiles

10% = 0.386 60% = 1.891
20% = 0.632 70% = 3.092
30% = 0.858 80% = 4.667
40% = 1.065 90% = 7.574
50% = 1.331 95% = 10.98

Dia	Vol%	Width
4.724	39%	7.125
0.864	61%	0.983

Volume Distribution; X-100 System

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	92.96	2.52						
592.0	100.00	0.00	7.778	90.44	3.05						
497.8	100.00	0.00	6.541	87.39	3.58						
418.6	100.00	0.00	5.500	83.81	4.02						
352.0	100.00	0.00	4.625	79.79	4.27						
296.0	100.00	0.00	3.889	75.52	4.22						
248.9	100.00	0.00	3.270	71.30	3.87						
209.3	100.00	0.00	2.750	67.43	3.47						
176.0	100.00	0.00	2.312	63.96	3.38						
148.0	100.00	0.00	1.945	60.58	3.94						
124.5	100.00	0.00	1.635	56.64	5.38						
104.7	100.00	0.00	1.375	51.26	7.33						
88.00	100.00	0.00	1.156	43.93	8.37						
74.00	100.00	0.00	0.972	35.56	7.50						
62.23	100.00	0.00	0.818	28.06	5.80						
52.33	100.00	0.00	0.688	22.26	4.43						
44.00	100.00	0.00	0.578	17.83	3.63						
37.00	100.00	0.00	0.486	14.20	3.20						
31.11	100.00	0.00	0.409	11.00	2.97						
26.16	100.00	0.39	0.344	8.03	2.79						
22.00	99.61	0.64	0.289	5.24	2.42						
18.50	98.97	0.97	0.243	2.82	1.74						
15.00	98.00	1.31	0.204	1.08	1.08						
12.00	96.69	1.67	0.172	0.00	0.00						
10.00	95.02	2.06	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-05

Date: 05/19/89 Meas #: 00164
Time: 18:04 Pres #: 01

CUF AZ-102 Waste

Sample: CUF- AZ102-05

In AZ-102-05 Simulated Supernatant

60ml/s, 2nd Sonication 40 watts 90 seconds

Number Distribution; X-100 System

Summary

mv = 2.971
mn = 0.320
ma = 0.951
cs = 6.312
sd = 0.109

Percentiles

10% = 0.187 60% = 0.279
20% = 0.200 70% = 0.314
30% = 0.215 80% = 0.373
40% = 0.233 90% = 0.515
50% = 0.254 95% = 0.708

Dia Vol% Width

0.254 100% 0.217

- Size (microns) -

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	0.03						
176.0	100.00	0.00	2.312	99.93	0.05						
148.0	100.00	0.00	1.945	99.88	0.10						
124.5	100.00	0.00	1.635	99.78	0.22						
104.7	100.00	0.00	1.375	99.56	0.51						
88.00	100.00	0.00	1.156	99.05	0.98						
74.00	100.00	0.00	0.972	98.07	1.48						
62.23	100.00	0.00	0.818	96.59	1.93						
52.33	100.00	0.00	0.688	94.66	2.48						
44.00	100.00	0.00	0.578	92.18	3.41						
37.00	100.00	0.00	0.486	88.77	5.05						
31.11	100.00	0.00	0.409	83.72	7.89						
26.16	100.00	0.00	0.344	75.83	12.50						
22.00	100.00	0.00	0.289	63.33	18.21						
18.50	100.00	0.00	0.243	45.12	21.97						
15.56	100.00	0.00	0.204	23.15	23.15						
13.08	100.00	0.00	0.172	0.00	0.00						
11.00	100.00	0.00	0.145	0.00	0.00						

Particle Size Analysis

CUF AZ-102 Waste
CUF-AZ102-05

Date: 05/20/89 Meas #: 00011
Time: 10:31 Pres #: 01

CUF AZ-102 Waste

UPA

Sample: CUF-AZ102-05

Summary

Percentiles

Dia	Vol%	Width
-----	------	-------

4	100%	.6983
---	------	-------

```
mv = 1.830
mn = 1.636
ma = 1.763
cs = 3.404
sd = .3491
```

10% = 1.402	60% = 1.883
20% = 1.526	70% = 1.986
30% = 1.625	80% = 2.113
40% = 1.711	90% = 2.302
50% = 1.794	95% = 2.482

1.794 100% .6983

1. Volume Distribution; UPA System

[illegible]

Particle Size Analysis

00626

Time: 10:31 Pres #: 01

[illegible]

Particle Size Analysis

CUF AZ-102 Waste
CUF-AZ102-05 SON

Date: 05/20/89 Meas #: 00017
Time: 10:59 Pres #: 01

CUF AZ-102 Waste

UPA

Sample: CUF-AZ102-05 Sonicated

Summary

Percentiles

Dia	Vol%	Width
-----	------	-------

Vol%	Width
0	0
10	0
20	0
30	0
40	0
50	0
60	0
70	0
80	0
90	0
100	0

Width

$$mv = 1.078$$

10% = .3875 60% = 1.466

$$mn = .4429$$

20% = .4342 70% = 1.570

$$ma = .7249$$

30% = .4819 80% = 1.677

CS = 8.277

40% = .5610 90% = 1.803

sd = .6535

50% = 1.318 95% = 1.888

Dia	Vol%	Width
-----	------	-------

1.602 .54% .4211

.4479 46% .1740

Volume Distribution; MPA System

[illegible]

Particle Size Distribution Plots
For Concentrated Slurry: Sample CUF-AZ-102-005 DUP
Replicate No: 2

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-05 DUP

Date: 05/18/89 Meas #: 00036
Time: 14:11 Pres #: 01

AZ-102 Waste
Sample CUF-AZ102-05 DUP
In AZ-102-05 Simulated Supernatant
40 mL/s

Summary

mv = 3.237
mn = 0.329
ma = 1.115
cs = 5.380
sd = 2.418

Percentiles

10% = 0.510 60% = 1.860
20% = 0.869 70% = 2.726
30% = 1.074 80% = 4.718
40% = 1.262 90% = 7.374
50% = 1.491 95% = 11.62

Dia	Vol%	Width
6.236	27%	8.730
1.271	66%	1.194
0.286	7%	0.148

Volume Distribution ; X-100 System

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	93.08	2.19						
592.0	100.00	0.00	7.778	90.89	3.22						
497.8	100.00	0.00	6.541	87.67	4.07						
418.6	100.00	0.00	5.500	83.60	4.04						
352.0	100.00	0.00	4.625	79.56	3.40						
296.0	100.00	0.00	3.889	76.16	2.96						
248.9	100.00	0.00	3.270	73.20	3.03						
209.3	100.00	0.00	2.750	70.17	3.68						
176.0	100.00	0.00	2.312	66.49	4.93						
148.0	100.00	0.00	1.945	61.66	6.87						
124.5	100.00	0.00	1.635	54.69	9.32						
104.7	100.00	0.00	1.375	45.37	10.93						
88.00	100.00	0.00	1.156	34.44	9.79						
74.00	100.00	0.00	0.972	24.65	6.65						
62.23	100.00	0.00	0.818	18.00	3.97						
52.33	100.00	0.00	0.688	14.03	2.56						
44.00	100.00	0.00	0.578	11.47	1.98						
37.00	100.00	0.50	0.486	9.49	1.81						
31.11	99.50	0.50	0.409	7.68	1.83						
26.16	99.00	0.58	0.344	5.85	1.89						
22.00	98.42	0.72	0.289	3.96	1.78						
18.50	97.70	0.87	0.243	2.18	1.35						
15.56	96.83	1.03	0.204	0.83	0.83						
13.08	95.80	1.20	0.172	0.00	0.00						
11.00	94.60	1.52	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-05 DUP

