

WTP-RPT-198 Rev 0  
PNNL-18291



Prepared for the U.S. Department of Energy  
under Contract DE-AC05-76RL01830

# Results of Aging Tests of Vendor-Produced Blended Feed Simulant

RL Russell  
WC Buchmiller  
KJ Cantrell

RA Peterson  
DE Rinehart

April 2009



## DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor Battelle Memorial Institute, nor any of their employees, makes **any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights**. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or Battelle Memorial Institute. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

PACIFIC NORTHWEST NATIONAL LABORATORY

*operated by*

BATTELLE

*for the*

UNITED STATES DEPARTMENT OF ENERGY

*under Contract DE-AC05-76RL01830*

**Printed in the United States of America**

**Available to DOE and DOE contractors from the**

**Office of Scientific and Technical Information,**

**P.O. Box 62, Oak Ridge, TN 37831-0062;**

**ph: (865) 576-8401**

**fax: (865) 576 5728**

**email: reports@adonis.osti.gov**

**Available to the public from the National Technical Information Service,  
U.S. Department of Commerce, 5285 Port Royal Rd., Springfield, VA 22161**

**ph: (800) 553-6847**

**fax: (703) 605-6900**

**email: orders@nits.fedworld.gov**

**online ordering: <http://www.ntis.gov/ordering.htm>**

# Results of Aging Tests of Vendor-Produced Blended Feed Simulant

RL Russell            RA Peterson  
WC Buchmiller      DE Rinehart  
KJ Cantrell

April 2009

Test specification: 24590-WTP-TSP-RT-07-004, Rev 0

Test plan: TP-RPP-WTP-509, Rev. 0.5

Test exceptions: 24590-WTP-TEF-RT-08-00013

R&T focus area: Pretreatment

Test Scoping Statement: None

Prepared for the U.S. Department of Energy  
under Contract DE-AC05-76RL01830

Pacific Northwest National Laboratory  
Richland, Washington 99352

## ***Completeness of Testing***

*This report describes the results of work and testing specified by Test Specification, 24590-WTP-TSP-RT-07-004, Rev 0, Test Plan TP-RPP-WTP-509, Rev 0, and Test Exception 24590-WTP-TEF-RT-08-00013 Rev 0. The work followed the quality assurance requirements outlined in the Test Specification and Test Plan. The descriptions provided in this 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 has been reviewed and verified.*

**Approved:**



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

5/1/09  
Date

# Contents

Abbreviations and Acronyms .....	v
Testing Summary .....	vii
Objective.....	vii
Success Criteria.....	ix
Test Exceptions.....	x
Quality Requirements .....	xi
R&T Test Conditions.....	xii
Simulant Use.....	xiv
Discrepancies and Follow-on Tests .....	xiv
1.0 Introduction.....	1.1
2.0 Aging Results.....	2.1
2.1 Particle-Size Distribution.....	2.1
2.2 Settling Tests .....	2.9
2.3 Rheology.....	2.10
2.4 Solids Content.....	2.11
2.5 Supernatant Composition.....	2.12
3.0 Conclusions.....	3.1
4.0 References.....	4.1
Appendix A: PSD Plots .....	A.1
Appendix B: Rheology Flow Curves .....	B.1
Appendix C: Chemical Analysis.....	C.1

## Figures

2.1.	PSD (Volume Percent) for the Stored-Outside Samples .....	2.2
2.2.	PSD (Number Percent) for the Stored-Outside Samples .....	2.3
2.3.	PSD (Volume Percent) for the Two Well Stirred Samples .....	2.4
2.4.	PSD (Number Percent) for the Two Well Stirred Samples .....	2.4
2.5.	PSD (Volume Percent) for the Stored Inside Samples .....	2.5
2.6.	PSD (Number Percent) for the Stored Inside Samples .....	2.6
2.7.	PSD (Volume Percent) for the Temperature Cycled Samples.....	2.7
2.8.	PSD (Number Percent) for the Temperature Cycled Samples .....	2.7
2.9.	PSD (Volume Percent) for the Last Sample Collected for Each Aging Scenario .....	2.8
2.10.	PSD (Number Percent) for the Last Sample Collected for Each Aging Scenario .....	2.9
2.11.	Volume-Percent Solids as a Function of Time .....	2.10
2.12.	Average Viscosity as a Function of Time .....	2.11

