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Revision 0

SCOPING STUDIES TO DEVELOP A METHOD TO DETERMINE PARTICLE SIZE IN SIMULANT SLUDGE SLURRIES BY SIEVING

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February 7, 2005

Analytical Development Section
Savannah River National Laboratory
Aiken, SC 29808

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SAVANNAH RIVER NATIONAL LABORATORY

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EXECUTIVE SUMMARY

A physical separation method (i.e. sieving) was investigated to determine particle size distribution in non-radioactive sludge slurry simulants with the goal of implementation into the SRNL (Savannah River National Laboratory) shielded cells for use with radioactive sludge slurries. The investigation included obtaining the necessary experimental equipment, developing accessory equipment for use with the sieve shaker (to be able to sieve simulant slurries with aqueous solutions), sieving three different simulant slurries through a number of sieves and determining the particle size distribution gravimetrically, and developing a sufficient cleaning protocol of the sieves for re-use.

The experimental protocol involved successive sieving of a NIST standard (to check the particle size retention of the sieves) and three non-radioactive slurry simulants (Batch 3 Tank 40 Test 3, Tank 40 Drum 3 and CETL Sludge Batch 2, which had been previously characterized by Microtrac analysis) through smaller and smaller sieves (150 microns $\rightarrow$ 5 microns) *via* use of the 'wet' sieving system or by hand. For each of the three slurries, duplicate experiments were carried out using filtered supernate and DI water (to check the accuracy of the method versus Microtrac data) to sieve the slurry.

Particle size determinations using the wet sieving system with DI water agree well with Microtrac data on a volume basis and in some cases the sieving data may be more accurate particularly if the material sieved had large particles. A correction factor had to be applied to data obtained from experiments done with supernate due to the dissolved solids which dried upon the sieves in the drying stage of the experiments. Upon subtraction of the correction factors, the experimental results were very similar to those obtained with DI water. It should be noted that approximately 250 mL of each of three simulant slurries was necessary to have enough filtered supernate available to carry out the experiments. The experimental results for the slurries are below with Microtrac data.

- Batch 3 Tank 40 Test 3
  - ~ 85% of the particles are less than 6 microns *via* Microtrac
  - ~78% of the particles are less than 6 microns *via* sieving with supernate
  - ~68% of the particles are less than 6 microns *via* sieving with water

- Tank 40 Drum 3
  - ~71% of the particles are less than 6 microns *via* Microtrac
  - ~91% of the particles are less than 6 microns *via* sieving with supernate
  - ~85% of the particles are less than 6 microns *via* sieving with water

- CETL Sludge Batch 2
  - ~30% of the particles are less than 6 microns *via* Microtrac
  - ~30% of the particles are less than 6 microns *via* sieving with supernate
  - ~30% of the particles are less than 6 microns *via* sieving with water

The design of the experimental equipment was sufficient initially, but some pieces of the equipment began failing over time due to the caustic nature of the supernate and the vibrations from the sieve shaker. It is therefore recommended that upgrades to the experimental equipment be done before implementation into the SRNL shielded cells. Theses upgrades include using manipulator friendly connections, changing brass parts for stainless steel parts, using Teflon rather than polycarbonate, and possibly a change of pumps used to re-circulate the sieving fluid.

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LIST OF ACRONYMS

DWPF	Defense Waste Processing Facility
SB	Sludge Batch
SRNL	Savannah River National Laboratory
HDPE	High Density Polyethylene
SEM	Scanning Electron Microscopy
DI	De-Ionized
NIST	National Institute of Standards and Technology

1.0 INTRODUCTION AND BACKGROUND

For each sludge batch that is processed in DWPF (Defense Waste Processing Facility), SRNL (Savannah River National Laboratory) performs a characterization of the sludge slurry. The sludge slurry is analyzed for chemical composition, radionuclide composition and physical characteristics (weight percent solids, rheology). The information obtained from this characterization is used in radioactive demonstrations of the DWPF process (after is has been done on non-radioactive sludge simulants) to evaluate potential processing issues and to identify a DWPF processing strategy.

As stated above, there are several characteristics that can influence the processing of a sludge batch, and one of these is the rheology of the sludge slurry. Rheology measurements are performed to ensure that the slurry can be transferred and mixed properly. The flow curves (shear rate vs. shear stress) generated from the rheology measurements are modeled to obtain the yield stresses and consistencies of these samples. The yield stresses and the consistencies are then compared to the design bases for the DWPF to determine if the feed may pose potential processing problems (i.e. pumping/ transfer problems).

To help SRNL/DWPF understand the rheology (yield stress and consistency of a sludge slurry sample) of a given sludge batch the parameters that influence the rheology must be measured on both non-radioactive and radioactive sludge slurry samples (pH, temperature, chemical composition, particle size, etc.). Particle size has been shown to affect rheological behavior in studies using non-radioactive sludge slurry.[1]

Currently, there is an abundance of particle size data (via Microtrac) for the non-radioactive sludge slurries, but very little particle size information is available for radioactive sludge slurries. The information that is available for the radioactive sludge slurry has been obtained using a Microtrac instrument. During Microtrac analysis a laser beam is projected through a transparent sample cell that contains a stream of moving particles suspended in a fluid. Light rays that strike particles are scattered through angles inversely proportional to particle size. The photodetector arrays measure the quantity of light (flux) at predetermined angles. Electrical signals proportional to the measured light flux values are then processed by the computer to form a multichannel histogram of the particle size distribution. The Microtrac data obtained thus far on radioactive sludge slurries is potentially suspect, due to the large dilution required to remove the radioactive sludge slurry samples from the Shielded Cells. It is thought that this large dilution may alter the true particle size of the sludge slurry. Although Microtrac analysis may still be pursued, the need to find an alternative means to determine particle size remains. Successive sieving of fine solid samples or liquid samples containing solids through smaller and smaller sieves to separate the particles by size and obtain a distribution is a viable mechanical method. To date, there has been one sieving experiment performed on radioactive sludge slurry to determine the particle size.[2] The experiment was somewhat successful, but the majority of particles (~60%) were smaller than 20 microns. This document will describe scoping tests performed in order to develop a sieving method for determining particle size and distribution in non-radioactive sludge simulant for eventual application to radiological samples.

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2.0 EXPERIMENTAL

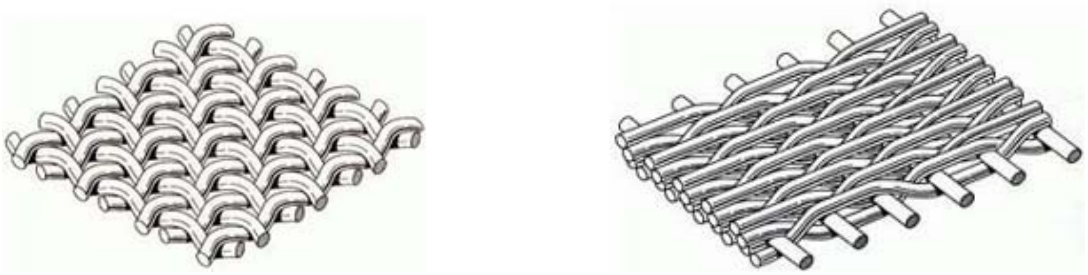
Apparatus

The Analytical Development Section of SRNL developed a wet sieving system to sieve simulant slurry. The experimental equipment is shown in Figure 2.1**.**

Figure 2.1 Experimental apparatus developed by ADS for sieving wet simulant slurry.

The system works by re-circulating slurry supernate or water, which has passed through the sieves and collected in the high density polyethylene (HDPE) sieve holder, back up to the top sieve by the use of a peristaltic pump while the sieves are being shaken by a sieve shaker. The HDPE sieve holder has a 150 mL reservoir and a hole for the use of vacuum (back left of holder). The vacuum hole is protected by a funnel inside the sieve holder so that the sieved liquid drains into the reservoir and does not get sucked up into the vacuum. There is also a small hole in the side near the bottom of the sieve holder which serves as a drain (front right of holder) so that supernate/water can be pumped out and re-circulated. The spacer ring (left of sieve holder) has two brass nozzles opposite 180º which helps to spread out the supernate/water that is re-circulating into the sieves to aid in the sieving process. The vibratory sieve

shaker is an Analysette 3 model manufactured by Fritsch. The shaker can be operated in two modes; normal and micro mode. The micro mode is used for sieves 20 microns and smaller. The sieves used during these experiments were made by Newark Wire Cloth Company and conform to ASTM specification E-11. The plain weave mesh of the sieves size 20 microns and greater result in square shape openings and below 20 microns the twill Dutch weave of the mesh results in openings which are probably more wedged or elliptical in shape. The wedged shape openings of mesh less than 20 microns makes it more difficult for particles to pass through. It is for this reason that the sieve holder was developed with a vacuum attachment. The difference in the weave of the wires that make up the mesh of the two sieves is shown pictorially in Figure 2.2.



A B

Figure 2.2 A is representative of a plain weave mesh used with sieves 20 microns and larger and results in square openings in the mesh. B is an example of the twill Dutch weave which results in wedged shaped or elliptical type openings in the mesh.

Material

The simulant sludge slurries given to ADS to perform sieving on were labeled Batch 3 Tank 40 Test 3, Tank 40 Drum 3 and CETL Sludge Batch 2. The insoluble solids content of the simulant slurries were measured and found to be 11.75%, 14.32% and 18.05%, respectively. The elemental composition of the simulant slurries are shown below in Table 2.1. The NIST 1984 Microtrac standard was provided by Wilson Smith (ADS) and is a mixture of tungsten carbide and cobalt, ranging in particle size 9.5 to 26.5 microns.

Table 2.1 Elemental composition of Batch 3 Tank 40 Test 3, Tank 40 Drum 3 and CETL SB-2 simulant slurries.

Batch 3 Tank 40 Test 3 Simulant		Tank 40 Drum 3 Simulant		CETL Sludge Batch 2 Simulant	
Element	Concentration (μg/g of slurry)	Element	Concentration (μg/g of slurry)	Element	Concentration (μg/g of slurry)
Ag	43.6	Al	12400	Al	15200
Al	12970	Ba	240	Ba	<15.0
Ba	88.5	Ca	4850	Ca	5400
Ca	3220	Cr	380	Cu	280
Cd	160	Cu	240	Fe	56900
Ce	180	Fe	47400	K	120
Cr	300	K	34.4	Mg	190
Cu	52	Mg	270	Mn	7900
Fe	34560	Mn	4280	Na	5900
Gd	91.3	Na	8610	Ni	1940
K	370	Ni	530	Si	1850
La	71.7	Pb	390	Zn	530
Li	200	Si	1900	Zr	1000
Mg	2980	Ti	<13.0		
Mn	7330	Zn	430		
Na	26550	Zr	880		
Ni	1970	Ru	<130		
P	730				
Pb	8.43				
S	280				
Sb	190				
Si	790				
Sn	<280				
Sr	710				
Ti	42.2				
Zn	71.7				
Zr	9.84				

Sieving Procedures

NIST 1984 Standard

Experiment 1.1

The NIST 1984 standard was mixed by rotating and turning the bottle end-over-end twenty times and a 1.0442 g portion was weighed into a beaker. The contents of the beaker were transferred into a 20 micron sieve which was the top sieve of a stack consisting of a pre-weighed 20 micron sieve, 16-18 micron sieve (165x1400 mesh), 12-14 micron sieve (200x1400 mesh) and an 8-9 micron sieve (325x2300 mesh), respectively. The vibratory sieve shaker was put in micro mode, set at an interval time of 10 seconds and a total sieve time of 45 minutes. No water or vacuum was used. Each sieve was re-weighed after the sieving time had ended.

Experiment 1.2

The NIST 1984 standard was mixed by rotating and turning the bottle end-over-end twenty times and a 1.0760 g portion was weighed into a beaker. The contents of the beaker were transferred into a 20 micron sieve which was the top sieve of a stack consisting of a pre-weighed and pre-wetted 20 micron sieve, 16-18 micron sieve (165x1400 mesh), 12-14 micron sieve (200x1400 mesh) and an 8-9 micron sieve (325x2300 mesh), respectively. The vibratory sieve shaker was put in micro mode, set at an interval time of 10 seconds and a total sieve time of 45 minutes. Water was re-circulated over the sieves for this experiment and vacuum was used periodically. Each sieve was dried overnight at 50 ºC and re-weighed. 50 ºC was the maximum drying temperature recommended by the manufacturer.

Experiment 1.3

The NIST 1984 standard was mixed by rotating and turning the bottle end-over-end twenty times and a 1.0286 g portion was weighed into a beaker. The contents of the beaker were transferred into a 25 micron sieve which was the top sieve of a stack consisting of a pre-weighed and pre-wetted 25 micron sieve, 20 micron sieve, 12-14 micron sieve (200x1400 mesh), 11-12 micron sieve (250x1400 mesh) and an 8-9 micron sieve (325x2300 mesh), respectively. The vibratory sieve shaker was put in micro mode, set at an interval time of 10 seconds and a total sieve time of 10 min. At the end of 10 minutes, the top sieve was rinsed and removed from the stack. The sieve system was re-assembled and sieving was commenced for an additional 10 minutes. This pattern was repeated down to the last sieve. Water was re-circulated over the sieves for this experiment and vacuum was used periodically. Each sieve was dried overnight at 50 ºC and re-weighed. Solids collected were submitted for Microtrac/SEM analysis.

Experiment 1.4

The NIST 1984 standard was mixed by rotating and turning the bottle end-over-end twenty times and a 1.1460 g portion was weighed into a beaker. The contents of the beaker were transferred into a pre-weighed 25 micron sieve. The sieve was shaken by hand and rinsed with ~ 50 mL of DI water. The particles that went through were collected in the beaker and then poured onto the next sieve, a pre-weighed 20 micron sieve. This pattern was continued through a pre-weighed 16-18 micron sieve (165x1400 mesh), 12-14 micron sieve (200x1400 mesh), 11-12 micron sieve (250x1400 mesh) and an 8-9 micron sieve (325x2300 mesh), respectively. The last two sieves were set in the HDPE holder and required the use of vacuum to get the water to pass through the sieves but they were still hand shaken. Each sieve was dried overnight at 50 ºC and re-weighed. Solids collected were submitted for Microtrac/SEM analysis.

Batch 3 Tank 40 Test 3

Experiment 2.1

5.1150 g of simulant slurry was added to a weighing cup, diluted in 50 mL of water and poured into a pre-weighed pre-wetted 38 micron sieve which was the top sieve of a stack consisting of the pre-weighed and pre-wetted (with DI water) 38 micron sieve, a 32 micron sieve and a 25 micron sieve. The vibratory sieve shaker was put in micro mode, set at an interval time of 10 secs and a total sieve time of 10 min while water was re-circulating. The top sieve was then rinsed and removed. The wet sieving system was re-assembled and the sieves where shaken for an additional 10 mins. This pattern was repeated down to the last sieve in the three sieve stack. The particles in the slurry which collected in the HDPE reservoir were then sieved through another stack of pre-weighed and pre-wetted sieves which began with a 20 micron sieve, then a 16-18 micron sieve (165x1400 mesh), and then a 12-14 micron sieve (200x1400 mesh),

respectively. The process was continued through the last three sieves, an 11-12 micron sieve (250x1400 mesh), an 8-9 micron sieve (325x2300 mesh), and ending with a 5-6 micron sieve (510x3600 mesh), respectively. Water was re-circulated over the sieves for this experiment and vacuum was used periodically. Each sieve was dried overnight at 50 ºC and re-weighed.

Experiment 2.2

5.0528 g of slurry simulant was added to a weighing cup, diluted in 50 mL of water and then poured into a pre-weighed pre-wetted 38 micron sieve which was the top sieve of a stack consisting of the pre-weighed and pre-wetted 38 micron sieve, a 32 micron sieve and a 25 micron sieve. The vibratory sieve shaker was put in the micro mode, set at an interval time of 10 secs and a total sieve time of 10 min while water was re-circulating. The top sieve was then rinsed and removed. The wet sieving system was re-assembled and the sieves where shaken for an additional 10 mins. This pattern was repeated down to the last sieve in a three sieve stack. The particles in the slurry which collected in the HDPE reservoir were then poured into a clean sieve that was the size of the last sieve screen in the previous stack (25 microns) and then sieved through another stack of pre-weighed and pre-wetted sieves beginning with 20 micron sieve, a 16-18 micron sieve (165x1400 mesh), and a 12-14 micron sieve (200x1400 mesh), respectively. The sieves were shaken for 10 min while water was re-circulating and then the top sieve was rinsed and removed. The wet sieving system was re-assembled and the sieves where shaken for 10 mins again. This pattern was repeated down to the last sieve in a three sieve stack. A similar pattern was repeated for the last three sieves (11-12 micron sieve (250x1400 mesh), an 8-9 micron sieve (325x2300 mesh), a 5-6 micron sieve (510x3600 mesh)) except that 0.5983 g of simulant slurry was diluted in 50 mL of water. Each sieve was dried overnight at 50 ºC and re-weighed.

Experiment 2.3

5.2097 g of slurry simulant was added to a weighing cup, diluted in 30 mL of water and then poured into a pre-weighed, pre-wetted 38 micron sieve held over a beaker. The sieve was shaken by hand and rinsed. The particles that went through were collected in the beaker and then poured onto the next sieve, a pre-weighed, pre-wetted 32 micron sieve and then a 25 micron sieve. 1.147 g was used for the next three sieves which consisted of a 20 micron sieve, a 16-18 micron sieve (165x1400 mesh), and a 12-14 micron sieve (200x1400 mesh). 0.5312 g of slurry simulant were used for the last three sieves which consisted of an 11-12 micron sieve (250x1400 mesh), an 8-9 micron sieve (325x2300 mesh), and a 5-6 micron sieve (510x3600 mesh), respectively. The last three sieves were set in the HDPE holder and required use of vacuum to get the water to pass through the sieves but they were still hand shaken. Each sieve was dried overnight at 50 ºC and re-weighed. Data obtained from experiments 2.1 -2.3 are contained in Table 1.

Experiment 3.1

5.0433 g of slurry simulant was added to a weighing cup and diluted in 15 mL of previously filtered supernate (by use of a 0.43 micron filter) and then poured into a pre-weighed pre-wetted 38 micron sieve which was the top sieve of a stack consisting of the pre-weighed and pre-wetted 38 micron sieve, a 32 micron sieve and a 25 micron sieve. The vibratory sieve shaker was put in the micro mode, set at an interval time of 10 secs and a total sieve time of 10 min. Supernate was re-circulated during the sieving. After 10 mins the top sieve was rinsed with ~10 mL of fresh previously filtered supernate and then removed. The wet sieving system was re-assembled and the sieves where shaken for 10 mins again. This pattern was repeated down to the last sieve in a three sieve stack. The slurry solution which collected in the HDPE reservoir was mixed with an additional 20 mL of filtered supernate and then sieved through another stack of pre-weighed and pre-wetted sieves beginning with a 20 micron sieve, then a 16-18 micron sieve (165x1400 mesh), and then a 12-14 micron sieve (200x1400 mesh), respectively. The same

process was repeated with the last three sieves; an 11-12 micron sieve (250x1400 mesh), an 8-9 micron sieve (325x2300 mesh), and ending with a 5-6 micron sieve (510x3600 mesh). Vacuum was used periodically for the smaller sized sieves. The bottom of each sieve screen was dabbed with a Kimwipe™ to absorb excess supernate on the sieve screen before drying overnight at 50 ºC. The sieves were then re-weighed.

