

# Results of Small-Scale Particle Cloud Tests and Non-Newtonian Fluid Cavern Tests

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November 2003

Prepared for Bechtel National, Inc.  
under Contract 24590-101-TSA-W0000-0004

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 11/19/03  
for W. Tamsaitis

November 2003

**ACCEPTED FOR  
WTP PROJECT USE**

Test Specification: none  
Test Plan: TP-PNNL-WTP-051  
Test Exception: 24590-WTP-TEF-RT-02-057  
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## Completeness of Testing

This report describes the results of work and testing specified by Test Plan TP-PNNL-WTP-051. The work and any associated testing followed the quality assurance requirements outlined in the Test Specification and Test Plan. The descriptions provided in this test report are an accurate account of both the conduct of the work and the data collected. Test plan results are reported. Also reported are any unusual or anomalous occurrences that are different from expected results. The test results and this report have been reviewed and verified.

Approved:



Gordon H. Beeman, Manager  
WTP R&T Support Project

11/17/03  
Date

# Summary

## Objectives

The objective of these tests performed by Battelle – Pacific Northwest Division (PNWD) was to generate experimental data to be used by Bechtel National, Inc. (BNI) in their effort to evaluate the FLUENT computer code models.

The objective of the cloud height tests was to obtain experimental measurements of the effective mixing heights for BNI to use in benchmarking the FLUENT computer code. The cloud height measurements were obtained for a single steady-state jet directed downward in an elliptical bottom tank. The cloud tests used glass beads in water to evaluate the height of the suspended slurry as a function of jet velocity.

The objective of the cavern tests was to obtain experimental data to validate the non-Newtonian fluid modeling capabilities of the computer code for fluid properties similar to those of certain tank wastes. A transparent material that exhibited a yield stress and shear thinning behavior was used to obtain measurements of steady-state cavern heights as a function of jet velocity. The simulant also exhibited time-dependent behavior. To evaluate the influence of the time-dependent behavior, constant shear rate tests were carried out. The measured shear stresses dropped continually for the first 20 minutes. After approximately 20 minutes, the change in shear stress was less than 1%. The magnitude of the change in rheological properties at steady-state conditions over the time steady-state measurements were made was negligible.

This document summarizes the tests and presents the experimental results produced at the small-scale PJM test setup in the Applied Process Engineering Laboratory (APEL). The scope of work for this effort was defined by Table 1, items 8, 9, and 10 of R&T Test Exception 24590-WTP-TEF-RT-02-057. All of the goals defined by the test exception for this testing effort were met.

## Conduct of Testing

The test setup consisted of a 33.5 inch-diameter, 300-gallon, cylindrical, clear acrylic tank with an elliptical bottom and a mockup PJM that was centered in the tank. The PJM was fitted with a 1-inch tube down the center to be used for the vertical jet nozzle. The downward-directed jet exit was 8.6 inches above the tank bottom. Four equally spaced 1-inch-outer diameter suction lines were situated at the tank's outer wall. The suction lines were in a vertical orientation with the inlets facing downward. The inlet elevation for the suction lines was approximately 9 inches above the tank bottom. Continuous flow was achieved using a centrifugal pump and monitored with an inline flow meter.

For the cloud test, separate tests were conducted with size-sorted, spherical, soda-lime glass beads and water simulants having nominal bead diameters of 35 (small diameter) and 71  $\mu\text{m}$ . (large diameter). Both bead sizes were washed to remove the bulk of the fine particulate. These beads had a specific gravity of 2.5 and median particle diameters of 48 (small) and 66  $\mu\text{m}$  (large), respectively, based on volume distribution after washing.

For each bead size, testing was conducted using an aqueous slurry with nominal 10 and 20 wt% beads, and the jet was operated at approximately 10 to 19 ft/sec. The tank was filled with a total volume of approximately 110 gallons for each cloud test. Testing began by setting the flow to the lowest velocity to be tested. The cloud height was monitored until a steady-state condition was achieved. The resulting steady-state cloud height of suspended particulate was measured at the tank wall and videotaped. The velocity was then increased to the next test condition.

Grab samples of the slurry cloud and other areas of the tank were collected at each simulant loading and jet velocity. Particle size distributions (PSD) were measured for a select number of the collected samples. The PSD results were obtained for information only and are included in an appendix.

Cavern testing used a transparent non-Newtonian fluid to observe the formation of a cavern of mobilized material due to the shear-sensitive properties of the fluid. The test simulant was 2.08 wt% Laponite in water with a measured shear strength of 48 Pa for the undisturbed material. By fitting rheogram data for initially undisturbed material to a Bingham plastic model, a yield stress of 12.3 Pa and a plastic viscosity of 6.8 mPa-s were obtained.

To ensure a homogeneous mixture of Laponite, the material was mixed in a separate tank and allowed to sit for approximately 20 hours. The material was then remixed and approximately 160 gallons transferred to the test tank, where it was allowed to set up for another 40 hours to establish the undisturbed shear strength. The Laponite mixture exhibits time dependence with respect to rheology. Rheological measurements were made for the undisturbed material and for material having experienced a high degree of shear for an extended time. These measurements were able to provide bounding rheological properties for the test simulant over the range of shear rates predicted to occur within the cavern. Shear vane tests demonstrated that the material set up to its original strength after being remixed.

Flow in the circulating loop was initiated in a bypass line, where dye was added to the flow loop to enhance visual measurement of the interface between the flowing versus non-yielding stagnant material. Flow was diverted to the discharge nozzle, and the cavern height was monitored. After observing a steady-state cavern height, cavern heights were recorded at every 45 degrees of tank circumference. The jet velocity was then increased to the next test condition.

## Results and Performance Against Objectives

Cloud testing revealed that the glass bead material was suspended in the tank quickly and developed a distinctive height for each combination of flow rate and particulate size tested. As expected, the solids loading had minimal impact on the cloud height for a given particle size. Cloud heights of 10 to 25 inches were obtained for the small-diameter beads and 10 to 14 inches for the large diameter beads. During all of the cloud tests, the surface of the tank remained totally calm, indicating that the slurry was dissipating the mixing energy of the relatively high-velocity jet. Table S.1 provides a summary of the results for the cloud height tests.

For the non-Newtonian fluid cavern tests, distinct cavern volumes were readily developed for the four velocities tested, which ranged from 12 to 26 ft/sec. The average maximum cavern heights ranged from 14 to 40 inches above the center of the tank bottom for the range of velocities tested. A linear relationship was observed to exist between cavern height and nozzle velocity. For each steady-state cavern size, samples of the circulating cavern material were collected for rheological characterization of the fluid. Table S.2 summarizes the results of the cavern tests.

**Table S.1.** Cloud Height Test Results

<b>Median Particle Diameter Based on Volume Distribution</b> ( $\mu\text{m}$ )	<b>Concentration</b> (wt%)	<b>Average Nozzle Discharge Velocity</b> <sup>(a)</sup> (ft/sec)	<b>Average Nozzle Flow Rate</b> <sup>(b)</sup> (gpm)	<b>Average Cloud Height from Tank Bottom</b> <sup>(c)</sup> (in.)
48	11.1 $\pm$ 0.8	10.9 Stdv =0.1	20.3 Stdv =0.2	9.8 Stdv =0.0
48	11.1 $\pm$ 0.8	17.9 Stdv =0.1	33.2 Stdv =0.2	16.5 Stdv =0.5
48	11.1 $\pm$ 0.8	19.1 Stdv =0.1	35.4 Stdv =0.2	24.8 Stdv =0.0
48	21.9 $\pm$ 1.0	9.6 Stdv =0.1	17.9 Stdv =0.2	10.8 Stdv =0.0
48	21.9 $\pm$ 1.0	15.9 Stdv =0.0	29.5 Stdv =0.1	16.8 Stdv =0.0
48	21.9 $\pm$ 1.0	19.0 Stdv =0.1	35.2 Stdv =0.2	23.8 Stdv =0.0
66	12.0 $\pm$ 0.6	10.8 Stdv =0.0	20.1 Stdv =0.1	9.8 Stdv =0.0
66	12.0 $\pm$ 0.6	18.8 Stdv =0.1	34.8 Stdv =0.1	11.8 Stdv =0.0
66	20.5 $\pm$ 0.8	10.3 Stdv =0.1	19.2 Stdv =0.1	9.8 Stdv =0.0
66	20.5 $\pm$ 0.8	18.5 Stdv =0.0	34.2 Stdv =0.1	13.8 Stdv =0.0

(a) Calculated from flow rate measurements; uncertainty associated with single values is  $\pm 4\%$  of value.  
 (b) Uncertainty associated with single measurements is  $\pm 3\%$  of reading based on calibration.  
 (c) The uncertainty associated with single measurements is  $\pm 0.6$  inch.

**Table S.2.** Cavern Test Steady-State Results for 2.08 wt% Laponite

Average Nozzle Flow Rate <sup>(a)</sup> (gpm)	Average Nozzle Discharge Velocity <sup>(b)</sup> (ft/sec)	Avg Max Cavern Ht above Bottom Center of Tank <sup>(c)</sup> (in.)	Avg Cavern Ht at Tank Wall above Bottom Center of Tank <sup>(c)</sup> (in.)	Cavern Fluid Temperature (±0.5°F)	Shear strength at beginning of test (Pa)	Consistency (cP)
23.4 Stdv=0.0	12.6 Stdv=0.0	13.8 Stdv=1.2	9.7 Stdv=2.0	77	44 Stdv=2	7.9
32.0 Stdv=0.2	17.3 Stdv=0.1	22.0 Stdv=1.2	20.3 Stdv=2.3	78	44 Stdv=2	8.1
42.8 Stdv=0.2	23.1 Stdv=0.1	34.9 Stdv=0.9	29.7 Stdv=1.7	80	44 Stdv=2	6.7
48.9 Stdv=0.2	26.4 Stdv=0.1	40.1 Stdv=1.3	34.3 Stdv=1.6	80	44 Stdv=2	6.3
(a) The uncertainty associated with single measurements is ±2.0% of reading based on calibration.						
(b) Calculated from flow rate measurements. Uncertainty associated with single values is ±3.0% of value.						
(c) The uncertainty associated with single measurements is ±0.6 inches.						

## Quality Requirements

PNWD implements the RPP-WTP quality requirements by performing work in accordance with the Waste Treatment Plant Support Project quality assurance project plan (QAPjP) approved by the RPP-WTP Quality Assurance (QA) organization. This QA manual is a web-based manual managed by the PNWD WTP QA engineer. This work was performed to the quality requirements of NQA-1-1989 Part I, “Basic and Supplementary Requirements,” and NQA-2a-1990, Part 2.7. These quality requirements were implemented through PNWD’s *Waste Treatment Plant Support Project (WTPSP) Quality Assurance Requirements and Description Manual*. The analytical requirements are implemented through PNWD’s *Conducting Analytical Work in Support of Regulatory Programs*.

For calculating the cloud and cavern height dimensions, independent measurements were performed by two individuals. PNWD addressed verification activities by conducting an independent technical review of the final data report in accordance with procedure QA-RPP-WTP-604. This review verified that the reported results were traceable, that inferences and conclusions were soundly based, and the reported work satisfied the test plan objectives. The review procedure is part of PNWD’s WTPSP Manual.

## Issues

These results were obtained to support the BNI computer modeling effort using the FLUENT computer code. All testing was conducted with a continuous flowing jet, and no attempt was made to compensate for scaling issues. These test results should not be used to directly predict WTP performance or make technical decisions regarding plant design or operation.

## Acronyms and Definitions

APEL	Advanced Process Engineering Laboratory
Avg	average
BNI	Bechtel National Inc.
°C	degrees Celsius
DAS	data acquisition system
°F	degrees Fahrenheit
ft/sec	feet per second
gpm	gallons per minute
ht	height
in.	inches
L/min	liters per minute
LRB	laboratory record book
Max	maximum
M&TE	measuring and testing equipment
mPa-s	milliPascal –seconds
Pa	Pascal
PJM	pulse jet mixer
PNWD	Battelle – Pacific Northwest Division
PSD	particle size distribution
QAPjP	Quality Assurance Project Plan
R&T	Research and Technology
RPP	River Protection Program
s or sec	seconds
Stdv	standard deviation
WTP	Waste Treatment Plant
wt%	weight percent
µm	micrometers (micron)



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# 1.0 Introduction

This work was performed for Bechtel National Inc. (BNI) by Battelle – Pacific Northwest Division (PNWD) in the small-scale pulse-jet mixer (PJM) test setup at the Applied Process Engineering Laboratory (APEL). The effort is part of the work scope covered by test plan TP-PNNL-WTP-051 (Bontha 2001).

Testing was conducted according to test procedure TPR–RPP-WTP-220 (Bontha 2002). The data for the cavern tests were collected according to test instruction TI–RPP-WTP-240 (Enderlin 2003). Data associated with this task were recorded in the following laboratory record books (LRB): BNW-14125 and BNW-14314.

Section 1.1 provides some background for the testing. The scope of testing is described in Section 1.2.

## 1.1 Background

BNI is performing work to validate the FLUENT computer code model of PJMs in tanks with the same configuration as those to be installed in the Hanford Waste Treatment Plant (WTP). Previous work associated with obtaining experimental data for the validation of the FLUENT computer code to model PJM systems has been conducted in both the large-scale (336 Building) and small-scale (APEL) test setups. The glass particles used for the work described in this report have also been used for large-scale testing (Bontha 2003). The associated work is documented in WTP-RPT-077 (Johnson 2002) and WTP-RPT-081 (Bontha 2003).

## 1.2 Scope of Work

The objective of the cloud test was to obtain experimental data for BNI to benchmark the computer code FLUENT. The objective of the cavern tests was to obtain experimental data that could be used by BNI to benchmark the non-Newtonian fluid modeling capability of the FLUENT computer code for predicting PJM mixing system performance.

The cloud test task was conducted to obtain steady-state experimental data that characterized the suspended slurry produced by a steady-state jet directed vertically downward in a cylindrical tank with an elliptical bottom. The jet was produced by a closed-loop system that recirculated flow from the tank through four symmetrical suction lines. The test matrix was performed for a fixed geometry and varied jet velocity, mean particle diameter, and solids concentration.

Two sizes of glass bead media were tested with water as the carrier fluid. For each size medium, at least two concentrations were tested. During testing, the flow rate through the loop was set and the system monitored until a steady-state cloud height was observed. Data were collected on the flow rate, tank temperature, and suspended cloud (slurry) height. The jet velocity was then

increased and the data recorded for a new steady-state cloud height. At least two data sets were recorded for each solids concentration tested. Particle size distributions (PSD) of the suspended slurries were measured.

The purpose of the cavern tests was to establish a submerged region of flowing material (the cavern) within a shear-thinning fluid that exhibited yield stress behavior and to quantify the steady-state dimensions of the cavern (the flowing region of the tank) using the same geometrical configuration as the cloud test. The simulant also exhibited time-dependent behavior. To evaluate the influence of the time-dependent behavior, constant shear rate tests were carried out. As can be seen in Section 4 (Figure 4.5), rheology tests were performed for different constant shear rates over a minimum of 60 minutes. The measured shear stress drops continually for the first 20 minutes. Between 20 and 30 minutes after the start of the test, the change in shear stress was less than 0.5%. The average values between 20 and 30 minutes are 10.3, 17 and 18.7 Pa for shear rates of 100, 800, and 1150  $s^{-1}$  for 2.08 wt% Laponite. The magnitude of the change in rheological properties was negligible at steady-state conditions over the time steady-state measurements were made.

The interface between the flowing region of the tank and the stagnant, non-sheared material produces a map of constant shear rate within the tank. This was done by preparing a transparent, water-based material with additives that produced shear-sensitive behavior. A centrifugal pump was used to induce flow through the closed-loop test setup that had the same internal tank geometry as that used for the cloud tests. As with the cloud tests, the flow rate through the loop was set and the system monitored until a steady-state interface between the sheared (flowing) fluid and the unsheared or jelled material was observed. Data were collected on the flow rate, in-line density, tank temperature, and cavern (interface) height. The jet velocity was then increased and data recorded for a new steady-state cavern.

The selection of the additive and its concentration was based on both rheological and optical properties. Rheological characterization was performed on the test material.

The test setup is presented in Section 2. The results of the cloud tests are described in Section 3 and those for the cavern tests in Section 4. Conclusions are presented in Section 5, and cited references are listed in Section 6. Number and volume distributions are depicted graphically in an appendix. This effort was tasked with collecting and transmitting the experimental data to BNI. Minimal analyses of the data have been performed.

## 2.0 Test Setup

All experiments were performed in a 300-gallon, 33.5-inch-diameter, optically transparent acrylic PJM test tank in the high-bay area of the APEL. The tank has a 2:1 elliptical bottom. Its internal configuration was the same for both the cloud and cavern tests. The jet nozzle consisted of a straight section of 1-inch tubing with a 0.065-inch-thick wall. Therefore, the nozzle diameter was 0.87 inch. Figure 2.1 presents a schematic of the internal configuration and dimensions for the test tank.

The system consists of an acrylic tank with a transparent scaled PJM suspended in the center. Previous testing mixed the fluid in the tank by alternating pressure and vacuum to draw fluid into the PJM and then expel it. For the two tests reported here, the test system was modified to allow continuous flow operation by retrofitting the PJM with a 1-inch tube centered in a plug that sealed the existing 2-inch orifice at the bottom of the PJM. The discharge of the 1-inch tube was 0.5 inch below the PJM bottom and 8.6 inches above the tank bottom (see E and L in Figure 2.1).

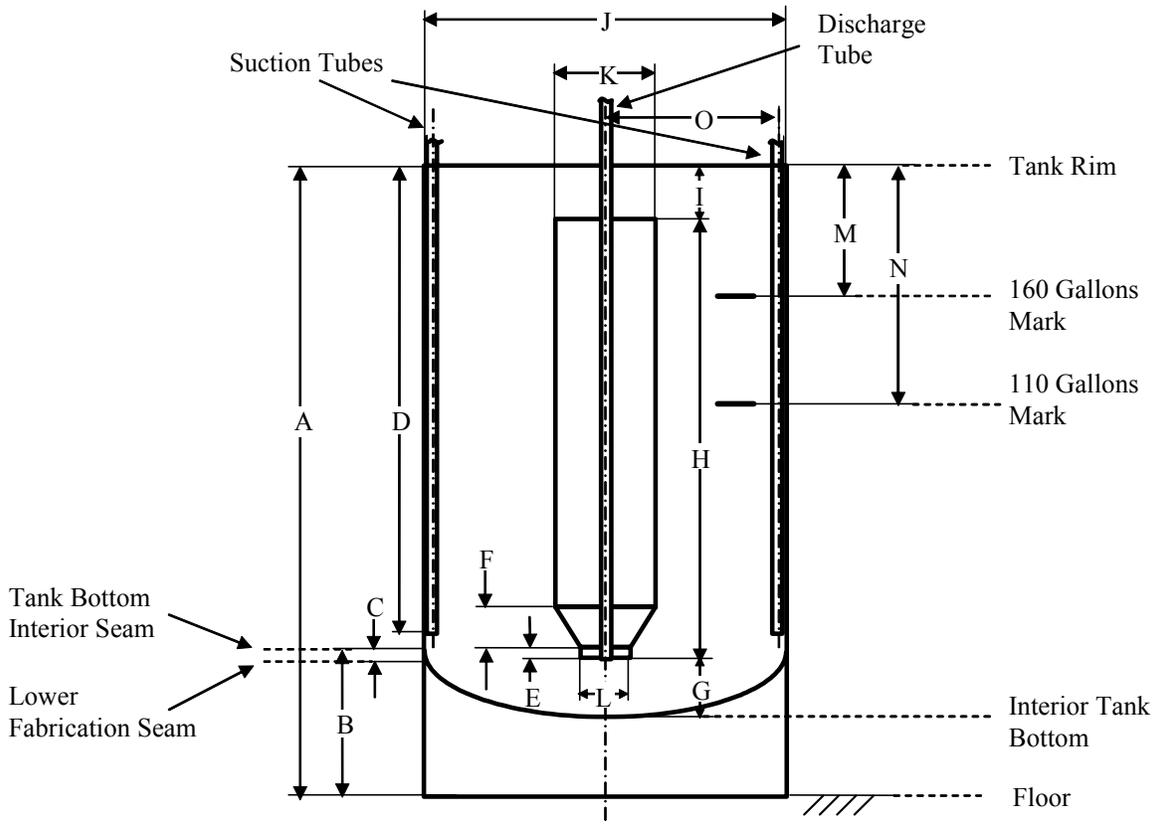
At the top of the PJM, the 1-inch tube was connected to the discharge from a centrifugal circulating pump. During the experiments, return flow was drawn through four symmetrical suction lines with inlets located 9 and 10 inches above the tank center bottom for the cloud and cavern tests, respectively. The inlets were situated at the tank wall with a centerline radius of 16.2 inches. A manifold at the top of the tank combined the four suction lines into a common return line that was routed to the inlet of the centrifugal pump. An in-line flow meter was installed in the pump discharge line.

While the internal tank configuration was the same for both the cavern and the cloud tests, the flow loop and instrumentation were modified after the cloud test to meet the needs of the cavern tests. Sections 2.1 and 2.2 describe the test setups and operations unique to the cloud and cavern tests, respectively.

### 2.1 Cloud Test Setup and Operation

The flow loop for the cloud tests was powered by a 2-hp centrifugal pump mounted on a level of scaffolding near the top of the tank. Flow was controlled with a ball valve located just downstream of the pump discharge. Figure 2.2 is a schematic of the system.

Data acquisition consisted of nozzle flow rate, loop temperature, manually measured cloud height taken from the side of the tank, and 50-mL grab samples taken from several locations within the tank. Grab samples were extracted from elevations 3 inches above the cloud, 3 inches below the top of the cloud, the bottom of the tank, and the recirculation loop. A thermocouple measured the liquid temperature during testing, and the measurement was recorded in the laboratory record book (LRB). The thermocouple met the project data quality requirements.



Schematic: Not Drawn to Scale

A=96.0 in.	B*=20.9 to 21.5 in.	C*=1.6 to 1.9 in.	D <sup>1+</sup> =74.8 in.	D <sup>2+</sup> =73.5 in.
E=0.6 in.	F=7.0 in.	G=8.6 in.	H=50.0 in.	I=25.0 in.
J**=33.5 in.		K°=11.1 in.	L°=3.0 in.	M=34.5 in.
N=49.0 in.		O°=16.2 in.		