## Tables

S.1.	Test Objectives from TP-RPP-WTP-509, Rev. 0 .....	vii
S.2.	Results and Performance against Success Criteria of TP-RPP-WTP-509, Rev. 0.....	ix
S.3.	Test Exceptions to Test Plan TP-RPP-WTP-509.....	xi
S.4.	R&T Test Conditions from 24590-WTP-TSP-RT-07-004, Rev. 0.....	xii
2.1.	PSD Samples Collected During the Aging Experiment .....	2.1
2.2.	Aging Test PSD Volume Percent Distribution.....	2.1
2.3.	Aging Test PSD Number Percent Distribution.....	2.2
2.4.	Rheology Results for the Aging Test Samples .....	2.11
2.5.	Percent Total, Dissolved, and Undissolved Solids Content .....	2.12
2.6.	Centrifuged Solids Content .....	2.12
2.7.	Supernatant Composition Results for Stored-Outside Aging Scenario (mg/kg solution) .....	2.13
2.8.	Supernatant Composition Results for Stirred Aging Scenario (mg/kg solution).....	2.13
3.1.	Summary of Aging Effects .....	3.2

## Abbreviations and Acronyms

BNI	Bechtel National, Inc.
DIW	De-Ionized Water
DOE	U.S. Department of Energy
IC	Ion Chromatography
ICP-AES	Inductively Coupled Plasma-Atomic Emission Spectroscopy
PEP	Pretreatment Engineering Platform
PNNL	Pacific Northwest National Laboratory
PSD	Particle-Size Distribution
QAM	Quality Assurance Manual
QAP	Quality Assurance Program
QARD	Quality Assurance Requirements and Description (document)
RPP	River Protection Project
R&T	Research and Technology
SBMS	Standards-Based Management System
SWRI	Southwest Research Institute
WTP	Hanford Tank Waste Treatment and Immobilization Plant



## Testing Summary

This report includes the results of the simulant aging tests performed in accordance with the test plan TP-RPP-WTP-509, Rev 0<sup>(1)</sup> (WTP Doc. No. 24590-101-TSA-W000-0004-72-00019 Rev 00A) prepared and approved in response to the Test Specification 24590-WTP-TSP-RT-07-004, Rev 0 (Sundar 2007), and the Test Exception 24590-WTP-TEF-RT-08-00013 Rev 0 (Sundar 2008).

### Objective

The test objectives for the work addressed in TP-RPP-WTP-509, Rev 0 (Daniel and Shimskey 2007) are summarized in Table S.1 along with a discussion of how the objectives were met. The overall objective of the work from the test plan described in this report was to determine the effect of aging on the physical characteristics of the filtration simulant.

**Table S.1.** Test Objectives from TP-RPP-WTP-509, Rev. 0

Test Objective	Objective Met? (Y/N)	Discussion
1) Determine the effect of initial aluminate ion concentration on the rate of boehmite leaching in caustic solutions and in the presence of soluble anions in a waste. The anions to be considered are those that are typically present in the Hanford Tank Farm wastes in significant amounts. This includes carbonate, free-hydroxide, nitrate, nitrite, oxalate, phosphate, and sulfate.	NA	This objective was addressed in report WTP-RPT-184, Rev. 0.
2) Determine the sensitivity of the rate of dissolution of boehmite to soluble anions through a limited number of laboratory tests. The anions to be considered are those that are typically present in the Hanford Tank Farm wastes in significant amounts. This includes carbonate, free-hydroxide, nitrate, nitrite, oxalate, phosphate, and sulfate.	NA	As of March 20, 2008, Pacific Northwest National Laboratory (PNNL) has been released from this objective by Test Exception, 24590-WTP-TEF-RT-07-00016.
3) Determine the effect of scaling the length of the ultrafilter element from 2 ft to 8 ft on the filtrate flux over the expected operating range of the ultrafilter using the crossflow ultrafiltration unit.	NA	This objective was addressed in report WTP-RPT-168, Rev. 0.