Experiment 3.2

5.0454 g of slurry simulant was added to a weighing cup, diluted in 15 mL of previously filtered supernate and then poured into a pre-weighed pre-wetted 38 micron sieve which was the top sieve of a stack consisting of the pre-weighed and pre-wetted 38 micron sieve, a 32 micron sieve and a 25 micron sieve. The vibratory sieve shaker was put in the micro mode, set at an interval time of 10 secs and a total sieve time of 10 min. The slurry solution that collected in the HDPE holder was filtered through a 0.45 micron filter and then re-circulated through the sieves. This eliminated the need to keep using additional filtered supernate and the sieving was easier with 'clean' supernate solution. After 10 mins the top sieve was removed. The wet sieving system was re-assembled and the sieves where shaken for 10 mins again. This pattern was repeated down to the last sieve in a three sieve stack. The final resulting slurry solution which collected in the HDPE reservoir was filtered through a 0.45 micron filter and used with the next set of sieves. Next, 1.0427 g of slurry simulant was added to a weighing cup, diluted in 15 mL of previously filtered supernate and then poured into a clean sieve, that was the size of the last sieve screen in the previous stack (25 microns), which was the top sieve of a four sieve stack which also had a pre-weighed, pre-wetted 20 micron sieve, a 16-18 micron sieve (165x1400 mesh), and a 12-14 micron sieve (200x1400 mesh), respectively. The pattern was repeated using the last three sieves (11-12 micron sieve (250x1400 mesh)), an 8-9 micron sieve (325x2300 mesh), and a 5-6 micron sieve (510x3600 mesh) except 0.5198 g of slurry was diluted in 15 mL of previously filtered supernate and then sieved. Vacuum was used periodically for the smaller sized sieves. The bottom of each sieve screen was dabbed with a Kimwipe™ to absorb excess supernate on the sieve screen before drying overnight at 50 ºC. The sieve was then re-weighed.

Experiment 3.3

5.1435 g of slurry simulant was added to a weighing cup, diluted in 15 mL of previously filtered supernate and then poured into a pre-weighed pre-wetted 38 micron sieve which was the top sieve of a stack consisting of the pre-weighed and pre-wetted 38 micron sieve, a 32 micron sieve and a 25 micron sieve. The vibratory sieve shaker was put in the micro mode, set at an interval time of 10 secs and a total sieve time of 10 min. The slurry solution that collected in the HDPE holder was filtered through a 0.45 micron filter and then re-circulated through the sieves. This eliminated the need to keep using additional filtered supernate and the sieving was easier with 'clean' supernate solution. After 10 mins the top sieve was removed. The wet sieving system was re-assembled and the sieves where shaken for 10 mins again. This pattern was repeated down to the last sieve in a three sieve stack. The final resulting slurry solution which collected in the HDPE reservoir was filtered through a 0.45 micron filter and used with the next set of sieves. This pattern was repeated for the next three sieves (20 micron sieve, a 16-18 micron sieve (165x1400 mesh), and a 12-14 micron sieve (200x1400 mesh)) except 1.3053 g of slurry simulant was diluted in 15 mL of previously filtered supernate and then sieved. This same pattern was repeated for the final three sieves (11-12 micron sieve (250x1400 mesh), an 8-9 micron sieve (325x2300 mesh), and a 5-6 micron sieve (510x3600 mesh)) except 0.5694 g of slurry simulant was added to a weighing cup, diluted in 15 mL of previously filtered supernate and then sieved. The bottom of each sieve screen was dabbed with a Kimwipe™ to absorb excess supernate on the sieve screen before drying overnight at 50 ºC. The sieve was then re-weighed. Note: In experiment 3.3, each time more slurry was sieved it was not sieved

through the smallest screen of the previous stack. A mathematical subtraction was used to account for the insoluble solids that should be removed from the prior sieve screen.

Experiments 3.3, 4.1-4.2, 5.1-5.2, 6.1-6.2 and 7.1-7.2 which were performed with Batch 3 Tank 40 Test 3, Tank 40 Drum 3 or CETL Sludge Batch 2 slurry simulants were done in a manner analogous to Experiment 3.3 except that the sieving done with water as the re-circulating fluid was not done using the sieve shaker but by hand. Experiments 4.2-4.2 and 5.1-5.3 were done with Tank 40 Drum 3 simulant and the following sieves in microns were used : 32, 25, 20, 16-18 (165x1400 mesh), 12-14 (200x1400 mesh), 11-12 (250x1400 mesh), 8-9 (325x2300 mesh), 6-7 (450x2750 mesh) and a 5-6 (510x3600 mesh). CETL Sludge Batch 2 was sieved with the following size sieves (in microns): 150, 75, 45, 32, 25, 16-18 (165x1400 mesh), 12-14(200x1400 mesh), 11-12 (250x1400 mesh), 8-9 (325x2300 mesh), 6-7 (450x2750 mesh) and a 5-6 (510x3600 mesh). See Appedices A-D for details.

Experiments 3.4, 5.3 and 7.3

The results from Experiments 3.1-3.3, 5.1-5.2 and 7.1-7.3 were skewed due to the excess weight of the dissolved solids which became stuck on the sieves when they were dried after the sieving was complete. Therefore, a dissolved solids correction factor was needed and arrived at from the following experiments.

Pre-weighed sieves of the same size as those used in experiments 3.1-3.3, 5.1-5.2 and 7.1-7.2 were stacked and then corresponding supernate from Batch 3 Tank 40 Test 3, USC Tank 40 or CETL Sludge batch 2 simulant was poured through the sieves and allowed to drain out. The sieves were shaken off by hand then dabbed with a Kimwipe™ before being dried overnight at 50ºC. The sieves were re-weighed. This progression was repeated twice. The average weight of dissolved solids that collected on each size sieve was then subtracted from the difference of weight obtained before and after sieving the simulant slurry with supernate except for Experiment 5.3 in which case only one set of data from the dissolved solids experiment was used. Please see the appropriate data table in Appendix B for specific details.

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3.0 RESULTS

NIST 1984 Standard

Because of the different weave of the mesh of the sieves it was necessary to 'calibrate' the sieves or determine the size of particles being retained on each of the different sieves, if possible. The NIST 1984 standard, which is used for Microtrac analysis, was chosen because of its size distribution, irregularly shaped particle morphology, resistance to fracture on handling and low level of aggregation. The micro sieves used in experiments 1.1-1.4 include the 25 micron, 20 micron, 16-18 micron (165x1400 mesh), 12-14 micron (200x1400 mesh), 11-12 micron (250x1400 mesh) and 8-9 micron (325x2300 mesh) size sieves. Solids collected from the 12-14 micron sieve (200x1400 mesh), 11-12 micron sieve (250x1400 mesh), and 8-9 micron sieve (325x2300 mesh) (from experiments 1.3 or 1.4) were submitted for Microtrac analysis and the results are contained in Appendix D. There were not enough solids on the 16-18 micron sieve to submit for Microtrac or SEM analysis. An additional experiment was done in which the NIST standard was sieved through the 8-9 micron sieve and then through a 5-6 micron sieve to test the retention of the 5-6 micron sieve. Particles collected on the 5-6 micron sieve were submitted for Microtrac analysis. Microtrac data indicated the following:

- For the 20 micron sieve ~90 of the particles were larger than 21 microns on a volume basis.
- For the 12-14 micron sieve 100% of the particles were above 15 microns on a volume basis.
- For the 11-12 micron sieve ~90% of the particles were above 12 microns on a volume basis.
- For the 8-9 micron sieve ~90% of the particles were above 9 microns on a volume basis.
- For the 5-6 micron sieve 100% of the particles were above 6 microns on a volume basis.

SEM data collected on the solids on the 25 micron sieve indicates 90% particles (based on 50 randomly chosen and measured particles) were greater than 25 microns. This is illustrated in Figure Figure 3.1.

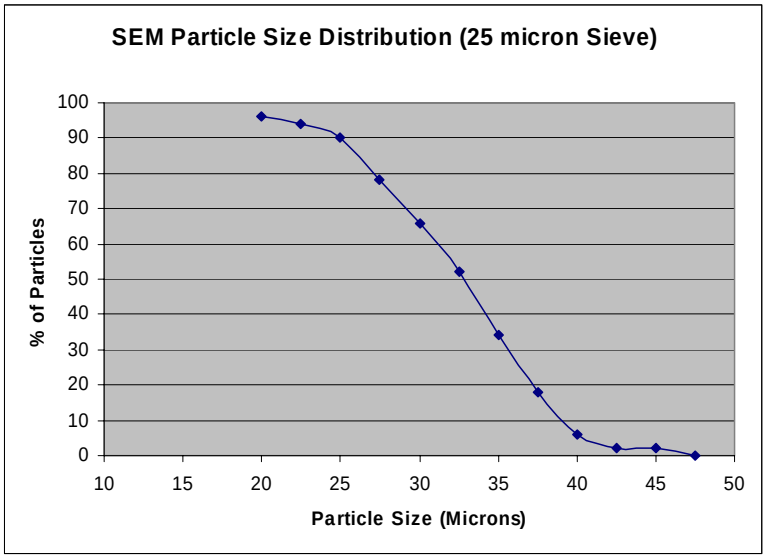

Figure 3.1 SEM analysis of particles collected on 25 micron sieve. ~90% of the particles are above 25 microns. Fifty randomly chosen particles were measured and graphed.

Shown in Figure 3.2 is a SEM of the 1984 NIST standard in which one can clearly see the different morphology of the particles. This lends more credence for using the NIST standard to calibrate the size of particles being retained by each sieve because the particles in sludge slurry are probably not all round.

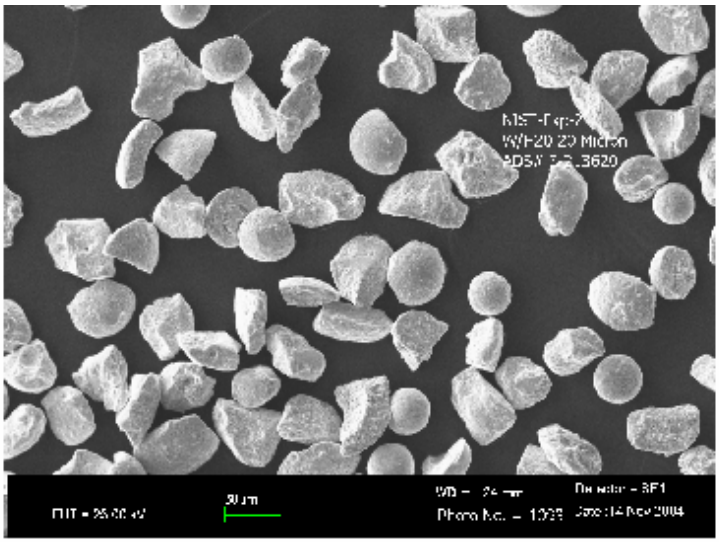

Figure 3.2 SEM of particles collected on 20 micron sieve screen.

A picture of the dried sieves used in Experiment 1.3 is shown in Figure 3.3 below and the experimental results obtained from sieving the NIST 1984 standard are shown in Figure 3.4.

Figure 3.3 Dried sieves used in experiment 1.3. From top left to right, 25 micron sieve, 20 micron sieve, 12-14 micron sieve (200x1400 mesh), 11-12 micron sieve (250x1400 mesh) and an 8-9 micron sieve (325x2300 mesh), respectively.

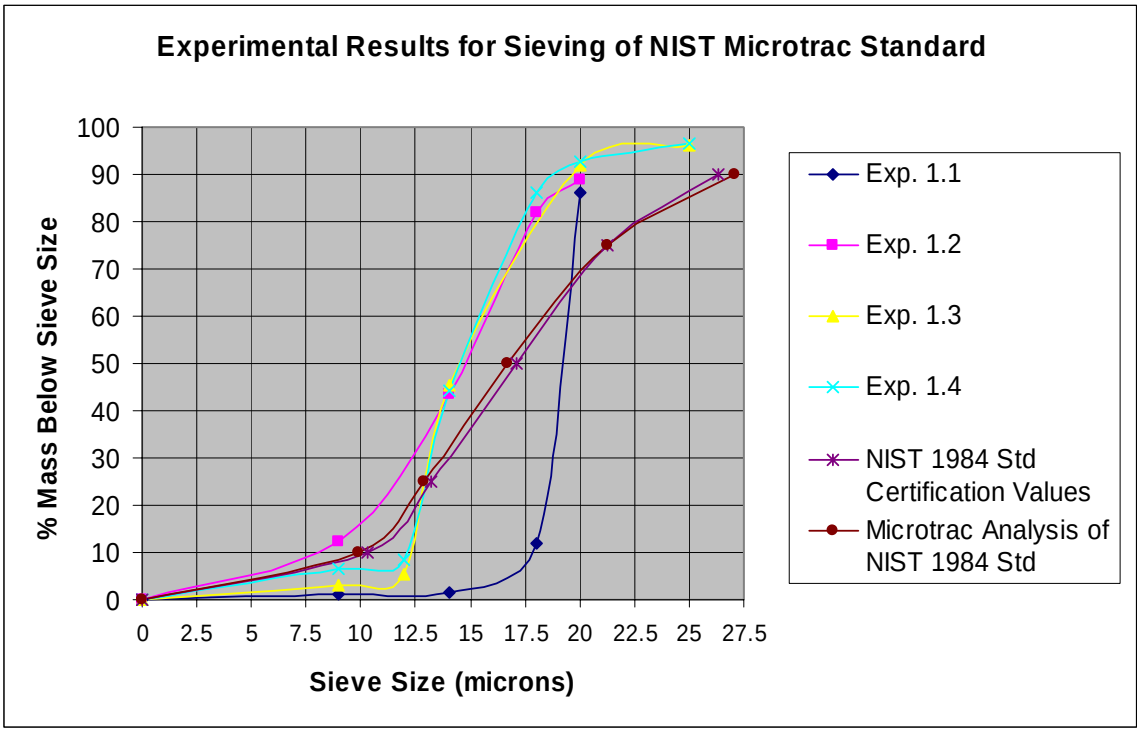

Figure 3.4 Experimental results obtained from sieving NIST1984 standard.

Experiment 1.1 was performed without the use of water during the sieving process. Experiments 1.2-1.4 were performed with water and the time of sieving varied. However, the results were consistent regardless of sieving time. The sieve sizes used to graph experiments 1.1-1.4 is based upon the manufacturer's upper size limit given for the sieve and is supported by Microtrac data. In other words, the sieve size used for the 12-14 micron sieve was 14. At larger micron values, the graph in Figure 4 indicates that the % mass fraction of the sample below 20 microns is higher than Microtrac data indicates. One possible reason for this may be that the standard is being broken into smaller particles during the

sieving process. However, these experiments were done both by hand and with the use of a sieve shaker and because the results obtained were very similar this seems unlikely. The important point is that the sieves are retaining particles larger than the specified micron size of the sieves.

Batch 3 Tank 40 Test 3 Simulant Slurry

Experiments 2.1-2.2 were performed with the use of a sieve shaker. Experiment 2.3 was performed by hand using water to rinse the slurry through the sieves. For specific details see the experimental section and data contained in Appendix A. The graph in Figure 3.5 highlights three important findings. The first is that a sieving experiment using over five grams of slurry blinded the smaller size sieves (Exp. 2.1) and vacuum was employed which tended to stick the particles to the sieve and no amount of rinsing could loosen them. The second important feature is that the experimental results are reproducible whether they are done with the use of a sieve shaker or by hand. The third feature is that in experiments 2.1-2.3 ~18% of the insoluble solids were larger than 38 microns yet this is not reflected in the Microtrac data. There are two possible reasons for this. One is that a representative sample was not put into the Microtrac instrument. The other possible reason is the large dilution done for Microtrac analysis. The large dilution changes the pH of the sample and begins to dissolve $M_n(OH)_x$ and M_nO_x species. If a large dilution of the slurry is done and more 'marbles' are created as compared to the number of 'boulders' present, then the volume data will be skewed and indicate there is a larger volume of small particles present. Smaller particles also cause more laser light scattering which skew the data further. The sieving experiments, on the other hand were performed with a much larger sample size and a much smaller dilution. The boulders in the present case are sand and are similar to the solids collected on the largest sieve shown in Figure 3.6.

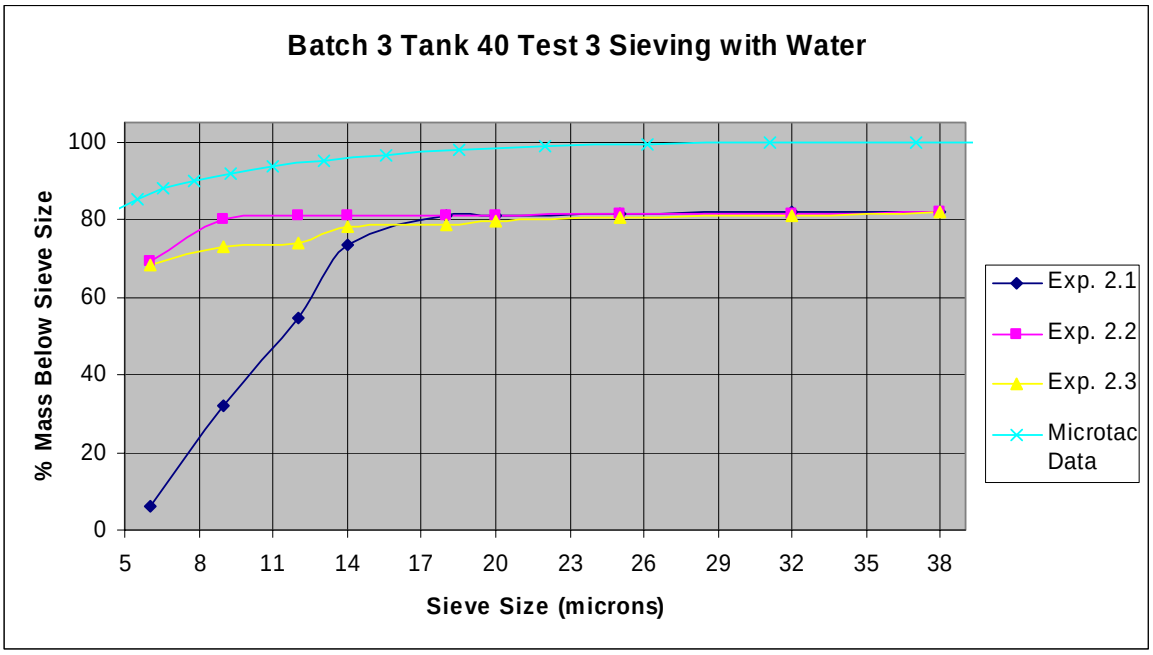

Figure 3.5 Batch 3 Tank 40 Test 3 simulant slurry sieving results using water as re-circulating fluid or as the rinsing solution if done by hand (% Mass Below Sieve Size *vs* Sieve Size (microns)).

Experiments 3.1-3.3 were performed using a sieve shaker and supernate as the re-circulating fluid. For specific details see the experimental section. A picture of the dried sieves used in experiment 3.2 is shown in Figure 3.6 and the experimental results are graphed in Figure 3.7.

Figure 3.6 Sieves used in experiment 3.2. From top left to right 38 micron sieve, 32 micron sieve, 25 micron sieve, 20 micron sieve, 16-18 micron sieve (165x1400 mesh), 12-14 micron sieve (200x1400 mesh), 11-12 micron sieve (250x1400 mesh), 8-9 micron sieve (325x2300 mesh), and a 5-6 micron sieve (510x3600 mesh), respectively.