Discharge Tube: 1 in. OD, 0.065 in. wall thickness

Suction Tubes (4 total): 1 in. OD, 0.048 in. wall thickness

Measurements ±0.3 in. except where noted

D<sup>1+</sup> Cloud Testing Suction Inlet Position; Uncertainty ±0.4 in.

D<sup>2+</sup> Cavern Testing Suction Inlet Position; Uncertainty ±0.3 in.

\* = Varies with azimuthally position

\*\* = Inner Diameter

° = Outer Diameter

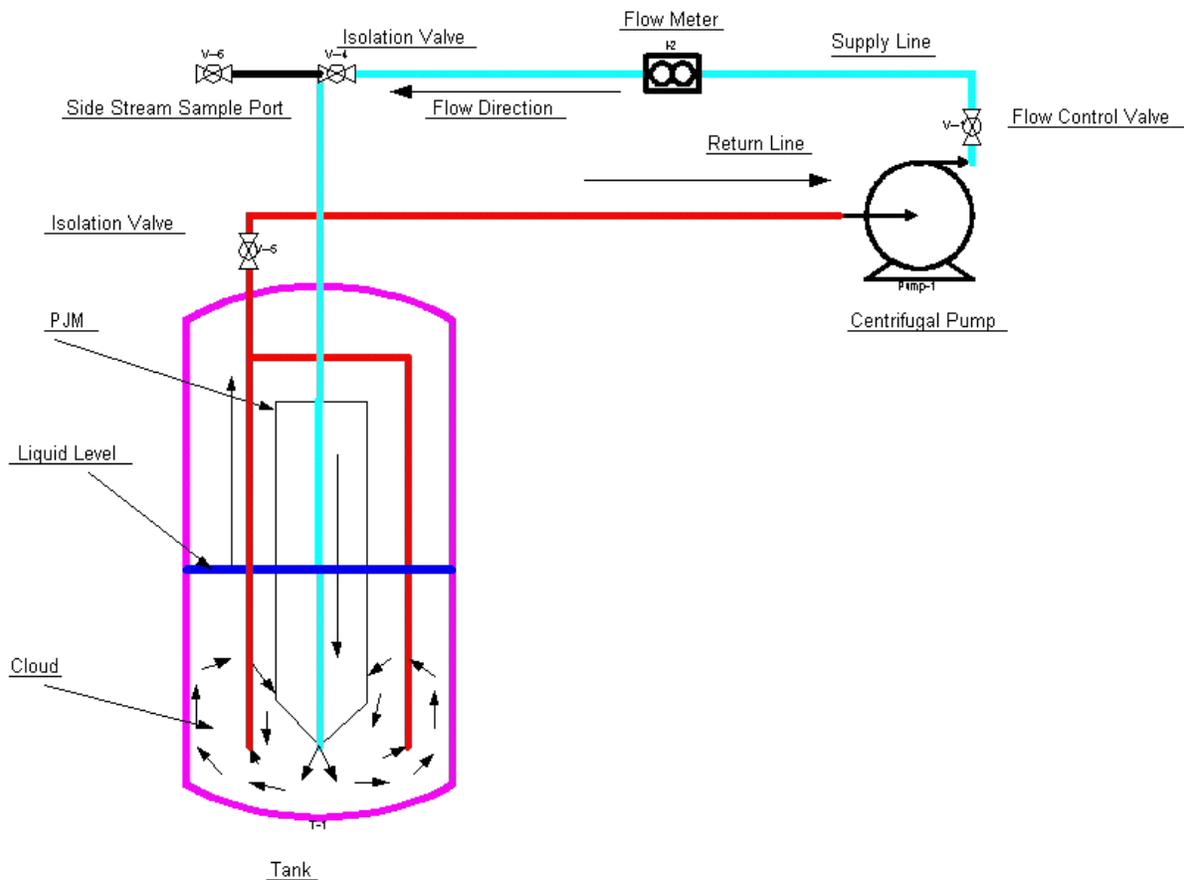
°° = Uncertainty ±0.4 in.

The zero elevation datum is the interior bottom of the tank.

Tank manufactured at Reynolds Polymer Technology, INC.

Drawing # 309954; Rev. original; Date: 07/25/01

**Figure 2.1.** Schematic of Tank Internal Configuration Used for Cloud and Cavern Tests (not drawn to scale)



**Figure 2.2.** Schematic of the Cloud Test Flow Loop

The data acquisition system (DAS) consisted of a desktop computer running Strawberry Tree DAS software and calibrated per measuring and testing equipment (M&TE) requirements as specified by WTP support quality assurance requirement and described in QA implementing procedure QA-RPP-WTP-1201. Temperature data were read from the DAS computer screen and recorded to the LRB. No electronic data files were recorded for the cloud tests (electronic data files were recorded for the cavern tests). A pipe tee and valve were installed downstream of the flow meter to allow the removal of a flow stream (grab) sample during operation.

An Inotek Instruments vortex flow meter monitored fluid flow through the pump discharge line. The flow rate was read from the digital readout on the dedicated flow meter. This type of flow meter uses a bluff body placed in the flow and tracks the vortices it induces in the flow. A post-test calibration was conducted to verify flow meter accuracy, and it demonstrated that the meter readings were  $\pm 3\%$  of reading for the range of application.

A digital videotape of all the cloud levels and activity in the tank was recorded. Measurements of the cloud heights were taken using a standard commercial measuring tape with the data recorded in the assigned LRB. One measurement was taken using a measuring tape affixed to the outer tank wall. The observed cloud interfaces were consistent around the tank. Therefore,

the single point measurement was sufficient to quantify cloud heights within the  $\pm 0.5$ -inch accuracy of the measurement technique.

Each test consisted of two or three variations of the circulating flow rate in the tank for each glass bead size and solids loading. The flow rates tested were determined as a result of the observed mixing and were selected as the test sequence progressed. Initial intentions were to establish a cloud height approximately 1 inch above the suction line inlets for the first data point. The second was to be a cloud height approximately 8 inches above the suction line inlets, and the third was a cloud level approximately 17 inches above the suction line inlets, or the maximum height achievable at full-loop flow rate.

At each flow rate, a minimum hold time of at least 30 minutes was stipulated to allow the system to come to a steady-state condition (steady state defined as less than a 0.3-inch change in 30 minutes).. At any set flow rate, at least five flow readings were taken at five-minute intervals to ensure that the reading was constant. After the steady-state time, samples were extracted from the cloud with a 50-mL syringe suspended on a rod. The samples were taken ~3 inches below the top of the cloud and at the bottom of the tank. In addition, at the request of the sponsor, two other samples were taken, one 3 inches above the cloud and the other a slipstream sample from the supply line to the outlet of the PJM.

The samples were drawn approximately in the center of the annular space between the PJM and the tank wall. Each sample was drawn into a 50 mL syringe that was quickly raised out of the tank and the contents expelled into a labeled bottle. The tip of the syringe had been modified to open the inlet hole approximately 0.25 inch in diameter. The contents were analyzed according to project requirements at a later date for PSD at each respective level, and a percent weight procedure determined the ratio of solids to liquid in each sample. The solids level was observed through the tank wall and recorded on the data sheet, which was placed permanently in the LRB at the close of each testing sequence or test day.

### **2.1.1 Simulant Preparation**

The glass beads used in this test were part of a bulk order of beads purchased from Potter Industries for the 336 Building laboratory large-scale test program (Bontha 2003). The beads are listed as A-Glass, Soda-Lime by Potter Industries. Some difficulties encountered while working with 10- $\mu\text{m}$  beads led to selecting beads nominally 35- and 71- $\mu\text{m}$  in size. The nominal 35- and 71- $\mu\text{m}$  beads will be referred to as small- and large-diameter beads, respectively.

The material has a fairly wide size distribution, and initial PSD examination showed a range from 5 to 100  $\mu\text{m}$  in particle diameter. Because small beads have very long settling times and tend to diminish visibility into the tank, a washing procedure was performed to remove a significant portion of the finer end of the size spectrum. To do this, a 30-gallon, open-top container was placed on a calibrated scale and an arbitrary fill line made on the inside of the container about 7/8 of the way up the side. The container was then filled with water to the fill

line and weighed. The barrel was emptied and approximately 60 to 100 lb of beads placed in it; water was added to the fill line and the weight was recorded. The mixture was stirred for a minimum of 10 minutes and 5 minutes for the small and large diameter beads, respectively.

The liquid above the beads was decanted until the liquid surface was approximately 2 inches above the settled bead layer. The washing process was repeated five times for the small-diameter material and three times for the large-diameter material. After the final wash, the barrel was refilled to the fill line and weighed to determine the quantity of glass beads remaining in the washed batch. This weight was recorded in the LRB. Multiple washed batches were required for each test. The total quantities of glass beads used for each test are provided in Table 2.1. The test tank was filled with approximately 110 gallons of aqueous slurry for each test.

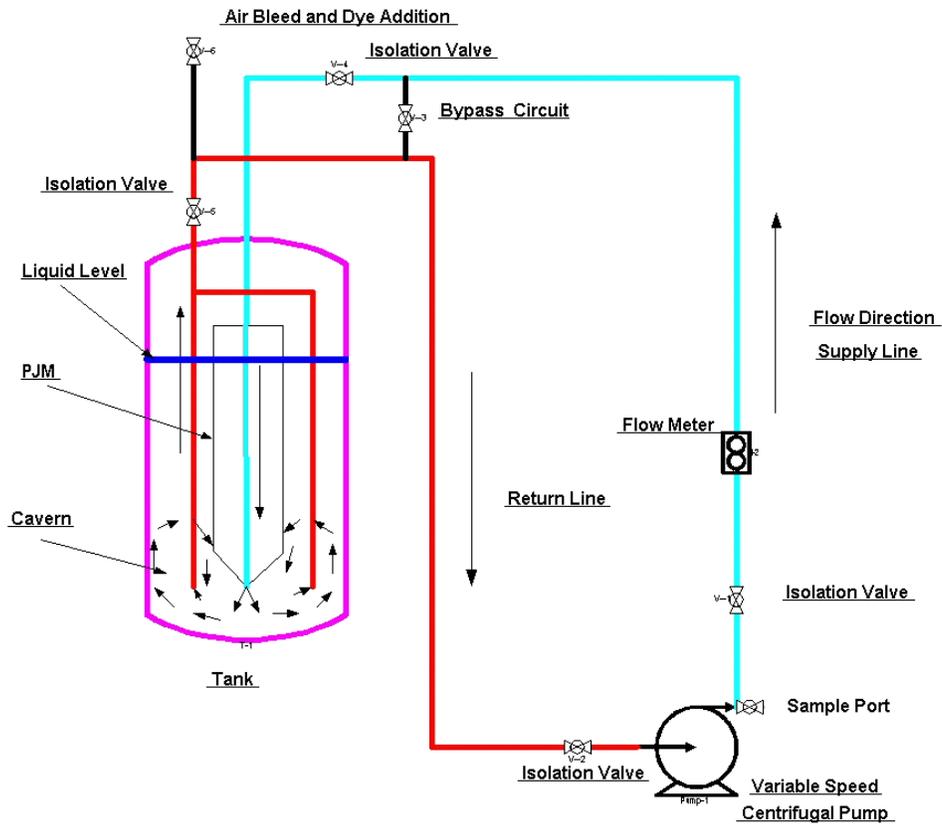
PSDs were measured for the initial glass beads before washing and for the final material in the barrel. The PSDs were obtained using a Microtrac Model S3000 particle size analyzer (property No. N830468). Measurements were conducted according to the manufacturers operating manual. Performance checks of the instrument were made before testing using 10- and 100- $\mu\text{m}$  standards. Good agreement was obtained between the specifications for the standards and the instrument measurements. Data sheets for the PSDs of the standards are included as an appendix. (Note: The PSD information discussed here is for informational purposes only; the data were taken before there was an approved procedure, and the samples may be reanalyzed with the approved procedure if required. However, based on the results from the performance check with the 5- and 100- $\mu\text{m}$  standards, no difference in the results is expected.)

**Table 2.1.** Final Weights of Glass Beads Used

<b>Test No.</b>	<b>Median Particle Size (<math>\mu\text{m}</math>)</b>	<b>Weight of Beads (lb)</b>	<b>Particle Concentration (wt%)</b>
1	48	108 $\pm$ 7	11.1 $\pm$ 0.8
2	48	230 $\pm$ 10	21.9 $\pm$ 1.0
3	66	117 $\pm$ 5	12.0 $\pm$ 0.6
4	66	213 $\pm$ 8	20.5 $\pm$ 0.8

## 2.2 Cavern Test Setup and Operation

The cavern test setup required some modification to the flow loop configuration used for the cloud test work. The loop circulation pump was replaced with a 3-hp variable-speed unit. The pump was relocated from the scaffold near the top of the test tank to the floor to provide increased net positive suction head to the pump inlet. Additional isolation valves were installed to facilitate startup procedures and allow for dye injection. A bypass line was installed between the inlet and outlet lines to allow the system to be primed and circulated before the discharge at the recirculation line was valved into the tank. A sampling port was installed at the pump discharge. A schematic of the test loop is presented in Figure 2.3.



**Figure 2.3.** Schematic of the Cavern Test Loop

The cavern tests monitored flow and fluid density in the discharge line with a Micromotion Coriolis meter, which replaced the Inotek vortex meter. Coriolis meters measure both mass flow rate and density directly with the parameters of mass, time, and length as the bases of all measurements. Mass flow measurements are accurate under conditions of changing viscosity, conductivity, density, and temperature of the fluid. A change in the type of flow meter was required due to the non-Newtonian behavior of the Laponite mixture. The flow rate through the test loop was controlled by adjusting the pump speed with the variable-frequency drive.

Several materials were considered for use as a simulant for the cavern tests, including organic polymer solutions (i.e., Carbopol) and inorganic slurries such as Bentonite clay and Laponite. Because resources and time were limited, it was determined that the cavern needed to be monitored visually as opposed to using sensors and instrumentation; therefore, the selection focused on transparent materials. Carbopol was considered, but it does not possess true yield stress and behaves as a power law fluid, which only approximates a yield stress material. Scoping tests with Laponite demonstrated the material had measurable shear strength, was shear thinning, and possessed good clarity.

The first attempt to produce a cavern test fluid used a solution of 2.5 wt% Laponite, which was far too viscous and exhibited a yield stress greater than that predicted for the waste. The

2.5-wt% material also did not provide good clarity (due to incomplete hydration) for observing the interface. As a scoping study, the 2.5 wt% solution was then diluted to ~1.9 wt% and the loop operated again briefly to see how to best record the visual data. In that scoping test, a small amount of intense blue dye was introduced into the flowing part of the system, and it quickly altered the color of the flowing material in contrast to the unyielded, jelled material. When the concentration of the final solution was tested (2.08 wt%), this same procedure was used. The resulting cavern was very discernable against the transparent, uncolored, nonflowing (unyielded) material.

The loop flow rate, solution density, and solution temperature were recorded to an electronic data file using a desktop computer running Strawberry Tree DAS. Data were stored on digital media. All instruments were on the system's M&TE list. Physical measurements of the cavern size were performed manually with standard measuring tapes, and all data were recorded on data sheets placed in the LRB. Tests were also recorded on digital videotape.

An approximate volume of 160 gallons of material was loaded into the test tank and allowed to sit undisturbed for approximately 40 hours (minimum of 20 hours required by procedure). The loop was started by circulating flow around the bypass loop. Concentrated dye was injected into the bypass loop and, after the recirculation flow was adjusted to approximately 16 gpm (60 L/m), the circulation valves were opened and the bypass line closed.

At startup, flow was unsymmetrical through the return lines and initiated in only two of the four suction tubes. Because Laponite is extremely shear sensitive and flows when a sufficient pressure drop is induced, long rods with a right-angle flat on the end were lowered manually into the tank and the already flowing tube inlets temporarily blocked to place additional pressure drop across the stagnant tube inlets. This initiated flow in the other suction lines, which continued when the other lines were unblocked. The loop flow rate was then adjusted to the desired magnitude with flow through all four return tubes.

The cavern height was measured at 90-degree increments about the tank perimeter approximately every 15 minutes. At each measurement location, two heights were recorded. The maximum cavern height (interface between the flowing and nonflowing regions of the tank) observed on a radial line and the cavern height at the tank wall were measured by visual inspection and recorded on data sheets that were taped into LRB 14314. Each cavern height measurement was repeated independently by two individuals. After observing constant cavern heights over a minimum of 30 minutes, cavern height measurements were repeated at 45-degree increments around the tank perimeter. After obtaining steady-state measurements, the nozzle velocity was increased by increasing the pump speed, and the cavern height was again monitored until a new steady-state cavern was obtained. The results of the cavern tests are presented in Section 4.

### 2.2.1 Simulant Rheology Measurements

The rheology tests were conducted in accordance with guidelines 24590-WTP-GPG-RTD-001 (Smith and Prindiville 2002). All measurements were performed on a Haake M5 rheometer, the same instrument that is used in the PNWD hot cells for characterizing WTP samples. Rheograms were obtained using an MV1 cup and sensor. A performance check of the rheometer was conducted with a Brookefield calibration standard before making rheological measurements on test samples. The standard was certified to be 98 mPa-s at 25°C. On the day of the cavern tests, a viscosity of 104 mPa-s was measured for the calibration standard at a temperature of 23°C. The accepted calibration limit for the machine and WTP approved rheology procedure is 10%. The measured value was within 6% of the standard viscosity; therefore, no temperature correction was evaluated for the standard.

Shear vane tests were conducted according to Section 5.5 of 24590-WTP-GPG-RTD-001 (Smith and Prindiville 2002) using a shear vane with a diameter and height of 0.87 inch. The rotational rate was 0.3 rpm.

To select the concentration of Laponite to be used for the cavern test simulant, rheograms and shear vane tests were performed on small samples of Laponite ranging from 1.5 to 2.4 wt% Laponite. The Laponite samples had been left undisturbed for approximately 38 hours before the shear vane tests began and for approximately 100 hours when the rheograms were produced.

The shear strength is a direct measurement obtained via a shear vane test. The yield stress was calculated from a flow curve by fitting the rheogram data to a Bingham Plastic model. The failure mode is not assumed to be due to pure shear. For a Bingham Plastic, the shear stress,  $\tau$ , is related to shear rate,  $\dot{\gamma}$ , by

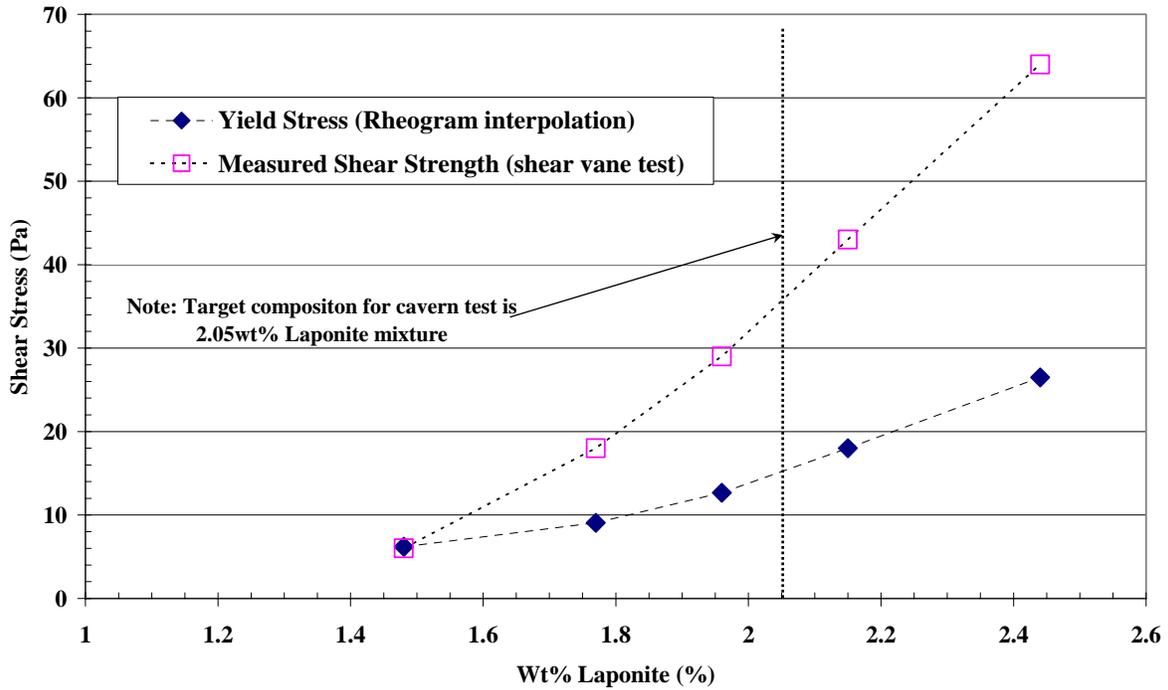
$$\dot{\gamma} = 0 \text{ for } \tau < \tau_y$$

$$\tau = \eta\dot{\gamma} + \tau_y \text{ for } \tau \geq \tau_y$$

The rheograms for the Laponite samples were obtained by ramping the shear rate linearly from 0 to 1150 s<sup>-1</sup> over 5 minutes. Because Laponite exhibits time-dependent behavior, the values for yield stress can be affected by the shear history of the sample. Performing the ramp-up of shear rate over a different time period could affect the values calculated for yield stress. The measured shear strengths and predicted yield stress as a function of Laponite concentration are plotted in Figure 2.4 and presented in Table 2.2.

Based on tank waste characterization data, BNI personnel specified that the yield stress of the cavern test material was to be between 6 and 20 Pa, with 20 Pa being the preferred yield stress for testing. The data presented in Figure 2.2 along with the optical quality of the samples were considered in selecting the 2.05 wt% Laponite target concentration (actual mixture = 2.08 wt%)

### Shear Strength and Yield Stress vs Wt% Laponite



**Figure 2.4.** Plots of Measured Shear Strength and Calculated Yield Strength as a Function of wt% Laponite

to be used for testing. The target value for the test simulant was a  $\tau_y$  of approximately 15 Pa,  $\eta$  of approximately 7 mPa-s, and a shear strength of 36 Pa.