Date: 05/18/89- Meas #: 00036
Time: 14:11 Pres #: 01

AZ-102 Waste
Sample CUF- AZ102-05 DUP
In AZ-102-05 Simulated Supernatant
40 mL/s

Summary

mv = 3.237
mn = 0.329
ma = 1.115
cs = 5.380
sd = 0.105

Percentiles

10% = 0.187 60% = 0.271
20% = 0.199 70% = 0.304
30% = 0.212 80% = 0.361
40% = 0.229 90% = 0.543
50% = 0.248 95% = 0.849

Dia Vol% Width
0.248 100% 0.210

Number Distribution; X-100 System

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	0.04						
176.0	100.00	0.00	2.312	99.91	0.10						
148.0	100.00	0.00	1.945	99.81	0.23						
124.5	100.00	0.00	1.635	99.58	0.53						
104.7	100.00	0.00	1.375	99.05	1.05						
88.00	100.00	0.00	1.156	98.00	1.58						
74.00	100.00	0.00	0.972	96.42	1.80						
62.23	100.00	0.00	0.818	94.62	1.81						
52.33	100.00	0.00	0.688	92.81	1.97						
44.00	100.00	0.00	0.578	90.84	2.55						
37.00	100.00	0.00	0.486	88.29	3.92						
31.11	100.00	0.00	0.409	84.37	6.66						
26.16	100.00	0.00	0.344	77.71	11.61						
22.00	100.00	0.00	0.289	66.10	18.36						
18.50	100.00	0.00	0.243	47.74	23.37						
15.56	100.00	0.00	0.204	24.37	24.37						
8	100.00	0.00	0.172	0.00	0.00						
7.00	100.00	0.00	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-05 DUP

Date: 05/18/89- Meas #: 00040
Time: 14:22 Pres #: 01

AZ-102 Waste
Sample CUF- AZ102-05 DUP
In AZ-102-05 Simulated Supernatant
60 mL/s

Summary

mv = 3.254
mn = 0.336
ma = 1.044
cs = 5.747
sd = 2.605

Percentiles

10% = 0.461 60% = 1.803
20% = 0.752 70% = 2.966
30% = 0.951 80% = 5.041
40% = 1.138 90% = 7.604
50% = 1.375 95% = 11.88

Dia	Vol%	Width
5.949	31%	8.122
1.031	69%	1.184

Volume Distribution; X-100 System

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	92.78	2.37						
592.0	100.00	0.00	7.778	90.41	3.56						
497.8	100.00	0.00	6.541	86.85	4.55						
418.6	100.00	0.00	5.500	82.30	4.42						
352.0	100.00	0.00	4.625	77.88	3.53						
296.0	100.00	0.00	3.889	74.35	2.84						
248.9	100.00	0.00	3.270	71.51	2.69						
209.3	100.00	0.00	2.750	68.82	3.02						
176.0	100.00	0.00	2.312	65.80	3.78						
148.0	100.00	0.00	1.945	62.02	5.05						
124.5	100.00	0.00	1.635	56.97	6.97						
104.7	100.00	0.00	1.375	50.00	9.08						
88.00	100.00	0.00	1.156	40.92	9.77						
74.00	100.00	0.00	0.972	31.15	8.14						
62.23	100.00	0.00	0.818	23.01	5.65						
52.33	100.00	0.00	0.688	17.36	3.80						
44.00	100.00	0.00	0.578	13.56	2.79						
37.00	100.00	0.44	0.486	10.77	2.34						
31.11	99.56	0.49	0.409	8.43	2.18						
26.16	99.07	0.59	0.344	6.25	2.14						
22.00	98.48	0.75	0.289	4.11	1.93						
18.50	97.73	0.94	0.243	2.18	1.38						
15.56	96.79	1.10	0.204	0.80	0.80						
13.08	95.69	1.28	0.172	0.00	0.00						
11.00	94.41	1.63	0.145	0.00	0.00						

00 6126

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-05 DUP

Date: 05/18/89 Meas #: 00040
Time: 14:22 Pres #: 01

AZ-102 Waste
Sample CUF-AZ102-05 DUP
In AZ-102-05 Simulated Supernatant
60 mL/s

Summary

mv = 3.254
mn = 0.336
ma = 1.044
cs = 5.747
sd = 0.121

Percentiles

10% = 0.188 60% = 0.282
20% = 0.202 70% = 0.319
30% = 0.217 80% = 0.387
40% = 0.236 90% = 0.581
50% = 0.256 95% = 0.817

Dia	Vol%	Width
0.256	100%	0.242

umber Distribution; X-100 System

%PASS

%CHAN

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	0.07						
148.0	100.00	0.00	1.945	99.85	0.16						
124.5	100.00	0.00	1.635	99.69	0.37						
104.7	100.00	0.00	1.375	99.32	0.81						
88.00	100.00	0.00	1.156	98.51	1.46						
74.00	100.00	0.00	0.972	97.05	2.04						
62.23	100.00	0.00	0.818	95.01	2.38						
52.33	100.00	0.00	0.688	92.63	2.70						
44.00	100.00	0.00	0.578	89.93	3.33						
37.00	100.00	0.00	0.486	86.60	4.69						
31.11	100.00	0.00	0.409	81.91	7.36						
26.16	100.00	0.00	0.344	74.55	12.18						
22.00	100.00	0.00	0.289	62.37	18.45						
18.50	100.00	0.00	0.243	43.92	22.15						
15.6	100.00	0.00	0.204	21.77	21.77						
8	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

AZ-102 Waste
CUF-AZ102-05 DUP

Date: 05/18/89 Meas #: 00044
Time: 14:35 Pres #: 01

AZ-102 Waste
SampleCUF-AZ102-05 DUP
In AZ-102-05 Simulated Supernatant
60 mLs, Sonicated 40 W, 90 sec

Summary
mv = 3.486
mn = 0.327
ma = 0.988
cs = 6.076
sd = 2.836

Percentiles
10% = 0.396
20% = 0.642
30% = 0.874
40% = 1.108
50% = 1.434
60% = 2.058
70% = 3.551
80% = 5.433
90% = 8.094
95% = 13.21