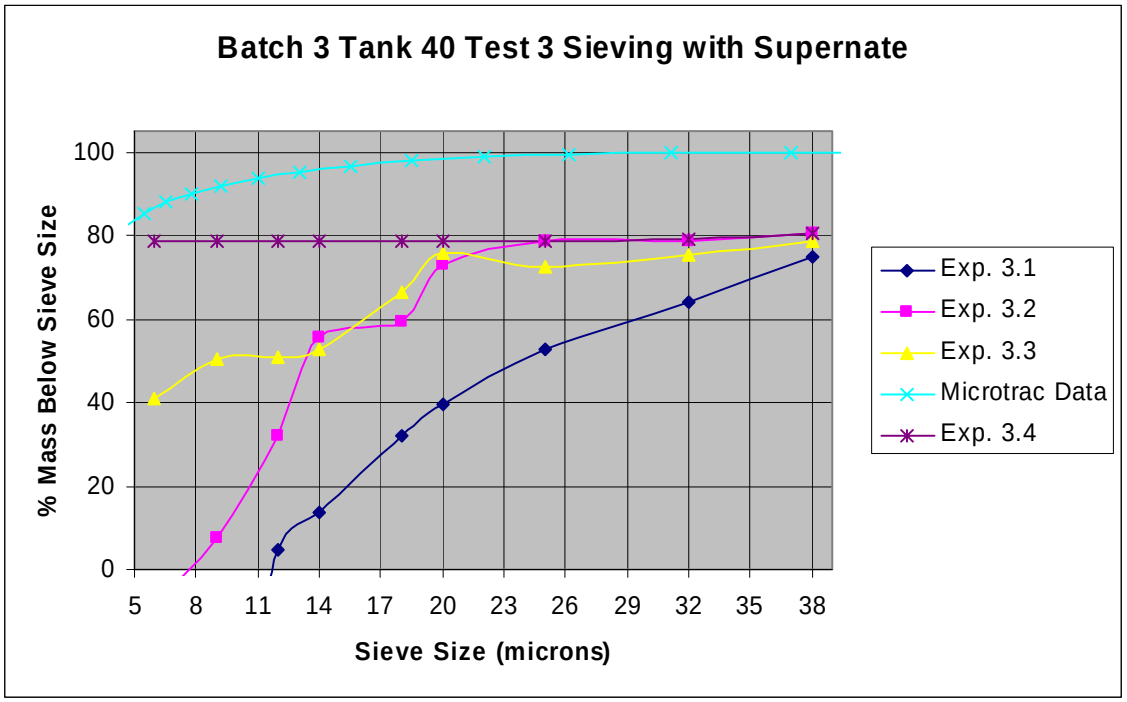

Figure 3.7 Batch 3 Tank 40 Test 3 simulant slurry sieving results using supernate as re-circulating fluid (% Mass Below Sieve Size *vs* Sieve Size (microns)). Experiment 3.4 is the corrected and averaged data based upon soluble solids in the supernate.

The results from experiment 3.1 indicate that too much slurry was used and when vacuum was employed it tended to stick the particles to the sieve and no amount of rinsing could loosen them. The results from all three experiments (3.1-3.3) were poor due to residual supernate solids being deposited on the sieves. The extra mass is attributed to dissolved solids present in the supernate. The dissolved solids become even more of a problem with smaller sieves. It should be noted that if a negative value was obtained for the weight difference between the clean dry sieve and the dry sieve after sieving, its contribution was counted as zero for the % of insoluble solids on the sieve. However, once the dissolved solids are subtracted out, the results appear to be much better. Exp. 3.4 is an average of experiments 3.1 and 3.2 with the average (from two experiments) dissolved solids subtracted out. It should be noted that when the dissolved solids were subtracted out, if a negative value was obtained it was counted as zero when the mass loss was subtracted from 100 to obtain the % mass below a specific sieve size (y-axis). This may be a point of debate (see Appendix C). The resulting data from Experiment 3.4 is closer to the results obtained when sieving with water (see Experiments 2.2-2.3 in Figure 3.5). Microtrac data is added for comparison.

Tank 40 Drum 3 Simulant Slurry

Experiments 4.1-4.2 and 5.1-5.3 were performed using Tank 40 Drum 3 simulant. This simulant slurry has a bimodal distribution of particles (based on Microtrac data) and there was interest in determining if the results obtained from sieving of the simulant slurry would reflect this type of distribution. Experiments 4.1-4.2 were done in a similar manner to Experiments 2.1-2.2; the slurry was rinsed through the sieves using DI H_2O. For specific details see the experimental section and Appendices A-C. The experimental results are graphed in Figure 3.8.

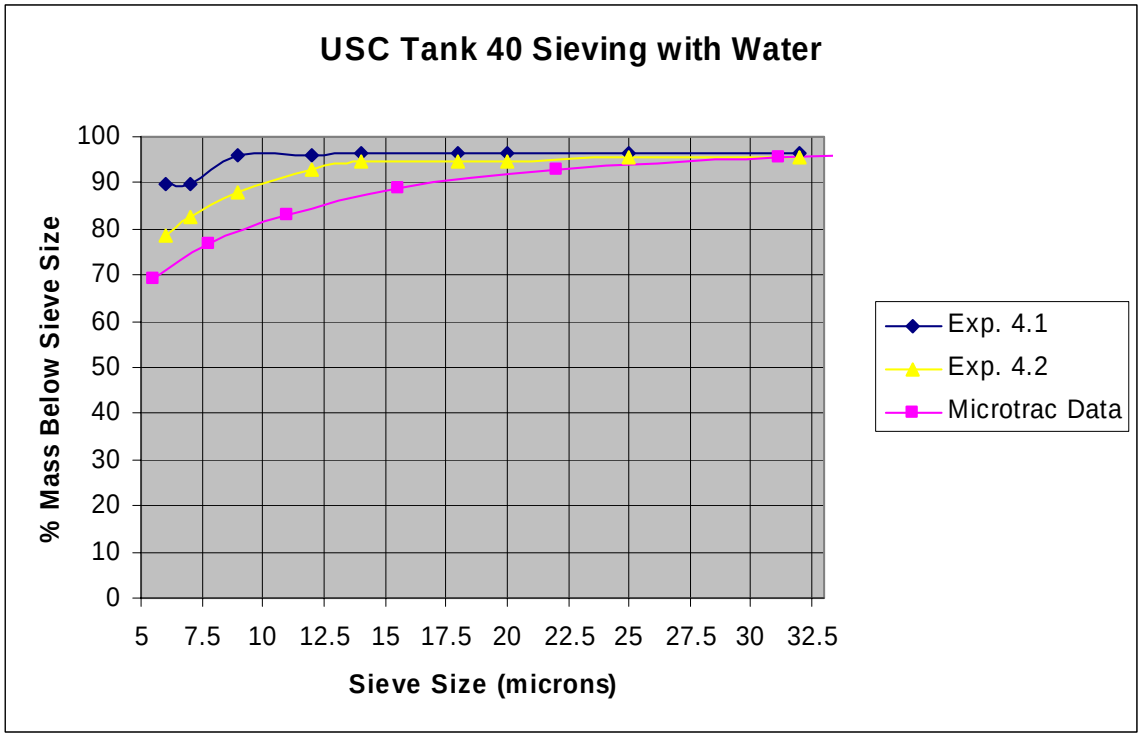

Figure 3.8 Tank 40 Drum 3 simulant sieving results using water as re-circulating fluid (% Mass Below Sieve Size *vs* Sieve Size (microns)).

Experiments 4.1 and 4.2 show good reproducibility and trend in the same direction as the Microtrac data. As stated earlier this need not be the case because the conditions of analysis are very different. Unfortunately, the smallest sieve available was the 5-6 micron sieve and the second node of the bimodal distribution was under 5 microns and it not reflected in Figure 3.8.

Experiments 5.1-5.2 were done in an analogous manner to Experiments 4.1 and 4.2 except a sieve shaker was used during the sieving process and supernate was used as the re-circulating fluid. Experiment 5.3 was done a similar manner to Experiment 3.4. For specific details see Appendices A-C and the experimental section. The experimental results are graphed in Figure 3.9.

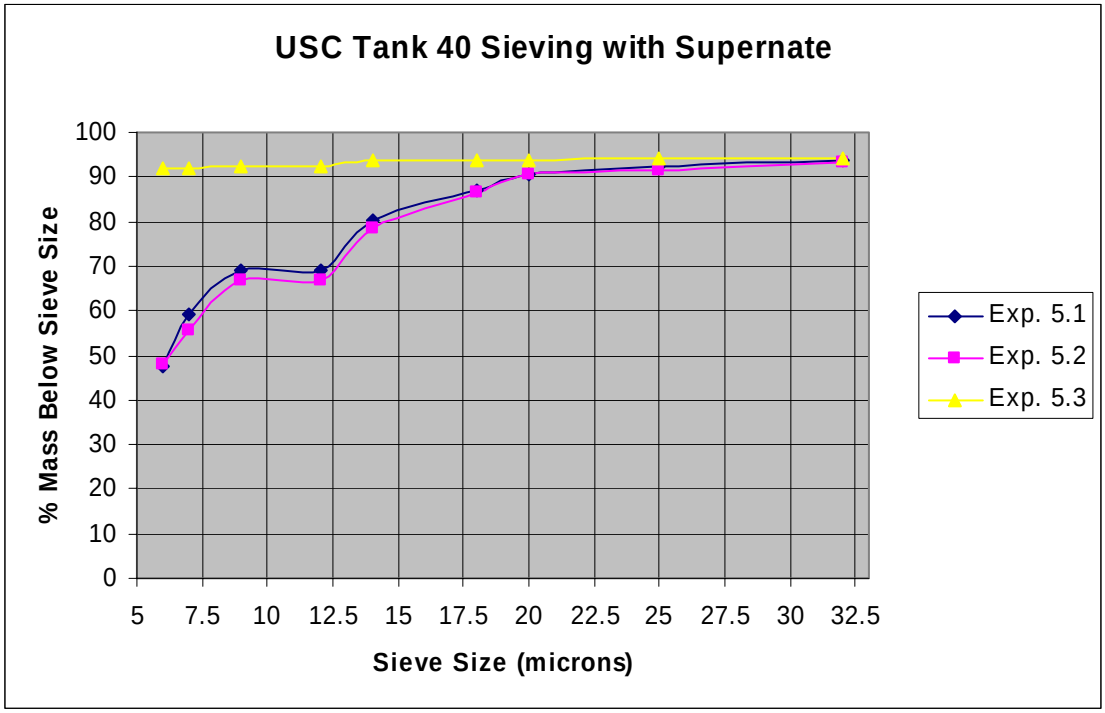

Figure 3.9 Tank 40 Test 3 simulant sieving results using supernate as re-circulating fluid (% Mass Below Sieve Size *vs* Sieve Size (microns)). Experiment 5.3 is the corrected and averaged data based upon soluble solids in the supernate.

Experiments 5.1-5.2 show good reproducibility, which also means the sieves are being cleaned well between uses. Again in Figure 11, the bimodal size distribution of particles in Tank 40 Drum 3 simulant slurry is not evident. The substantial mass increase on the smaller sieves due to the dissolved solids in the samples is evident. Once the data was corrected for dissolved solids (Exp. 5.3) the resulting % mass below sieve size (y-axis) increased substantially. The corrected data is more similar to that seen in Experiments 4.1-4.2. The corrected average is still within 20% of the Microtrac data.

CETL Sludge Batch 2 Slurry Simulant

Experiments 6.1-6.2 and 7.1-7.3 were performed using CETL Sludge Batch 2 slurry simulant. This simulant slurry also has a bimodal size distribution of particles. Experiments 6.1-6.2 were performed by hand using water to rinse the re-circulating fluid through the sieves. Also, ~ 10 grams of simulant slurry was used for the first three sieves because of their larger size and the larger particle size based on

Microtrac data. The experimental results are graphed in Figure 3.10. For specific details see Appendices A-C and the experimental section.

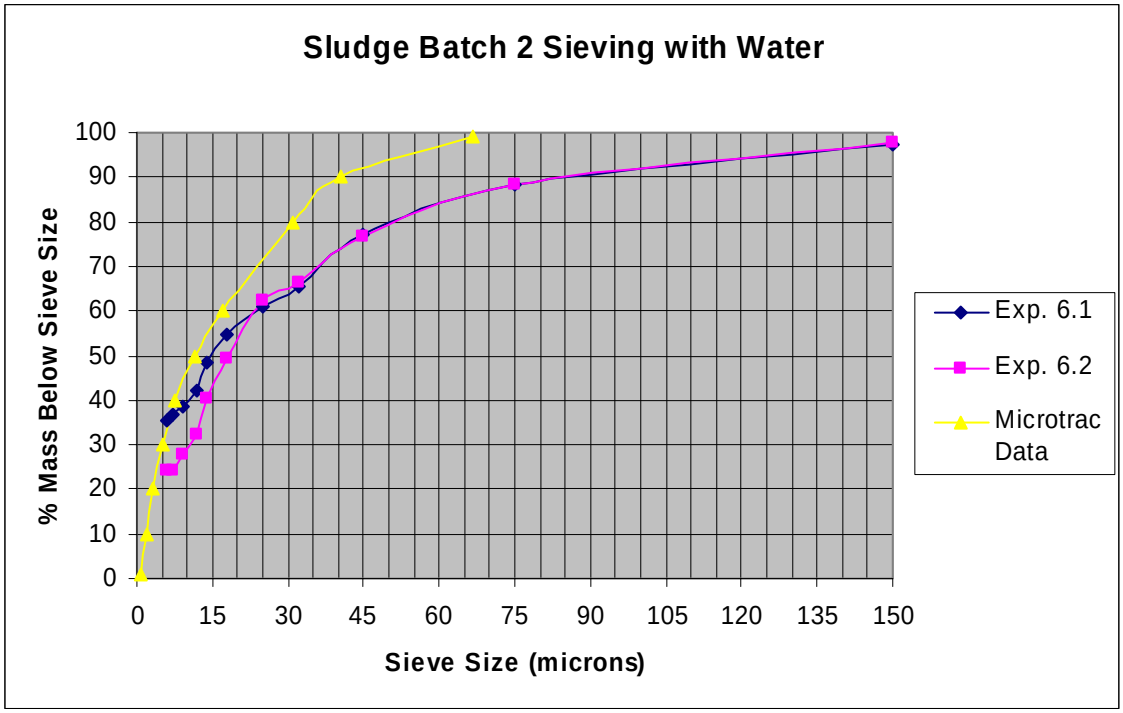

Figure 3.10 CETL Sludge Batch 2 simulant sieving results using water as re-circulating fluid (% Mass Below Sieve Size *vs* Sieve Size (microns)).

Good reproducibility was also seen with this simulant slurry in Experiments 6.1-6.2. The bimodal distribution may be slightly evident in the decrease of the slope in the lines between ~32 microns and 25 microns. This decrease reflects the same decrease in seen in the % channel information from Microtrac data. However, if we did not have advance knowledge of the bimodal distribution, it would be impossible to pick out such a subtlety and reliably say that is was due to a bimodal distribution. Again, a majority of the particles in the second node of the bimodal distribution were smaller than the smallest size sieve.

Experiments 7.1-7.2 were performed in a similar manner, and using the same size sieves, as Experiments 6.1-6.2 except the sieve shaker was used and supernate was used as the re-circulating fluid. In addition, ~5 grams of slurry was used for the largest three sieves (150 microns, 75 microns, 45 microns, 32 microns) instead of ~10 grams. The weight of the slurry for the reaming sieving was similar to Experiments 6.1-6.2. The experimental results are graphed in **Figure 3.11**. For specific details see Appendices A-C

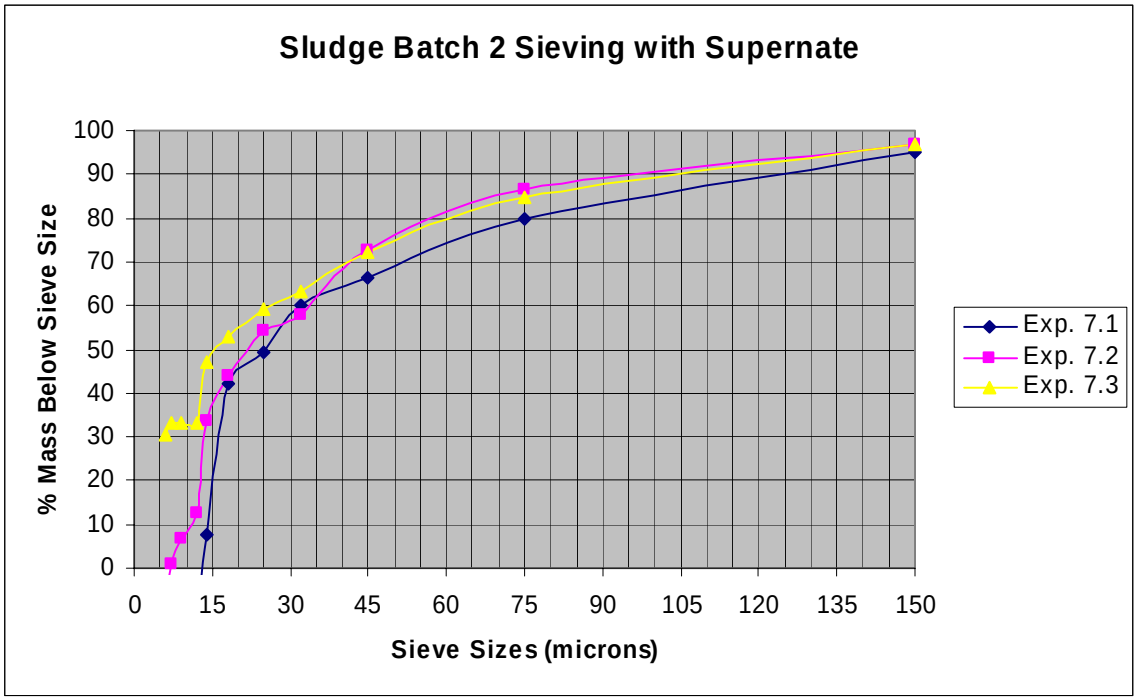

Figure 3.11 CETL Sludge Batch 2 simulant sieving results using supernate as re-circulating fluid (% Mass Below Sieve Size *vs* Sieve Size (microns)).

Experimental data from Experiments 7.1-7.2 contained large negative percentages for the % mass below the smallest sieve size due to the dissolved solids drying on the sieves when using supernate as the re-circulating fluid. However upon subtraction of the dissolved solids, the average line (Exp. 7.3) is in good agreement with Microtrac data and the data from the experiments done using water as the re-circulating fluid. The bimodal distribution of particles in the simulant slurry cannot be seen in Experiments 7.1-7.2 nor is it evident in the dissolved solids corrected data (Exp. 7.3).

Sieve Cleaning

The sieves were cleaned by removing as much dried slurry as possible using a water rinse. Then, the sieves were submerged in 2 volume % nitric acid solution for a period of five minutes, rinsed with DI water and transferred to a 1 M oxalic acid solution for five minutes. The sieves were then rinsed with DI water and placed sieve foil side up in a Sonicator bath for no longer than three minutes (damage may result to the sieves if left in Sonicator longer than three minutes). The sieves were again rinsed with DI water and if there was any resulting residue on the sieves, the process was repeated. In many cases the sieve weight did not change except in the fourth decimal place between experimental runs. Also, the repeatability of the experiments suggests the sieve cleaning protocol is sufficient and can be implemented in the shielded cells.