**Table 2.2.** Rheological Parameters for Initial Samples Ranging from 1.5 to 2.4 wt% Laponite

Laponite (wt%)	Measured Shear Strength (Pa)	$\tau_y$ for Bingham Plastic (Pa)	$\eta$ for Bingham Plastic (mPa-s)	$R^2$ for Bingham Plastic fit
1.48	6	6.2	5.9	0.97
1.77	18	9.1	6.9	0.97
1.96	29	12.7	7.0	0.95
2.15	43	18.0	7.4	0.92
2.44	64	25.0	7.9	0.87

## 2.2.2 Simulant Preparation

Preliminary work with the Laponite demonstrated that the mixing, if not done properly, could result in jelled, non-homogeneous lumps of material suspended in the solution. To mitigate the occurrence of the jelled lumps, the solution was mixed as follows. A large plastic tank was placed on a calibrated scale, and the amount of water required for the target Laponite concentration was weighed into the tank. The calculated quantity of Laponite was weighed out on a calibrated scale.

- A large mixer was positioned in the make-up tank and switched on to stir the water. The Laponite was added to the tank slowly using a sieve to separate the powdered material and ensure that the mixture had as few lumps as possible. This is important because the hydrating properties of the Laponite cause it to form lumps immediately on contact with water if it is not sufficiently dispersed, and even then it is difficult to prevent some lumps from forming.
- After completing the addition of the Laponite, the solution was stirred for a minimum of one hour to mix the solution as much as possible. The mixer was removed and the solution covered for a minimum of 12 hours to allow full hydration of the Laponite.
- After a minimum of 12 hours, the mechanical mixer was reinstalled in the make-up tank. The inlet and discharge hoses for a 1/3 hp centrifugal pump were placed into the make-up tank for closed-loop recirculation of the contents. The jelled Laponite was liquefied with the stirrer and the liquid circulated through the pump to homogenize the lumps. The fluid was agitated for at least one hour or until no lumps were visible.
- When mixing was complete, the Laponite was transferred to the acrylic test tank and allowed to stand for a minimum of 20 hours to permit it to re-establish its full yield stress. During the filling operation, the suction and feed lines were pre-filled and all air bled from the lines in preparation for the actual test.

## 3.0 Cloud Test Results

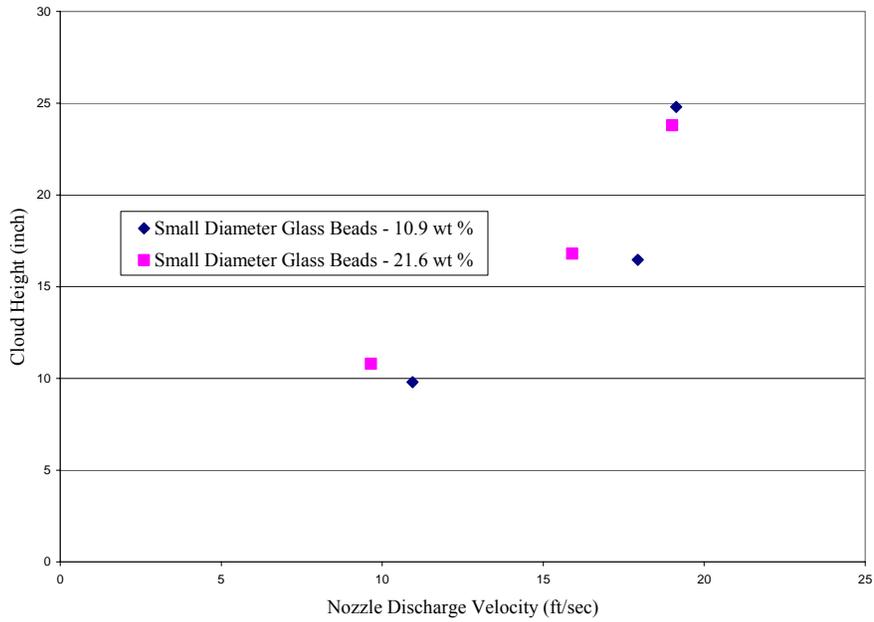
### 3.1 Observations

For a given bead size and solids loading, the cloud height was measured as a function of discharge velocity. The original intent was to measure at least three cloud heights per bead size and solids concentration. The large-diameter material at 20.5 wt% was found to produce only two distinct cloud heights with the available pump. The data presented here are reduced from the information recorded in LRB 14314. Table 3.1 provides a summary of the test matrix and lists the results of the cloud height tests. A value of zero is listed for the standard deviation for calculated averages of cloud height that are presented in Table 3.1. The interfaces measured consisted of wavy boundaries that are shown in Figures 3.3 and 3.4. The recorded value for a single measurement of a cloud height taken by a test operator was the visually averaged height of the interface. The zero value for the standard deviation indicated the visual average measured by the test operator remained constant for the number of observations recorded.

Cloud heights as a function of nozzle velocity for both 11.1 and 21.9 wt% solids loading are presented in Figure 3.1 for the small beads. Figure 3.2 presents cloud height as a function of nozzle velocity for both 12 and 20.5 wt% solids loading for the large-diameter beads. Figure 3.3 provides photographic images of steady-state cloud heights of the small-diameter glass beads at 11.1 wt% solids loading for the nozzle velocities tested. Figure 3.4 shows the same condition for

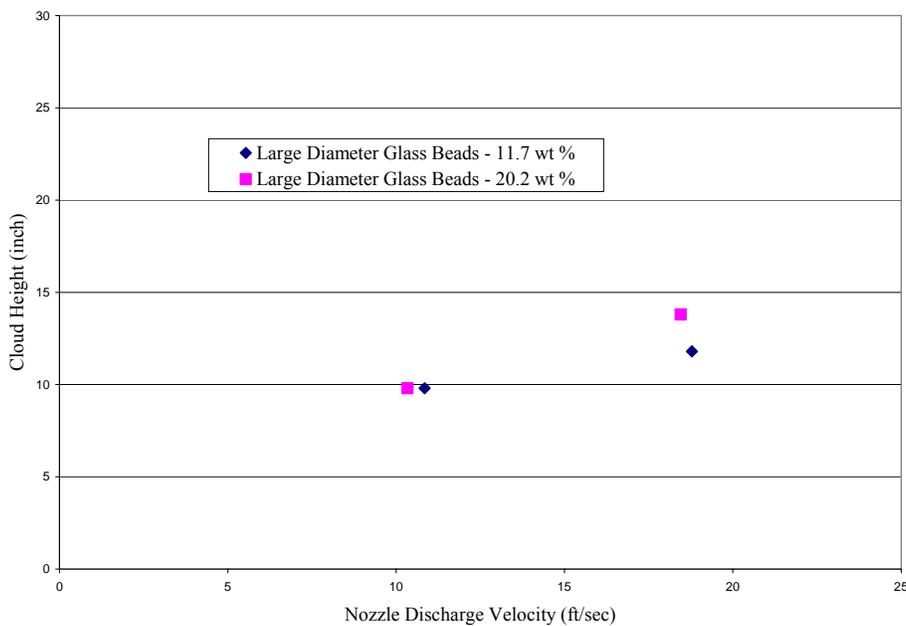
**Table 3.1.** Cloud Height Test Results

Median Particle Diameter Based on Volume Distribution ( $\mu\text{m}$ )	Concentration (wt%)	Average Nozzle Discharge Velocity <sup>(a)</sup> (ft/sec)	Average Nozzle Flow Rate <sup>(b)</sup> (gpm)	Average Cloud Height from Tank Bottom <sup>(c)</sup> (in.)
48	11.1 $\pm$ 0.8	10.9 Stdv =0.1	20.3 Stdv =0.2	9.8 Stdv =0.0
48	11.1 $\pm$ 0.8	17.9 Stdv =0.1	33.2 Stdv =0.2	16.5 Stdv =0.5
48	11.1 $\pm$ 0.8	19.1 Stdv =0.1	35.4 Stdv =0.2	24.8 Stdv =0.0
48	21.9 $\pm$ 1.0	9.6 Stdv =0.1	17.9 Stdv =0.2	10.8 Stdv =0.0
48	21.9 $\pm$ 1.0	15.9 Stdv =0.0	29.5 Stdv =0.1	16.8 Stdv =0.0
48	21.9 $\pm$ 1.0	19.0 Stdv =0.1	35.2 Stdv =0.2	23.8 Stdv =0.0
66	12.0 $\pm$ 0.6	10.8 Stdv =0.0	20.1 Stdv =0.1	9.8 Stdv =0.0
66	12.0 $\pm$ 0.6	18.8 Stdv =0.1	34.8 Stdv =0.1	11.8 Stdv =0.0
66	20.5 $\pm$ 0.8	10.3 Stdv =0.1	19.2 Stdv =0.1	9.8 Stdv =0.0
66	20.5 $\pm$ 0.8	18.5 Stdv =0.0	34.2 Stdv =0.1	13.8 Stdv =0.0
(a) Calculated from flow rate measurements; uncertainty associated with single values is $\pm 4\%$ of value.				
(b) Uncertainty associated with single measurements is $\pm 3\%$ of reading based on calibration.				
(c) The uncertainty associated with single measurements is $\pm 0.6$ inches.				

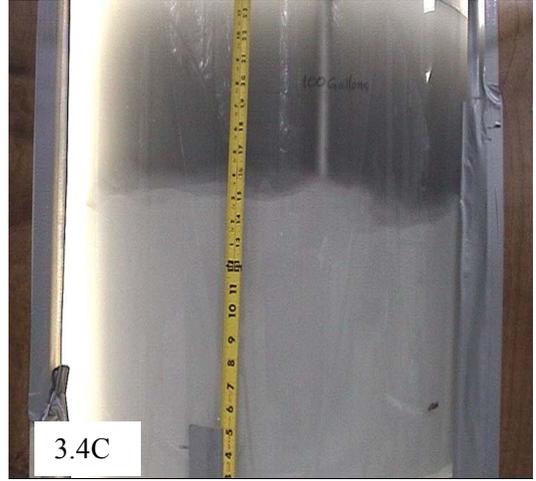
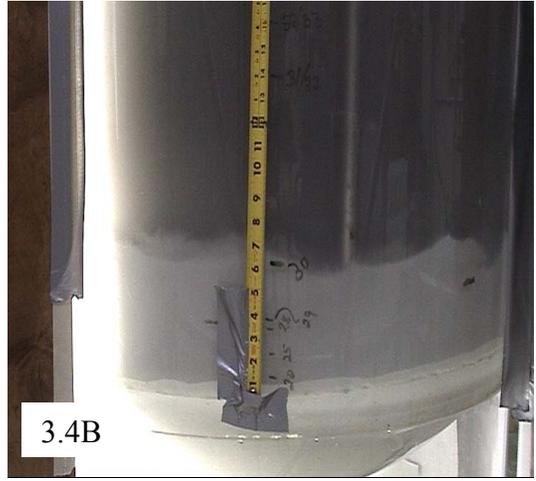
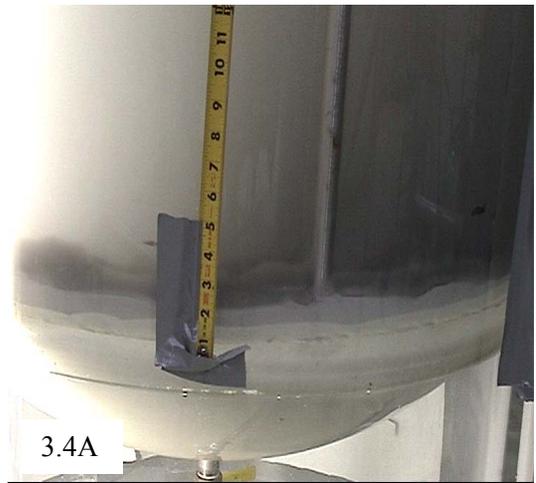


**Figure 3.1.** Measured Cloud Heights with Glass Beads Having a Median Particle Diameter of  $48 \mu\text{m}$  as a Function of Nozzle Discharge Velocity

21.9 wt% loading. Photographs of the large-diameter glass beads at solids loadings of 12 and 20.5 wt% are presented as Figures 3.5 through 3.7. In all of the figures, the observed interface is that between the suspended slurry cloud and the upper clear liquid. The top surface of the tank liquid is not shown in the photos.



**Figure 3.2.** Measured Cloud Heights with Glass Beads Having a Median Particle Diameter of  $66 \mu\text{m}$  as a Function of Nozzle Discharge Velocity



**Figure 3.3.** Cloud Tests Using Glass Beads with 48- $\mu\text{m}$  Median Particle Diameter at 11.1 wt% Solids Loading, Steady-State Conditions, and Nozzle Velocities of 10.9, 17.9, and 19.1 ft/sec

**Figure 3.4.** Cloud Tests Using Glass Beads with 48- $\mu\text{m}$  Median Particle Diameter at 21.9 wt% Solids Loading, Steady-State Conditions, and Nozzle Velocities of 9.6, 15.9, and 19.0 ft/sec



**Figure 3.5.** Photographs of Cloud Tests with Glass Beads Having a Median Particle Diameter of  $66\ \mu\text{m}$  at 12 wt% Solids Loading and Steady-State Conditions for Nozzle Velocities of 10.8 and 18.8 ft/sec



**Figure 3.6.** Photographs of Cloud Test with Glass Beads having a Median Particle Diameter of  $66\ \mu\text{m}$  at 20.5 wt% Solids Loading and Steady-State Conditions for the Settled Tank Prior to Testing



**Figure 3.7.** Photographs of Cloud Test with Glass Beads Having a Median Particle Diameter of  $66\ \mu\text{m}$  at 20.5 wt% Solids-Loading and Steady-State Conditions for Nozzle Velocities of 10.3 and 18.5 ft/sec

At each flow rate, a distinct cloud height was established, and the interface between the suspended slurry (cloud) and clear liquid could be seen and measured. The clouds were fairly constant in level and showed some level of increased turbulence as the flow rate was increased. The surface of the cloud did not display any preferential behavior that could be seen to repeat, except that at higher flow rates larger rolling waves would continuously appear and abate on the surface. For nozzle velocities above approximately 15 ft/sec, the surface of the cloud interface displayed constant undulations or waves. The reported cloud heights in Table 3.1 are visual averages of the wave peaks and valleys (see Figures 3.3 through 3.7).

### 3.2 Cloud Height Particle Size Data

For all cases tested, a condition of all solids suspended was achieved. The acrylic tank bottom allowed visual observation of the entire bottom of the tank. It was readily observed for both small- and large-diameter beads that no solids remained immobilized on the bottom of the tank for all nozzle velocities tested. Grab samples were collected and archived for all steady-state test conditions. Table 3.2 contains a summary of the PSD obtained for representative samples. The results in the table are based on volume count distribution. For the two conditions tested, the PSD results indicated a slight vertical stratification in particle size.

**Table 3.2.** Summary of Particle Size Distributions Based on Volume Count for Representative Grab Samples Taken During Steady-State Conditions

Sample No.	Median Bead Diameter Based on Volume Distribution (μm)	Solids Loading (wt%)	Nozzle Velocity (ft/s)	Relative Elevation of Grab Sample (±0.5in.)	Particle Size for 20% Passing Vol Count (μm)	Particle Size for 50% Passing Vol Count (μm)	Particle Size for 80% Passing Vol Count (μm)
B1W5b B3W5b B4W5b	48	Initial Material	NA	Average of 3 samples	33.7	48.4	77.5
B5W3 Real B6W3	66	Initial Material	NA	Average of 2 samples	55.6	66.5	82.5
T1V3S1	48	11.1±0.8	19.1Stdv=0.1	3 in. below top of cloud	35.7	47.3	68.2
T1V3S2	48	11.1±0.8	19.1Stdv=0.1	Bottom of tank	36.5	49.4	84.5
T3V2S1	66	12.0±0.6	18.8Stdv=0.1	3 in. below top of cloud	56.2	67.1	83.3
T3V2S2	66	12.0±0.6	18.8Stdv=0.1	Bottom of tank	57.6	68.7	85.2

The PSD data sheets for both volume and number count distributions for the representative samples and standards used for the PSD performance check are presented in the appendix. The PSD results were collected according to the instructions provided by the manufacturer of the PSD analyzer prior to the development of a WTP approved procedure and therefore are for information only. The samples may be reanalyzed with the approved procedure if required. However, based on the results from the performance check with standards, no difference is expected in the results (refer to Section 2.1.1).

## 4.0 Cavern Test Results

As can be seen in the series of still pictures taken from the videotape and presented in Figure 4.1, cavern volumes vary with flow rates and are clearly distinguishable. The cavern shape at the interface between the liquid and jelled régimes, however, is asymmetrical and undulating in shape. Several factors may contribute to this phenomenon:

- The discharge nozzle and tank drain port may be slightly misaligned within the tank so that the jet does not impact the exact center of the elliptical tank bottom.
- The test material may possess some degree of slight nonhomogeneity that would result in local spatial variations in the shear strength of the undisturbed material. The undulations may provide an indication of an interface of constant strength.
- The random, chaotic nature of the turbulent flow/eddies at the initial transient or start of cavern formation can be amplified to exist at the steady-state condition. For example, a restart would provide the same bulk average height but different local contour variations.

Even though the undulating shape appears, the shape is constant in time. The cavern shape is formed by shearing between the liquid and jelled portions, which achieves an equilibrium point, and thus the shape remains constant after a sufficient period of time (more than 30 minutes). The cavern heights obtained from the tests are presented in Table 4.1, and the results are plotted as a function of discharge velocity in Figure 4.2.

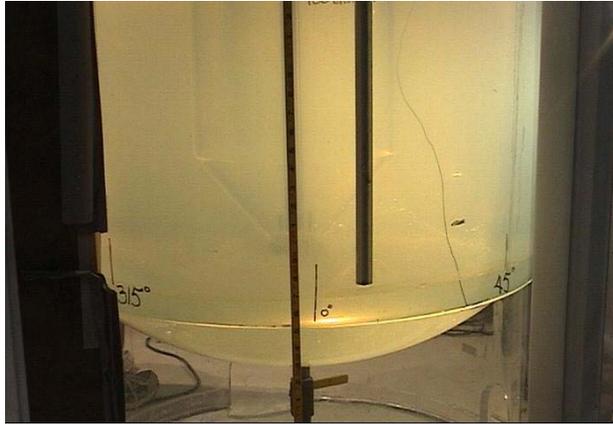
### 4.1 Rheological Measurements

This section presents the results of the rheological characterization of the Laponite test fluid. All of the samples used for material characterization were drawn from the same batch of material used in the test tank. Section 2.2 discusses the material selection and preparation.

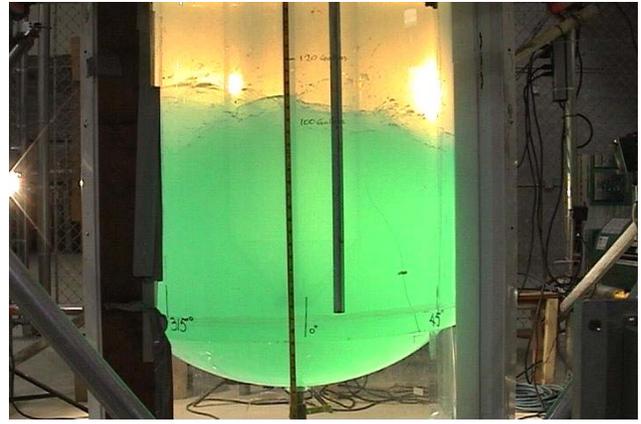
The 2.08 wt% Laponite and water mixture is a shear-thinning material that exhibits a yield stress. However, the Laponite material also exhibits time-dependent behavior when sheared. The rheological characterization was performed to obtain bounding data for both the undisturbed (stagnant) and sheared (flowing) material that existed within the tank at the time steady-state cavern measurements were made. Section 4.1.1 presents the rheology for the undisturbed material, and Section 4.1.2 presents the rheology for the highly sheared material.

#### 4.1.1 Rheology of Undisturbed Material

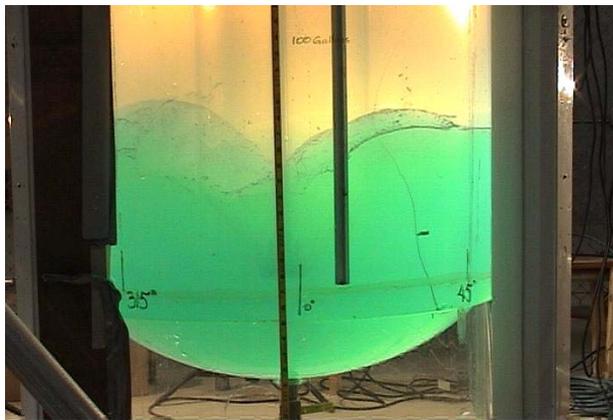
Figure 4.3 is a rheogram of the initially undisturbed material that was produced on the day of testing. The sample was drawn from the test batch of Laponite at the time the material was loaded into the test tank. The sample was left undisturbed for approximately the same period



A. Tank before flow started



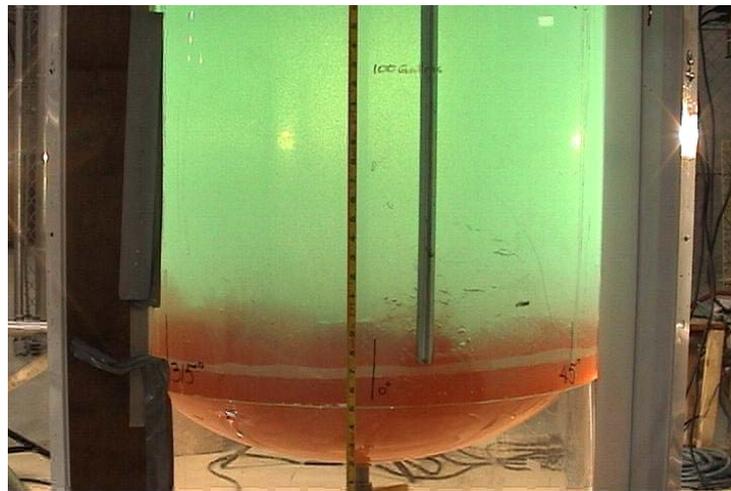
C. 42.8 gpm (162 L/m) nozzle velocity 23.1 ft/sec



B. 32 gpm (121 L/m), nozzle velocity 17.3 ft/sec



D. 48.9 gpm (185 L/m) nozzle velocity 26.4 ft/sec



E. 23 gpm (87 L/m), nozzle velocity 12.4 ft/sec

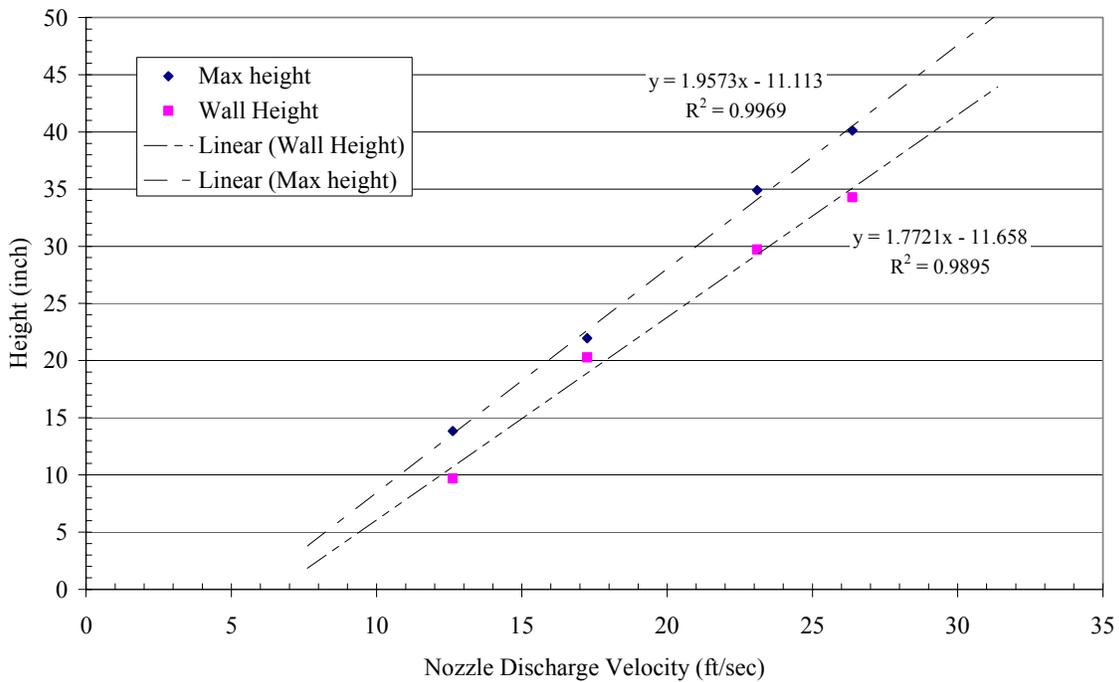
**Figure 4.1.** Cavern Test Conducted with 2.08 wt% Laponite at Various Stages of Testing. Photos A through D are steady-state conditions for the test conditions specified; photo E was taken after the Laponite with blue dye was allowed to set up again and a red dye was injected for demonstration purposes.