Dia Vol% Width
5.977 34% 8.595
0.938 66% 1.291

(1st Sonication)
Volume Distribution; X-100 System

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	91.84	2.52						
692.0	100.00	0.00	7.778	89.32	3.89						
497.8	100.00	0.00	6.541	85.43	5.06						
418.6	100.00	0.00	5.500	80.37	4.88						
352.0	100.00	0.00	4.625	75.49	3.83						
296.0	100.00	0.00	3.889	71.66	3.05						
248.9	100.00	0.00	3.270	68.61	2.89						
209.3	100.00	0.00	2.750	65.72	3.22						
176.0	100.00	0.00	2.312	62.50	3.81						
148.0	100.00	0.00	1.945	58.69	4.57						
124.5	100.00	0.00	1.635	54.12	5.58						
104.7	100.00	0.00	1.375	48.54	6.76						
88.00	100.00	0.00	1.156	41.78	7.40						
74.00	100.00	0.00	0.972	34.38	6.89						
62.23	100.00	0.00	0.818	27.49	5.62						
52.33	100.00	0.00	0.688	21.87	4.45						
44.00	100.00	0.00	0.578	17.42	3.67						
37.00	100.00	0.53	0.486	13.75	3.22						
31.11	99.47	0.59	0.409	10.53	2.96						
26.16	98.88	0.72	0.344	7.57	2.76						
22.00	98.16	0.90	0.289	4.81	2.35						
18.50	97.26	1.10	0.243	2.46	1.58						
15.56	96.16	1.23	0.204	0.88	0.88						
13.08	94.93	1.38	0.172	0.00	0.00						
11.00	93.55	1.71	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-05 DUP

Date: 05/18/89 Meas #: 00044
Time: 14:35 Pres #: 01

AZ-102 Waste

Sample CUF- AZ102-05 DUP

In AZ-102-05 Simulated Supernatant

60 mLs, Sonicated 40 W, 90 sec

(1st Sonication)

Number Distribution; X-100 System

Summary

mv = 3.486
mn = 0.327
ma = 0.988
cs = 6.076
sd = 0.113

Percentiles

10% = 0.189 60% = 0.287
20% = 0.204 70% = 0.323
30% = 0.221 80% = 0.384
40% = 0.240 90% = 0.526
50% = 0.261 95% = 0.711

Dia Vol% Width
0.261 100% 0.226

%PASS

%CHAN

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	0.01						
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	0.06						
148.0	100.00	0.00	1.945	99.86	0.12						
124.5	100.00	0.00	1.635	99.74	0.25						
104.7	100.00	0.00	1.375	99.49	0.51						
88.00	100.00	0.00	1.156	98.98	0.94						
74.00	100.00	0.00	0.972	98.04	1.47						
62.23	100.00	0.00	0.818	96.57	2.01						
52.33	100.00	0.00	0.688	94.56	2.69						
44.00	100.00	0.00	0.578	91.87	3.72						
37.00	100.00	0.00	0.486	88.15	5.48						
31.11	100.00	0.00	0.409	82.67	8.47						
26.16	100.00	0.00	0.344	74.20	13.33						
22.00	100.00	0.00	0.289	60.87	19.06						
18.50	100.00	0.00	0.243	41.81	21.50						
15.6	100.00	0.00	0.204	20.31	20.31						
8	100.00	0.00	0.172	0.00	0.00						
71.00	100.00	0.00	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-05 DUP

Date: 05/18/89 Meas #: 00048
Time: 14:46 Pres #: 01

AZ-102 Waste
Sample CUF- AZ102-05 DUP
In AZ-102-05 Simulated Supernatant
60 mLs, 2nd Sonication 40 W, 90 sec

Summary

mv = 3.528
mn = 0.323
ma = 0.986
cs = 6.084
sd = 2.870

Percentiles

10% = 0.389 60% = 2.134
20% = 0.635 70% = 3.668
30% = 0.876 80% = 5.502
40% = 1.123 90% = 8.176
50% = 1.472 95% = 13.38

Dia	Vol%	Width
5.973	35%	8.596
0.935	65%	1.320

Volume Distribution; X-100 System

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	91.70	2.55						
592.0	100.00	0.00	7.778	89.15	3.97						
497.8	100.00	0.00	6.541	85.18	5.19						
418.6	100.00	0.00	5.500	79.99	5.00						
352.0	100.00	0.00	4.625	74.99	3.89						
296.0	100.00	0.00	3.889	71.10	3.09						
248.9	100.00	0.00	3.270	68.01	2.96						
209.3	100.00	0.00	2.750	65.05	3.31						
176.0	100.00	0.00	2.312	61.74	3.90						
148.0	100.00	0.00	1.945	57.84	4.60						
124.5	100.00	0.00	1.635	53.24	5.51						
104.7	100.00	0.00	1.375	47.73	6.54						
88.00	100.00	0.00	1.156	41.19	7.10						
74.00	100.00	0.00	0.972	34.09	6.58						
62.23	100.00	0.00	0.818	27.51	5.41						
52.33	100.00	0.00	0.688	22.10	4.34						
44.00	100.00	0.00	0.578	17.76	3.65						
37.00	100.00	0.52	0.486	14.11	3.26						
31.11	99.48	0.59	0.409	10.85	3.04						
26.16	98.89	0.73	0.344	7.81	2.84						
22.00	98.16	0.93	0.289	4.97	2.41						
18.50	97.23	1.13	0.243	2.56	1.64						
15.56	96.10	1.27	0.204	0.92	0.92						
13.08	94.83	1.40	0.172	0.00	0.00						
11.00	93.43	1.73	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-05 DUP

Date: 05/18/89 Meas #: 00048
Time: 14:46 Pres #: 01

AZ-102 Waste
Sample CUF-AZ102-05 DUP
In AZ-102-05 Simulated Supernatant
60 mL/s, 2nd Sonication 40 W, 90 sec

Summary

mv = 3.528
mn = 0.323
ma = 0.986
cs = 6.084
sd = 0.109

Percentiles

10% = 0.189 60% = 0.285
20% = 0.203 70% = 0.320
30% = 0.220 80% = 0.378
40% = 0.238 90% = 0.513
50% = 0.259 95% = 0.694

Dia Vol% Width
0.259 100% 0.219

Number Distribution; X-100 System

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	0.06						
148.0	100.00	0.00	1.945	99.86	0.12						
124.6	100.00	0.00	1.635	99.74	0.24						
104.7	100.00	0.00	1.375	99.50	0.48						
88.00	100.00	0.00	1.156	99.02	0.88						
74.00	100.00	0.00	0.972	98.14	1.36						
62.23	100.00	0.00	0.818	96.78	1.89						
52.33	100.00	0.00	0.688	94.89	2.55						
44.00	100.00	0.00	0.578	92.34	3.61						
37.00	100.00	0.00	0.486	88.73	5.40						
31.11	100.00	0.00	0.409	83.33	8.48						
26.16	100.00	0.00	0.344	74.85	13.36						
22.00	100.00	0.00	0.289	61.49	19.05						
18.50	100.00	0.00	0.243	42.44	21.75						
15.56	100.00	0.00	0.204	20.69	20.69						
13.00	100.00	0.00	0.172	0.00	0.00						
11.00	100.00	0.00	0.145	0.00	0.00						

Meas #: 00013
Pres #: 01

	Vol%	Width
32	96%	.9169
65	4%	.1483

Article Size Analysis

CUF AZ-102 Waste
CUF-AZ102-05 DUP

Date: 05/20/89 Meas #: 00013
Time: 10:42 Pres #: 01

UP

tribution; UPA System

Summary

```
mv = 1.673
mn = .5358
ma = 1.365
cs = 4.396
sd = .3385
```

Percentiles

10% = .2615	60% = .3684
20% = .2759	70% = .4282
30% = .2911	80% = .6642
40% = .3104	90% = 1.300
50% = .3346	95% = 1.594