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4.0 CONCLUSIONS

Sieving simulant sludge slurries to determine approximate particle size distribution appears to be a viable option to Microtrac. However, only **volume** data can be obtained using the sieving method whereas Microtrac analysis gives both **volume** and **number** data. The experimental protocol which involved duplicate sieving experiments of a NIST standard (to check the particle size retention of the sieves) and three non-radioactive slurry simulants were carried out using DI water only and filtered supernate and DI water (to check the accuracy of the method versus Microtrac data), respectively.

Particle size determinations using the wet sieving method with simulant slurry and filtered supernate as the re-circulating fluid resulted in data which needed to be corrected.
However, upon subtracting out the average dissolved solids left on the sieves, as a result of the supernate, the results were comparable to that obtained from sieving with water. It should be noted that approximately 250 mL of each of three simulant slurries was necessary to have enough filtered supernate available to carry out the experiments. Therefore, depending upon the size of the sample using supernate to sieve may not be viable.

The design of the experimental equipment was sufficient initially but some pieces of the equipment began failing over time due to the caustic nature of the supernate and the vibrations from the sieve shaker. It is therefore recommended that upgrades to the experimental equipment be done before implementation into the SRNL shielded cells. Theses upgrades include using manipulator friendly connections, changing brass parts for stainless steel parts, using Teflon rather than polycarbonate, and possibly a change of pumps used to re-circulate the sieving fluid.

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5.0 RECOMMENDATIONS/PATH FORWARD

Based upon the results obtained, it is recommended that this method be implemented in the shielded cells of SRNL upon upgrades and approval from ITS and it should be used whenever particle size determination is necessary on a volume basis. Consideration should also be given to combining the sieving of particles with SEM analysis. By doing this, a better particle size distribution of the particles collected on the sieves can be attained. However, this may not be possible when working with samples that pose a radiological hazard.

The upgrades necessary before this can be done include using manipulator friendly hose connections, changing brass parts for stainless steel parts, using Teflon rather than polycarbonate, and possibly a change of pumps used to re-circulate the sieving fluid.

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6.0 REFERENCES

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[2]T.L. Fellinger, "Results of the Shielded Cells Sludge Batch 2 Phase II Testing SRT-GDP-2003-00078.

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7.0 ACKNOWLEDGEMENTS

The author would like to acknowledge ITS (Immobilization Technology Section) for use of lab space to perform the experiments, Terri Fellinger (for weight percent solids information, technical review and helpful discussions), Frank Pennebaker (spreadsheet review), David Koopman (for material characterization data), Wilson Smith (for providing Microtrac analysis and discussions), Mike Summer (for providing SEM data and discussions) and Don Blankenship (for helpful discussions).

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APPENDIX A

DATA TABLES FROM EXPERIMENTS 2.1-2.3, 3.1-3.3, 4.1-4.2, 5.1-5.2, 6.1-6.2 AND 7.1-7.2

Table A.1 Experimental Data from Experiments 2.1 - 2.3.

Exp. 2.1 Sieved with Water Using Shaker			Total Sample Used (g) = Insoluble Solids (g) =		5.1150 0.6010
Sieve Size (microns or mesh)	Dry Wt of Sieve (g)	Wt. After Sieving Slurry and Drying (g)	Dry Wt of Sieve (g) - Wt. After Sieving Slurry and Drying (g)	% of Insoluble Solids Collected on Sieve	% of Solids Below Sieve Size
38	80.5326	80.6399	0.1073	17.85	82.15
32	80.5693	80.5718	0.0025	0.42	81.73
25	72.9787	72.9808	0.0021	0.35	81.38
20	71.4555	71.4567	0.0012	0.20	81.18
16-18 (165x1400)	84.3403	84.3402	-0.0001	0.00	81.18
12-14 (200x1400)	75.3484	75.3960	0.0476	7.92	73.28
11-12 (250x1400)	81.2208	81.3317	0.1109	18.45	54.83
8-9 (325x2300)	75.5930	75.7301	0.1371	22.81	32.01
5-6 (510x3600)	80.6794	80.8347	0.1553	25.84	6.17
Exp. 2.2 Sieved with Water Using Shaker			Total Sample Used (g) = Insoluble Solids (g) =		5.0528 0.5937
Sieve Size (microns or mesh)	Dry Wt of Sieve (g)	Wt. After Sieving Slurry and Drying (g)	Dry Wt of Sieve (g) - Wt. After Sieving Slurry and Drying (g)	% of Insoluble Solids Collected on Sieve	% of Solids Below Sieve Size
38	80.0424	80.1506	0.1082	18.22	81.78
32	81.1155	81.1168	0.0013	0.22	81.56
25	72.9318	72.9318	0.0000	0.00	81.56
20	73.4064	73.4085	0.0021	0.35	81.20
16-18 (165x1400)	83.6445	83.6445	0.0000	0.00	81.20
12-14 (200x1400)	83.7140	83.7144	0.0004	0.07	81.14
			Total Sample Used (g) = Insoluble Solids (g) =		0.60 0.0703
11-12 (250x1400)	75.5404	75.5391	-0.0013	0.00	81.14
8-9 (325x2300)	75.5800	75.5809	0.0009	1.28	79.86
5-6 (510x3600)	80.6572	80.6646	0.0074	10.53	69.33

Table A.1 Continued. Experimental Data from Experiments 2.1 - 2.3.

Exp. 2.3			Total Sample Used (g) =		5.2097
Sieved with Water By Hand			Insoluble Solids (g) =		0.6121
Sieve Size (microns or mesh)	Dry Wt of Sieve (g)	Wt. After Sieving Slurry and Drying (g)	Dry Wt of Sieve (g) - Wt. After Sieving Slurry and Drying (g)	% of Insoluble Solids Collected on Sieve	% of Solids Below Sieve Size
38	80.5204	80.6323	0.1119	18.28	81.72
32	80.5624	80.5670	0.0046	0.75	80.97
25	80.5215	80.5232	0.0017	0.28	80.69
			Total Sample Used (g) =		1.1475
			Insoluble Solids (g) =		0.1348
20	71.4420	71.4438	0.0018	1.34	79.36
16-18 (165x1400)	75.1031	75.1040	0.0009	0.67	78.69
12-14 (200x1400)	75.3330	75.3339	0.0009	0.67	78.02
			Total Sample Used (g) =		0.5312
			Insoluble Solids (g) =		0.0624
11-12 (250x1400)	81.2040	81.2067	0.0027	4.33	73.69
8-9 (325x2300)	75.1270	75.1273	0.0003	0.48	73.21
5-6 (510x3600)	80.7907	80.7939	0.0032	5.13	68.09

Table A.2 Experimental Data from Experiments 3.1 - 3.3.

Exp. 3.1			Total Sample Used (g) =		5.0433
Sieved with Supernate			Insoluble Solids (g) =		0.5926
Sieve Size (microns or mesh)	Dry Wt of Sieve (g)	Wt. After Sieving Slurry and Drying (g)	Dry Wt of Sieve (g) - Wt. After Sieving Slurry and Drying (g)	% of Insoluble Solids Collected on Sieve	% of Solids Below Sieve Size
38	80.5605	80.7081	0.1476	24.91	75.09
32	80.5823	80.6477	0.0654	11.04	64.06
25	80.5381	80.6054	0.0673	11.36	52.70
20	73.4327	73.51	0.0773	13.04	39.65
16-18 (165x1400)	84.3837	84.4291	0.0454	7.66	31.99
12-14 (200x1400)	75.422	75.5317	0.1097	18.51	13.48
11-12 (250x1400)	75.5882	75.6403	0.0521	8.79	4.69
8-9 (325x2300)	75.6283	75.9945	0.3662	61.80	-57.11
5-6 (510x3600)	80.732	81.1475	0.4155	70.12	-127.22
Exp. 3.2			Total Sample Used (g) =		5.0454
Sieved with Supernate			Insoluble Solids (g) =		0.5928
Sieve Size (microns or mesh)	Dry Wt of Sieve (g)	Wt. After Sieving Slurry and Drying (g)	Dry Wt of Sieve (g) - Wt. After Sieving Slurry and Drying (g)	% of Insoluble Solids Collected on Sieve	% of Solids Below Sieve Size
38	80.0745	80.1906	0.1161	19.58	80.42
32	81.1278	81.1375	0.0097	1.64	78.78
25	72.9554	72.9576	0.0022	0.37	78.41
			Total Sample Used (g) =		1.0427
			Insoluble Solids (g) =		0.1225
20	71.8797	71.8861	0.0064	5.22	73.19
16-18 (165x1400)	83.6655	83.6827	0.0172	14.04	59.15
12-14 (200x1400)	84.7724	84.7766	0.0042	3.43	55.72
			Total Sample Used (g) =		0.5198
			Insoluble Solids (g) =		0.0611
11-12 (250x1400)	81.8233	81.8378	0.0145	23.74	31.98
8-9 (325x2300)	73.7102	73.7251	0.0149	24.40	7.58
5-6 (510x3600)	80.8067	80.8165	0.0098	16.05	-8.46

Table A.2 Continued. Experimental Data from Experiments 3.1 - 3.3.

Exp. 3.3 Sieved with Supernate			Total Sample Used (g) = Insoluble Solids (g) =		5.1435 0.6044
Sieve Size (microns or mesh)	Dry Wt of Sieve (g)	Wt. After Sieving Slurry and Drying (g)	Dry Wt of Sieve (g) - Wt. After Sieving Slurry and Drying (g)	% of Insoluble Solids Collected on Sieve	% of Solids Below Sieve Size
38	80.4293	80.558	0.1287	21.30	78.70
32	80.4921	80.5135	0.0214	3.54	75.16
25	80.4471	80.4625	0.0154	2.55	72.62
			Total Sample Used (g) = Insoluble Solids (g) =		1.3053 0.1534
20	71.3248	71.362	-0.0048	0.00	72.62
16-18 (165x1400)	75.0208	75.0355	0.0147	9.58	63.03
12-14 (200x1400)	83.6747	83.6952	0.0205	13.37	49.67
			Total Sample Used (g) = Insoluble Solids (g) =		0.5694 0.0669
11-12 (250x1400)	81.1583	81.1966	0.0013	1.93	47.74
8-9 (325x2300)	73.608	73.6085	0.0005	0.75	46.99
5-6 (510x3600)	80.7602	80.7665	0.0063	9.42	37.57

Table A.3 Experimental Data from Experiments 4.1 - 4.2.

Exp. 4.1			Total Sample Used (g) =		5.1737
Sieved with Water				Insoluble Solids (g) =	0.7409
Sieve Size (microns or mesh)	Dry Wt of Sieve (g)	Wt. After Sieving Slurry and Drying (g)	Dry Wt of Sieve (g) - Wt. After Sieving Slurry and Drying (g)	% of Insoluble Solids Collected on Sieve	% of Solids Below Sieve Size
32	81.028	81.0535	0.0255	3.44	96.56
25	72.8381	72.8381	0	0.00	96.56
20	71.8195	71.821	0.0015	0.20	96.36
			Total Sample Used (g) =		1.2653
				Insoluble Solids (g) =	0.1812
16-18 (165x1400)	83.628	83.631	-0.0036	0.00	96.36
12-14 (200x1400)	75.2727	75.2727	0	0.00	96.36
11-12 (250x1400)	75.503	75.5034	0.0004	0.22	96.13
			Total Sample Used (g) =		0.506
				Insoluble Solids (g) =	0.0725
8-9 (325x2300)	75.052	75.0531	-0.0017	0.00	96.13
6-7(450x2750)	72.7433	72.7481	0.0048	6.62	89.51
5-6 (510x3600)	80.5538	80.5538	0	0.00	89.51
Exp. 4.2			Total Sample Used (g) =		5.0348
Sieved with Water				Insoluble Solids (g) =	0.7210
Sieve Size (microns or mesh)	Dry Wt of Sieve (g)	Wt. After Sieving Slurry and Drying (g)	Dry Wt of Sieve (g) - Wt. After Sieving Slurry and Drying (g)	% of Insoluble Solids Collected on Sieve	% of Solids Below Sieve Size
32	73.7485	73.781	0.0325	4.51	95.49
25	72.8195	72.82	0.0005	0.07	95.42
20	73.322	73.3275	0.0055	0.76	94.66
			Total Sample Used (g) =		1.0813
				Insoluble Solids (g) =	0.154842
16-18 (165x1400)	84.3005	84.3012	-0.0076	0.00	94.66
12-14 (200x1400)	84.6939	84.6939	0	0.00	94.66
11-12 (250x1400)	81.6365	81.6395	0.003	1.94	92.72
			Total Sample Used (g) =		0.5036
				Insoluble Solids (g) =	0.0721
8-9 (325x2300)	75.483	75.4916	0.0034	4.65	88.07
6-7(450x2750)	74.5777	74.5817	0.004	5.55	82.53
5-6 (510x3600)	75.021	75.024	0.003	4.16	78.37

Table A.4 Experimental Data from Experiments 5.1 - 5.2.

Exp. 5.1			Total Sample Used (g) =		5.0368
Sieved with Supernate			Insoluble Solids (g) =		0.7213
Sieve Size (microns or mesh)	Dry Wt of Sieve (g)	Wt. After Sieving Slurry and Drying (g)	Dry Wt of Sieve (g) - Wt. After Sieving Slurry and Drying (g)	% of Insoluble Solids Collected on Sieve	% of Solids Below Sieve Size
32	80.4422	80.489	0.0468	6.49	93.51
25	72.7478	72.7555	0.0077	1.07	92.44
20	73.2633	73.2754	0.0121	1.68	90.77
			Total Sample Used (g) =		1.0938
				Insoluble Solids (g) =	0.1566
16-18 (165x1400)	84.271	84.2915	0.006037031	3.85	86.91
12-14 (200x1400)	75.26	75.2705	0.0105	6.70	80.21
11-12 (250x1400)	81.1215	81.139	0.0175	11.17	69.04
			Total Sample Used (g) =		0.5879
				Insoluble Solids (g) =	0.0842
8-9 (325x2300)	74.9575	74.9667	-0.016867974	0.00	69.04
6-7(450x2750)	74.7879	74.7963	0.0084	9.98	59.06
5-6 (510x3600)	80.5448	80.5546	0.0098	11.64	47.42
Exp. 5.2			Total Sample Used (g) =		5.0799
Sieved with Supernate			Insoluble Solids (g) =		0.7274
Sieve Size (microns or mesh)	Dry Wt of Sieve (g)	Wt. After Sieving Slurry and Drying (g)	Dry Wt of Sieve (g) - Wt. After Sieving Slurry and Drying (g)	% of Insoluble Solids Collected on Sieve	% of Solids Below Sieve Size
32	81.0065	81.0568	0.0503	6.91	93.09
25	80.4064	80.4173	0.0109	1.50	91.59
20	71.2716	71.2788	0.0072	0.99	90.60
			Total Sample Used (g) =		1.1443
				Insoluble Solids (g) =	0.1639
16-18 (165x1400)	74.9406	74.963	0.006992193	4.27	86.33
12-14 (200x1400)	83.622	83.6346	0.0126	7.69	78.64
11-12 (250x1400)	75.4684	75.488	0.0196	11.96	66.68
			Total Sample Used (g) =		0.5705
				Insoluble Solids (g) =	0.081
8-9 (325x2300)	73.5325	73.5501	0	0.00	66.68
6-7(450x2750)	72.7239	72.733	0.0091	11.14	55.54
5-6 (510x3600)	80.5343	80.5405	0.0062	7.59	47.95

Table A.5 Experimental Data from Experiments 6.1 - 6.2.

Exp. 6.1			Total Sample Used (g) =		10.3127
Sieved with Water			Insoluble Solids (g) =		1.8614
Sieve Size (microns or mesh)	Dry Wt of Sieve (g)	Wt. After Sieving Slurry and Drying (g)	Dry Wt of Sieve (g) - Wt. After Sieving Slurry and Drying (g)	% of Insoluble Solids Collected on Sieve	% of Solids Below Sieve Size
150	73.3513	73.3994	0.0481	2.58	97.42
75	80.0686	80.2415	0.1729	9.29	88.13
45	80.3802	80.581	0.2008	10.79	77.34
			Total Sample Used (g) =		5.4166
			Insoluble Solids (g) =		0.9777
32	81.0435	81.3794	0.1144	11.70	65.64
25	72.8489	72.8947	0.0458	4.68	60.96
			Total Sample Used (g) =		0.5977
			Insoluble Solids (g) =		0.1079
16-18 (165x1400)	84.298	84.347	0.0069	6.38	54.58
12-14 (200x1400)	84.7025	84.7089	0.0064	5.93	48.65
11-12 (250x1400)	75.5133	75.5203	0.007	6.49	42.16
			Total Sample Used (g) =		0.5404
			Insoluble Solids (g) =		0.0975
8-9 (325x2300)	75.5048	75.5675	0.0035	3.63	38.53
6-7(450x2750)	74.7202	74.7218	0.0016	1.64	36.89
5-6 (510x3600)	80.585	80.5865	0.0015	1.54	35.36
Exp. 6.2			Total Sample Used (g) =		10.2919
Sieved with Water			Insoluble Solids (g) =		1.8577
Sieve Size (microns or mesh)	Dry Wt of Sieve (g)	Wt. After Sieving Slurry and Drying (g)	Dry Wt of Sieve (g) - Wt. After Sieving Slurry and Drying (g)	% of Insoluble Solids Collected on Sieve	% of Solids Below Sieve Size
150	74.3015	74.3391	0.0376	2.02	97.98
75	74.0275	74.2031	0.1756	9.45	88.52
45	75.0689	75.2855	0.2166	11.66	76.86
			Total Sample Used (g) =		5.6663
			Insoluble Solids (g) =		1.0228
32	73.7805	74.1222	0.1051	10.27	66.59
25	72.8969	72.9403	0.0434	4.24	62.35
			Total Sample Used (g) =		0.6108
			Insoluble Solids (g) =		0.1102
16-18 (165x1400)	83.644	83.7	0.0145	13.14	49.21
12-14 (200x1400)	75.2882	75.2982	0.01	9.07	40.14
11-12 (250x1400)	81.6427	81.6515	0.0088	7.98	32.15
			Total Sample Used (g) =		0.5622
			Insoluble Solids (g) =		0.1015
8-9 (325x2300)	75.0734	75.1394	0.0045	4.48	27.68
6-7(450x2750)	72.8665	72.87	0.0035	3.45	24.23
5-6 (510x3600)	75.1047	75.1049	0.0002	0.20	24.03

Table A.6 Experimental data from Experiments 7.1-7.2.