**Table 4.1.** Cavern Test Steady-State Results for 2.08 wt% Laponite

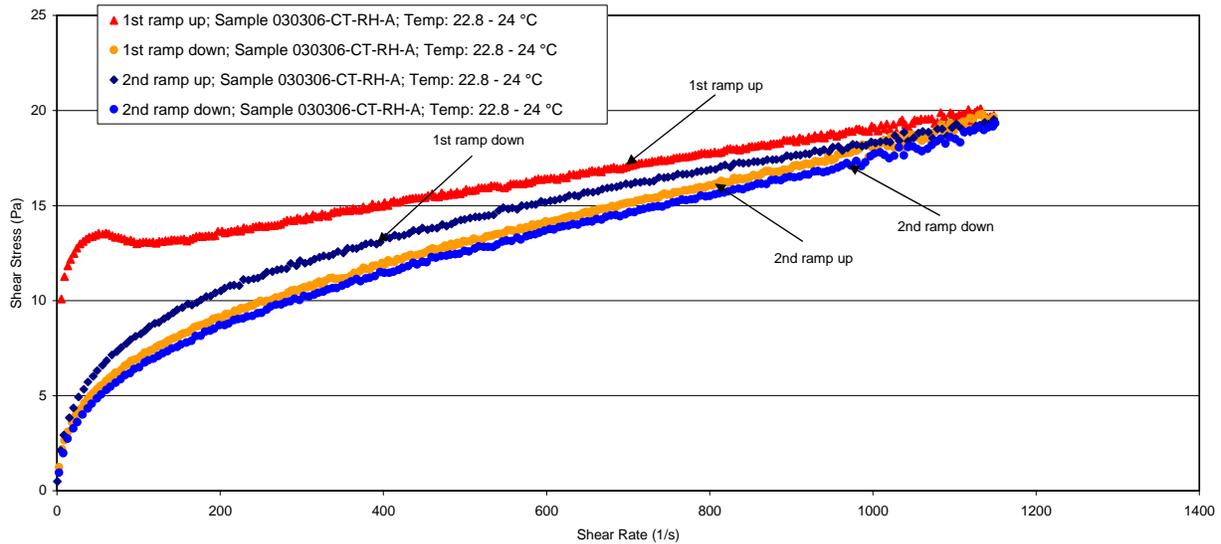
Average Nozzle Flow Rate <sup>(a)</sup> (gpm)	Average Nozzle Discharge Velocity <sup>(b)</sup> (ft/sec)	Avg Max Cavern Ht above Bottom Center of Tank <sup>(c)</sup> (in.)	Avg Cavern Ht at Tank Wall above Bottom Center of Tank <sup>(c)</sup> (in.)	Cavern Fluid Temperature (±0.5°F)	Shear strength at beginning of test (Pa)	Consistency (cP)
23.4 Stdv=0.0	12.6 Stdv=0.0	13.8 Stdv=1.2	9.7 Stdv=2.0	77	44 Stdv=2	7.9
32.0 Stdv=0.2	17.3 Stdv=0.1	22.0 Stdv=1.2	20.3 Stdv=2.3	78	44 Stdv=2	8.1
42.8 Stdv=0.2	23.1 Stdv=0.1	34.9 Stdv=0.9	29.7 Stdv=1.7	80	44 Stdv=2	6.7
48.9 Stdv=0.2	26.4 Stdv=0.1	40.1 Stdv=1.3	34.3 Stdv=1.6	80	44 Stdv=2	6.3

(a) The uncertainty associated with single measurements is ±2.0% of reading based on calibration.  
 (b) Calculated from flow rate measurements. Uncertainty associated with single values is ±3.0% of value.  
 (c) The uncertainty associated with single measurements is ±0.6 inches.

Adjusted Cavern Heights, Referenced to Tank Bottom



**Figure 4.2.** Steady-State Cavern Height as a Function of Nozzle Discharge Velocity for 2.08 wt% Laponite



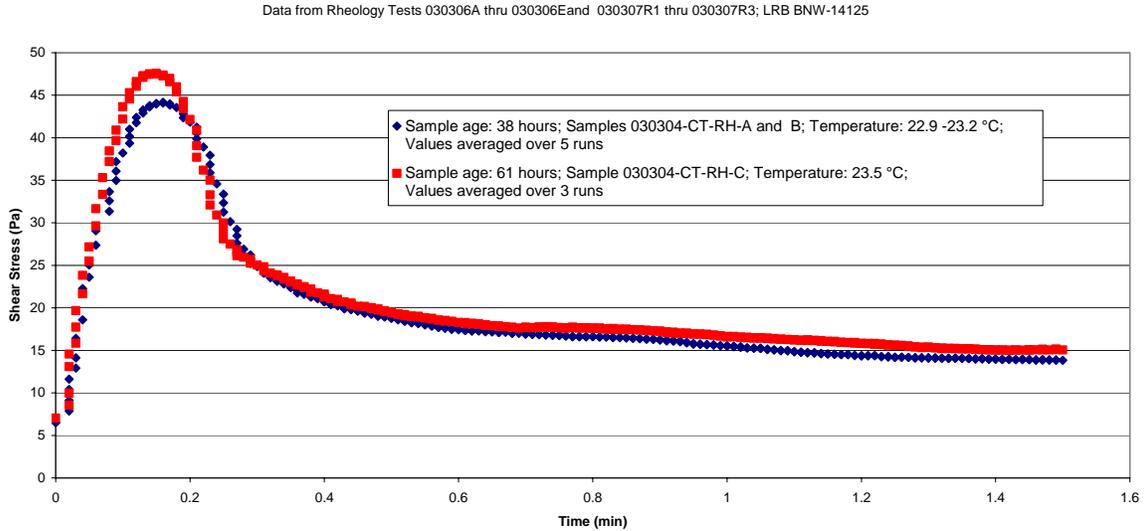
**Figure 4.3.** Rheogram of Undisturbed 2.08 wt% Laponite Obtained by Linearly Ramping Shear Rate from 0 to  $1150 \text{ s}^{-1}$  for 5 min. Test runs were performed as one continuous test in the order of first ramp-up, first ramp-down, second ramp-up, and second ramp-down, with no pause between runs.

( $\approx 40$  hr) as the material in the test tank. The rheogram was produced by linearly ramping the shear rate from 0 to  $1150 \text{ s}^{-1}$  over a period of 5 minutes, followed immediately by a ramp-down of the same duration. Test runs 1 and 2 were performed as one continuous test with no pause between runs. The first ramp-up represents the undisturbed material. The fact that the first and second ramp-downs do not yield identical results indicates the material is time dependent. The rheogram indicates that the apparent viscosity reduces with time for a constant shear rate, which implies thixotropy. Testing demonstrated that the thixotropy exhibited by the Laponite is reversible because the material eventually regained its original strength when the shear rate was reduced or eliminated.

The data for the rheogram produced by the first ramp-up (Figure 4.3) were used to calculate yield stress ( $\tau_y$ ), which is calculated by fitting the data to a rheological model. Fitting the data for the first ramp-up of the undisturbed material to a Bingham Plastic model ( $\tau = \tau_y + \eta \dot{\gamma}$ ) produced a  $\tau_y$  of 12.3 Pa and  $\eta$  of 6.8 mPa s for the undisturbed material at 23°C.

The yield stress of the material is not a direct measurement nor does it define the mode of failure exhibited by the material. For time-dependent material, the calculated yield stress can be affected by the rate at which shear rate is changed. Shear vane tests were conducted to assess whether the shear strength of the material was changing as a function of time. (Section 2.2 has a description of the shear vane tests.) Shear vane tests were run on samples drawn from the test batch of Laponite when the material was loaded into the test tank. The Laponite mixture was

approximately 24 hours old when it was loaded into the test tank. The material was left undisturbed for an additional 38 hours before testing began. Shear vane tests were performed on several samples when the cavern test began and were repeated on undisturbed samples approximately 23 hours after the test began. Average shear strengths of 44 Pa (stdev = 2 Pa) and 48 Pa (stdev = 1 Pa) were obtained for measurements at the initiation of the cavern tests and 23 hours later, respectively. Sample temperatures were approximately 23°C during the shear vane tests. Figure 4.4 shows average shear rate as a function of time obtained from the shear vane tests.



**Figure 4.4.** Plots of Shear Stress Versus Time Obtained During Shear Vane Tests on Undisturbed 2.08 wt% Laponite at the Initiation of Cavern Tests (38 hr of setup) and ~23 hr after Testing Began (61 hr of setup). The plots were developed by averaging data from several runs.

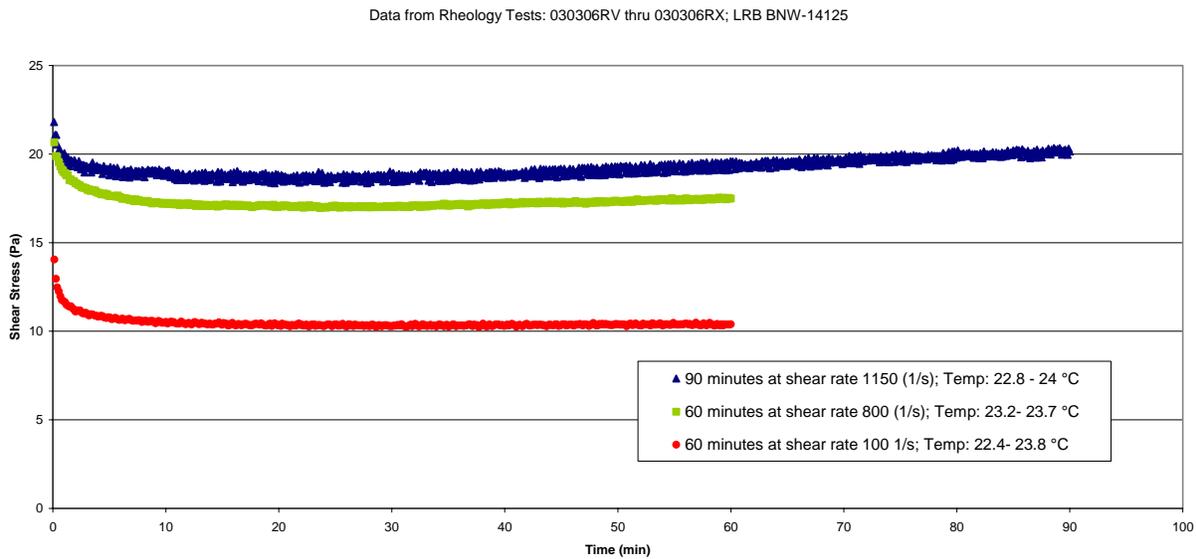
#### 4.1.2 Rheology of Material after a High Degree of Shear

The material passing through the centrifugal pump was subjected to a high degree of shearing. An order of magnitude for the minimum range of the peak shear rate within the nozzle was approximated by assuming a no-slip condition at the nozzle wall and the average nozzle velocity at the nozzle centerline. Granted the flow profile at the nozzle discharge will be nonlinear, but based on the nozzle diameter and range of discharge velocities tested, the maximum shear rate for the four discharge velocities was estimated to be on the order of 500 to 1000  $s^{-1}$ . Depending on the flow distribution of the impacting jet on the tank bottom, the peak shear rates within the cavern may occur along the bottom of the tank. The shear rates imposed on the material passing through the centrifugal pump were potentially an order of magnitude higher than the peak shear rates existing within the cavern region of the tank.

Because of the time-dependent behavior of the Laponite, the disturbed material was characterized and evaluated as a function of time. The objective was to determine whether the

Laponite mixture obtained constant behavior after being subjected to a high degree of shear for an extended time period. The nozzle velocity would need to be held constant for at least the minimum time required for the rheological behavior of the material to reach a constant to ensure that a steady-state cavern had been obtained.

Jump tests were conducted on undisturbed material. A jump test consists of subjecting the material to a constant shear rate for an extended period and measuring the resulting shear stress. Tests were conducted at shear rates of 100, 800, and 1150  $\text{s}^{-1}$  for a minimum of 60 minutes. Figure 4.5 plots shear stress versus time for the jump tests performed on the undisturbed material.



**Figure 4.5.** Plot of Shear Stress Versus Time for Jump Tests Performed on Undisturbed Material

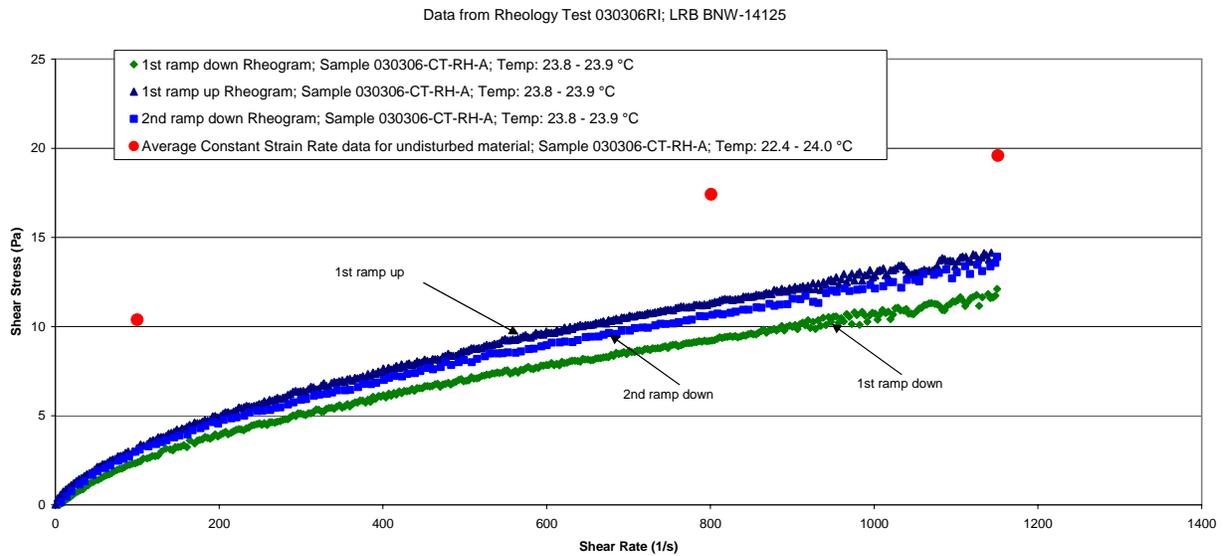
The measured shear stress for the material continually decreased for approximately the first 20 minutes of each test (see Figure 4.5). Between 20 and 30 minutes, the change in shear stress was less than 0.5%, followed by an observed increase in measured shear stress. The increase in shear stress can be explained by either evaporation of liquid from the sample over a prolonged measurement period or heterogeneity in the test sample created by the centrifuging the Laponite particles in the rheometer at the high rotational rates. The observed increase in shear stress as a function of time that occurs after 30 minutes increases with applied shear rate. The 10-minute average values for shear stress occurring between 20 and 30 minutes of each test run were 10.3, 17.0, and 18.7 Pa for shear rates of 100, 800, and 1150  $\text{s}^{-1}$ , respectively.

The intent of the rheological characterization was to bound the material behavior in the flowing cavern for each discharge velocity tested. The material passing through the centrifugal pump has experienced a shear rate far greater than any existing within the cavern. After entering the

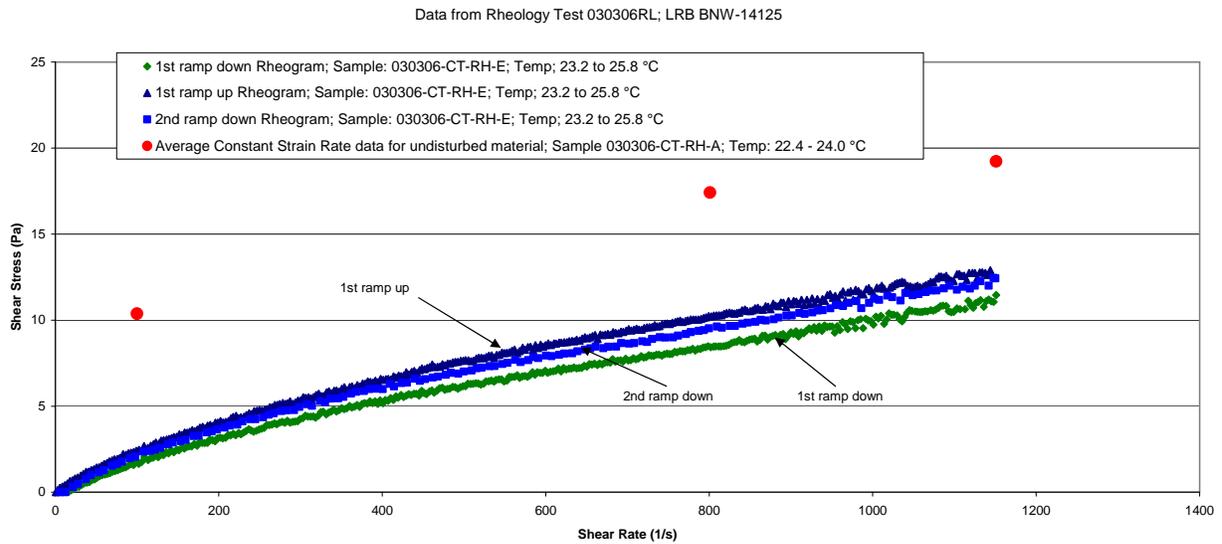
cavern, the material begins to regain strength, approaching a constant magnitude if subjected to a constant shear rate for a sufficient time (approximately 20 minutes based on the results of Figure 4.5). The jump tests performed with the undisturbed material provide the upper bound on the time-dependent shear stress that existed within the cavern.

To obtain a lower bound for the time-independent shear stress, rheograms were obtained using samples drawn from the pump discharge once each cavern evaluated had reached steady state. The samples were immediately loaded into the rheometer and tested. The pump was required to run for a minimum of 30 minutes at a constant flow rate before a cavern could be declared at steady state based on cavern height measurements. The rheograms for samples drawn from each steady-state cavern are shown in Figures 4.6 through 4.9. The material temperature for each sample is included on the rheograms. The data points obtained from the jump tests on the undisturbed material are presented on each rheogram.

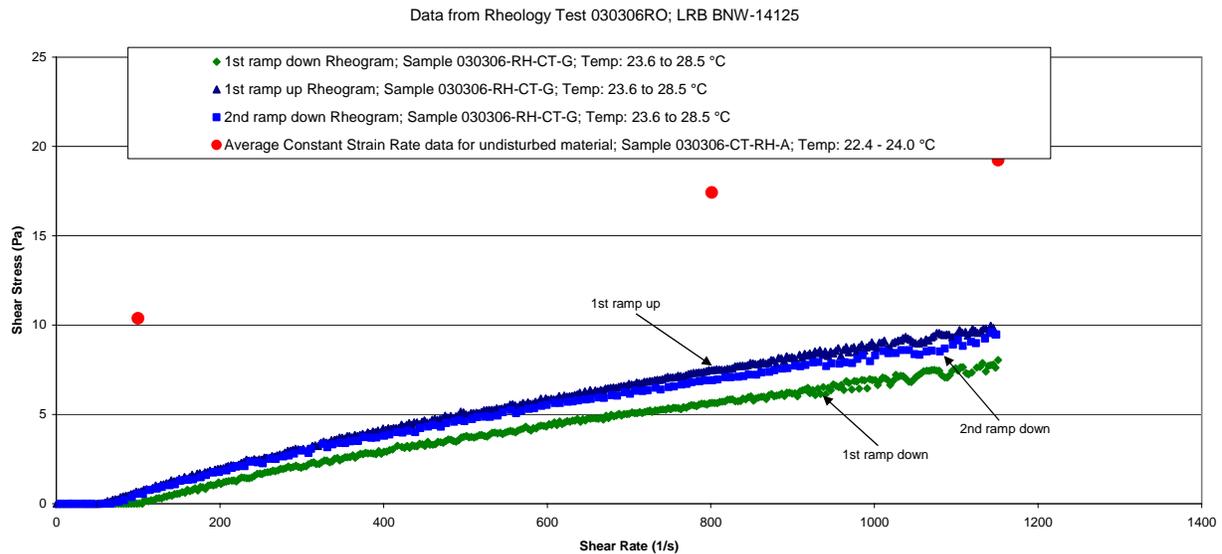
The initial ramp-down for each case provides the lower bound for the time-dependent shear stress that exists within the cavern. The lower bound measurements are made on a fluid that has been subjected to a high degree of shear and is regaining strength within the rheometer as it is subjected to a lower range of shear rates. The relaxation of the material and increase in apparent viscosity with time are observed by comparing the first and second rheograms for each cavern size evaluated (Figures 4.6 through 4.9).