Dia	Vol%	Width
-----	------	-------

1.310	20%	.8324
.3108	80%	.1425

<u>%PASS</u>	<u>%CHAN</u>
--------------	--------------

[illegible]

Particle Size Analysis

CUF AZ-102 Waste
CUF-AZ102-05 DUP SON

Date: 05/20/89 Meas #: 00015
Time: 10:53 Pres #: 01

CUF AZ-102 Waste

LUPA

Sample: CUF-AZ102-05DUP SON

Volume Distribution; UPA System

Summary

```
mv = .8834
mn = .8014
ma = .8559
cs = 7.010
sd = .1569
```

Percentiles

10% = .6909	60% = .9137
20% = .7492	70% = .9576
30% = .7953	80% = 1.012
40% = .8359	90% = 1.095
50% = .8743	95% = 1.158

Dia	Vol%	Width
-----	------	-------

.8743 100% .3138

[illegible]

Particle Size Analysis

CUF AZ-102 Waste
CUF-AZ102-05 DUP SON

Date: 05/20/89 Meas #: 00015
Time: 10:53 Pres #: 01

CUF AZ-102 Waste
UPA
Sample: CUF-AZ102-05DUP SON

Summary

Percentiles

Dia	Vol%	Width
-----	------	-------

```
mv = .8834
mn = .8014
ma = .8559
cs = 7.010
sd = .1469
```

10% = .6106	60% = .8251
20% = .6695	70% = .8679
30% = .7126	80% = .9181
40% = .7499	90% = .9899
50% = .7866	95% = 1.055

.7866 100% .2937

Number Distribution; UPA System.

[illegible]

Particle Size Distribution Plots
For Final Slurry: Sample CUF-AZ-102-024
Replicate No: 1

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-24

Date: 05/19/89 Meas #: 00132
Time: 16:16 Pres #: 01

CUF AZ-102 Waste
Sample: CUF- AZ102-24
In AZ-102-24 Simulated Supernatant
40ml/s

Summary

mv = 1.531
mn = 0.314
ma = 0.700
cs = 8.568
sd = 0.666

Percentiles

10% = 0.305 60% = 1.149
20% = 0.432 70% = 1.299
30% = 0.667 80% = 1.531
40% = 0.876 90% = 2.611
50% = 1.019 95% = 5.789

Dia Vol% Width

6.525 9% 7.377
1.112 69% 0.789
0.319 22% 0.179

Volume Distribution - X-100 System

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	97.69	0.91						
592.0	100.00	0.00	7.778	96.78	1.02						
497.8	100.00	0.00	6.541	95.76	1.08						
418.6	100.00	0.00	5.500	94.68	1.08						
352.0	100.00	0.00	4.625	93.60	1.06						
296.0	100.00	0.00	3.889	92.54	1.05						
248.9	100.00	0.00	3.270	91.49	1.12						
209.3	100.00	0.00	2.750	90.37	1.37						
176.0	100.00	0.00	2.312	89.00	2.13						
148.0	100.00	0.00	1.945	86.87	4.18						
124.5	100.00	0.00	1.635	82.69	8.53						
104.7	100.00	0.00	1.375	74.16	13.65						
88.00	100.00	0.00	1.156	60.51	14.10						
74.00	100.00	0.00	0.972	46.41	9.74						
62.23	100.00	0.00	0.818	36.67	5.88						
52.33	100.00	0.00	0.688	30.79	4.15						
44.00	100.00	0.00	0.578	26.64	3.81						
37.00	100.00	0.00	0.486	22.83	4.24						
31.11	100.00	0.00	0.409	18.59	4.94						
26.16	100.00	0.00	0.344	13.65	5.26						
22.00	100.00	0.00	0.289	8.39	4.44						
18.50	100.00	0.37	0.243	3.95	2.67						
15.56	99.63	0.51	0.204	1.28	1.28						
13.08	99.12	0.65	0.172	0.00	0.00						
11.00	98.47	0.78	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-24

Date: 05/19/89 Meas #: 00132
Time: 16:16 Pres #: 01

CUF AZ-102 Waste
Sample: CUF- AZ102-24
In AZ-102-24 Simulated Supernatant
40ml/s

Summary

mv = 1.531
mn = 0.314
ma = 0.700
cs = 8.568
sd = 0.090

Percentiles

10% = 0.192 60% = 0.283
20% = 0.207 70% = 0.311
30% = 0.224 80% = 0.353
40% = 0.242 90% = 0.454
50% = 0.261 95% = 0.657

Dia Vol% Width
0.261 100% 0.179

umber Distribution; X-100 System

%PASS

%CHAN

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	0.02						
148.0	100.00	0.00	1.945	99.97	0.07						
124.5	100.00	0.00	1.635	99.90	0.24						
104.7	100.00	0.00	1.375	99.66	0.63						
88.00	100.00	0.00	1.156	99.03	1.10						
74.00	100.00	0.00	0.972	97.93	1.27						
62.23	100.00	0.00	0.818	96.66	1.29						
52.33	100.00	0.00	0.688	95.37	1.54						
44.00	100.00	0.00	0.578	93.83	2.38						
37.00	100.00	0.00	0.486	91.45	4.44						
31.11	100.00	0.00	0.409	87.01	8.70						
26.16	100.00	0.00	0.344	78.31	15.63						
22.00	100.00	0.00	0.289	62.68	22.15						
18.50	100.00	0.00	0.243	40.53	22.36						
15.56	100.00	0.00	0.204	18.17	18.17						
	100.00	0.00	0.172	0.00	0.00						
	100.00	0.00	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-24

Date: 05/19/89 Meas #: 00136
Time: 16:33 Pres #: 01

CUF AZ-102 Waste
Sample: CUF- AZ102-24
In AZ-102-24 Simulated Supernatant
60ml/s

Summary

mv = 1.432
mn = 0.314
ma = 0.683
cs = 8.783
sd = 0.609

Percentiles

10% = 0.302 60% = 1.106
20% = 0.425 70% = 1.244
30% = 0.650 80% = 1.443
40% = 0.849 90% = 2.197
50% = 0.985 95% = 5.147

Dia	Vol%	Width
6.488	7%	7.258
1.081	69%	0.738
0.318	24%	0.178

Volume Distribution; X-100 System

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	98.02	0.80						
592.0	100.00	0.00	7.778	97.22	0.90						
497.8	100.00	0.00	6.541	96.32	0.95						
418.6	100.00	0.00	5.500	95.37	0.96						
352.0	100.00	0.00	4.625	94.41	0.94						
296.0	100.00	0.00	3.889	93.47	0.92						
248.9	100.00	0.00	3.270	92.55	0.96						
209.3	100.00	0.00	2.750	91.59	1.16						
176.0	100.00	0.00	2.312	90.43	1.79						
148.0	100.00	0.00	1.945	88.64	3.57						
124.5	100.00	0.00	1.635	85.07	7.74						
104.7	100.00	0.00	1.375	77.33	13.44						
88.00	100.00	0.00	1.156	63.89	14.98						
74.00	100.00	0.00	0.972	48.91	10.84						
62.23	100.00	0.00	0.818	38.07	6.51						
52.33	100.00	0.00	0.688	31.56	4.40						
44.00	100.00	0.00	0.578	27.16	3.88						
37.00	100.00	0.00	0.486	23.28	4.25						
31.11	100.00	0.00	0.409	19.03	4.98						
26.16	100.00	0.00	0.344	14.05	5.40						
22.00	100.00	0.00	0.289	8.65	4.60						
18.50	100.00	0.31	0.243	4.05	2.75						
15.56	99.69	0.43	0.204	1.30	1.30						
13.08	99.26	0.56	0.172	0.00	0.00						
11.00	98.70	0.68	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-24