Exp. 7.1			Total Sample Used (g) =		4.9980
Sieved with Supernate			Insoluble Solids (g) =		0.9021
Sieve Size (microns or mesh)	Dry Wt of Sieve (g)	Wt. After Sieving Slurry and Drying (g)	Dry Wt of Sieve (g) - Wt. After Sieving Slurry and Drying (g)	% of Insoluble Solids Collected on Sieve	% of Solids Below Sieve Size
150	73.3006	73.34444	0.04384	4.86	95.14
75	80.0242	80.1635	0.1393	15.44	79.70
45	80.3355	80.455	0.1195	13.25	66.45
			Total Sample Used (g) =		1.0012
			Insoluble Solids (g) =		0.1807
32	73.7058	73.7777	0.0113	6.24	60.21
25	72.7662	72.786	0.0198	10.96	49.26
			Total Sample Used (g) =		0.5557
			Insoluble Solids (g) =		0.1003
16-18 (165x1400)	83.5363	83.5945	0.0073	7.28	41.98
12-14 (200x1400)	84.6671	84.7014	0.0343	34.20	7.78
11-12 (250x1400)	81.6089	81.6313	0.0224	22.33	-14.55
			Total Sample Used (g) =		0.5217
			Insoluble Solids (g) =		0.0942
8-9 (325x2300)	75.0086	75.0139	0.0006	0.61	-15.16
6-7(450x2750)	74.849	74.8551	0.0061	6.48	-21.64
5-6 (510x3600)	74.8719	74.8881	0.0162	17.20	-38.84
Exp. 7.2			Total Sample Used (g) =		5.0318
Sieved with Supernate			Insoluble Solids (g) =		0.9082
Sieve Size (microns or mesh)	Dry Wt of Sieve (g)	Wt. After Sieving Slurry and Drying (g)	Dry Wt of Sieve (g) - Wt. After Sieving Slurry and Drying (g)	% of Insoluble Solids Collected on Sieve	% of Solids Below Sieve Size
150	73.2305	73.2585	0.028	3.08	96.92
75	79.951	80.0445	0.0935	10.29	86.62
45	80.2665	80.3943	0.1278	14.07	72.55
			Total Sample Used (g) =		1.0932
			Insoluble Solids (g) =		0.1973
32	80.3525	80.4358	0.0291	14.77	57.78
25	72.6565	72.6635	0.007	3.55	54.24
			Total Sample Used (g) =		0.5380
			Insoluble Solids (g) =		0.0971
16-18 (165x1400)	74.8935	74.9478	0.0099	10.15	44.08
12-14 (200x1400)	75.228	75.238	0.01	10.30	33.79
11-12 (250x1400)	75.4313	75.4518	0.0205	21.11	12.68
			Total Sample Used (g) =		0.5744
			Insoluble Solids (g) =		0.1037
8-9 (325x2300)	75.2831	75.349	0.0060	5.75	6.92
6-7(450x2750)	72.6199	72.626	0.0061	5.88	1.04
5-6 (510x3600)	80.51	80.522	0.012	11.57	-10.54

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APPENDIX B

Data tables for Experiments 3.4, 5.3, and 7.3

Table B.1 Experimental data from Experiment 3.4 - Sieving of supernate only to obtain dissolved solids correction factor.

Exp. 1 (Sieve Size)	Dry Wt of Sieve (g)	Wt. After Sieving Supernate and Drying (g)	Dissolved Solids Correction Factor (Dry Wt of Sieve (g) - Wt. After Sieving Supernate and Drying (g)
38	79.9695	79.9833	0.0138
32	80.9752	80.9875	0.0123
25	80.3718	80.3868	0.015
20	71.2203	71.2321	0.0118
16-18 (165x1400)	84.2383	84.2685	0.0302
12-14 (200x1400)	83.599	83.6235	0.0245
11-12 (250x1400)	81.1	81.1566	0.0566
8-9 (325x2300)	73.4505	73.4712	0.0207
5-6 (510x3600)	74.8134	74.8342	0.0208
Exp. 2 (Sieve Size)	Dry Wt of Sieve (g)	Wt. After Sieving Supernate and Drying (g)	Dissolved Solids Correction Factor (Dry Wt of Sieve (g) - Wt. After Sieving Supernate and Drying (g)
38	80.3783	80.3878	0.0095
32	73.6687	73.6807	0.012
25	72.7271	72.7418	0.0147
20	73.2064	73.2221	0.0157
16-18 (165x1400)	74.8781	74.9093	0.0312
12-14 (200x1400)	75.2211	75.2463	0.0252
11-12 (250x1400)	75.4064	75.4598	0.0534
8-9 (325x2300)	75.2612	75.2833	0.0221
5-6 (510x3600)	80.4926	80.5192	0.0266
(Sieve Size)	Average Dissolved Solids on Each Sieve From Two Experiments (g)	Standard Deviation	%RSD
38	0.0117	0.0030	26.0992
32	0.0121	0.0002	1.7459
25	0.0149	0.0002	1.4285
20	0.0138	0.0028	20.0561
16-18 (165x1400)	0.0307	0.0007	2.3033
12-14 (200x1400)	0.0249	0.0005	1.9919
11-12 (250x1400)	0.0550	0.0023	4.1141
8-9 (325x2300)	0.0214	0.0010	4.6259
5-6 (510x3600)	0.0237	0.0041	17.3047

Table B.2 Dissolved solids corrected data from Experiments 3.1-3.3 using values from Table A.2. The plot in Figure 3.7 (Exp. 3.4) is the average concentration from Experiments 3.2 and 3.3.

Exp.1				Total Sample Used (g) =			5.0433
Sieved with Supernate				Insoluble Solids (g) =			0.5926
Sieve Size (microns or mesh)	Dry Wt of Sieve (g)	Wt. After Sieving Slurry and Drying (g)	Dry Wt of Sieve (g) - Wt. After Sieving Slurry and Drying (g)	Dissolved Solids (g) – From Table B.1	Delta-Dissolved Solids (g) or Corrected Mass of Solids on Sieve (g)	% of Insoluble Solids Collected on Sieve	% of Solids Below Sieve Size
38	80.5605	80.7081	0.1476	0.01165	0.1359	22.94	77.06
32	80.5823	80.6477	0.0654	0.01215	0.0532	8.99	68.07
25	80.5381	80.6054	0.0673	0.01485	0.0525	8.85	59.22
20	73.4327	73.51	0.0773	0.01375	0.0636	10.72	48.50
16-18 (165x1400)	84.3837	84.4291	0.0454	0.0307	0.0147	2.48	46.02
12-14 (200x1400)	75.422	75.5317	0.1097	0.02485	0.0849	14.32	31.70
11-12 (250x1400)	75.5882	75.6403	0.0521	0.055	0.0000	0.00	31.70
8-9 (325x2300)	75.6283	75.9945	0.3662	0.0214	0.3448	58.19	-26.49
5-6 (510x3600)	80.732	81.1475	0.4155	0.0237	0.3918	66.12	-92.60
Exp.2				Total Sample Used (g) =			5.0454
Sieved with Supernate				Insoluble Solids (g) =			0.5928
Sieve Size (microns or mesh)	Dry Wt of Sieve (g)	Wt. After Sieving Slurry and Drying (g)	Dry Wt of Sieve (g) - Wt. After Sieving Slurry and Drying (g)	Dissolved Solids (g) – From Table B.1	Delta-Dissolved Solids (g) or Corrected Mass of Solids on Sieve (g)	% of Insoluble Solids Collected on Sieve	% of Solids Below Sieve Size
38	80.0745	80.1906	0.1161	0.01165	0.1045	19.58	80.42
32	81.1278	81.1375	0.0097	0.01215	-0.0024	1.64	78.78
25	72.9554	72.9576	0.0022	0.01485	-0.0127	0.37	78.41
				Total Sample Used (g) =			1.0427
				Insoluble Solids (g) =			0.1225
20	71.8797	71.8861	0.0064	0.01375	-0.0074	0.00	78.41
16-18 (165x1400)	83.6655	83.6827	0.0172	0.0307	-0.0135	0.00	78.41
12-14 (200x1400)	84.7724	84.7766	0.0042	0.02485	-0.0207	0.00	78.41
				Total Sample Used (g) =			0.5198
				Insoluble Solids (g) =			0.0611
11-12 (250x1400)	81.8233	81.8378	0.0145	0.055	-0.0405	0.00	78.41
8-9 (325x2300)	73.7102	73.7251	0.0149	0.0214	-0.0065	0.00	78.41
5-6 (510x3600)	80.8067	80.8165	0.0098	0.0237	-0.0139	0.00	78.41

Table B.2 Continued. Dissolved solids corrected data from Experiments 3.1-3.3 using values from Table A.2. The plot (Exp. 3.4) in Figure 3.7 is the average concentration from Experiments 3.2 and 3.3.

Exp.3			Total Sample Used (g) =				5.1435
Sieved with Supernate				Insoluble Solids (g) =			0.6044
Sieve Size (microns or mesh)	Dry Wt of Sieve (g)	Wt. After Sieving Slurry and Drying (g)	Dry Wt of Sieve (g) - Wt. After Sieving Slurry and Drying (g)	Dissolved Solids (g) – From Table B.1	Delta-Dissolved Solids (g) or Corrected Mass of Solids on Sieve (g)	% of Insoluble Solids Collected on Sieve	% of Solids Below Sieve Size
38	80.4293	80.558	0.1287	0.01165	0.11705	19.37	80.63
32	80.4921	80.5135	0.0214	0.01215	0.00925	1.53	79.10
25	80.4471	80.4625	0.0154	0.01485	0.00055	0.09	79.01
				Total Sample Used (g) =			1.3053
				Insoluble Solids (g) =			0.1534
20	71.3248	71.362	0.005008	0.01375	-0.00874	0.00	79.01
16-18 (165x1400)	75.0208	75.0355	0.0147	0.0307	-0.016	0.00	79.01
12-14 (200x1400)	83.6747	83.6952	0.0205	0.02485	-0.00435	0.00	79.01
				Total Sample Used (g) =			0.5694
				Insoluble Solids (g) =			0.0669
11-12 (250x1400)	81.1583	81.1966	0.000538	0.055	-0.05446	0.00	79.01
8-9 (325x2300)	73.608	73.6085	0.0005	0.0214	-0.0209	0.00	79.01
5-6 (510x3600)	80.7602	80.7665	0.0063	0.0237	-0.0174	0.00	79.01

Table B.3 Experimental data from Experiment 5.3 - Sieving of supernate only to obtain dissolved solids correction factor.

Exp. 1 (Sieve Size)	Dry Wt of Sieve (g)	Wt. After Sieving Supernate and Drying (g)	Dissolved Solids Correction Factor (Dry Wt of Sieve (g) - Wt. After Sieving Supernate and Drying (g)
32	80.3484	80.36	0.0116
25	80.3722	80.3849	0.0127
20	71.7913	71.8001	0.0088
16-18 (165x1400)	74.8796	74.8968	0.0172
12-14 (200x1400)	75.2215	75.2388	0.0173
11-12 (250x1400)	75.4069	75.4403	0.0334
8-9 (325x2300)	74.9486	74.9649	0.0163
6-7(450x2750)	74.7697	74.7858	0.0161
5-6 (510x3600)	80.4941	80.5134	0.0193
Exp. 2 (Sieve Size)	Dry Wt of Sieve (g)	Wt. After Sieving Supernate and Drying (g)	Dissolved Solids Correction Factor (Dry Wt of Sieve (g) - Wt. After Sieving Supernate and Drying (g)
32	80.3486	80.3559	0.0073
25	72.6372	72.6448	0.0076
20	71.7891	71.7946	0.0055
16-18 (165x1400)	83.4963	83.511	0.0147
12-14 (200x1400)	84.6375	84.6516	0.0141
11-12 (250x1400)	81.5862	81.5985	0.0123
8-9 (325x2300)	74.9476	74.9544	0.0068
6-7(450x2750)	74.7681	74.7768	0.0087
5-6 (510x3600)	80.5047	80.5154	0.0107
(Sieve Size)	Used Only Values From Experiment 2 (g)	Standard Deviation	%RSD
32	0.0073	NA	NA
25	0.0076	NA	NA
20	0.0055	NA	NA
16-18 (165x1400)	0.0147	NA	NA
12-14 (200x1400)	0.0141	NA	NA
11-12 (250x1400)	0.0123	NA	NA
8-9 (325x2300)	0.0068	NA	NA
6-7(450x2750)	0.0087	NA	NA
5-6 (510x3600)	0.0107	NA	NA

Table B.4 Dissolved solids corrected data from Experiments 5.1 and 5.2 using values from Table A.4. The plot in Figure 3.9 (Exp. 5.3) is the average concentration from Experiments 5.1 and 5.2.

Exp.1					Total Sample Used (g) =		5.0368
Sieved with Supernate					Insoluble Solids (g) =		0.7212
Sieve Size (microns or mesh)	Dry Wt of Sieve (g)	Wt. After Sieving Slurry and Drying (g)	Dry Wt of Sieve (g) - Wt. After Sieving Slurry and Drying (g)	Dissolved Solids (g) – From Table B.3	Delta-Dissolved Solids (g) or Corrected Mass of Solids on Sieve (g)	% of Insoluble Solids Collected on Sieve	% of Solids Below Sieve Size
32	80.4422	80.489	0.0468	0.0073	0.0395	5.48	94.52
25	72.7478	72.7555	0.0077	0.0076	0.0001	0.01	94.51
20	73.2633	73.2754	0.0121	0.0055	0.0066	0.92	93.59
					Total Sample Used (g) =		1.0938
					Insoluble Solids (g) =		0.1566
16-18 (165x1400)	84.271	84.2915	0.0205	0.0147	-0.00423	0.00	93.59
12-14 (200x1400)	75.26	75.2705	0.0105	0.0141	-0.0036	0.00	93.59
11-12 (250x1400)	81.1215	81.139	0.0175	0.0123	0.0052	3.32	95.75
					Total Sample Used (g) =		0.5879
					Insoluble Solids (g) =		0.0842
8-9 (325x2300)	74.9575	74.9667	0.0092	0.0068	-0.00118	0.00	95.75
6-7(450x2750)	74.7879	74.7963	0.0084	0.0087	-0.0003	0.00	95.75
5-6 (510x3600)	80.5448	80.5546	0.0098	0.0107	-0.0009	0.00	95.75
Exp.2					Total Sample Used (g) =		5.0799
Sieved with Supernate					Insoluble Solids (g) =		0.7274
Sieve Size (microns or mesh)	Dry Wt of Sieve (g)	Wt. After Sieving Slurry and Drying (g)	Dry Wt of Sieve (g) - Wt. After Sieving Slurry and Drying (g)	Dissolved Solids (g) – From Table B.3	Delta-Dissolved Solids (g) or Corrected Mass of Solids on Sieve (g)	% of Insoluble Solids Collected on Sieve	% of Solids Below Sieve Size
32	81.0065	81.0568	0.0503	0.0073	0.043	5.91	94.09
25	80.4064	80.4173	0.0109	0.0076	0.0033	0.45	93.64
20	71.2716	71.2788	0.0072	0.0055	0.0017	0.23	93.40
					Total Sample Used (g) =		1.0932
					Insoluble Solids (g) =		0.1639
16-18 (165x1400)	74.9406	74.963	0.0224	0.0147	-0.0047	0.00	93.40
12-14 (200x1400)	83.622	83.6346	0.0126	0.0141	-0.0015	0.00	93.40
11-12 (250x1400)	75.4684	75.488	0.0196	0.0123	0.0073	4.45	88.95
					Total Sample Used (g) =		0.5705
					Insoluble Solids (g) =		0.0817
8-9 (325x2300)	73.5325	73.5501	0.0176	0.0068	-0.0709	0.00	88.95
6-7(450x2750)	72.7239	72.733	0.0091	0.0087	0.0004	0.49	88.46
5-6 (510x3600)	80.5343	80.5405	0.0062	0.0107	-0.0045	0.00	88.46

Table B.5 Experimental data from Experiment 7.3 - Sieving of supernate only to obtain dissolved solids correction factor.

Exp. 1 (Sieve Size)	Dry Wt of Sieve (g)	Wt. After Sieving Supernate and Drying (g)	Dissolved Solids Correction Factor (Dry Wt of Sieve (g) - Wt. After Sieving Supernate and Drying (g)
150	74.2656	74.2727	0.0071
75	73.9841	73.991	0.0069
45	75.0312	75.0375	0.0063
32	80.9755	80.9827	0.0072
25	72.6394	72.6435	0.0041
16-18 (165x1400)	84.24	84.2471	0.0071
12-14 (200x1400)	83.6006	83.6067	0.0061
11-12 (250x1400)	81.5867	81.5924	0.0057
8-9 (325x2300)	73.4513	73.4569	0.0056
6-7(450x2750)	72.6008	72.6076	0.0068
5-6 (510x3600)	80.5042	80.517	0.0128
Exp. 2 (Sieve Size)	Dry Wt of Sieve (g)	Wt. After Sieving Supernate and Drying (g)	Dissolved Solids Correction Factor (Dry Wt of Sieve (g) - Wt. After Sieving Supernate and Drying (g)
150	81.3019	81.3099	0.008
75	73.9657	73.975	0.0093
45	73.9397	73.9476	0.0079
32	73.6682	73.6774	0.0092
25	72.7268	72.7338	0.007
16-18 (165x1400)	83.4961	83.5085	0.0124
12-14 (200x1400)	84.639	84.6484	0.0094
11-12 (250x1400)	81.102	81.1118	0.0098
8-9 (325x2300)	75.261	75.2679	0.0069
6-7(450x2750)	74.3548	74.3601	0.0053
5-6 (510x3600)	74.8136	74.8241	0.0105
(Sieve Size)	Average Dissolved Solids on Each Sieve From Two Experiments (g)	Standard Deviation	%RSD
150	0.00755	0.00755	8.4291
75	0.0081	0.0081	20.9513
45	0.0071	0.0071	15.9348
32	0.0082	0.0082	17.2465
25	0.00555	0.00555	36.9479
16-18 (165x1400)	0.00975	0.00975	38.4376
12-14 (200x1400)	0.00775	0.00775	30.1091
11-12 (250x1400)	0.00775	0.00775	37.4082
8-9 (325x2300)	0.00625	0.00625	14.7078
6-7(450x2750)	0.00605	0.00605	17.5316
5-6 (510x3600)	0.01165	0.01165	13.9600

Table B.6 Dissolved solids corrected data for Experiments 7.1 and 7.2 using values from Table A.5. The plot in Figure 3.11 (Exp. 7.3) is the average concentration from Experiments 7.1 and 7.2.