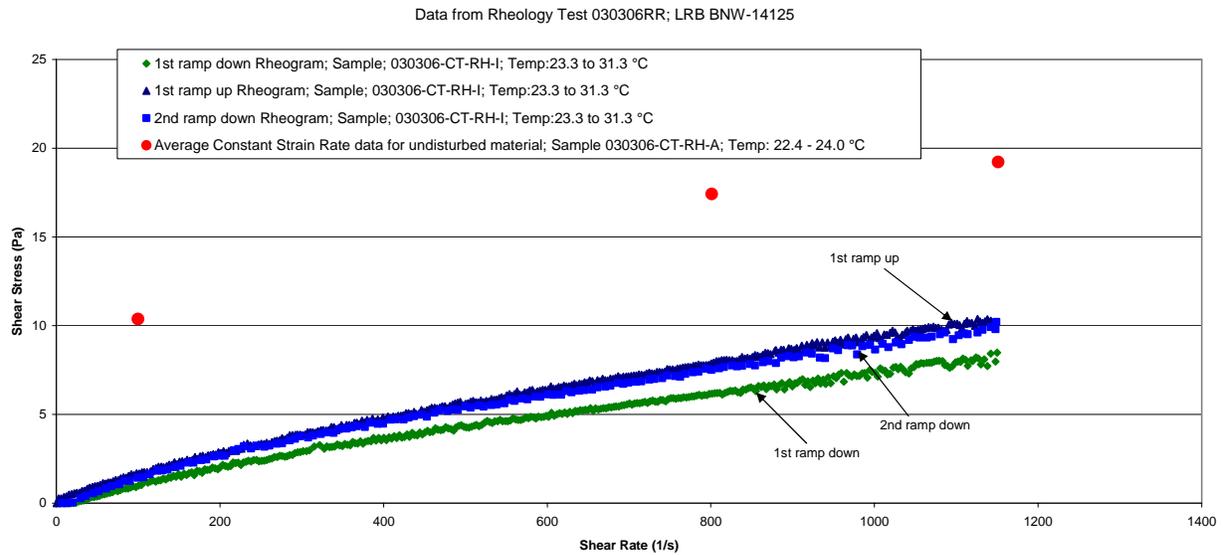
**Figure 4.6.** Rheogram for 2.08 wt% Laponite Sampled after a Steady-State Cavern Was Achieved with a Nozzle Discharge Velocity of 12.6 ft/sec



**Figure 4.7.** Rheogram for 2.08 wt% Laponite Sampled after a Steady-State Cavern Was Achieved with a Nozzle Discharge Velocity of 17.3 ft/sec



**Figure 4.8.** Rheogram for 2.08 wt% Laponite Sampled after a Steady-State Cavern Was Achieved with a Nozzle Discharge Velocity of 23.1 ft/sec



**Figure 4.9.** Rheogram for 2.08 wt% Laponite Sampled after a Steady-State Cavern Was Achieved with a Nozzle Discharge Velocity of 26.4 ft/sec

Minimal temperature variations were observed between the various samples tested. The temperature of the undisturbed material was between 22° and 24°C during rheological testing. The recirculating material experienced some heating during the test sequence. Sample temperatures used to obtain the rheograms of the disturbed material started at 24°C for the first cavern and increased to 28°C for the last cavern.

## 5.0 Conclusions

For the cloud tests, distinct cloud heights were obtained for all nozzle velocities tested. These tests produced full solids suspension with no solids material deposited on the bottom of the tank for all of the velocities tested. As expected, the cloud height was dependent on nozzle velocity and particle size and was nearly independent of solids loading for the range of conditions tested. For all cloud heights, the surface of the liquid in the tank remained totally calm.

The cavern tests produced well-defined interfaces between the flowing and undisturbed material that were observed with the aid of dye. The cavern height increased linearly as a function of nozzle discharge velocity, as was predicted prior to testing. Rheological characterization of the 2.08 wt% Laponite was performed to define the behavior of both the flowing and undisturbed regions of the tank.

The completion of the scope of work defined by Table 1, items 8, 9, and 10 of R&T Test Exception 24590-WTP-TEF-RT-02-057 is documented by this report. With the application of a CFD model, the data presented in this report are sufficient to meet the success criteria defined by Test Plan TP-PNNL-WTP-051 Rev. 0.

## 6.0 References

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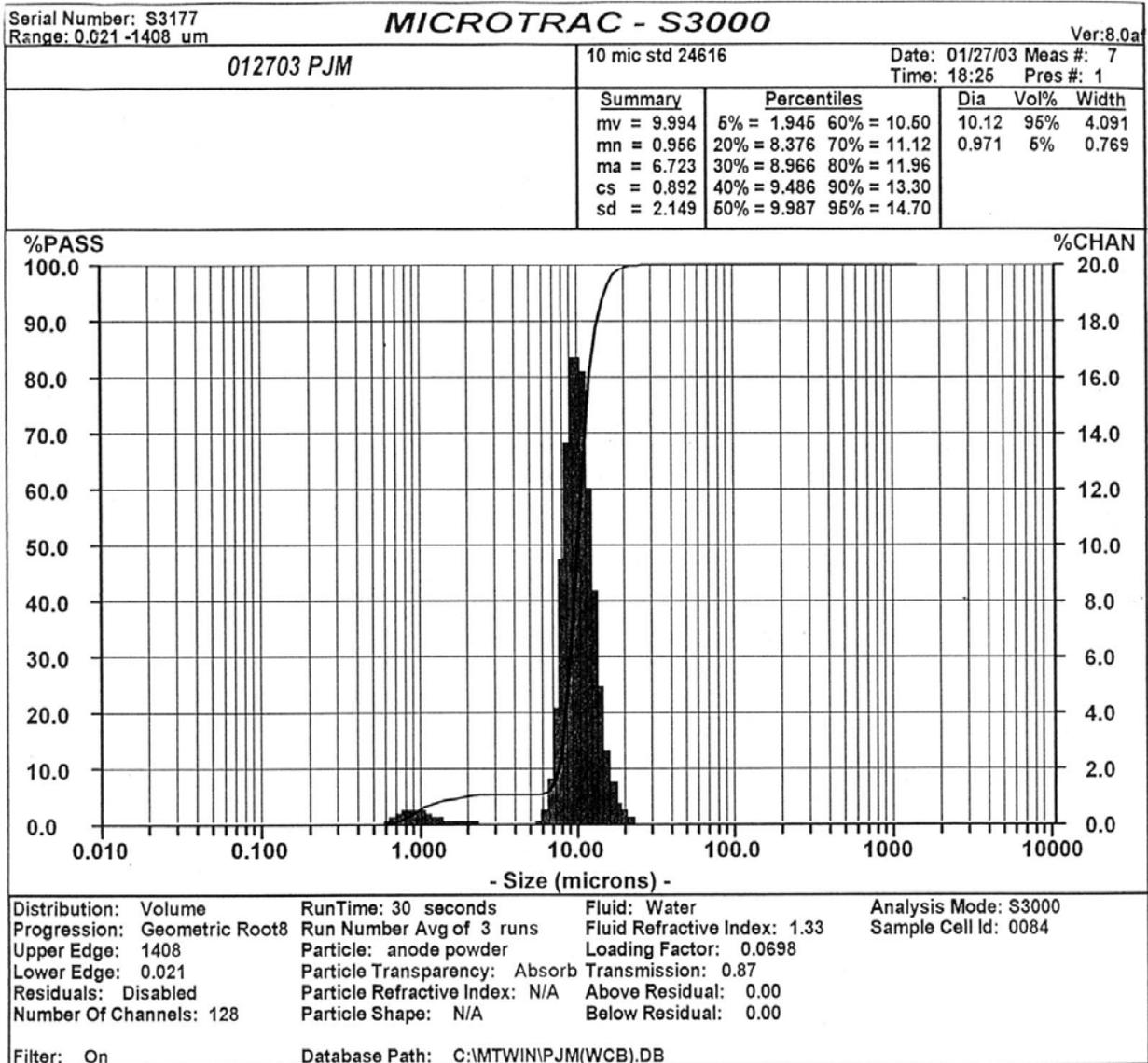
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Johnson MD, JR Bontha, and JM Bates. 2002. *Demonstration of Ability to Mix in a Small-Scale Pulsed-Jet Mixer Test Facility*. WTP-RPT-077, Battelle – Pacific Northwest Division, Richland, WA.

Smith GL and K Prindiville. 2002. *Performing Chemical, Physical, and Rheological Properties Measurements*. 24590-WTP-GPG-RTD-001 Rev. 0, Bechtel National Inc., Richland, WA.

## **Appendix**

### **Number and Volume Distributions**



**Figure A.1.** Particle Size Distribution by Volume of 10 Micron Standard 24616

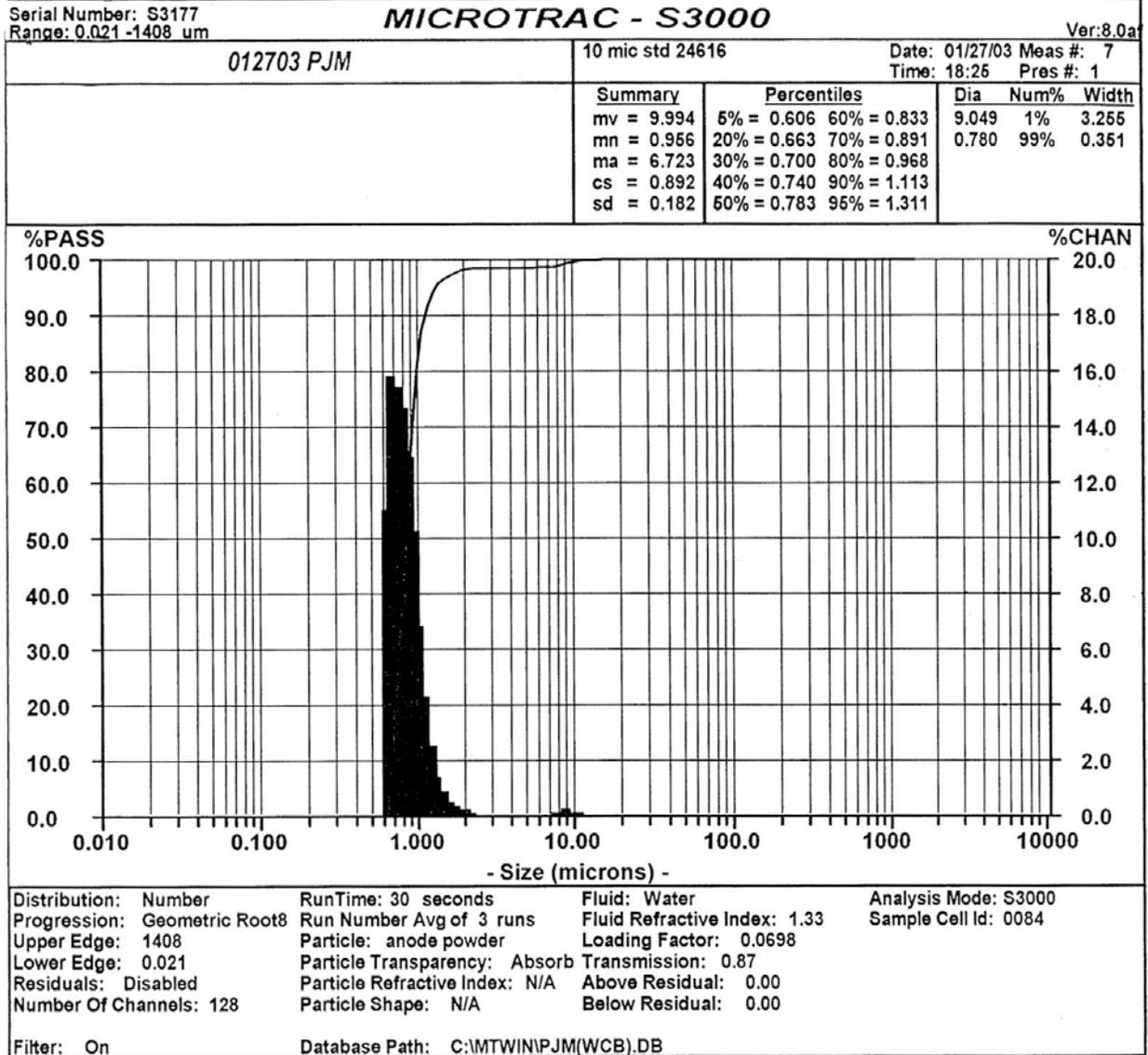
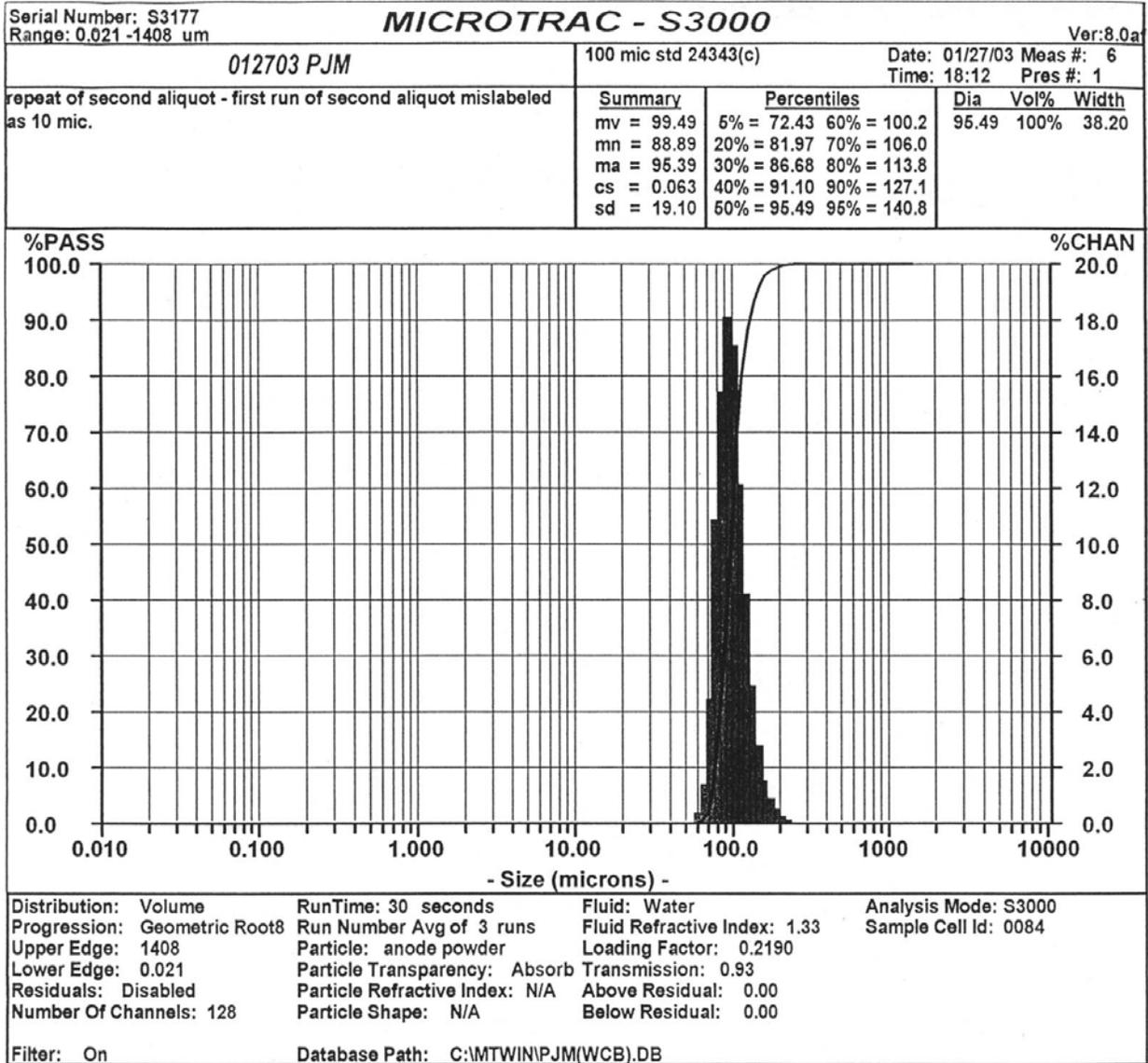
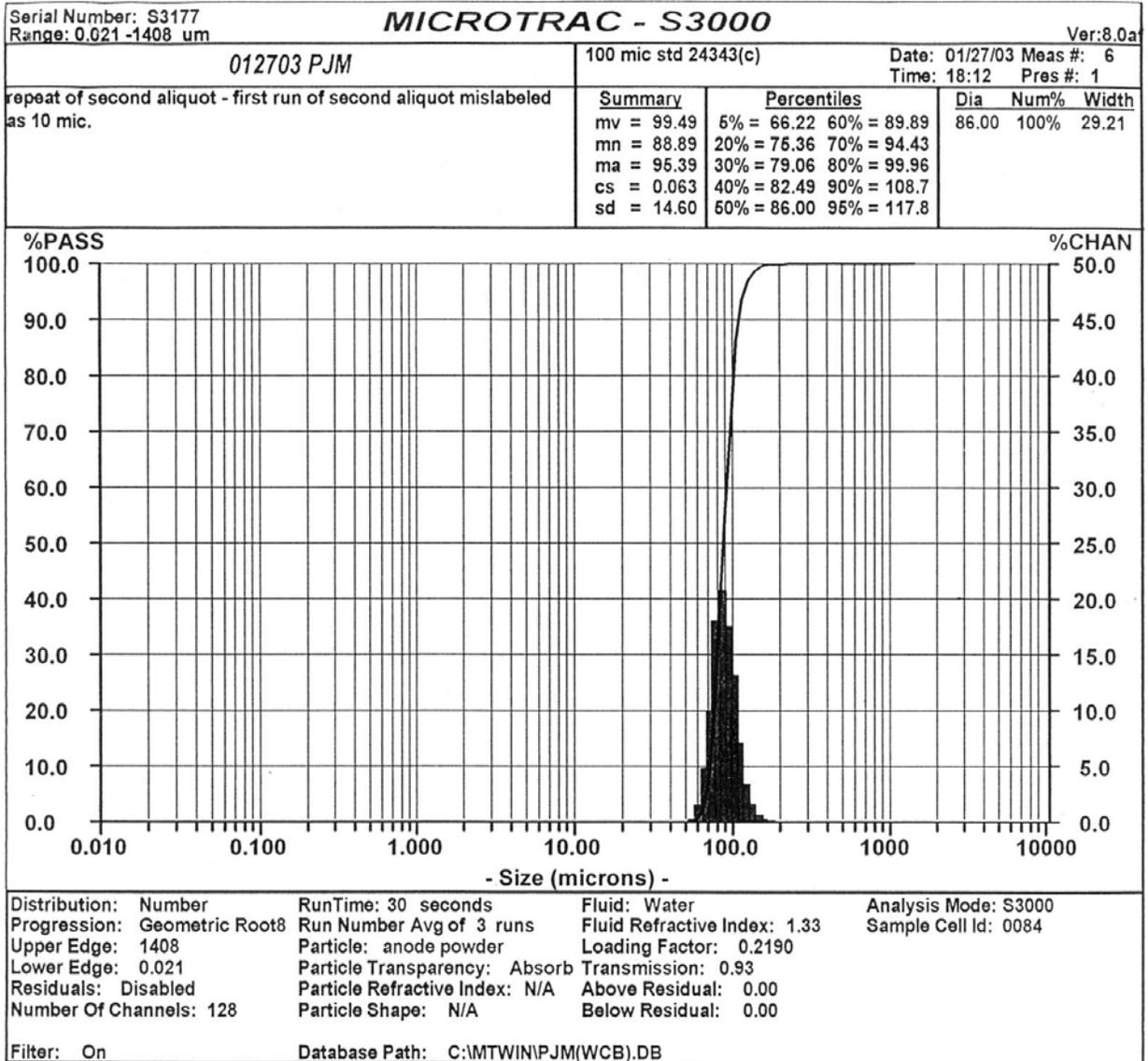