Date: 05/19/89 Meas #: 00140
Time: 16:45 Pres #: 01

CUF AZ-102 Waste

Sample: CUF- AZ102-24

In AZ-102-24 Simulated Supernatant

60ml/s, 1st Sonication, 40 W, 90 sec

ume Distribution; X-100 System

Summary

mv = 1.326
mn = 0.244
ma = 0.504
cs = 11.91
sd = 0.528

Percentiles

10% = 0.251 60% = 0.802
20% = 0.320 70% = 0.963
30% = 0.399 80% = 1.187
40% = 0.509 90% = 2.532
50% = 0.649 95% = 6.203

Dia	Vol%	Width
6.032	10%	7.654
0.862	51%	0.637
0.314	39%	0.201

%PASS

%CHAN

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	97.47	0.99						
692.0	100.00	0.00	7.778	96.48	1.12						
497.8	100.00	0.00	6.541	95.36	1.18						
418.6	100.00	0.00	5.500	94.18	1.15						
352.0	100.00	0.00	4.625	93.03	1.04						
296.0	100.00	0.00	3.889	91.99	0.89						
248.9	100.00	0.00	3.270	91.10	0.76						
209.3	100.00	0.00	2.750	90.34	0.72						
176.0	100.00	0.00	2.312	89.62	0.87						
148.0	100.00	0.00	1.945	88.75	1.41						
124.5	100.00	0.00	1.635	87.34	2.82						
104.7	100.00	0.00	1.375	84.52	5.52						
88.00	100.00	0.00	1.156	79.00	8.48						
74.00	100.00	0.00	0.972	70.52	9.47						
62.23	100.00	0.00	0.818	61.05	8.53						
52.33	100.00	0.00	0.688	52.52	7.39						
44.00	100.00	0.00	0.578	45.13	6.94						
37.00	100.00	0.00	0.486	38.19	7.18						
31.11	100.00	0.00	0.409	31.01	7.70						
26.16	100.00	0.00	0.344	23.31	7.79						
22.00	100.00	0.00	0.289	15.52	6.54						
18.50	100.00	0.40	0.243	8.98	4.11						
15.56	99.60	0.57	0.204	4.87	2.26						
8	99.03	0.71	0.172	2.61	1.42						
0.00	98.32	0.85	0.145	1.19	1.19						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-24

Date: 05/19/89 Meas #: 00140
Time: 16:45 Pres #: 01

CUF AZ-102 Waste
Sample: CUF- AZ102-24
In AZ-102-24 Simulated Supernatant
60ml/s, 1st Sonication, 40 W, 90 sec

Summary	Percentiles			Dia	Vol%	Width
mv = 1.326	10% = 0.134	60% = 0.236		0.257	66%	0.177
mn = 0.244	20% = 0.145	70% = 0.266		0.144	34%	0.029
ma = 0.504	30% = 0.163	80% = 0.306				
cs = 11.91	40% = 0.185	90% = 0.380				
sd = 0.094	50% = 0.209	95% = 0.479				

Number Distribution - X-100 System

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	0.11						
88.00	100.00	0.00	1.156	99.85	0.28						
74.00	100.00	0.00	0.972	99.57	0.53						
62.23	100.00	0.00	0.818	99.04	0.80						
52.33	100.00	0.00	0.688	98.24	1.17						
44.00	100.00	0.00	0.578	97.07	1.84						
37.00	100.00	0.00	0.486	95.23	3.20						
31.11	100.00	0.00	0.409	92.03	5.77						
26.16	100.00	0.00	0.344	86.26	9.85						
22.00	100.00	0.00	0.289	76.41	13.89						
18.50	100.00	0.00	0.243	62.52	14.65						
15.56	100.00	0.00	0.204	47.87	13.66						
13.08	100.00	0.00	0.172	34.21	14.23						
11.00	100.00	0.00	0.145	19.98	19.98						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-24

Date: 05/19/89 Meas #: 00144
Time: 16:55 Pres #: 01

CUF AZ-102 Waste
Sample: CUF- AZ102-24
In AZ-102-24 Simulated Supernatant
60ml/s, 2st Sonication, 40 W, 90 sec

Summary

mv = 1.358
mn = 0.238
ma = 0.496
cs = 12.11
sd = 0.545

Percentiles

10% = 0.246 60% = 0.790
20% = 0.315 70% = 0.959
30% = 0.392 80% = 1.198
40% = 0.498 90% = 2.883
50% = 0.635 95% = 6.468

Dia

6.038
0.856
0.312

Vol%

11%
50%
39%

Width

7.787
0.644
0.204

une Distribution - X-100 System

%PASS

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	97.27	1.03						
592.0	100.00	0.00	7.778	96.24	1.16						
497.8	100.00	0.00	6.541	95.08	1.23						
418.6	100.00	0.00	5.500	93.85	1.21						
352.0	100.00	0.00	4.625	92.64	1.10						
296.0	100.00	0.00	3.889	91.54	0.94						
248.9	100.00	0.00	3.270	90.60	0.81						
209.3	100.00	0.00	2.750	89.79	0.76						
176.0	100.00	0.00	2.312	89.03	0.90						
148.0	100.00	0.00	1.945	88.13	1.42						
124.5	100.00	0.00	1.635	86.71	2.75						
104.7	100.00	0.00	1.375	83.96	5.26						
88.00	100.00	0.00	1.156	78.70	7.99						
74.00	100.00	0.00	0.972	70.71	8.99						
62.23	100.00	0.00	0.818	61.72	8.28						
52.33	100.00	0.00	0.688	53.44	7.35						
44.00	100.00	0.00	0.578	46.09	7.02						
37.00	100.00	0.00	0.486	39.07	7.29						
31.11	100.00	0.00	0.409	31.78	7.77						
26.16	100.00	0.00	0.344	24.01	7.81						
22.00	100.00	0.00	0.289	16.20	6.58						
18.50	100.00	0.44	0.243	9.62	4.22						
15.56	99.56	0.62	0.204	5.40	2.42						
8	98.94	0.77	0.172	2.98	1.59						
...00	98.17	0.90	0.145	1.39	1.39						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-24

Date: 05/19/89 Meas #: 00144
Time: 16:55 Pres #: 01

CUF AZ-102 Waste

Sample: CUF- AZ102-24

In AZ-102-24 Simulated Supernatant

60ml/s, 2st Sonication, 40 W, 90 sec

Number Distribution; X-100 System

Summary

mv = 1.358
mn = 0.238
ma = 0.496
cs = 12.11
sd = 0.092

Percentiles

10% = 0.133 60% = 0.230
20% = 0.143 70% = 0.260
30% = 0.158 80% = 0.300
40% = 0.178 90% = 0.373
50% = 0.203 95% = 0.467