Exp.1					Total Sample Used (g) =		4.998
Sieved with Supernate					Insoluble Solids (g) =		0.9021
Sieve Size (microns or mesh)	Dry Wt of Sieve (g)	Wt. After Sieving Slurry and Drying (g)	Dry Wt of Sieve (g) - Wt. After Sieving Slurry and Drying (g)	Dissolved Solids (g) – From Table B.5	Delta-Dissolved Solids (g) or Corrected Mass of Solids on Sieve (g)	% of Insoluble Solids Collected on Sieve	% of Solids Below Sieve Size
150	73.3006	73.34444	0.04384	0.00755	0.03629	4.02	95.98
75	80.0242	80.1635	0.1393	0.0081	0.1312	14.54	81.43
45	80.3355	80.455	0.1195	0.0071	0.1124	12.46	68.97
					Total Sample Used (g) =		1.0012
					Insoluble Solids (g) =		0.1807
32	73.7058	73.7777	0.0719	0.0082	0.007632	4.22	64.75
25	72.7662	72.786	0.0198	0.00555	0.01425	7.89	56.87
					Total Sample Used (g) =		0.5557
					Insoluble Solids (g) =		0.1003
16-18 (165x1400)	83.5363	83.5945	0.0582	0.00975	0.005185	5.17	51.70
12-14 (200x1400)	84.6671	84.6842	0.0171	0.00775	0.00935	9.32	42.38
11-12 (250x1400)	81.6089	81.6313	0.0224	0.00775	0.01465	14.61	27.77
					Total Sample Used (g) =		0.5217
					Insoluble Solids (g) =		0.094167
8-9 (325x2300)	75.0086	75.0139	0.0053	0.00625	-0.06897	0.00	27.77
6-7(450x2750)	74.849	74.8551	0.0061	0.00605	5E-05	0.05	27.72
5-6 (510x3600)	74.8719	74.8881	0.0162	0.01165	0.00455	4.83	22.88
Exp.2					Total Sample Used (g) =		5.0318
Sieved with Supernate					Insoluble Solids (g) =		0.9082
Sieve Size (microns or mesh)	Dry Wt of Sieve (g)	Wt. After Sieving Slurry and Drying (g)	Dry Wt of Sieve (g) - Wt. After Sieving Slurry and Drying (g)	Dissolved Solids (g) – From Table B.5	Delta-Dissolved Solids (g) or Corrected Mass of Solids on Sieve (g)	% of Insoluble Solids Collected on Sieve	% of Solids Below Sieve Size
150	73.2305	73.2585	0.028	0.00755	0.02045	2.25	97.75
75	79.951	80.0445	0.0935	0.0081	0.0854	9.40	88.35
45	80.2665	80.3943	0.1278	0.0071	0.1207	13.29	75.06
					Total Sample Used (g) =		1.0932
					Insoluble Solids (g) =		0.1973
32	80.3525	80.4358	0.0833	0.0082	0.02588	13.12	61.94
25	72.6565	72.6635	0.007	0.00555	0.00145	0.73	61.21
					Total Sample Used (g) =		0.538
					Insoluble Solids (g) =		0.0971
16-18 (165x1400)	74.8935	74.9478	0.0543	0.00975	0.006877	7.08	54.12
12-14 (200x1400)	75.228	75.238	0.01	0.00775	0.00225	2.32	51.81
11-12 (250x1400)	75.4313	75.4518	0.0205	0.00775	0.01275	13.13	38.68
					Total Sample Used (g) =		0.5744
					Insoluble Solids (g) =		0.1037
8-9 (325x2300)	75.2831	75.349	0.0659	0.00625	-0.00393	0.00	38.68
6-7(450x2750)	72.6199	72.626	0.0061	0.00605	5E-05	0.05	38.63
5-6 (510x3600)	80.51	80.522	0.012	0.01165	0.00035	0.34	38.29

APPENDIX C
Sample Calculations

Table C.1 Sample calculations based on Experiment 6.1. The experimentally obtained data is in Table A.5 and the calculation formulas are in Table C.2. Calculations of this type were done on data collected from Experiments 3.3, 4.1-4.2, 5.1-5.2, 6.1-6.2 and 7.1-7.2.

	B	C	D	E	F	G	H
15	**Exp. 6.1**				Total Sample Used (g) =		10.3127
16	Sieved with Water					Insoluble Solids (g) =	1.8614
17	Sieve Size (microns or mesh)	Dry Wt of Sieve (g)	Wt. After Sieving Slurry and Drying (g)	Dry Wt of Sieve (g) - Wt. After Sieving Slurry and Drying (g)	% of Insoluble Solids Collected on Sieve	% of Solids Below Sieve Size	
18	150	73.3513	73.3994	0.0481	2.58	97.42	
19	75	80.0686	80.2415	0.1729	9.29	88.13	
20	45	80.3802	80.581	0.2008	10.79	77.34	
21					Total Sample Used (g) =		5.4166
22						Insoluble Solids (g) =	0.9777
23	32	81.0435	81.3794	0.1144	11.70	65.64	
24	25	72.8489	72.8947	0.0458	4.68	60.96	
25					Total Sample Used (g) =		0.5977
26						Insoluble Solids (g) =	0.1079
27	16-18 (165x1400)	84.298	84.347	0.0069	6.38	54.58	
28	12-14 (200x1400)	84.7025	84.7089	0.0064	5.93	48.65	
29	11-12 (250x1400)	75.5133	75.5203	0.007	6.49	42.16	
30					Total Sample Used (g) =		0.5404
31						Insoluble Solids (g) =	0.0975
32	8-9 (325x2300)	75.5048	75.5675	0.0035	3.63	38.53	
33	6-7(450x2750)	74.7202	74.7218	0.0016	1.64	36.89	
34	5-6 (510x3600)	80.585	80.5865	0.0015	1.54	35.36	

Table C.2 Sample calculations based on Experiment 6.1. Calculations of this type were done on data collected from Experiments 3.3, 4.1-4.2, 5.1-5.2, 6.1-6.2 and 7.1-7.2.

	B	C	D	E	F	G	H
15	**Exp. 6.1**					Total Sample Used (g) =	10.3127
16	Sieved with Water					Insoluble Solids (g) =	1.8614
17	Sieve Size (microns or mesh)	Dry Wt of Sieve (g)	Wt. After Sieving Slurry and Drying (g)	Dry Wt of Sieve (g) - Wt. After Sieving Slurry and Drying (g)	% of Insoluble Solids Collected on Sieve	% of Solids Below Sieve Size	
18	150	73.3513	73.3994	=D18-C18	=E18/$H$16*100	=100-F18	
19	75	80.0686	80.2415	=D19-C19	=E19/$H$16*100	=100-F18-F19	
20	45	80.3802	80.581	=D20-C20	=E20/$H$16*100	=100-F18-F19-F20	
21						Total Sample Used (g) =	5.4166
22						Insoluble Solids (g) =	0.9777
23	32	81.0435	81.3794	=(D23-C23)-(H22)*(100-G20)/100	=E23/$H$22*100	=100-F18-F19-F20-F23	
24	25	72.8489	72.8947	=D24-C24	=E24/$H$22*100	=100-F18-F19-F20-F23-F24	
25						Total Sample Used (g) =	0.5977
26						Insoluble Solids (g) =	0.1079
27	16-18 (165x1400)	84.298	84.347	=(D27-C27)-(H26)*(100-G24)/100	=E27/$H$26*100	=100-F18-F19-F20-F23-F24-F27	
28	12-14 (200x1400)	84.7025	84.7089	=D28-C28	=E28/$H$26*100	=100-F18-F19-F20-F23-F24-F27-F28	
29	11-12 (250x1400)	75.5133	75.5203	=D29-C29	=E29/$H$26*100	=100-F18-F19-F20-F23-F24-F27-F28-F29	
30						Total Sample Used (g) =	0.5404
31						Insoluble Solids (g) =	0.0975
32	8-9 (325x2300)	75.5048	75.5675	=(D32-C32)*(H31)*(100-G29)/100	=E32/$H$31*100	=100-F18-F19-F20-F23-F24-F27-F28-F29-F32	
33	6-7(450x2750)	74.7202	74.7218	=D33-C33	=E33/$H$31*100	=100-F18-F19-F20-F23-F24-F27-F28-F29-F32-F33	
34	5-6 (510x3600)	80.585	80.5865	=D34-C34	=E34/$H$31*100	=100-F18-F19-F20-F23-F24-F27-F28-F29-F32-F33-F34	

Table C.3 Sample calculations based on Experiment 7.3 – Determination of Dissolved Solids Correction Factor. The experimental numbers are in Table B.5 and B.6 and the calculation formulas are in Table C.4. Calculations of this type were done on data collected from Experiments 3.4, 4.3, 5.3, 6.3 and 7.3.

	N	O	P	Q	R	T	S	U	V
15	**Exp. 7.3**							Total Sample Used (g) =	4.9980
16	**Sieved with Supernate Only to Determine Correction Factor**							Insoluble Solids (g) =	0.9021
17	Sieve Size (microns or mesh)	Dry Wt of Sieve (g)	Wt. After Sieving Slurry and Drying (g)	Dry Wt of Sieve (g) - Wt. After Sieving Slurry and Drying (g)	Dissolved Solids (g) – From Table 11	Delta-Dissolved Solids (g) or Corrected Mass of Solids on Sieve (g)	% of Insoluble Solids Collected on Sieve	% of Solids Below Sieve Size	
18	150	73.3006	73.3444	0.04384	0.0076	0.03629	4.02	95.98	
19	75	80.0242	80.1635	0.1393	0.0081	0.1312	14.54	81.43	
20	45	80.3355	80.455	0.1195	0.0071	0.1124	12.46	68.97	
21								Total Sample Used (g) =	1.0012
22								Insoluble Solids (g) =	0.1807
23	32	73.7058	73.7777	0.0719	0.0082	0.0076	4.22	64.75	
24	25	72.7662	72.786	0.0198	0.00555	0.01425	7.89	56.87	
25								Total Sample Used (g) =	0.5557
26								Insoluble Solids (g) =	0.1003
27	16-18 (165x1400)	83.5363	83.5945	0.0582	0.00975	0.0052	5.17	51.70	
28	12-14 (200x1400)	84.6671	84.6842	0.0171	0.00775	0.00935	9.32	42.38	
29	11-12 (250x1400)	81.6089	81.6313	0.0224	0.00775	0.01465	14.61	27.77	
30								Total Sample Used (g) =	0.5217
31								Insoluble Solids (g) =	0.0942
32	8-9 (325x2300)	75.0086	75.0139	0.0053	0.00625	0.06897	0.00	27.77	
33	6-7(450x2750)	74.849	74.8551	0.0061	0.00605	5E-05	0.05	27.72	
34	5-6 (510x3600)	74.8719	74.8881	0.0162	0.01165	0.00455	4.83	22.88	

Table C.4 Sample calculations based on Experiment 7.3 – Determination of Dissolved Solids Correction Factor. The experimental numbers are in Table B.5 and B.6 and the calculation formulas are in Table C.5. Calculations of this type were done on data collected from Experiments 3.4, 4.3, 5.3, 6.3 and 7.3.

	N	O	P	Q	R	T	S	U	V
15	**Exp. 7.3**							Total Sample Used (g) =	4.998
16	**Sieved with Supernate Only to Determine Correction Factor**							Insoluble Solids (g) =	0.9021
17	Sieve Size (microns or mesh)	Dry Wt of Sieve (g)	Wt. After Sieving Slurry and Drying (g)	Dry Wt of Sieve (g) - Wt. After Sieving Slurry and Drying (g)	Dissolved Solids (g) – From Table 11	Delta-Dissolved Solids (g) or Corrected Mass of Solids on Sieve (g)	% of Insoluble Solids Collected on Sieve	% of Solids Below Sieve Size	
18	150	73.3006	73.3444	=P18-O18	0.00755	=Q18-R18	=S18/$V$16*100	=100-T18	
19	75	80.0242	80.1635	=P19-O19	0.0081	=Q19-R19	=S19/$H$16*100	=100-T18-T19	
20	45	80.3355	80.455	=P20-O20	0.0071	=Q20-R20	=S20/$H$16*100	=100-T18-T19-T20	
21								Total Sample Used (g) =	1.0012
22								Insoluble Solids (g) =	0.1807
23	32	73.7058	73.7777	=P23-O23	0.0082	=(Q23-R23)-(V22) *(100-U20)/100	=S23/$V$22*100	=100-T18-T19-T20-T23	
24	25	72.7662	72.786	=P24-O24	0.0056	=Q24-R24	=S24/$V$22*100	=100-T18-T19-T20-T23-T24	
25								Total Sample Used (g) =	0.5557
26								Insoluble Solids (g) =	0.1003
27	16-18 (165x1400)	83.5363	83.5945	=P27-O27	0.0098	=(Q27-R27)-(V26) *(100-U24)/100	=S27/$V$26*100	=100-T18-T19-T20-T23-T24 -T27	
28	12-14 (200x1400)	84.6671	84.6842	=P28-O28	0.0078	=Q28-R28	=S28/$V$26*100	=100-T18-T19-T20-T23-T24 -T27-T28	
29	11-12 (250x1400)	81.6089	81.6313	=P29-O29	0.0078	=Q29-R29	=S29/$V$26*100	=100-T18-T19-T20-T23-T24 -T27-T28-T29	
30								Total Sample Used (g) =	0.5217
31								Insoluble Solids (g) =	0.0942
32	8-9 (325x2300)	75.0086	75.0139	=P32-O32	0.0063	=(Q32-R32)-(V31) *(100-U29)/100	0	=100-T18-T19-T20-T23-T24 -T27-T28-T29-T32	
33	6-7 (450x2750)	74.849	74.8551	=P33-O33	0.0061	=Q33-R33	=S33/$V$31*100	=100-T18-T19-T20-T23-T24 -T27-T28-T29-T32-T33	
34	5-6 (510x3600)	74.8719	74.8881	=P34-O34	0.0116	=Q34-R34	=S34/$V$31*100	=100-T18-T19-T20-T23-T24 -T27-T28-T29-T32-T33-T34	

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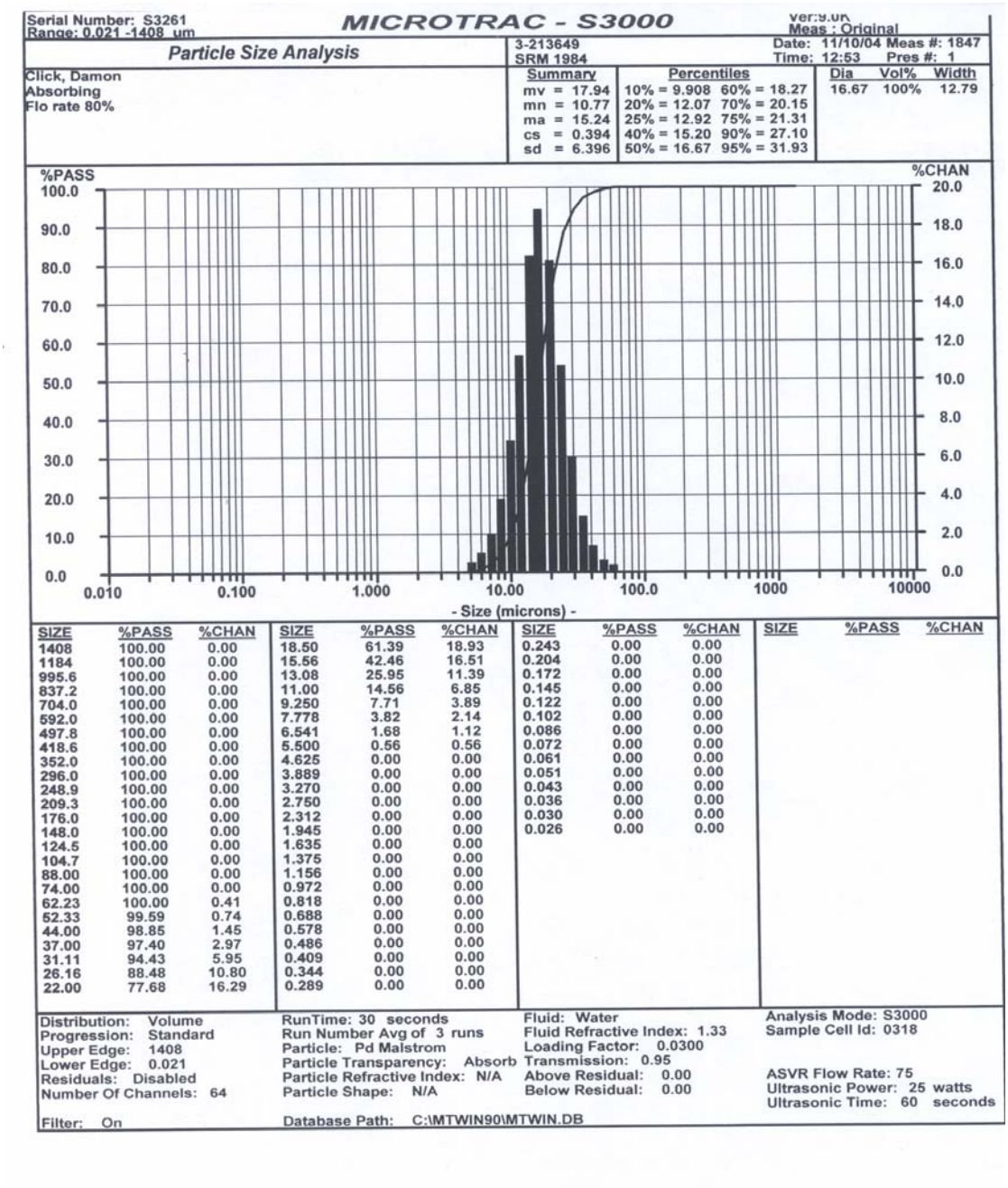
APPENDIX D

Microtrac Data

Figure D.1 Microtrac analysis of NIST 1984 standard on a volume basis.