Figure A.2. Particle Size Distribution by Number of 10 Micron Standard 24616



**Figure A.3.** Particle Size Distribution by Volume of 100 Micron Standard 24343(c)



**Figure A.4.** Particle Size Distribution by Number of 100 Micron Standard 24343(c)

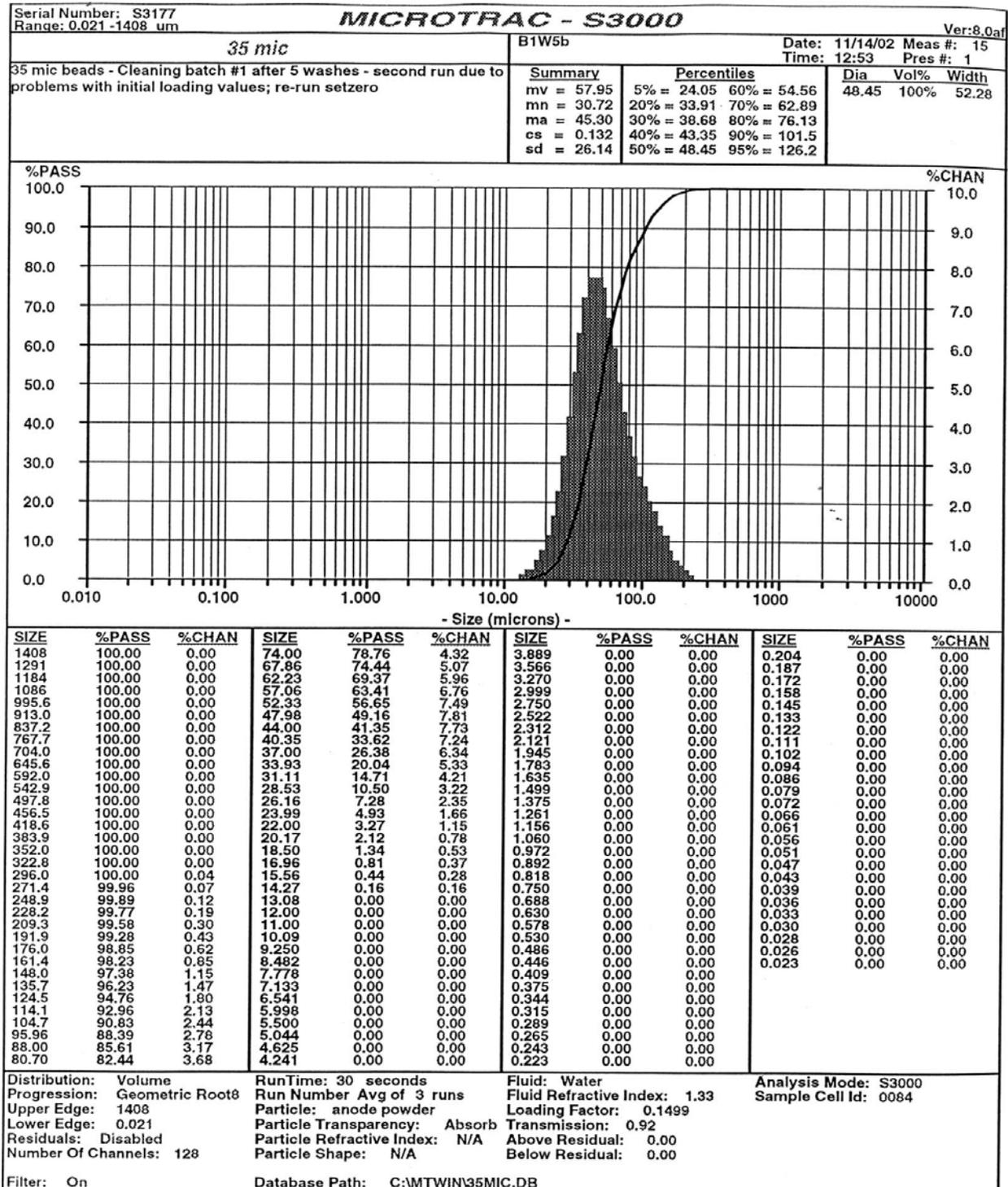


Figure A.5. Particle Size Distribution by Volume of Sample B1W5b

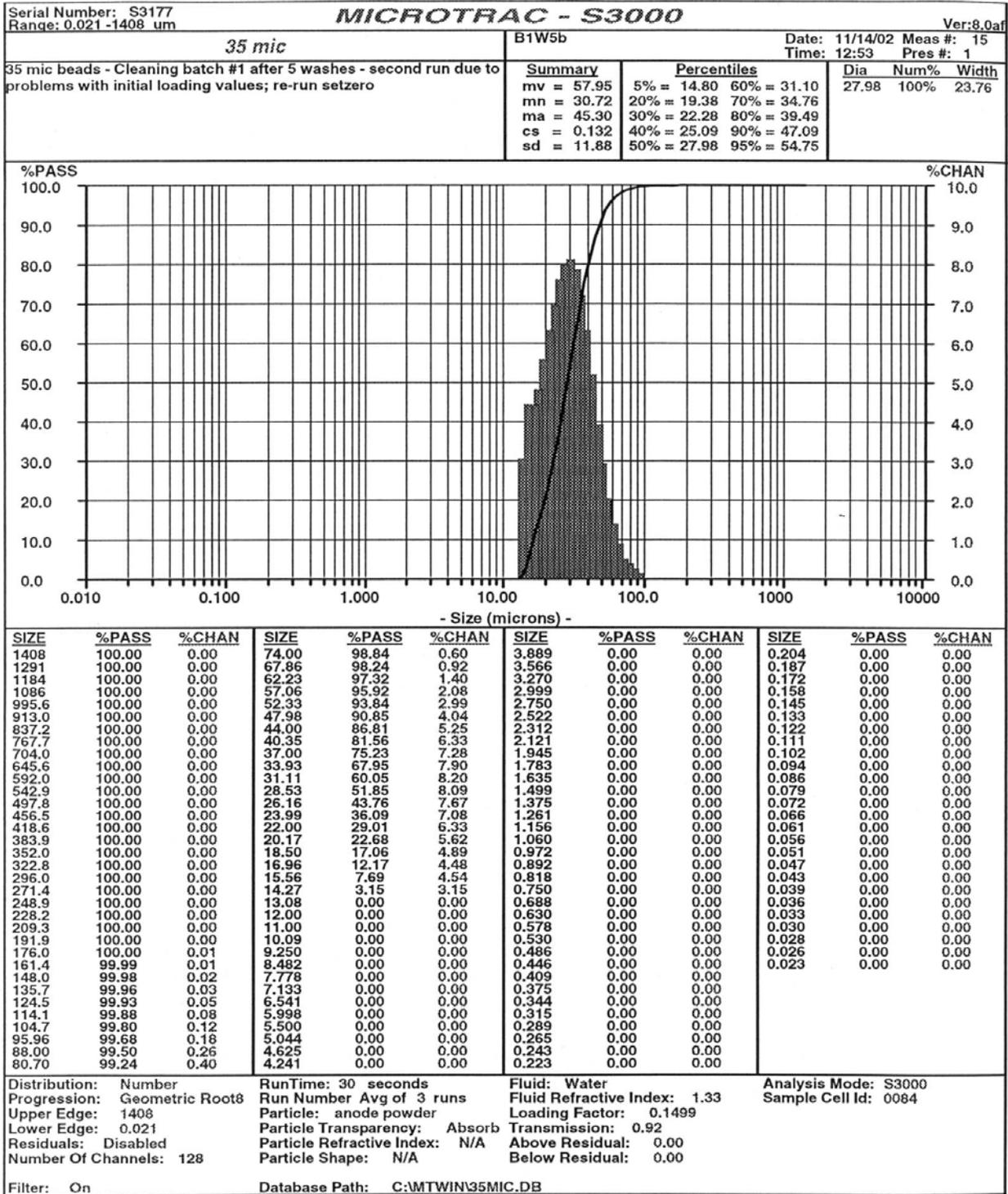


Figure A.6. Particle Size Distribution by Number of Sample B1W5b

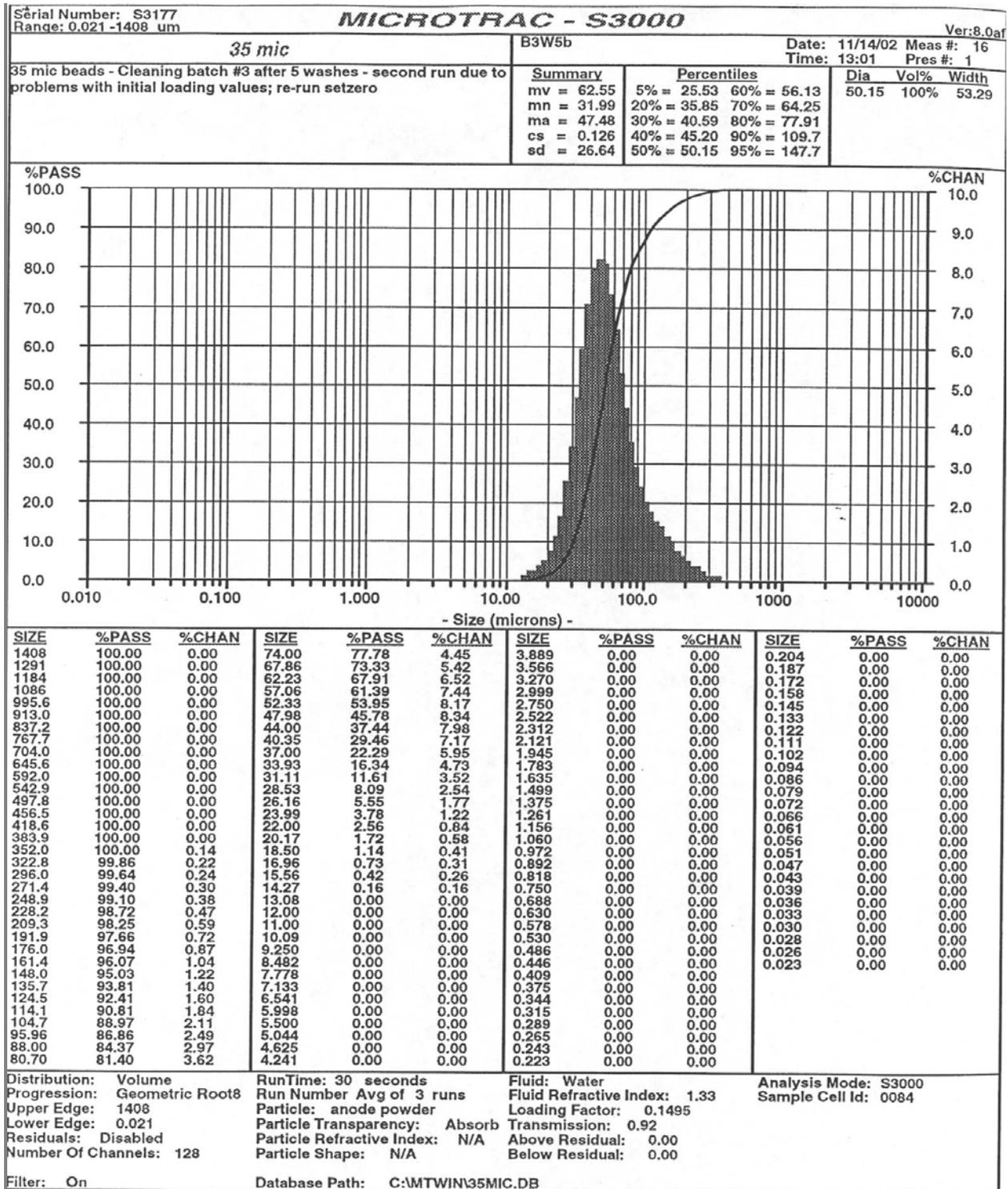


Figure A.7. Particle Size Distribution by Volume of Sample B3W5b

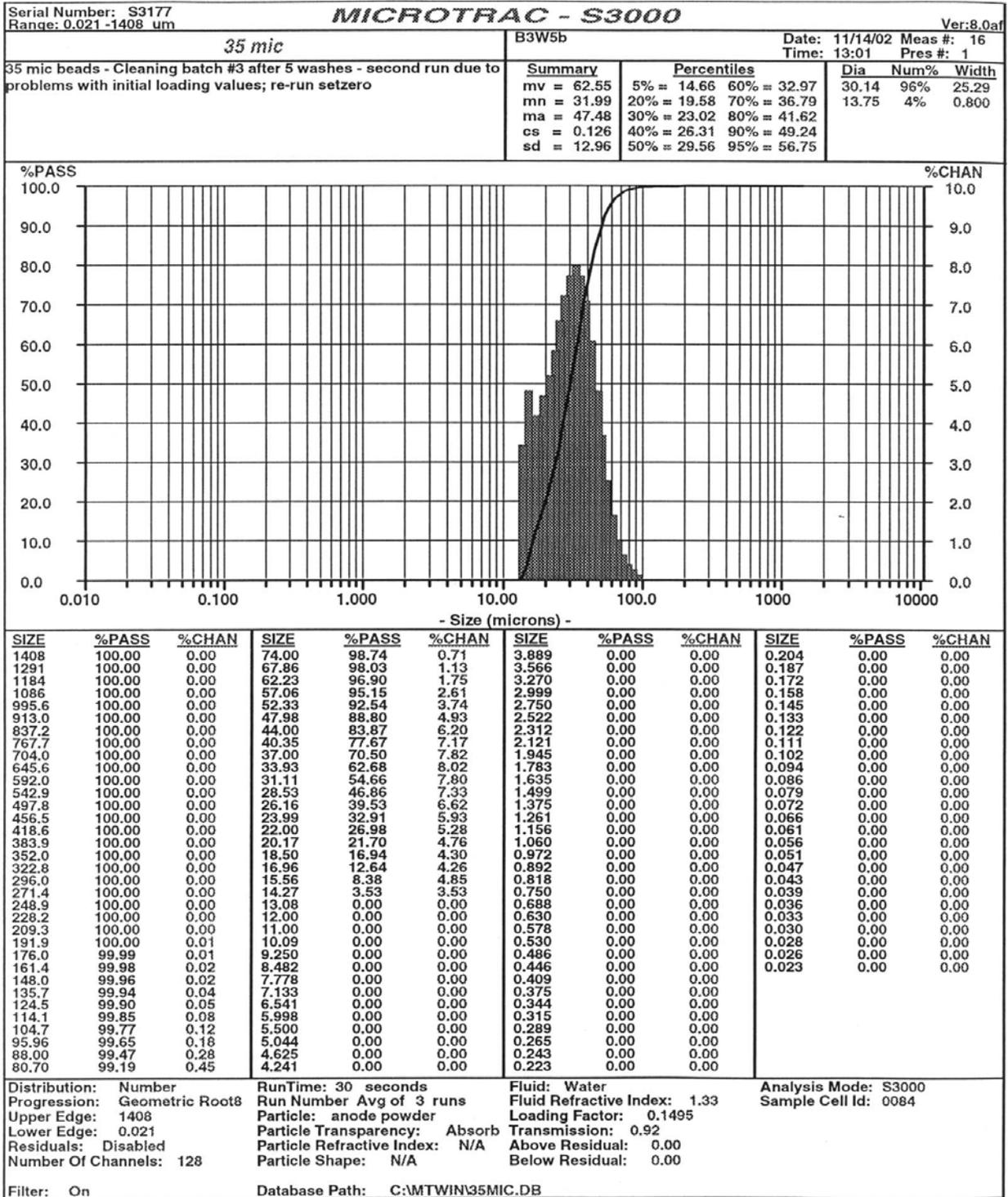


Figure A.8. Particle Size Distribution by Number of Sample B3W5b

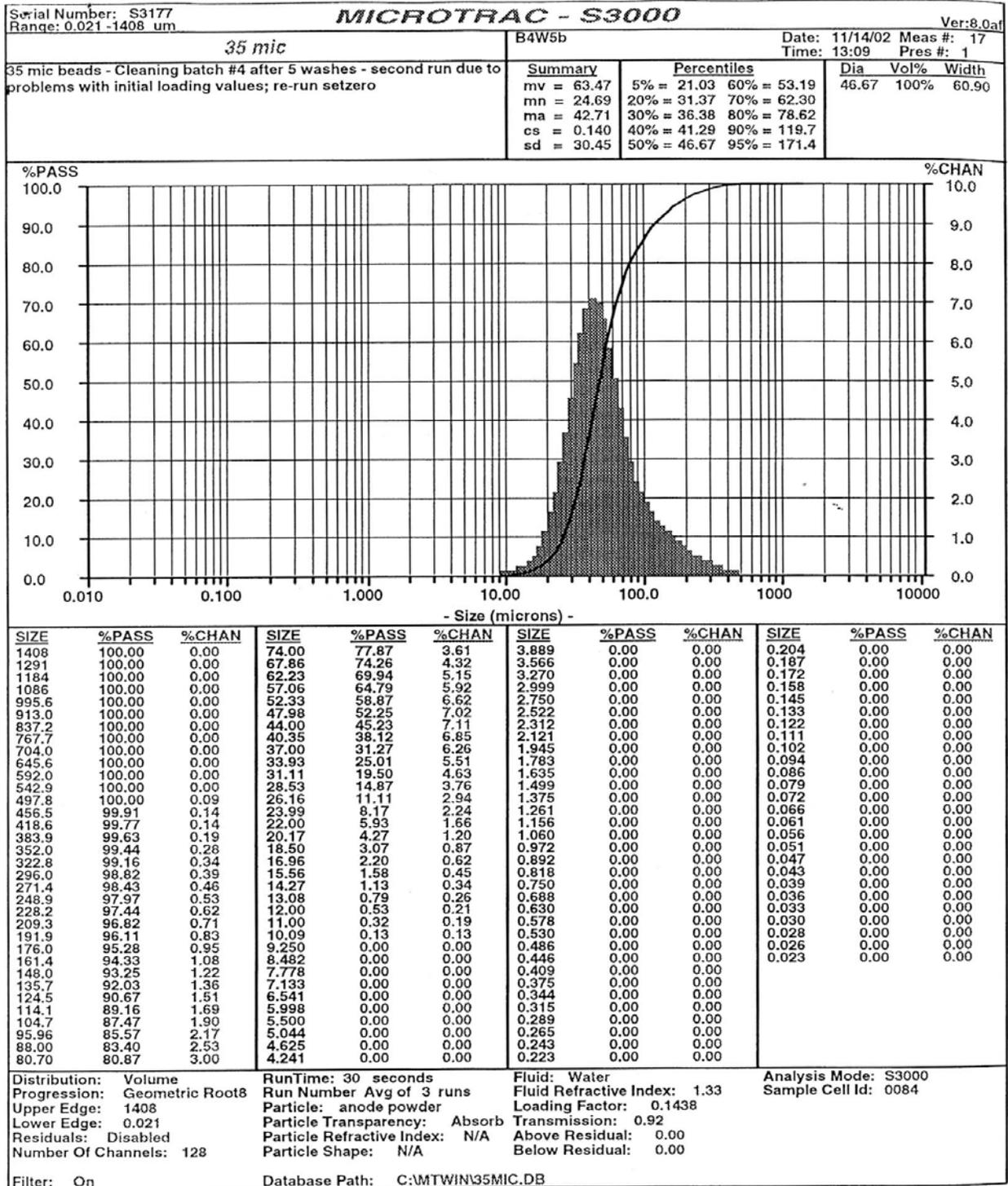


Figure A.9. Particle Size Distribution by Volume of Sample B4W5b