Dia Vol% Width

0.255 63% 0.176
0.143 37% 0.029

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						
124.6	100.00	0.00	1.635	99.99	0.03						
104.7	100.00	0.00	1.375	99.96	0.10						
88.00	100.00	0.00	1.156	99.86	0.25						
74.00	100.00	0.00	0.972	99.61	0.47						
62.23	100.00	0.00	0.818	99.14	0.73						
52.33	100.00	0.00	0.688	98.41	1.09						
44.00	100.00	0.00	0.578	97.32	1.75						
37.00	100.00	0.00	0.486	95.57	3.05						
31.11	100.00	0.00	0.409	92.52	5.46						
26.16	100.00	0.00	0.344	87.06	9.26						
22.00	100.00	0.00	0.289	77.80	13.11						
18.50	100.00	0.00	0.243	64.69	14.11						
15.56	100.00	0.00	0.204	50.58	13.72						
13.08	100.00	0.00	0.172	36.86	14.95						
11.00	100.00	0.00	0.145	21.91	21.91						

Particle Size Analysis

CUF AZ-102 Waste
UPA CUF-AZ102-24

Date: 05/20/89 Meas #: 00006
Time: 09:56 Pres #: 01

CUF AZ-102 Waste

U**P****A**

Sample: CUF-AZ102-24

Volume Distribution; ~~K210025~~ UPA System

Summary

Percentiles

```
mv = 2.434
mn = .2155
ma = 1.209
cs = 4.962
sd = 1.307
```

10% = .4871	60% = 2.863
20% = 1.354	70% = 3.097
30% = 2.050	80% = 3.372
40% = 2.382	90% = 3.762
50% = 2.634	95% = 4.099

<u>Dia</u>	<u>Vol%</u>	<u>Width</u>
2.826	83%	1.558
.4113	17%	.5335

[illegible]

Particle Size Analysis

CUF AZ-102 Waste
CUF-AZ102-24 SON

Date: 05/20/89 Meas #: 00009
Time: 10:14 Pres #: 01

CUF AZ-102 Waste

UPA

Sample: CUF-AZ102-24 Sonicated

Volume Distribution; UPA System

Summary

```
mv = 1.409
mn = .3173
ma = .7587
cs = 7.908
sd = .5947
```

Percentiles

10% = .3156	60% = 1.119
20% = .5968	70% = 1.220
30% = .8394	80% = 1.384
40% = .9497	90% = 4.075
50% = 1.035	95% = 4.421

<u>Dia</u>	<u>Vol%</u>	<u>Width</u>
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4.228	15%	.8392
1.044	67%	.4532
.3018	18%	.1621

[illegible]

Particle Size Analysis

CUF AZ-102 Waste
CUF-AZ102-24 SON

Date: 05/20/89 Meas #: 00009
Time: 10:14 Pres #: 01

CUF AZ-102 Waste

UPA

Sample: CUF-AZ102-24 Sonicated

Summary

Percentiles

Dia	Vol%	Width
-----	------	-------

$$mv = 1.409$$

10% = .1920 60% = .2751

.9029 8% .4337

$$mn = .3173$$

20% = .2075 70% = .2996

.10320	91%	.11661
.2490	92%	.1260

$$ma = .7587$$

30% = .2229 80% = .3374

.2400 6270 .11200

CS = 7.908

40% = .2389 90% = .4558

```
sd = .0806
```

50% = .2558 95% = .8247

Number Distribution; UPA System

[illegible]

Particle Size Distribution Plots
For Final Slurry: CUF-AZ-102-024 DUP
Replicate No: 2

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-24 DUP

Date: 05/18/89 Meas #: 00020
Time: 13:14 Pres #: 01

AZ-102 Waste
Sample CUF- AZ102-24 DUP
In AZ-102-24 Simulated Supernatant
40 mL/s

Summary

mv = 2.739
mn = 0.316
ma = 0.824
cs = 7.284
sd = 2.538

Percentiles

10% = 0.329 60% = 1.340
20% = 0.528 70% = 1.686
30% = 0.792 80% = 4.448
40% = 0.979 90% = 6.967
50% = 1.144 95% = 9.857

Dia	Vol%	Width
6.505	23%	7.417
1.103	58%	0.823
0.319	19%	0.182

Volume Distribution; X-100 System

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	94.45	2.23						
592.0	100.00	0.00	7.778	92.22	3.71						
497.8	100.00	0.00	6.541	88.51	4.54						
418.6	100.00	0.00	5.500	83.97	3.44						
352.0	100.00	0.00	4.625	80.53	1.85						
296.0	100.00	0.00	3.889	78.68	1.05						
248.9	100.00	0.00	3.270	77.63	0.90						
209.3	100.00	0.00	2.750	76.73	1.21						
176.0	100.00	0.00	2.312	75.52	2.19						
148.0	100.00	0.00	1.945	73.33	4.26						
124.5	100.00	0.00	1.635	69.07	7.58						
104.7	100.00	0.00	1.375	61.49	10.79						
88.00	100.00	0.00	1.156	50.70	11.10						
74.00	100.00	0.00	0.972	39.60	8.39						
62.23	100.00	0.00	0.818	31.21	5.50						
52.33	100.00	0.00	0.688	25.71	3.91						
44.00	100.00	0.00	0.578	21.80	3.39						
37.00	100.00	0.46	0.486	18.41	3.50						
31.11	99.54	0.43	0.409	14.91	3.89						
26.16	99.11	0.45	0.344	11.02	4.14						
22.00	98.66	0.53	0.289	6.88	3.60						
18.50	98.13	0.65	0.243	3.28	2.22						
15.56	97.48	0.77	0.204	1.06	1.06						
13.08	96.71	0.94	0.172	0.00	0.00						
11.00	95.77	1.32	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-24 DUP

Date: 05/18/89 Meas #: 00020
Time: 13:14 Pres #: 01

009Kb

AZ-102 Waste
Sample CUF- AZ102-24 DUP
In AZ-102-24 Simulated Supernatant
40 mL/s

Summary

mv = 2.739
mn = 0.316
ma = 0.824
cs = 7.284
sd = 0.092

Percentiles

10% = 0.191 60% = 0.282
20% = 0.207 70% = 0.310
30% = 0.224 80% = 0.356
40% = 0.241 90% = 0.468
50% = 0.260 95% = 0.678

Dia

0.260

Vol%

100%

Width

0.185

umber Distribution; X-100 System

%PASS

%CHAN

- Size (microns) -

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	0.03						
148.0	100.00	0.00	1.945	99.96	0.09						
124.5	100.00	0.00	1.635	99.87	0.25						
104.7	100.00	0.00	1.375	99.62	0.61						
88.00	100.00	0.00	1.156	99.01	1.06						
74.00	100.00	0.00	0.972	97.95	1.34						
62.23	100.00	0.00	0.818	96.61	1.48						
52.33	100.00	0.00	0.688	95.13	1.77						
44.00	100.00	0.00	0.578	93.36	2.58						
37.00	100.00	0.00	0.486	90.78	4.47						
31.11	100.00	0.00	0.409	86.31	8.36						
26.16	100.00	0.00	0.344	77.95	15.00						
22.00	100.00	0.00	0.289	62.95	21.91						
18.50	100.00	0.00	0.243	41.04	22.68						
15.56	100.00	0.00	0.204	18.36	18.36						
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