Serial Number: S3261
Range: 0.021 -1408 um

MICROTRAC - S3000

Ver:5.0h
Meas : Original

Particle Size Analysis

3-213649
SRM 1984

Date: 11/10/04 Meas #: 1847
Time: 12:53 Pres #: 1

Click, Damon
Absorbing
Flo rate 80%

Summary		Percentiles		Dia	Vol%	Width
mv = 17.94		10% = 9.908	60% = 18.27	16.67	100%	12.79
mn = 10.77		20% = 12.07	70% = 20.15			
ma = 15.24		25% = 12.92	75% = 21.31			
cs = 0.394		40% = 15.20	90% = 27.10			
sd = 6.396		50% = 16.67	95% = 31.93			

%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

0.010 0.100 1.000 10.00 100.0 1000 10000

- Size (microns) -

SIZE	%PASS	%CHAN
1408	100.00	0.00
1184	100.00	0.00
995.6	100.00	0.00
837.2	100.00	0.00
704.0	100.00	0.00
592.0	100.00	0.00
497.8	100.00	0.00
418.6	100.00	0.00
352.0	100.00	0.00
296.0	100.00	0.00
248.9	100.00	0.00
209.3	100.00	0.00
176.0	100.00	0.00
148.0	100.00	0.00
124.5	100.00	0.00
104.7	100.00	0.00
88.00	100.00	0.00
74.00	100.00	0.00
62.23	100.00	0.41
52.33	99.59	0.74
44.00	98.85	1.45
37.00	97.40	2.97
31.11	94.43	5.95
26.16	88.48	10.80
22.00	77.68	16.29

SIZE	%PASS	%CHAN
18.50	61.39	18.93
15.56	42.46	16.51
13.08	25.95	11.39
11.00	14.56	6.85
9.250	7.71	3.89
7.778	3.82	2.14
6.541	1.68	1.12
5.500	0.56	0.56
4.625	0.00	0.00
3.889	0.00	0.00
3.270	0.00	0.00
2.750	0.00	0.00
2.312	0.00	0.00
1.945	0.00	0.00
1.635	0.00	0.00
1.375	0.00	0.00
1.156	0.00	0.00
0.972	0.00	0.00
0.818	0.00	0.00
0.688	0.00	0.00
0.578	0.00	0.00
0.486	0.00	0.00
0.409	0.00	0.00
0.344	0.00	0.00
0.289	0.00	0.00

SIZE	%PASS	%CHAN
0.243	0.00	0.00
0.204	0.00	0.00
0.172	0.00	0.00
0.145	0.00	0.00
0.122	0.00	0.00
0.102	0.00	0.00
0.086	0.00	0.00
0.072	0.00	0.00
0.061	0.00	0.00
0.051	0.00	0.00
0.043	0.00	0.00
0.036	0.00	0.00
0.030	0.00	0.00
0.026	0.00	0.00

SIZE	%PASS	%CHAN

Distribution: Volume
Progression: Standard
Upper Edge: 1408
Lower Edge: 0.021
Residuals: Disabled
Number Of Channels: 64

Filter: On

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Pd Malstrom
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

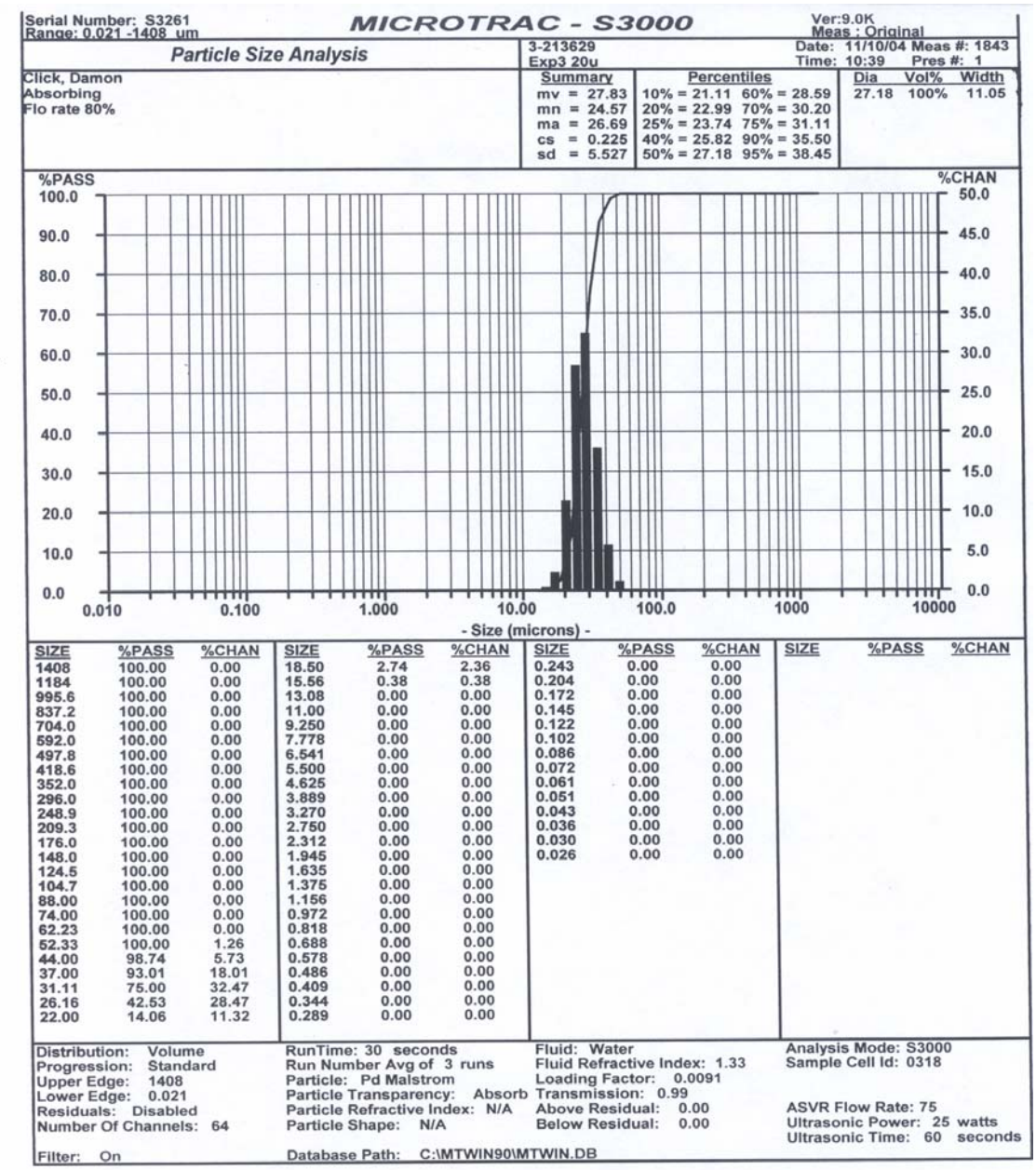
Database Path: C:\MTWIN90\MTWIN.DB

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.0300
Transmission: 0.95
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0318

ASVR Flow Rate: 75
Ultrasonic Power: 25 watts
Ultrasonic Time: 60 seconds

Figure D.2 **Microtrac analysis of solids collected on the 20 micron sieve from Exp 3 which was done with DI H_2O. Data is on a volume basis.**



Serial Number: S3261
Range: 0.021 -1408 um

MICROTRAC - S3000

Ver:9.0K
Meas : Original

Particle Size Analysis

3-213629
Exp3 20u

Date: 11/10/04 Meas #: 1843
Time: 10:39 Pres #: 1

Click, Damon
Absorbing
Flo rate 80%

Summary		Percentiles		Dia	Vol%	Width
mv = 27.83	10% = 21.11	60% = 28.59		27.18	100%	11.05
mn = 24.57	20% = 22.99	70% = 30.20				
ma = 26.69	25% = 23.74	75% = 31.11				
cs = 0.225	40% = 25.82	90% = 35.50				
sd = 5.527	50% = 27.18	95% = 38.45				

%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

0.010 0.100 1.000 10.00 100.0 1000 10000

- Size (microns) -

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1408	100.00	0.00	18.50	2.74	2.36	0.243	0.00	0.00			
1184	100.00	0.00	15.56	0.38	0.38	0.204	0.00	0.00			
995.6	100.00	0.00	13.08	0.00	0.00	0.172	0.00	0.00			
837.2	100.00	0.00	11.00	0.00	0.00	0.145	0.00	0.00			
704.0	100.00	0.00	9.250	0.00	0.00	0.122	0.00	0.00			
592.0	100.00	0.00	7.778	0.00	0.00	0.102	0.00	0.00			
497.8	100.00	0.00	6.541	0.00	0.00	0.086	0.00	0.00			
418.6	100.00	0.00	5.500	0.00	0.00	0.072	0.00	0.00			
352.0	100.00	0.00	4.625	0.00	0.00	0.061	0.00	0.00			
296.0	100.00	0.00	3.889	0.00	0.00	0.051	0.00	0.00			
248.9	100.00	0.00	3.270	0.00	0.00	0.043	0.00	0.00			
209.3	100.00	0.00	2.750	0.00	0.00	0.036	0.00	0.00			
176.0	100.00	0.00	2.312	0.00	0.00	0.030	0.00	0.00			
148.0	100.00	0.00	1.945	0.00	0.00	0.026	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	1.26	0.688	0.00	0.00						
44.00	98.74	5.73	0.578	0.00	0.00						
37.00	93.01	18.01	0.486	0.00	0.00						
31.11	75.00	32.47	0.409	0.00	0.00						
26.16	42.53	28.47	0.344	0.00	0.00						
22.00	14.06	11.32	0.289	0.00	0.00						

Distribution: Volume
Progression: Standard
Upper Edge: 1408
Lower Edge: 0.021
Residuals: Disabled
Number Of Channels: 64
Filter: On

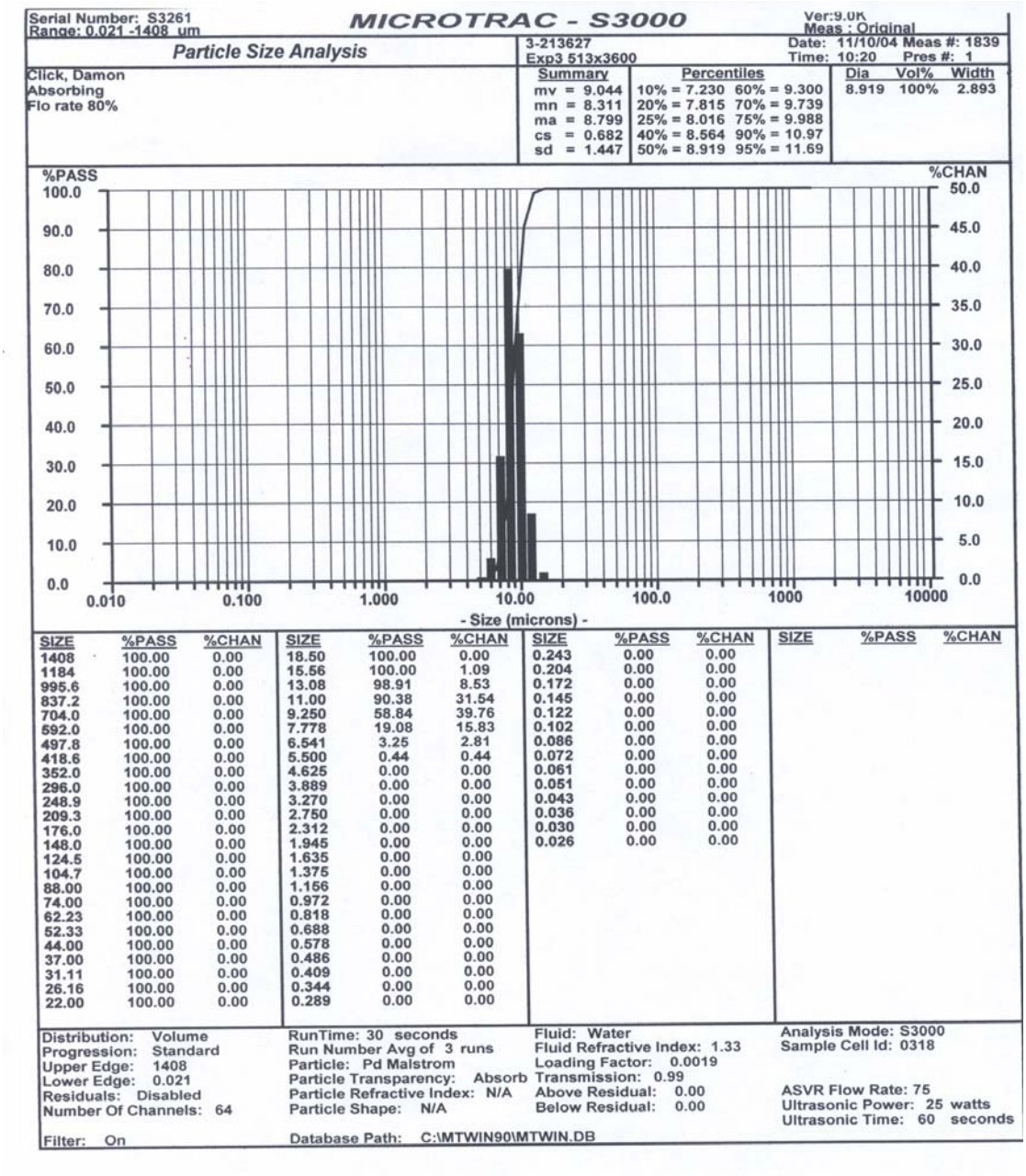
RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Pd Malstrom
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A
Database Path: C:\MTWIN90\MTWIN.DB

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.0091
Transmission: 0.99
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0318

ASVR Flow Rate: 75
Ultrasonic Power: 25 watts
Ultrasonic Time: 60 seconds

Figure D.3 Microtrac analysis of solids collected on the 5-6 micron sieve (510x3600 mesh). Data is on a volume basis.



Serial Number: S3261
Range: 0.021 -1408 um

MICROTRAC - S3000

Ver:9.0K
Meas : Original

Particle Size Analysis

3-213627
Exp3 513x3600

Date: 11/10/04 Meas #: 1839
Time: 10:20 Pres #: 1

Click, Damon
Absorbing
Flo rate 80%

Summary		Percentiles		Dia	Vol%	Width
mv = 9.044		10% = 7.230	60% = 9.300	8.919	100%	2.893
mn = 8.311		20% = 7.815	70% = 9.739			
ma = 8.799		25% = 8.016	75% = 9.988			
cs = 0.682		40% = 8.564	90% = 10.97			
sd = 1.447		50% = 8.919	95% = 11.69			

%PASS / %CHAN

100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0.0
50.0 45.0 40.0 35.0 30.0 25.0 20.0 15.0 10.0 5.0 0.0

0.010 0.100 1.000 10.00 100.0 1000 10000

- Size (microns) -

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1408	100.00	0.00	18.50	100.00	0.00	0.243	0.00	0.00			
1184	100.00	0.00	15.56	100.00	1.09	0.204	0.00	0.00			
995.6	100.00	0.00	13.08	98.91	8.53	0.172	0.00	0.00			
837.2	100.00	0.00	11.00	90.38	31.54	0.145	0.00	0.00			
704.0	100.00	0.00	9.250	58.84	39.76	0.122	0.00	0.00			
592.0	100.00	0.00	7.778	19.08	15.83	0.102	0.00	0.00			
497.8	100.00	0.00	6.541	3.25	2.81	0.086	0.00	0.00			
418.6	100.00	0.00	5.500	0.44	0.44	0.072	0.00	0.00			
352.0	100.00	0.00	4.625	0.00	0.00	0.061	0.00	0.00			
296.0	100.00	0.00	3.889	0.00	0.00	0.051	0.00	0.00			
248.9	100.00	0.00	3.270	0.00	0.00	0.043	0.00	0.00			
209.3	100.00	0.00	2.750	0.00	0.00	0.036	0.00	0.00			
176.0	100.00	0.00	2.312	0.00	0.00	0.030	0.00	0.00			
148.0	100.00	0.00	1.945	0.00	0.00	0.026	0.00	0.00			
124.5	100.00	0.00	1.635	0.00	0.00						
104.7	100.00	0.00	1.375	0.00	0.00						
88.00	100.00	0.00	1.156	0.00	0.00						
74.00	100.00	0.00	0.972	0.00	0.00						
62.23	100.00	0.00	0.818	0.00	0.00						
52.33	100.00	0.00	0.688	0.00	0.00						
44.00	100.00	0.00	0.578	0.00	0.00						
37.00	100.00	0.00	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						

Distribution: Volume
Progression: Standard
Upper Edge: 1408
Lower Edge: 0.021
Residuals: Disabled
Number Of Channels: 64

Filter: On

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Pd Malstrom
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Database Path: C:\MTWIN90\MTWIN.DB

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.0019
Transmission: 0.99
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0318

ASVR Flow Rate: 75
Ultrasonic Power: 25 watts
Ultrasonic Time: 60 seconds

Figure D.4 **Microtrac analysis of solids collected on the 8-9 micron sieve (325x2300 mesh) from Exp 3 which was done with DI H_2O. Data is on a volume basis.**

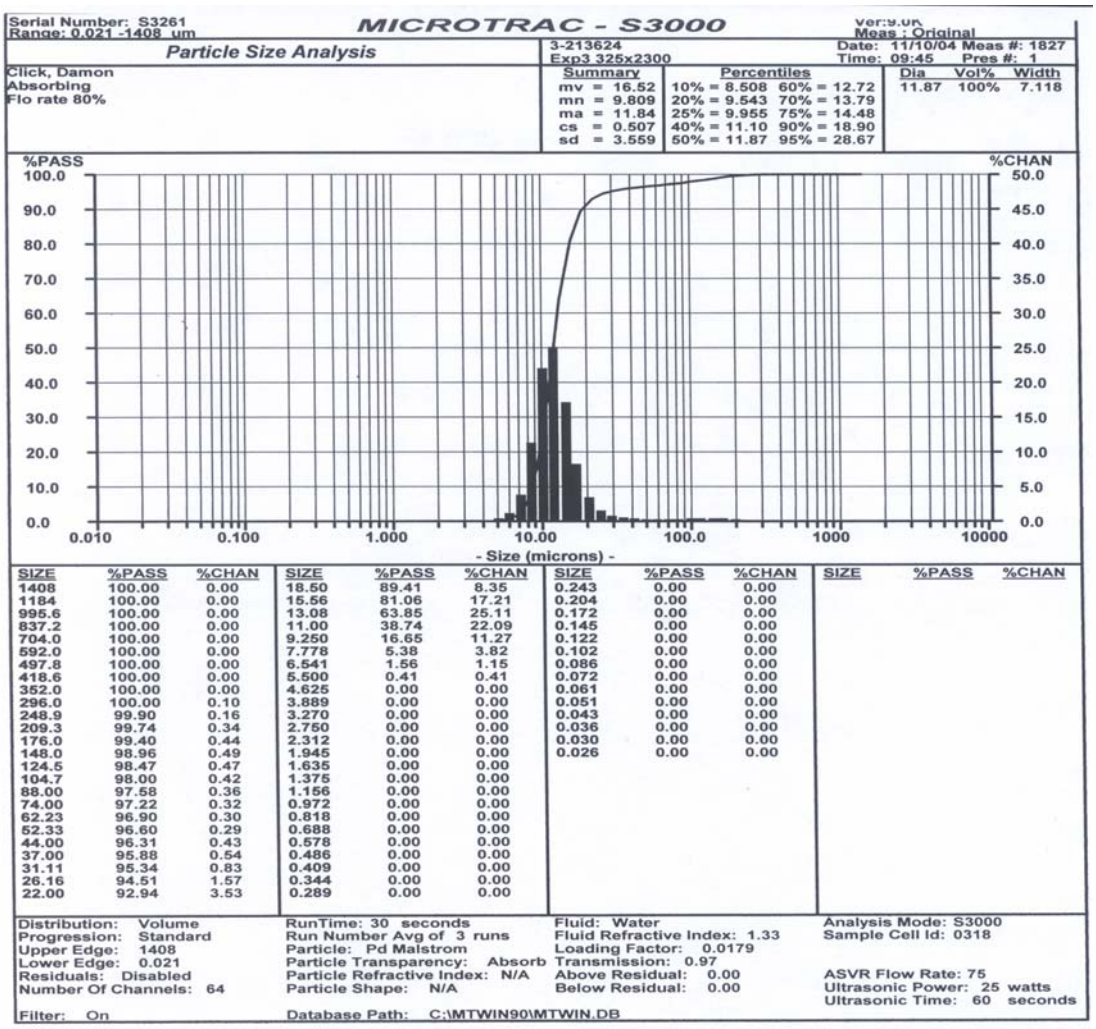
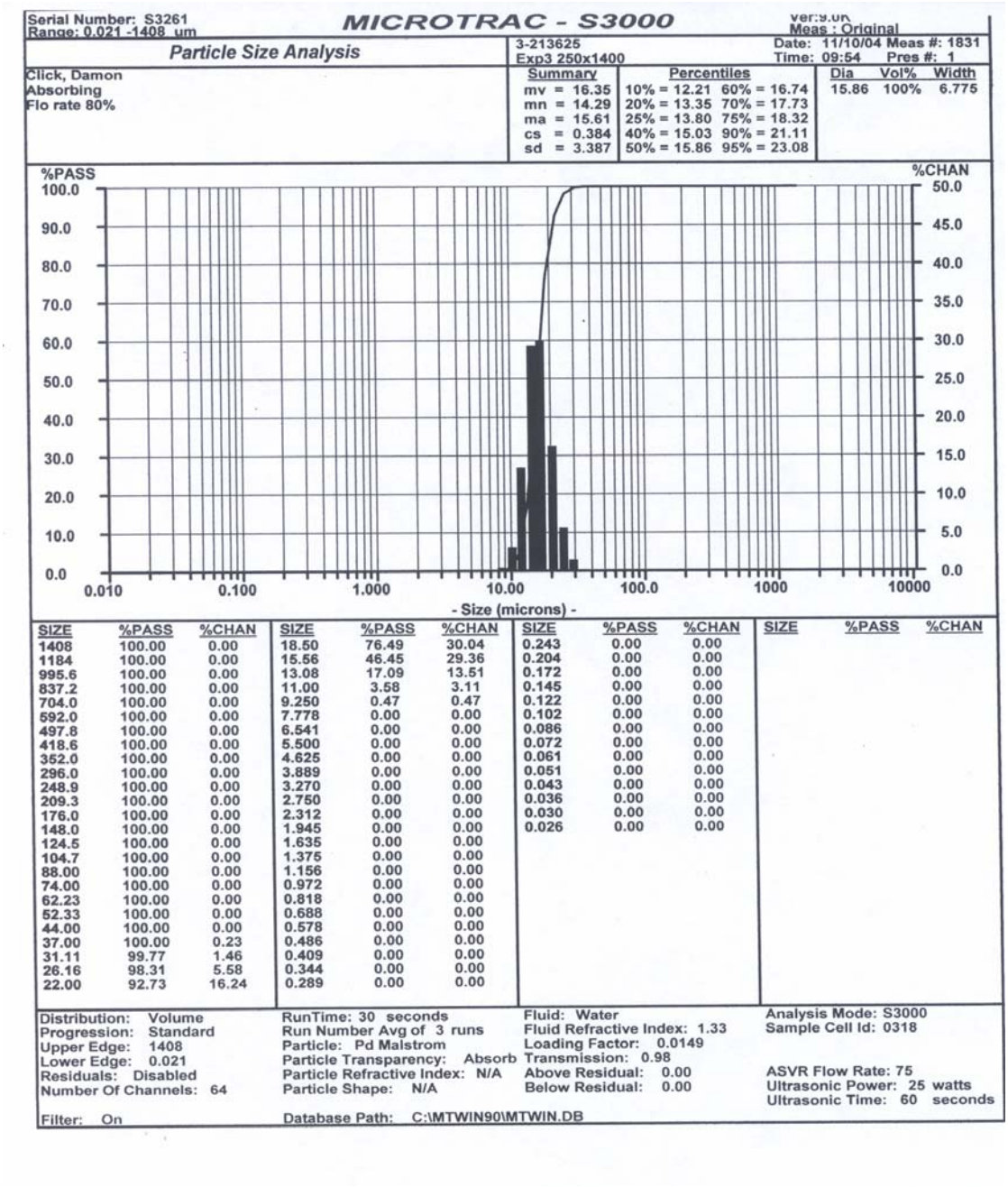