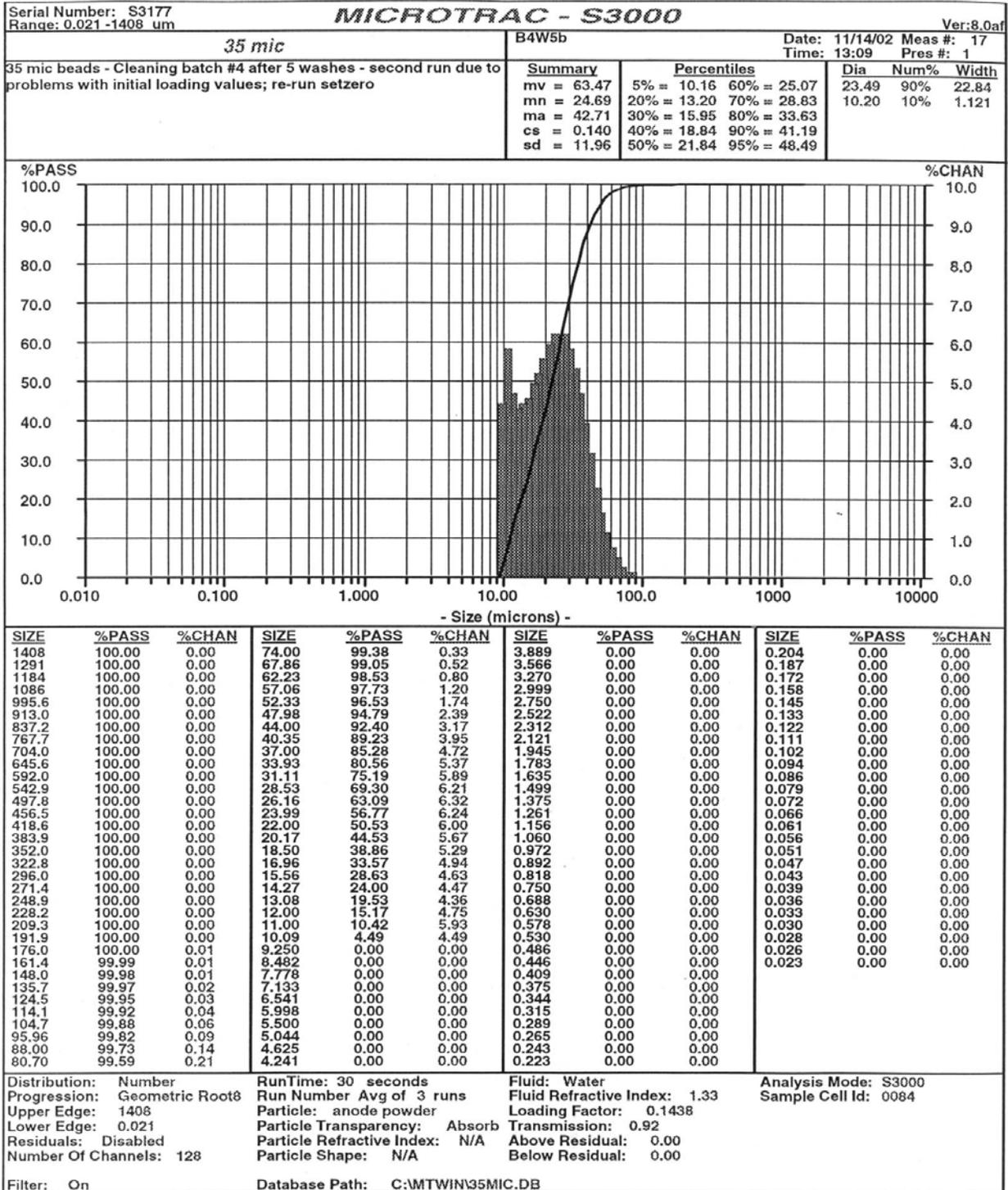


Figure A.10. Particle Size Distribution by Number of Sample B4W5b

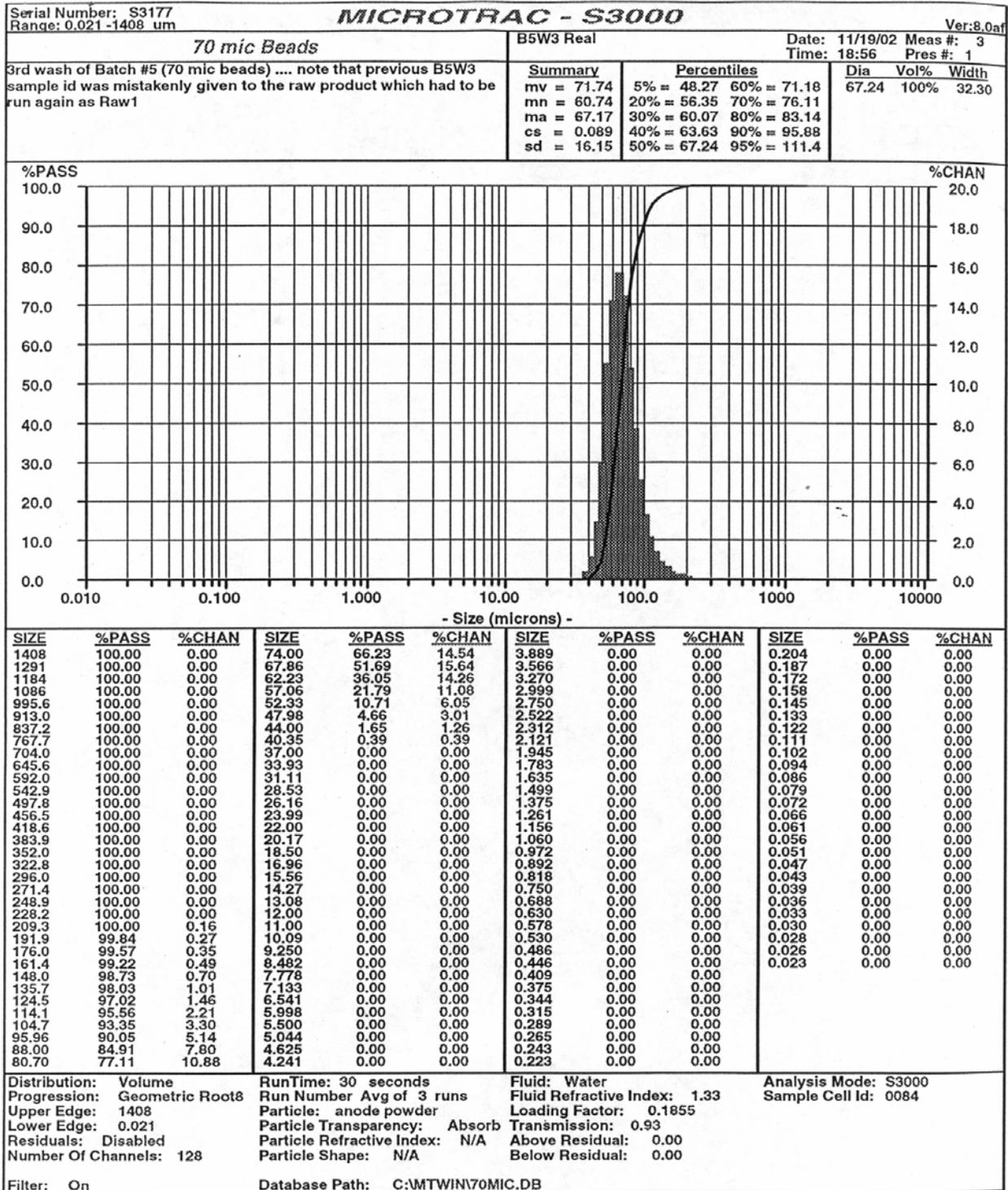


Figure A.11. Particle Size Distribution by Volume of Sample B5W3 Real

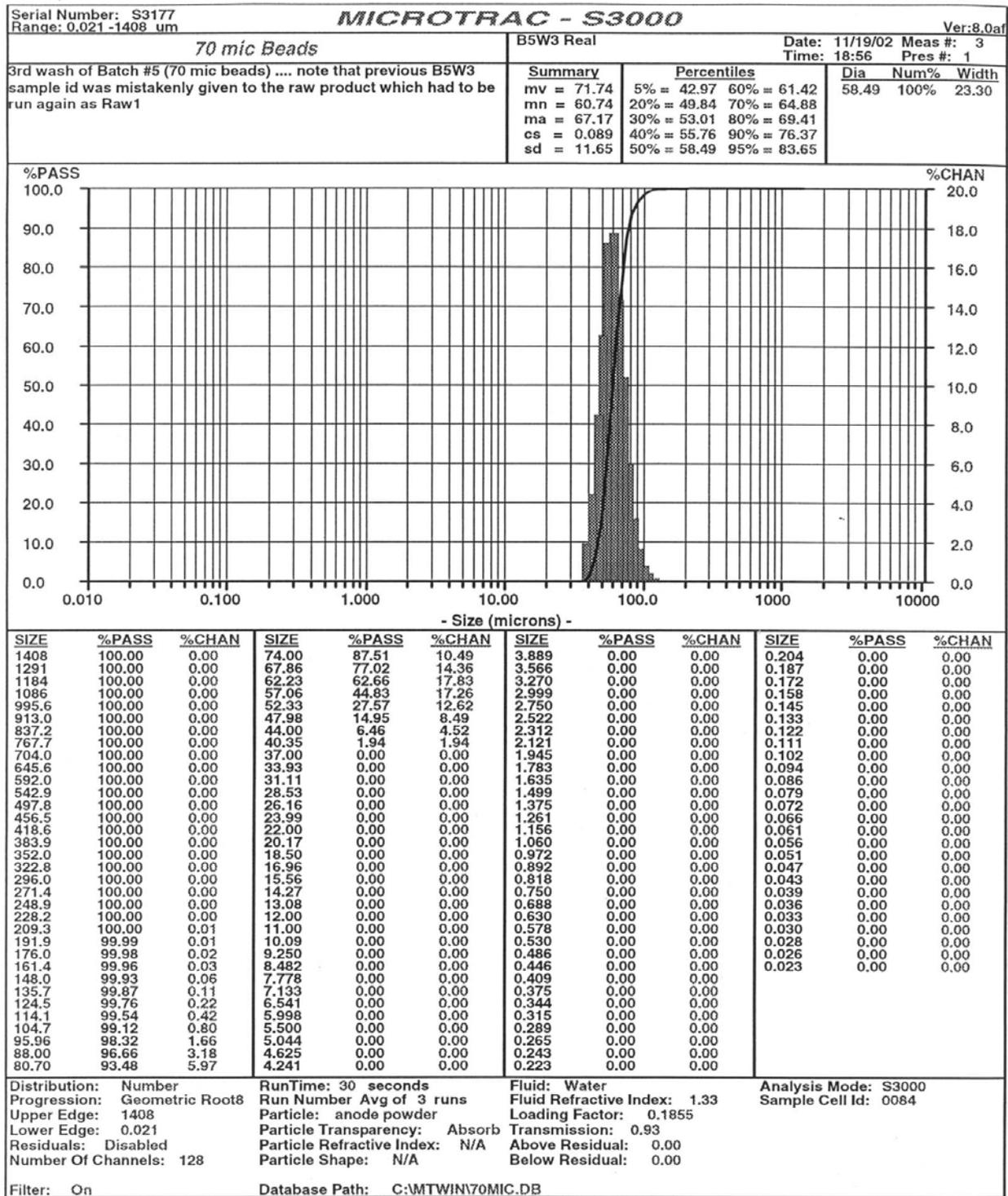


Figure A.12. Particle Size Distribution by Number of Sample B5W3 Real

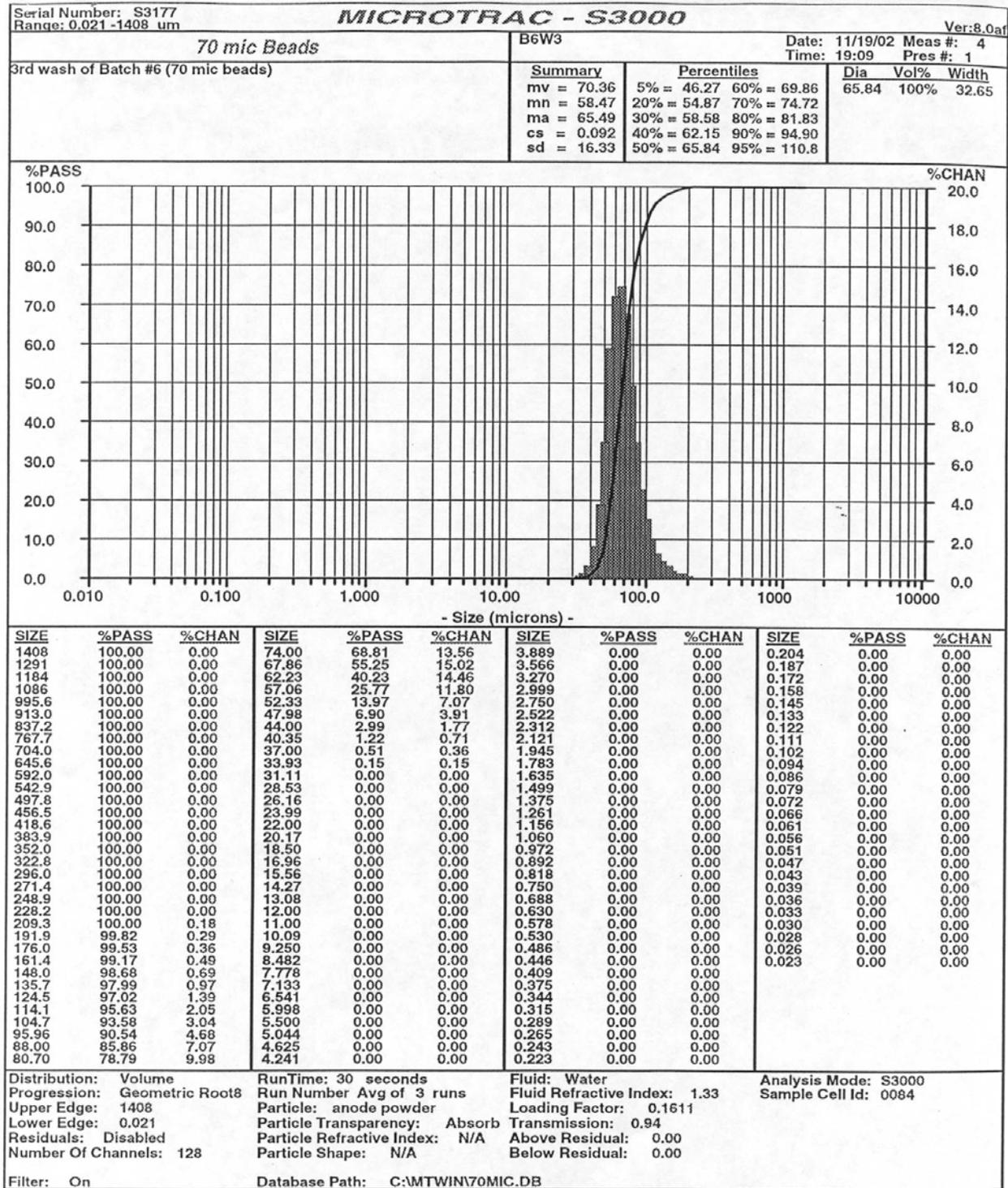


Figure A.13. Particle Size Distribution by Volume of Sample B6W3

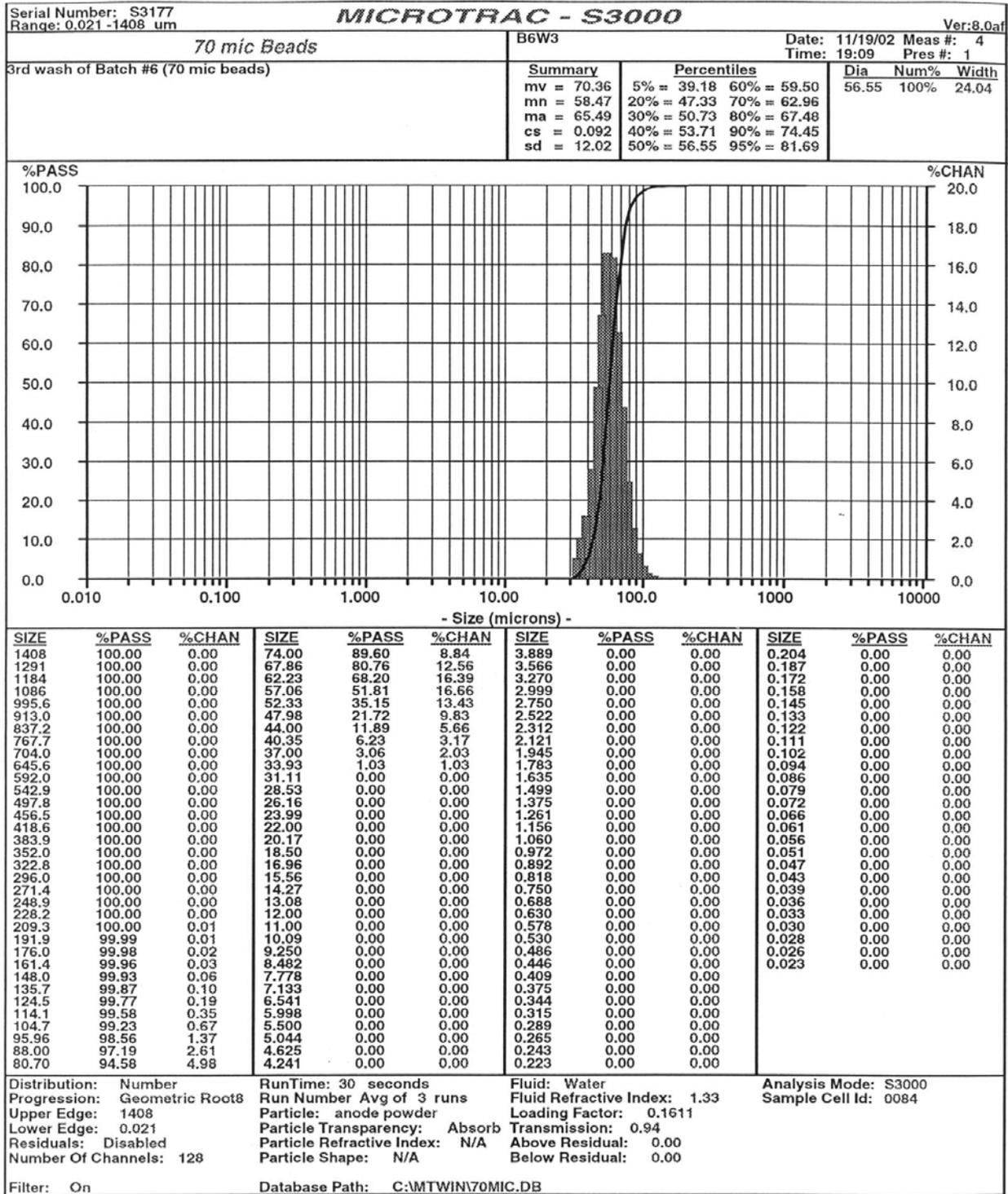


Figure A.14. Particle Size Distribution by Number of Sample B6W3

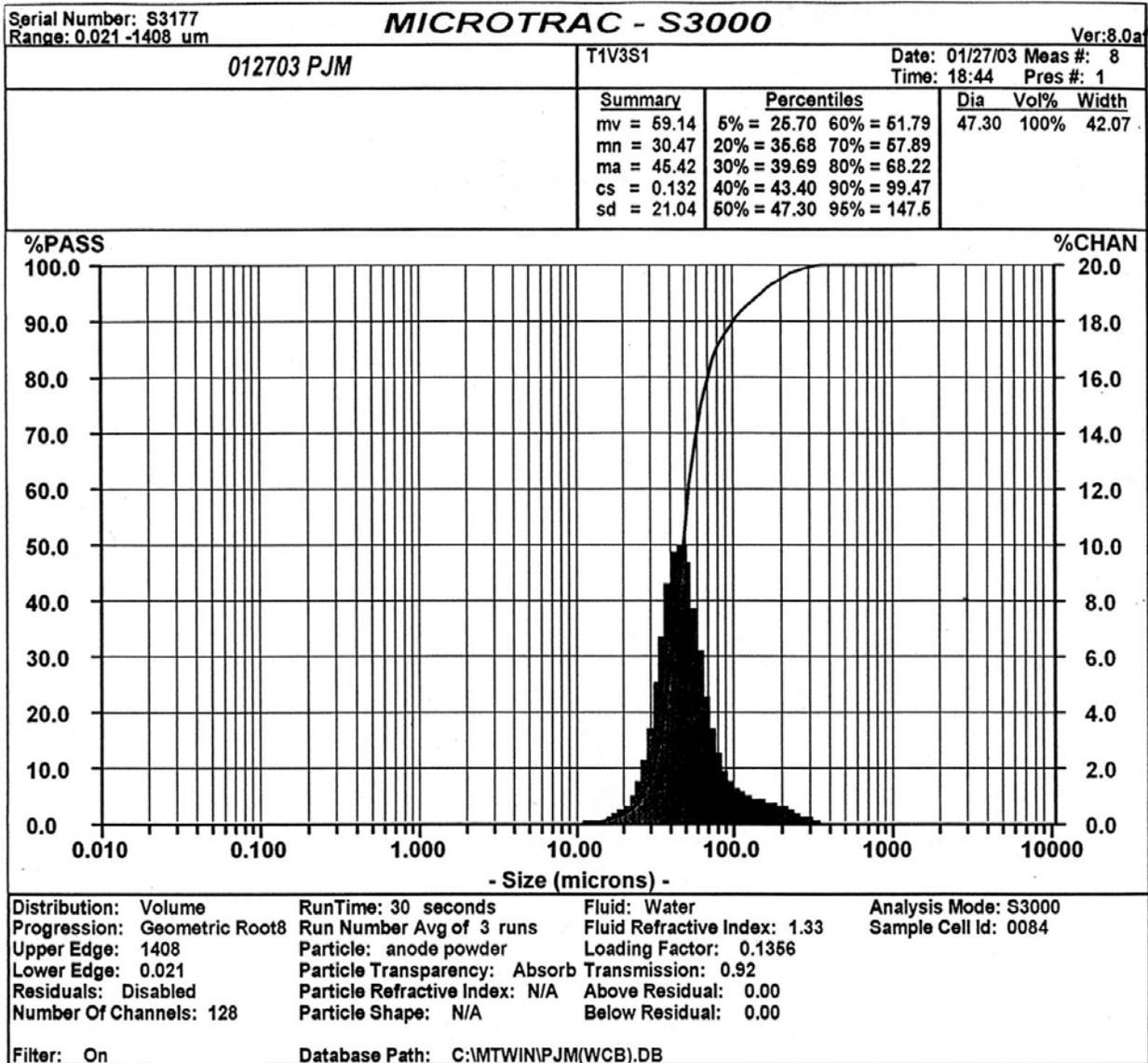


Figure A.15. Particle Size Distribution by Volume of Sample T1V3S1

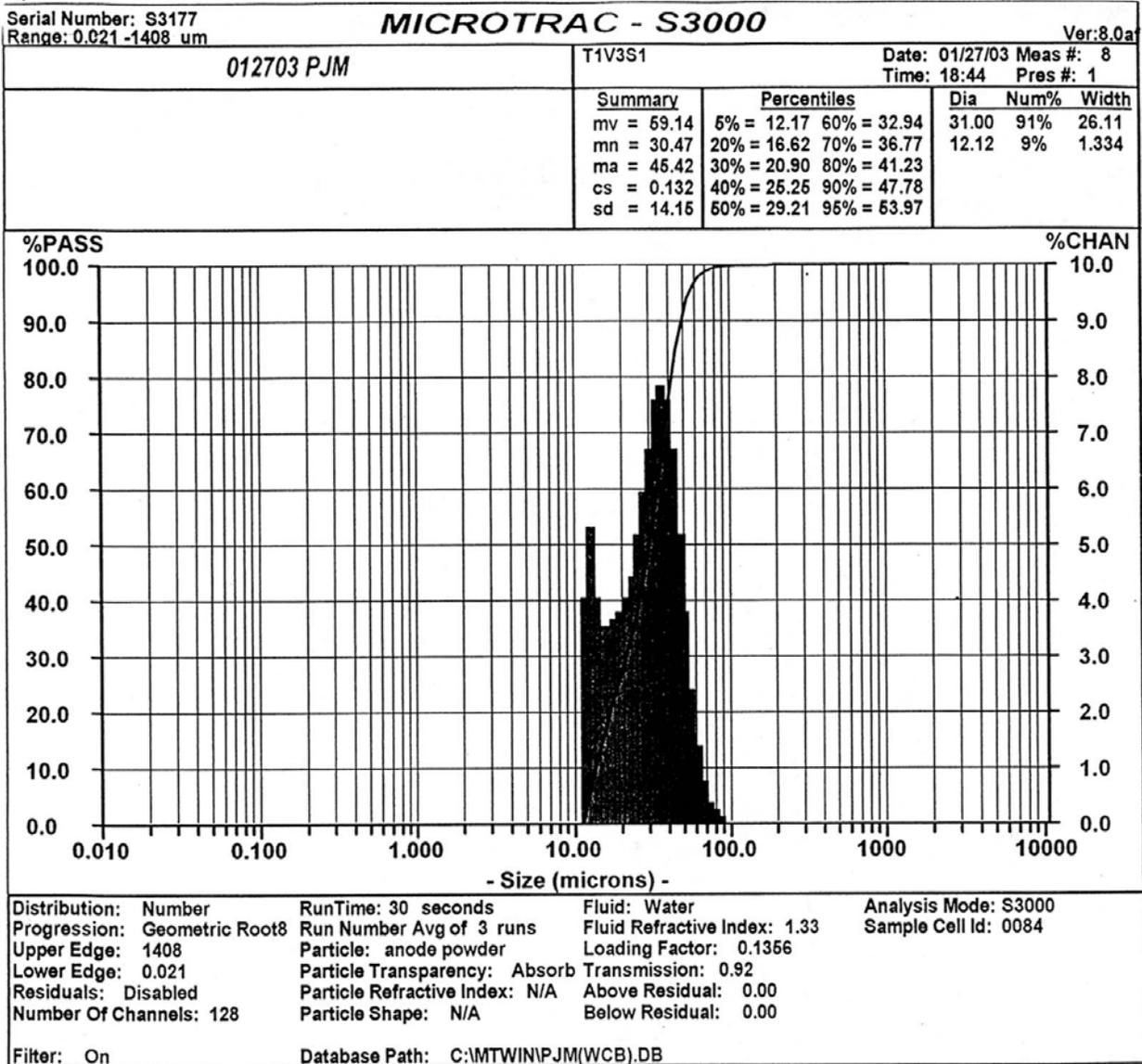