AZ-102 Waste
CUF-AZ102-24 DUP

Date: 05/18/89 Meas #: 00024
Time: 13:26 Pres #: 01

AZ-102 Waste
Sample CUF- AZ102-24 DUP
In AZ-102-24 Simulated Supernatant
60 mL/s

Summary
mv = 2.362
mn = 0.315
ma = 0.717
cs = 8.365
sd = 2.188

Percentiles
10% = 0.307 60% = 1.127
20% = 0.436 70% = 1.403
30% = 0.622 80% = 3.073
40% = 0.798 90% = 6.130
50% = 0.954 95% = 8.197

Dia Vol% Width
6.065 21% 6.679
0.936 61% 0.804
0.298 18% 0.136

Volume Distribution; X-100 System

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	95.90	1.45						
592.0	100.00	0.00	7.778	94.45	2.90						
497.8	100.00	0.00	6.541	91.55	4.31						
418.6	100.00	0.00	5.500	87.24	3.74						
352.0	100.00	0.00	4.625	83.50	2.08						
296.0	100.00	0.00	3.889	81.42	1.11						
248.9	100.00	0.00	3.270	80.31	0.84						
209.3	100.00	0.00	2.750	79.47	0.98						
176.0	100.00	0.00	2.312	78.49	1.54						
148.0	100.00	0.00	1.945	76.95	2.73						
124.5	100.00	0.00	1.635	74.22	4.89						
104.7	100.00	0.00	1.375	69.33	7.91						
88.00	100.00	0.00	1.156	61.42	10.26						
74.00	100.00	0.00	0.972	51.16	9.91						
62.23	100.00	0.00	0.818	41.25	7.74						
52.33	100.00	0.00	0.688	33.51	5.79						
44.00	100.00	0.00	0.578	27.72	4.80						
37.00	100.00	0.44	0.486	22.92	4.62						
31.11	99.56	0.40	0.409	18.30	4.91						
26.16	99.16	0.41	0.344	13.39	5.14						
22.00	98.75	0.45	0.289	8.25	4.42						
18.50	98.30	0.50	0.243	3.83	2.64						
15.56	97.80	0.53	0.204	1.19	1.19						
13.08	97.27	0.58	0.172	0.00	0.00						
11.00	96.69	0.79	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-24 DUP

Date: 05/18/89 Meas #: 00024
Time: 13:26 Pres #: 01

AZ-102 Waste
SampleCUF- AZ102-24 DUP
In AZ-102-24 Simulated Supernatant
60 mL/s

Summary

mv = 2.362
mn = 0.315
ma = 0.717
cs = 8.365
sd = 0.095

Percentiles

10% = 0.193 60% = 0.286
20% = 0.209 70% = 0.315
30% = 0.227 80% = 0.362
40% = 0.244 90% = 0.478
50% = 0.263 95% = 0.653

Dia Vol% Width
0.263 100% 0.190

Number Distribution; X-100 System

%PASS

%CHAN

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						
362.0	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	0.02						
148.0	100.00	0.00	1.945	99.97	0.05						
124.5	100.00	0.00	1.635	99.92	0.14						
104.7	100.00	0.00	1.375	99.78	0.37						
88.00	100.00	0.00	1.156	99.41	0.81						
74.00	100.00	0.00	0.972	98.60	1.31						
62.23	100.00	0.00	0.818	97.29	1.72						
52.33	100.00	0.00	0.688	95.57	2.16						
44.00	100.00	0.00	0.578	93.41	3.01						
37.00	100.00	0.00	0.486	90.40	4.87						
31.11	100.00	0.00	0.409	85.53	8.70						
26.16	100.00	0.00	0.344	76.83	15.37						
22.00	100.00	0.00	0.289	61.46	22.20						
18.50	100.00	0.00	0.243	39.26	22.25						
15.56	100.00	0.00	0.204	17.01	17.01						
8	100.00	0.00	0.172	0.00	0.00						
0	100.00	0.00	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-24 DUP

Date: 05/18/89 Meas #: 00028
Time: 13:41 Pres #: 01

AZ-102 Waste
Sample CUF- AZ102-24 DUP
In AZ-102-24 Simulated Supernatant
60 mL/s; Sonicated at 40 W, 90 sec

(1st Sonication)

Volume Distribution; X-100 System

Summary

mv = 3.061
mn = 0.282
ma = 0.613
cs = 9.790
sd = 2.274

Percentiles

10% = 0.277 60% = 1.034
20% = 0.357 70% = 1.375
30% = 0.461 80% = 2.563
40% = 0.608 90% = 7.161
50% = 0.801 95% = 18.37

Dia	Vol%	Width
23.05	7%	17.86
5.719	12%	3.524
1.015	42%	0.881
0.349	39%	0.243

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	91.95	1.10						
592.0	100.00	0.00	7.778	90.85	2.05						
497.8	100.00	0.00	6.541	88.80	2.95						
418.6	100.00	0.00	5.500	85.85	2.50						
352.0	100.00	0.00	4.625	83.35	1.40						
296.0	100.00	0.00	3.889	81.95	0.82						
248.9	100.00	0.00	3.270	81.13	0.75						
209.3	100.00	0.00	2.750	80.38	1.08						
176.0	100.00	0.00	2.312	79.30	1.86						
148.0	100.00	0.00	1.945	77.44	3.04						
124.5	100.00	0.00	1.635	74.40	4.41						
104.7	100.00	0.00	1.375	69.99	5.74						
88.00	100.00	0.00	1.156	64.25	6.66						
74.00	100.00	0.00	0.972	57.59	6.81						
62.23	100.00	0.00	0.818	50.78	6.43						
52.33	100.00	0.00	0.688	44.35	6.13						
44.00	100.00	0.00	0.578	38.22	6.20						
37.00	100.00	1.65	0.486	32.02	6.56						
31.11	98.35	1.23	0.409	25.46	6.94						
26.16	97.12	1.07	0.344	18.52	6.88						
22.00	96.05	1.01	0.289	11.64	5.67						
18.50	95.04	0.93	0.243	5.97	3.41						
15.56	94.11	0.79	0.204	2.56	1.70						
13.08	93.32	0.66	0.172	0.86	0.86						
11.00	92.66	0.71	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-24 DUP

Date: 05/18/89 Meas #: 00028
Time: 13:41 Pres #: 01

AZ-102 Waste

Sample CUF-AZ102-24 DUP

In AZ-102-24 Simulated Supernatant

60 mL/s; Sonicated at 40 W, 90 sec

(1st Sonication)
Number Distribution; X-100 System

Summary

mv = 3.061
mn = 0.282
ma = 0.613
cs = 9.790
sd = 0.093

Percentiles

10% = 0.166 60% = 0.269
20% = 0.184 70% = 0.298
30% = 0.205 80% = 0.339
40% = 0.225 90% = 0.420
50% = 0.246 95% = 0.524