Figure D.5 **Microtrac analysis of solids collected on the 11-12 micron sieve (250x1400) from Exp 3 which was done with DI H_2O. Data is on a volume basis.**



Serial Number: S3261
Range: 0.021 -1408 um

MICROTRAC - S3000

Ver:9.0h
Meas : Original

Particle Size Analysis

3-213625
Exp3 250x1400

Date: 11/10/04 Meas #: 1831
Time: 09:54 Pres #: 1

Click, Damon
Absorbing
Flo rate 80%

Summary		Percentiles		Dia	Vol%	Width
mv = 16.35		10% = 12.21	60% = 16.74	15.86	100%	6.775
mn = 14.29		20% = 13.35	70% = 17.73			
ma = 15.61		25% = 13.80	75% = 18.32			
cs = 0.384		40% = 15.03	90% = 21.11			
sd = 3.387		50% = 15.86	95% = 23.08			

%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

0.010 0.100 1.000 10.00 100.0 1000 10000

- Size (microns) -

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1408	100.00	0.00	18.50	76.49	30.04	0.243	0.00	0.00			
1184	100.00	0.00	15.56	46.45	29.36	0.204	0.00	0.00			
995.6	100.00	0.00	13.08	17.09	13.51	0.172	0.00	0.00			
837.2	100.00	0.00	11.00	3.58	3.11	0.145	0.00	0.00			
704.0	100.00	0.00	9.250	0.47	0.47	0.122	0.00	0.00			
592.0	100.00	0.00	7.778	0.00	0.00	0.102	0.00	0.00			
497.8	100.00	0.00	6.541	0.00	0.00	0.086	0.00	0.00			
418.6	100.00	0.00	5.500	0.00	0.00	0.072	0.00	0.00			
352.0	100.00	0.00	4.625	0.00	0.00	0.061	0.00	0.00			
296.0	100.00	0.00	3.889	0.00	0.00	0.051	0.00	0.00			
248.9	100.00	0.00	3.270	0.00	0.00	0.043	0.00	0.00			
209.3	100.00	0.00	2.750	0.00	0.00	0.036	0.00	0.00			
176.0	100.00	0.00	2.312	0.00	0.00	0.030	0.00	0.00			
148.0	100.00	0.00	1.945	0.00	0.00	0.026	0.00	0.00			
124.5	100.00	0.00	1.635	0.00	0.00						
104.7	100.00	0.00	1.375	0.00	0.00						
88.00	100.00	0.00	1.156	0.00	0.00						
74.00	100.00	0.00	0.972	0.00	0.00						
62.23	100.00	0.00	0.818	0.00	0.00						
52.33	100.00	0.00	0.688	0.00	0.00						
44.00	100.00	0.00	0.578	0.00	0.00						
37.00	100.00	0.23	0.486	0.00	0.00						
31.11	99.77	1.46	0.409	0.00	0.00						
26.16	98.31	5.58	0.344	0.00	0.00						
22.00	92.73	16.24	0.289	0.00	0.00						

Distribution: Volume
Progression: Standard
Upper Edge: 1408
Lower Edge: 0.021
Residuals: Disabled
Number Of Channels: 64

Filter: On

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Pd Malstrom
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

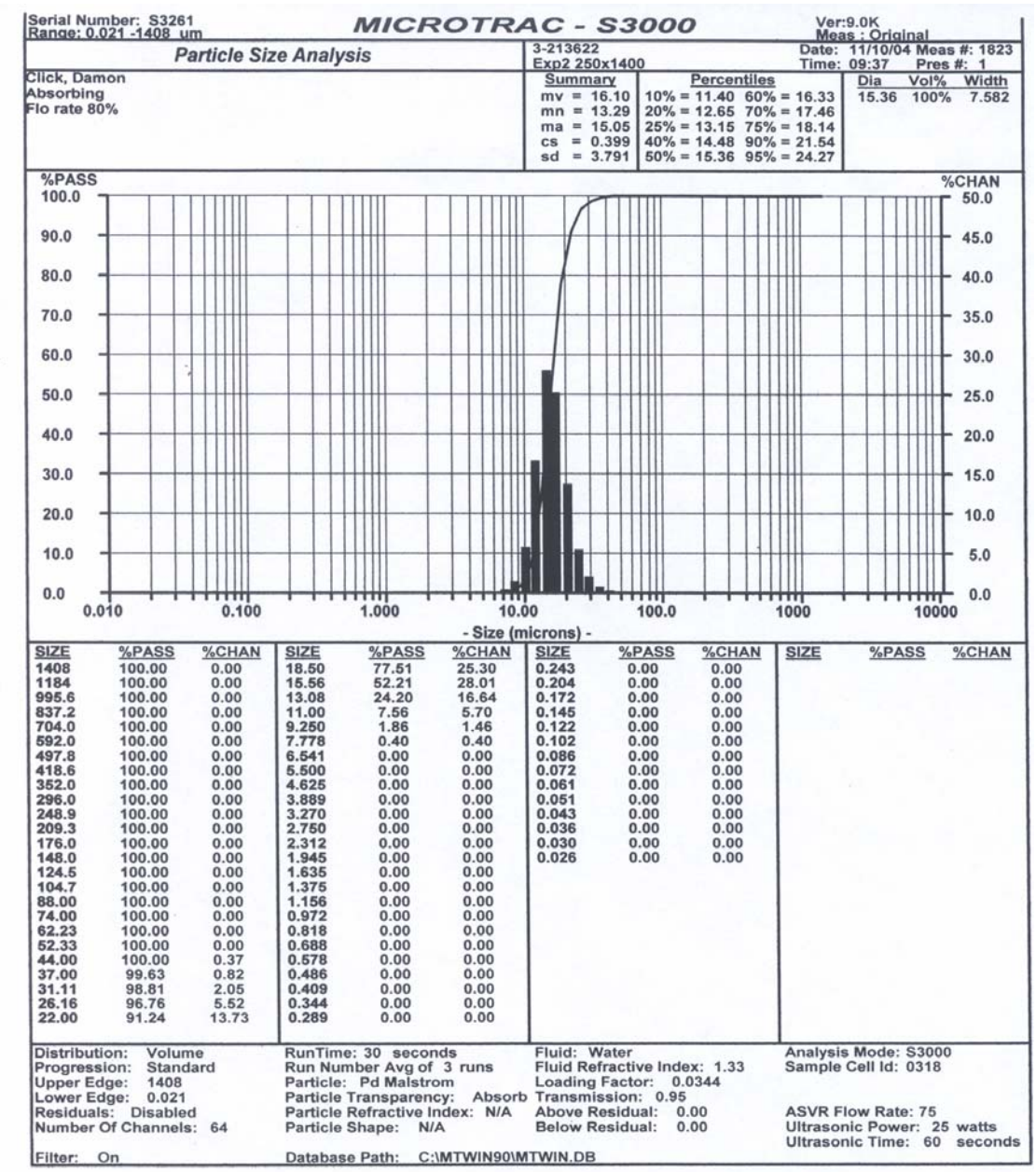
Database Path: C:\MTWIN90\MTWIN.DB

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.0149
Transmission: 0.98
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0318

ASVR Flow Rate: 75
Ultrasonic Power: 25 watts
Ultrasonic Time: 60 seconds

Figure D.6 **Microtrac analysis of solids collected on the 11-12 micron sieve (250x1400) from Exp 2 which was done with DI H_2O. Data is on a volume basis.**



Serial Number: S3261
Range: 0.021 -1408 um

MICROTRAC - S3000

Ver:9.0K
Meas : Original

Particle Size Analysis

3-213622
Exp2 250x1400

Date: 11/10/04 Meas #: 1823
Time: 09:37 Pres #: 1

Click, Damon
Absorbing
Flo rate 80%

Summary		Percentiles		Dia	Vol%	Width
mv = 16.10	10% = 11.40	60% = 16.33		15.36	100%	7.582
mn = 13.29	20% = 12.65	70% = 17.46				
ma = 15.05	25% = 13.15	75% = 18.14				
cs = 0.399	40% = 14.48	90% = 21.54				
sd = 3.791	50% = 15.36	95% = 24.27				

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1408	100.00	0.00	18.50	77.51	25.30	0.243	0.00	0.00			
1184	100.00	0.00	15.56	52.21	28.01	0.204	0.00	0.00			
995.6	100.00	0.00	13.08	24.20	16.64	0.172	0.00	0.00			
837.2	100.00	0.00	11.00	7.56	5.70	0.145	0.00	0.00			
704.0	100.00	0.00	9.250	1.86	1.46	0.122	0.00	0.00			
592.0	100.00	0.00	7.778	0.40	0.40	0.102	0.00	0.00			
497.8	100.00	0.00	6.541	0.00	0.00	0.086	0.00	0.00			
418.6	100.00	0.00	5.500	0.00	0.00	0.072	0.00	0.00			
352.0	100.00	0.00	4.625	0.00	0.00	0.061	0.00	0.00			
296.0	100.00	0.00	3.889	0.00	0.00	0.051	0.00	0.00			
248.9	100.00	0.00	3.270	0.00	0.00	0.043	0.00	0.00			
209.3	100.00	0.00	2.750	0.00	0.00	0.036	0.00	0.00			
176.0	100.00	0.00	2.312	0.00	0.00	0.030	0.00	0.00			
148.0	100.00	0.00	1.945	0.00	0.00	0.026	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	0.37	0.578	0.00	0.00						
37.00	99.63	0.82	0.486	0.00	0.00						
31.11	98.81	2.05	0.409	0.00	0.00						
26.16	96.76	5.52	0.344	0.00	0.00						
22.00	91.24	13.73	0.289	0.00	0.00						

Distribution: Volume
Progression: Standard
Upper Edge: 1408
Lower Edge: 0.021
Residuals: Disabled
Number Of Channels: 64
Filter: On

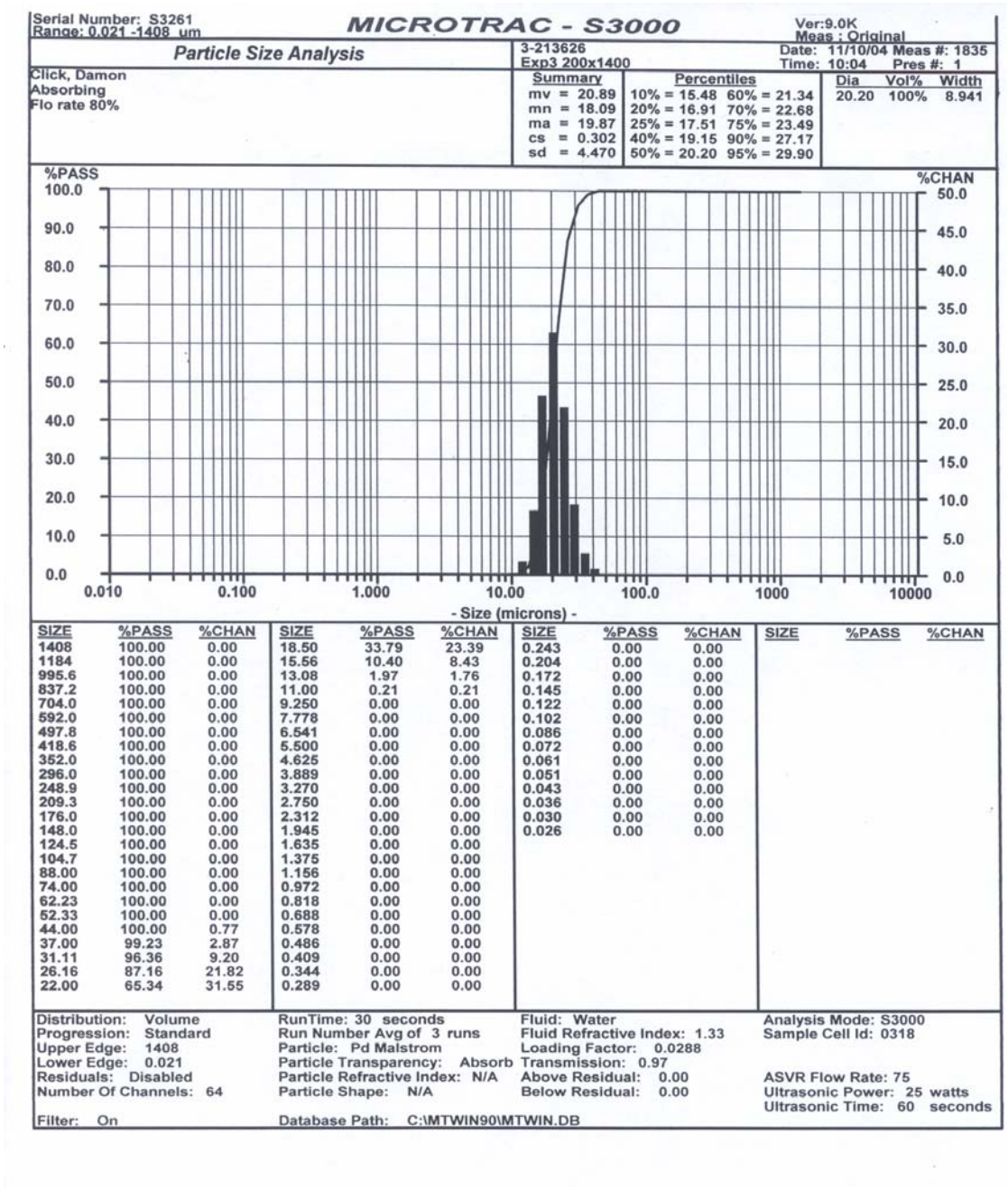
RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Pd Malstrom
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A
Database Path: C:\MTWIN90\MTWIN.DB

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.0344
Transmission: 0.95
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0318

ASVR Flow Rate: 75
Ultrasonic Power: 25 watts
Ultrasonic Time: 60 seconds

Figure D.7 **Microtrac analysis of solids collected on the 12-14 micron sieve (200x1400 mesh) from Exp 3 which was done with DI H_2O. Data is on a volume basis.**



Serial Number: S3261
Range: 0.021 -1408 um

MICROTRAC - S3000

Ver:9.0K
Meas : Original

Particle Size Analysis

3-213626
Exp3 200x1400

Date: 11/10/04 Meas #: 1835
Time: 10:04 Pres #: 1

Click, Damon
Absorbing
Flo rate 80%

Summary		Percentiles		Dia	Vol%	Width
mv	= 20.89	10% = 15.48	60% = 21.34	20.20	100%	8.941
mn	= 18.09	20% = 16.91	70% = 22.68			
ma	= 19.87	25% = 17.51	75% = 23.49			
cs	= 0.302	40% = 19.15	90% = 27.17			
sd	= 4.470	50% = 20.20	95% = 29.90			

%PASS
%CHAN
100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0.0
50.0 45.0 40.0 35.0 30.0 25.0 20.0 15.0 10.0 5.0 0.0
0.010 0.100 1.000 10.00 100.0 1000 10000
- Size (microns) -

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1408	100.00	0.00	18.50	33.79	23.39	0.243	0.00	0.00			
1184	100.00	0.00	15.56	10.40	8.43	0.204	0.00	0.00			
995.6	100.00	0.00	13.08	1.97	1.76	0.172	0.00	0.00			
837.2	100.00	0.00	11.00	0.21	0.21	0.145	0.00	0.00			
704.0	100.00	0.00	9.250	0.00	0.00	0.122	0.00	0.00			
592.0	100.00	0.00	7.778	0.00	0.00	0.102	0.00	0.00			
497.8	100.00	0.00	6.541	0.00	0.00	0.086	0.00	0.00			
418.6	100.00	0.00	5.500	0.00	0.00	0.072	0.00	0.00			
352.0	100.00	0.00	4.625	0.00	0.00	0.061	0.00	0.00			
296.0	100.00	0.00	3.889	0.00	0.00	0.051	0.00	0.00			
248.9	100.00	0.00	3.270	0.00	0.00	0.043	0.00	0.00			
209.3	100.00	0.00	2.750	0.00	0.00	0.036	0.00	0.00			
176.0	100.00	0.00	2.312	0.00	0.00	0.030	0.00	0.00			
148.0	100.00	0.00	1.945	0.00	0.00	0.026	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	0.77	0.578	0.00	0.00						
37.00	99.23	2.87	0.486	0.00	0.00						
31.11	96.36	9.20	0.409	0.00	0.00						
26.16	87.16	21.82	0.344	0.00	0.00						
22.00	65.34	31.55	0.289	0.00	0.00						

Distribution: Volume
Progression: Standard
Upper Edge: 1408
Lower Edge: 0.021
Residuals: Disabled
Number Of Channels: 64
Filter: On

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Pd Malstrom
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A
Database Path: C:\MTWIN90\MTWIN.DB

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.0288
Transmission: 0.97
Above Residual: 0.00
Below Residual: 0.00

Analysis Mode: S3000
Sample Cell Id: 0318

ASVR Flow Rate: 75
Ultrasonic Power: 25 watts
Ultrasonic Time: 60 seconds

Figure D.8 **Microtrac analysis of solids collected on the 12-14 micron sieve (200x1400) from Exp 2 which was done with DI H_2O. Data is on a volume basis.**