Figure A.16. Particle Size Distribution by Number of Sample T1V3S1

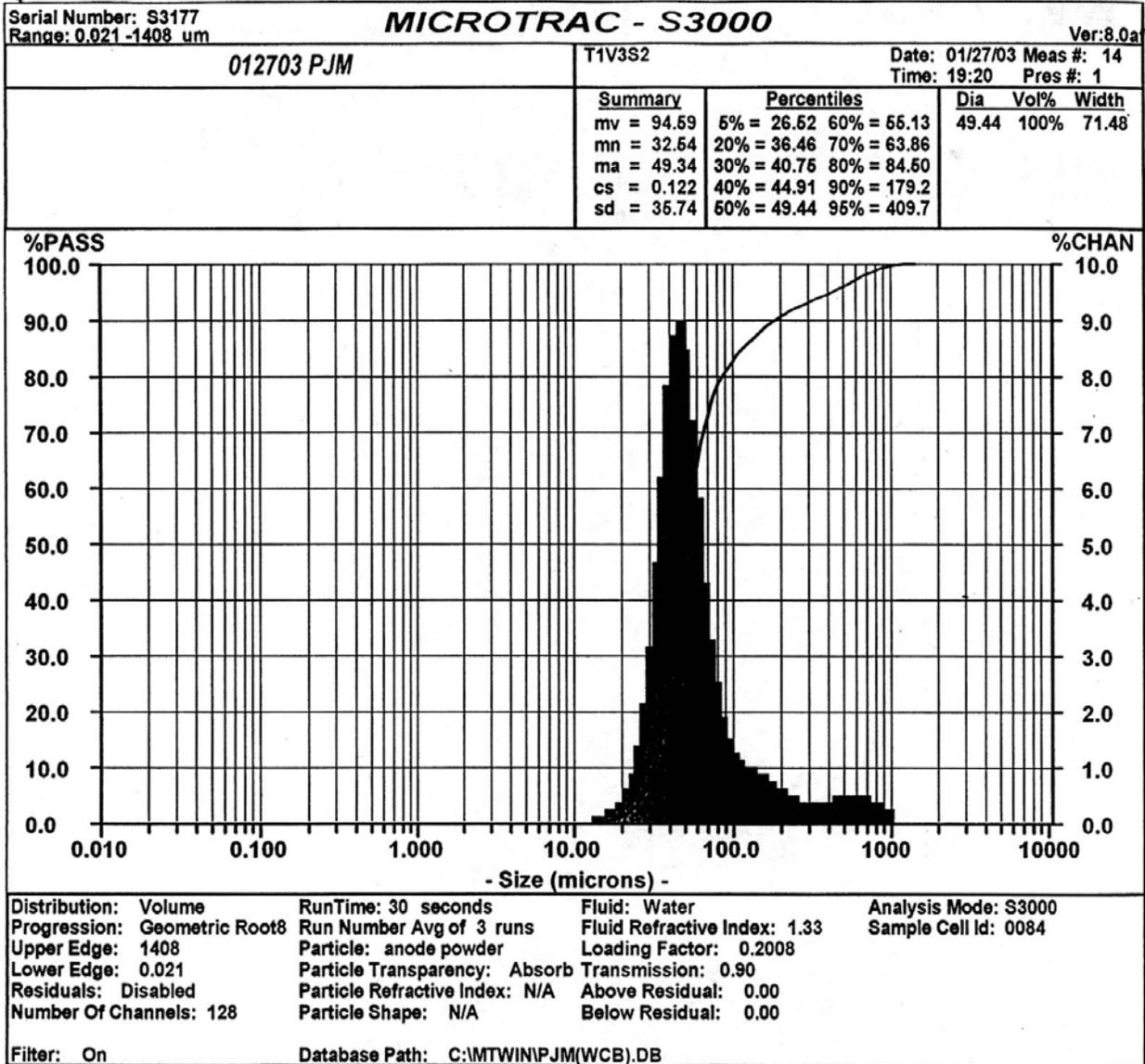


Figure A.17. Particle Size Distribution by Volume of Sample T1V3S2

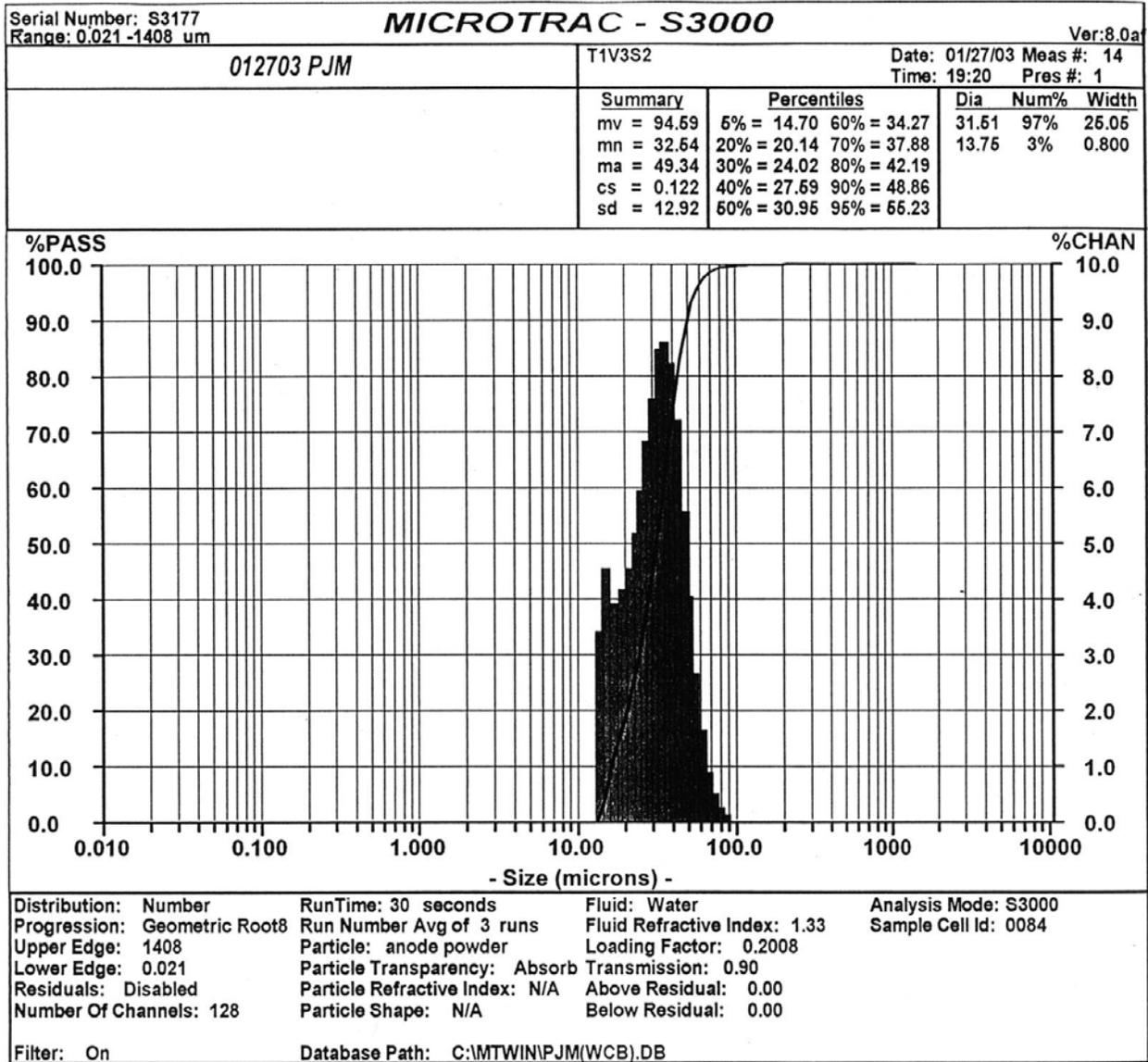
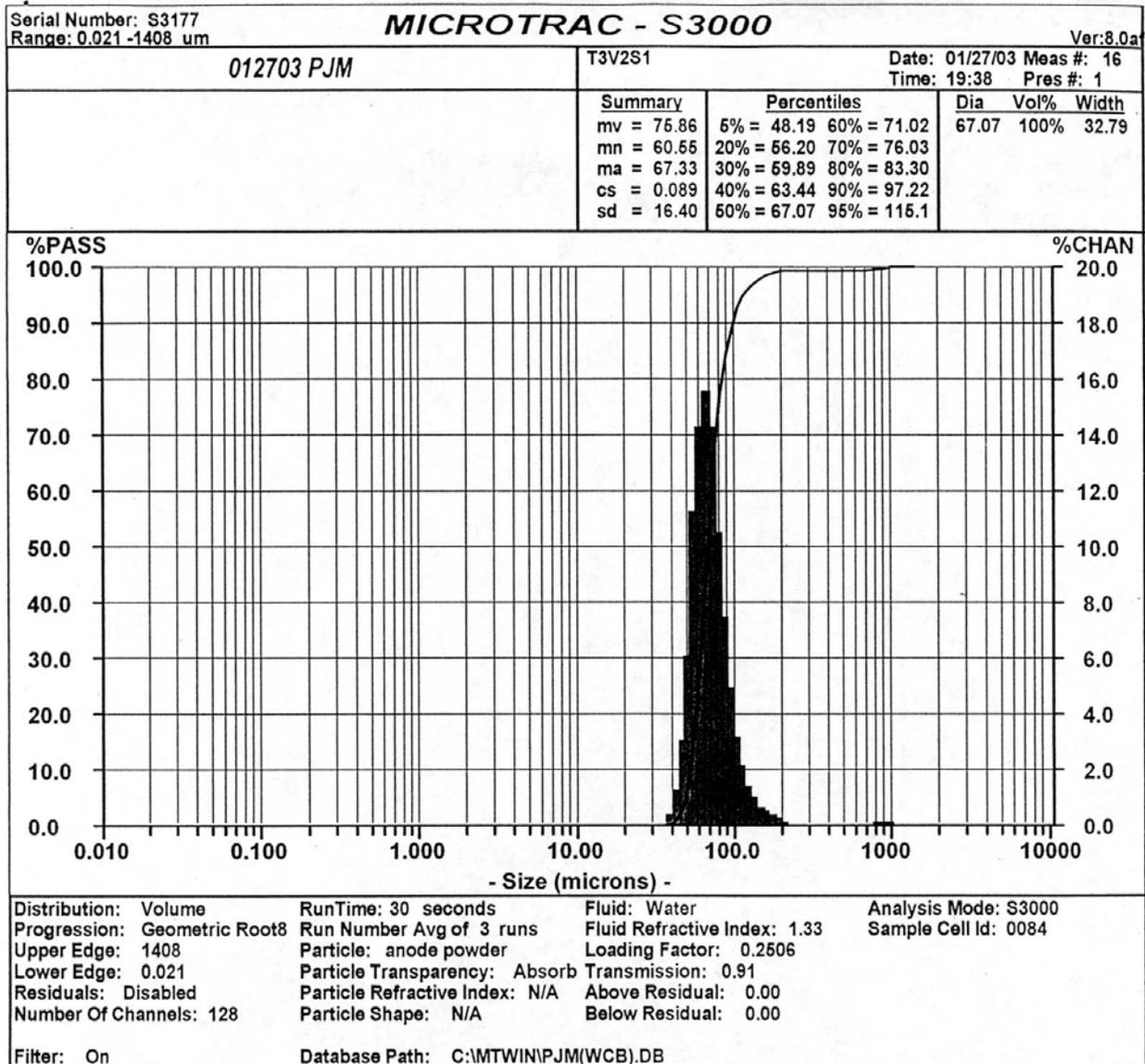
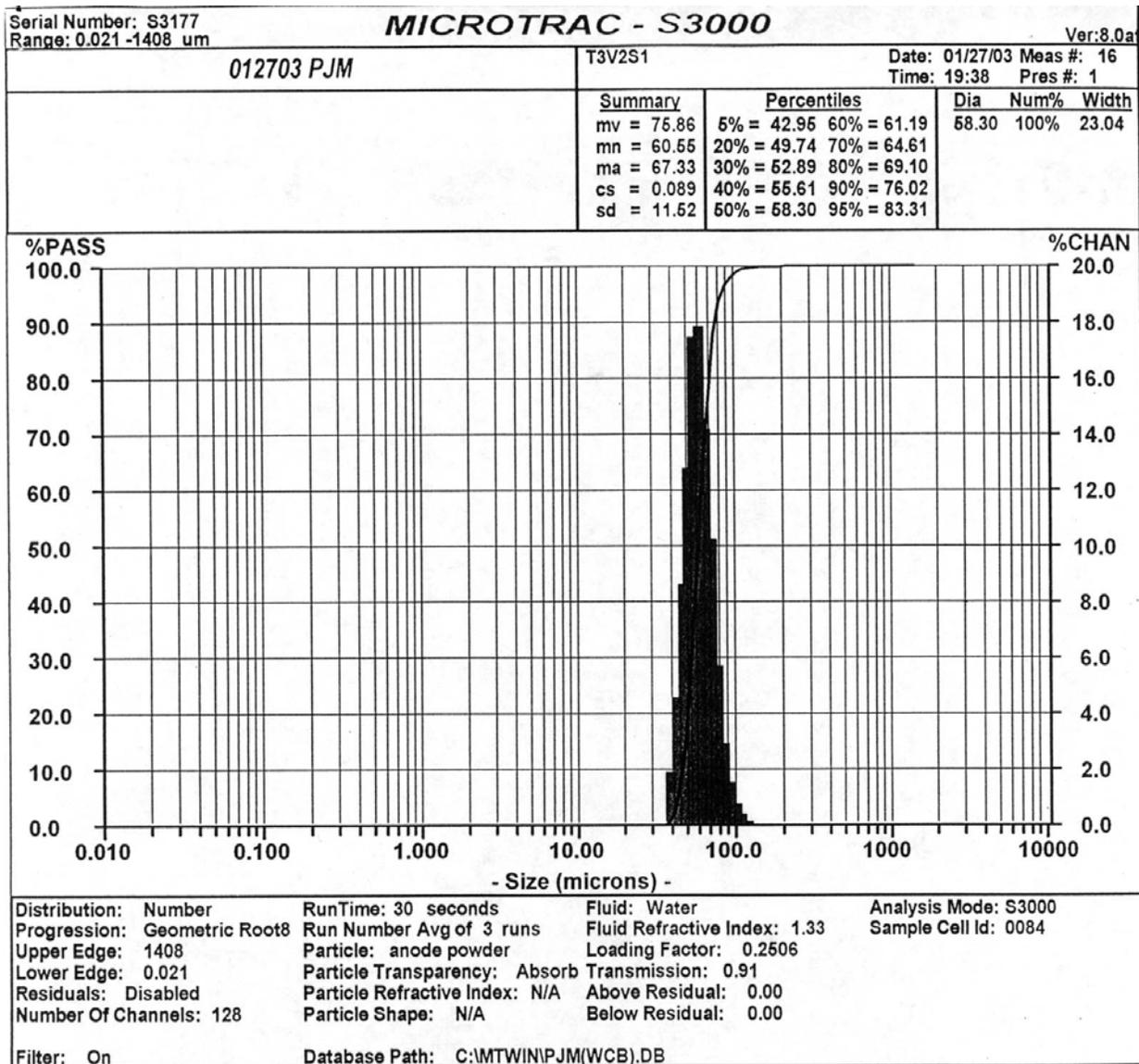


Figure A.18. Particle Size Distribution by Number of Sample T1V3S2



**Figure A.19.** Particle Size Distribution by Volume of Sample T3V2S1



**Figure A.20.** Particle Size Distribution by Number of Sample T3V2S1

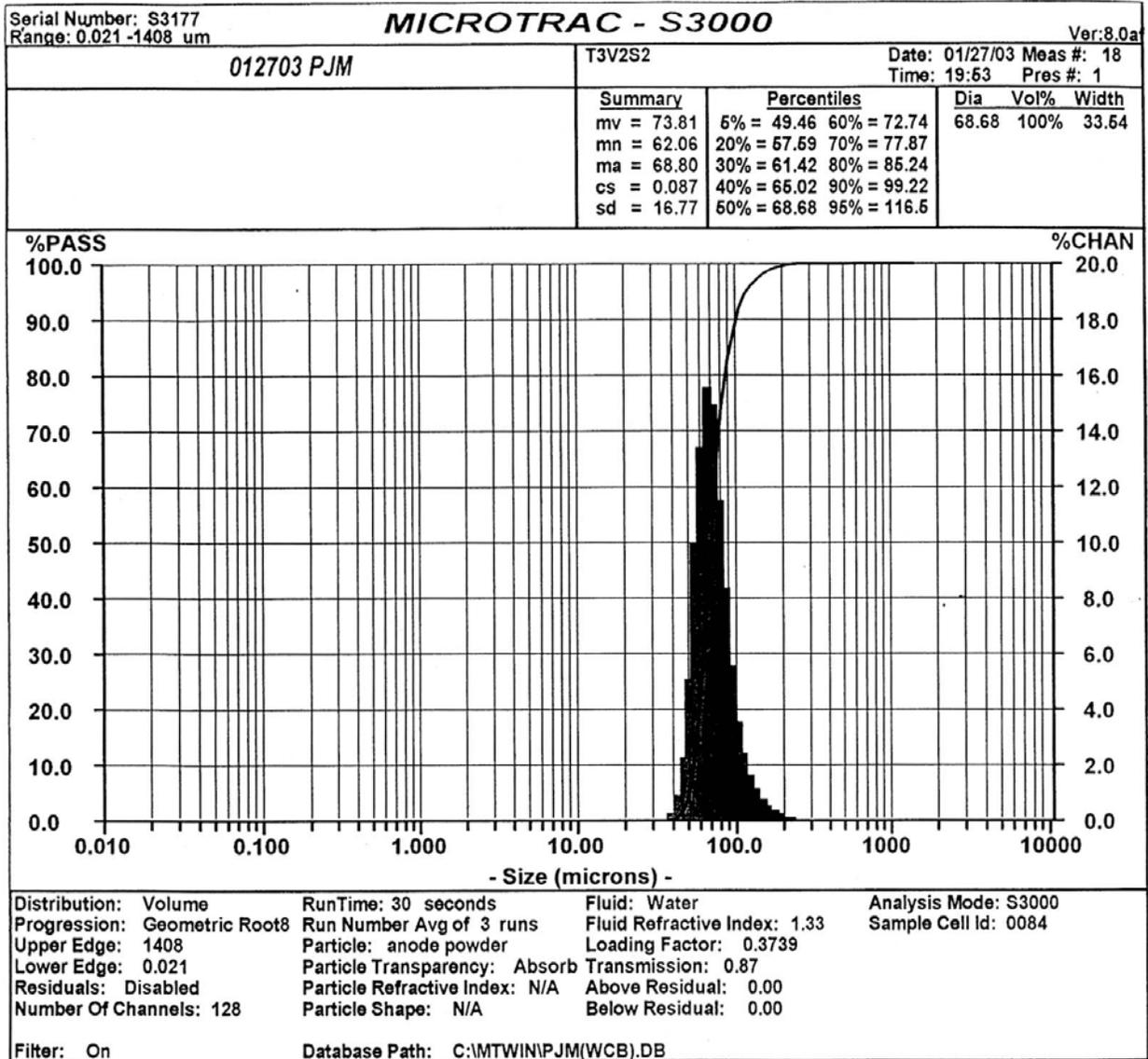


Figure A.21. Particle Size Distribution by Volume of Sample T3V2S2

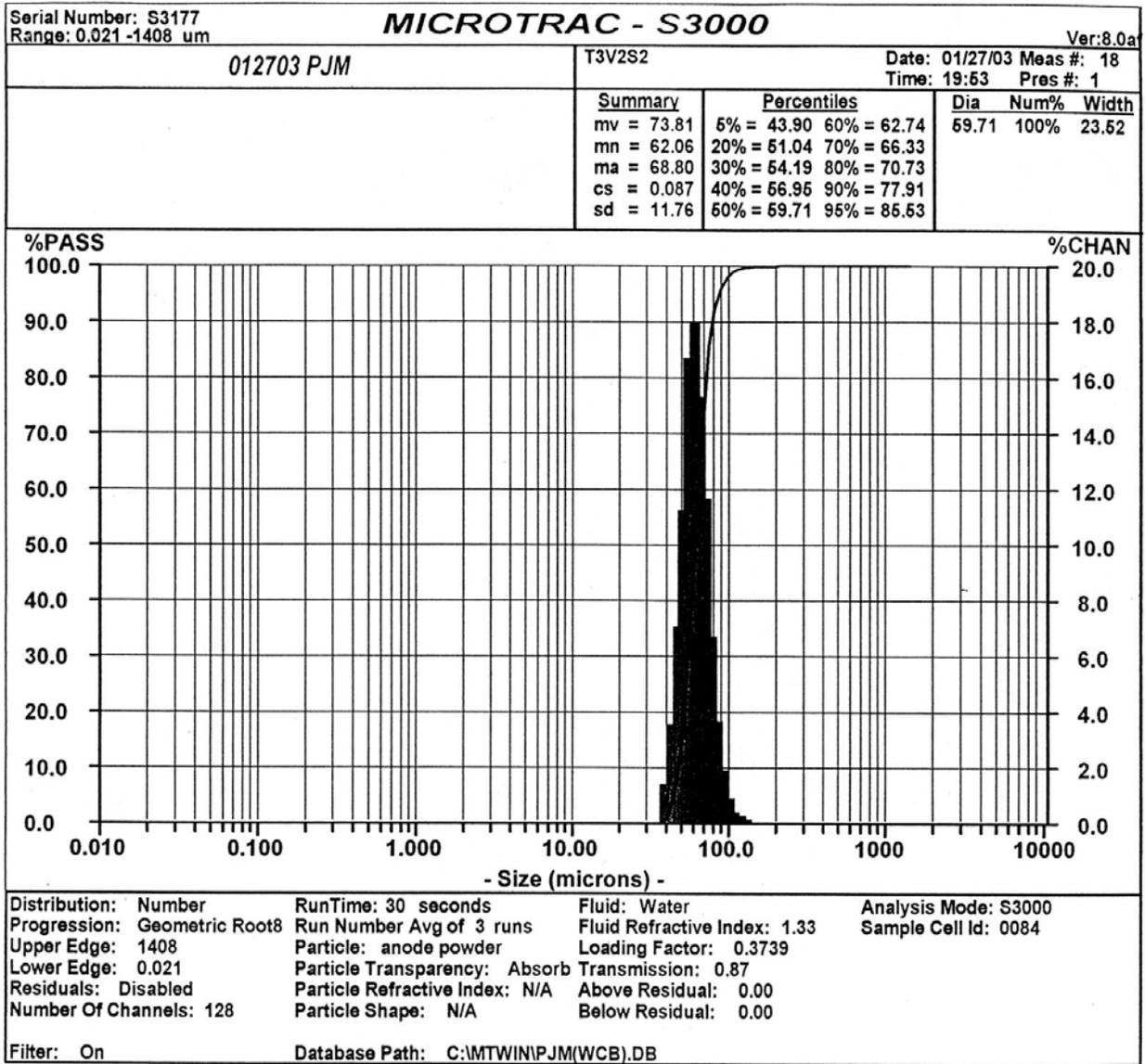


Figure A.22. Particle Size Distribution by Number of Sample T3V2S2

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