Dia

0.246

Vol%

100%

Width

0.186

%PASS

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	0.03						
124.5	100.00	0.00	1.635	99.96	0.08						
104.7	100.00	0.00	1.375	99.88	0.18						
88.00	100.00	0.00	1.156	99.70	0.35						
74.00	100.00	0.00	0.972	99.35	0.59						
62.23	100.00	0.00	0.818	98.76	0.94						
52.33	100.00	0.00	0.688	97.82	1.52						
44.00	100.00	0.00	0.578	96.30	2.57						
37.00	100.00	0.00	0.486	93.73	4.57						
31.11	100.00	0.00	0.409	89.16	8.14						
26.16	100.00	0.00	0.344	81.02	13.61						
22.00	100.00	0.00	0.289	67.41	18.84						
18.50	100.00	0.00	0.243	48.57	19.02						
15.56	100.00	0.00	0.204	29.55	16.07						
8	100.00	0.00	0.172	13.48	13.48						
0.00	100.00	0.00	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-24 DUP

Date: 05/18/89 Meas #: 00032
Time: 13:51 Pres #: 01

AZ-102 Waste
Sample CUF- AZ102-24 DUP
In AZ-102-24 Simulated Supernatant
60 mL/s; Sonicated at 40 W, 90 sec (2nd Sonication)
Volume Distribution; X-100 System

Summary	Percentiles		Dia	Vol%	Width
mv = 2.965	10% = 0.282	60% = 1.033	22.54	7%	17.80
mn = 0.286	20% = 0.365	70% = 1.365	5.730	12%	3.531
ma = 0.622	30% = 0.472	80% = 2.424	1.010	43%	0.875
cs = 9.650	40% = 0.619	90% = 6.971	0.354	38%	0.24/
sd = 2.209	50% = 0.807	95% = 17.23

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	92.30	1.11						
592.0	100.00	0.00	7.778	91.19	2.06						
497.8	100.00	0.00	6.541	89.13	2.96						
418.6	100.00	0.00	5.500	86.17	2.49						
352.0	100.00	0.00	4.625	83.68	1.39						
296.0	100.00	0.00	3.889	82.29	0.81						
248.9	100.00	0.00	3.270	81.48	0.75						
209.3	100.00	0.00	2.750	80.73	1.08						
176.0	100.00	0.00	2.312	79.65	1.88						
148.0	100.00	0.00	1.945	77.77	3.08						
124.5	100.00	0.00	1.635	74.69	4.46						
104.7	100.00	0.00	1.375	70.23	5.84						
88.00	100.00	0.00	1.156	64.39	6.83						
74.00	100.00	0.00	0.972	57.56	7.04						
62.23	100.00	0.00	0.818	50.52	6.68						
52.33	100.00	0.00	0.688	43.84	6.36						
44.00	100.00	0.00	0.578	37.48	6.37						
37.00	100.00	1.47	0.486	31.11	6.64						
31.11	98.53	1.14	0.409	24.47	6.89						
26.16	97.39	1.02	0.344	17.58	6.68						
22.00	96.37	0.98	0.289	10.90	5.37						
18.50	95.39	0.92	0.243	5.53	3.16						
15.56	94.47	0.78	0.204	2.37	1.56						
13.08	93.69	0.67	0.172	0.81	0.81						
11.00	93.02	0.72	0.145	0.00	0.00						

Particle Size Analysis

AZ-102 Waste
CUF-AZ102-24 DUP

Date: 05/18/89 Meas #: 00032
Time: 13:51 Pres #: 01

AZ-102 Waste
Sample CUF-AZ102-24 DUP
In AZ-102-24 Simulated Supernatant
60 mL/s; Sonicated at 40 W, 90 sec

Summary

mv = 2.965
mn = 0.286
ma = 0.622
cs = 9.650
sd = 0.097

Percentiles

10% = 0.166 60% = 0.273
20% = 0.185 70% = 0.302
30% = 0.206 80% = 0.345
40% = 0.228 90% = 0.430
50% = 0.249 95% = 0.538

Dia Vol% Width
0.249 100% 0.193

2nd Sonication
Distribution; X-100 System
Number

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	0.04						
124.5	100.00	0.00	1.635	99.95	0.09						
104.7	100.00	0.00	1.375	99.86	0.19						
88.00	100.00	0.00	1.156	99.67	0.37						
74.00	100.00	0.00	0.972	99.30	0.64						
62.23	100.00	0.00	0.818	98.66	1.03						
52.33	100.00	0.00	0.688	97.63	1.65						
44.00	100.00	0.00	0.578	95.98	2.78						
37.00	100.00	0.00	0.486	93.20	4.86						
31.11	100.00	0.00	0.409	88.34	8.48						
26.16	100.00	0.00	0.344	79.86	13.86						
22.00	100.00	0.00	0.289	66.00	18.71						
18.50	100.00	0.00	0.243	47.29	18.49						
15.56	100.00	0.00	0.204	28.80	15.48						
9	100.00	0.00	0.172	13.32	13.32						
0.00	100.00	0.00	0.145	0.00	0.00						

Particle Size Analysis

CUF AZ-102 Waste
UPA CUF-AZ102-24DUP

Date: 05/20/89 Meas #: 00004
Time: 09:44 Pres #: 01

CUF AZ-102 Waste

LIPA

Sample: CUF-AZ102-24 DUP

Sample: CUF-42 102-24 DUF

Volume Distribution; UPA System

Summary

```
mv = 1.457
mn = .0343
ma = .3630
cs = 16.53
sd = .9156
```

Percentiles

10% = .1731	60% = 1.696
20% = .6160	70% = 1.889
30% = 1.053	80% = 2.122
40% = 1.313	90% = 2.485
50% = 1.512	95% = 2.837

Dia	Vol%	Width
-----	------	-------

1.614	89%	1.426
.1633	3%	.0554
.0783	4%	.0452
.0341	4%	.0178

[illegible]

Particle Size Analysis

CUF AZ-102 Waste
UPA CUF-AZ102-24DUP

Date: 05/20/89 Meas #: 00004
Time: 09:44 Pres #: 01

CUF AZ-102 Waste

UPA

Sample: CUF-AZ102-24 DUP

Number Distribution; UPA System

Summary

```
mv = 1.457
mn = .0343
ma = .3630
cs = 16.53
sd = .0083
```

Percentiles

10% = .0232	60% = .0312
20% = .0246	70% = .0340
30% = .0259	80% = .0381
40% = .0275	90% = .0474
50% = .0292	95% = .0605

<u>Dia</u>	<u>Vol%</u>	<u>Width</u>
.0292	100%	.0167

[illegible]

Particle Size Analysis

CUF AZ-102 Waste
CUF-AZ102-24DUP SON

Date: 05/20/88 Meas #: 00007
Time: 10:04 Pres #: 01

CUF AZ-102 Waste
UPA

Sample: CUF-AZ102-24DUP Sonicated

Number Distribution ; UPA System

Summary

```

mv = .7494
mn = .5038
ma = .5991
cs = 10.02
sd = .0623

```

Percentiles

10% = .4183	60% = .5024
20% = .4370	70% = .5220
30% = .4532	80% = .5448
40% = .4689	90% = .5752
50% = .4848	95% = .6060

[illegible]

1.478	1%	.3440
.4840	99%	.1223

[illegible]

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