**Table S.1 (Contd)**

<b>Test Objective</b>	<b>Objective Met? (Y/N)</b>	<b>Discussion</b>
4) Use an 8-ft-long filter element in the crossflow ultrafiltration unit to determine the effect of temperature on the filtration of a waste simulant over the range of temperature conditions for the leaching processes.	NA	This objective was addressed in report WTP-RPT-168, Rev. 0.
5) Use a 2-ft-long filter element in the crossflow ultrafiltration unit to evaluate the effect of the fine-particle fraction in the ultrafiltration simulant on fouling of the filter element over the range of concentrations of operating solids. The fine-particle fraction is defined as those particles with diameters smaller than the 10 <sup>th</sup> percentile (i.e., the dp10) of the particle-size number distribution.	NA	This objective was addressed in report WTP-RPT-183, Rev. 0.
6) Perform various simulant aging tests to understand the changes that may occur to the simulant in storage and to confirm the adequacy of the simulant for use in the Pretreatment Engineering Platform (PEP).	Y	Aging tests were performed under various circumstances, including temperature cycling, stirring, storing in the laboratory, and storing outside in a tote. It was found that there was no significant aging effect on the filtration simulant under these conditions in the time frame tested.
7) Perform Cr-simulant leaching tests to establish that the Cr-simulant from the larger batch exhibits similar or better leaching behavior than the initial trial batch during caustic and oxidative leaching operations.	NA	These results will be presented in a report that is yet to be released.
8) Perform leaching tests to determine the mass loss and aluminum and chromium dissolution rates during caustic leaching under varying temperature-processing conditions without aeration in both UFP-1A/B and UFP-2A/B vessels as well as to measure the effect of aeration on chromium leaching in UFP-2A/B.	NA	These results will be presented in a report that is yet to be released.
9) Perform leaching tests to develop an accurate model for the dissolution of boehmite.	NA	These results will be presented in a report that is yet to be released.

**Table S.1 (Contd)**

<b>Test Objective</b>	<b>Objective Met? (Y/N)</b>	<b>Discussion</b>
10) Perform leaching tests to verify the effect of aluminate ions on the performance of the boehmite component B3 during caustic leach at temperatures lower than 100°C and to determine the effect of temperature on the dissolution rate of boehmite component B7.	NA	These results will be presented in a report that is yet to be released.
11) Perform leaching tests to determine the extent of boehmite conversion one would expect under leaching conditions during the planned testing in PEP.	NA	These results will be presented in a report that is yet to be released.

## Success Criteria

This work meets the sixth success criterion described in TP-RPP-WTP-509, Rev. 0 (Daniel and Shimskey 2007), which is listed in Table S.2.

**Table S.2.** Results and Performance against Success Criteria of TP-RPP-WTP-509, Rev. 0

<b>List Success Criteria</b>	<b>Explain How the Tests Did or Did Not Meet the Success Criteria</b>
1) Development of empirical information that allows the effect of initial aluminate ion concentration on the kinetics of boehmite leaching in a waste simulant to be determined.	This criterion is addressed in report WTP-RPT-184, Rev. 0.
2) Determination of the sensitivity of boehmite leaching to carbonate, free-hydroxide, nitrate, nitrite, oxalate, phosphate, and sulfate anions in a waste-simulant solution.	As of March 20, 2008, PNNL has been released from this objective by Test Exception, 24590-WTP-TEF-RT-07-00016.
3) Determination of the effect of scaling the length of the ultrafilter element from 2 ft to 8 ft on the performance of the filter over the expected process operating range in transmembrane pressure, axial velocity, and ultrafiltration temperature.	This criterion is addressed in report WTP-RPT-168, Rev. 0.
4) Determination of the effect of temperature on the filtration flux for the waste simulant over the range of solid concentrations and temperature conditions for the leaching processes.	This criterion is addressed in report WTP-RPT-168, Rev. 0.
5) Determination of the effect of fine-particle concentration on the propensity of the waste simulant to foul the ultrafilter element over the range of concentrations of operating solids in the waste simulant.	This criterion is addressed in report WTP-RPT-183, Rev. 0.
6) Determine the effect of longer term outdoor and indoor storage, prolonged mixing under ambient conditions, and the effect of temperature cycling on the characteristics of the vendor produced PEP	The results of these tests discussed in Section 2 showed that there was no significant effect of storage on the physical characteristics of the PEP simulant after the aging period under outdoor storage, indoor storage at

**Table S.2 (Contd)**

simulant (Test Exception 24590-WTP-TEF-RT-08-00013 Rev 0).	ambient conditions, prolonged mixing under ambient conditions, and temperature cycling from 70°F to 113°F. Rheology at 5 wt% UDS was measured; however, rheology was not measured at 20 wt% UDS due to the results obtained at 5 wt% UDS and the lack of resources which was a deviation from the Test Exception,
7) Verification of Cr-simulant produced both in the small batch (15 kg of Cr) and the production batch (57 kg of Cr) exhibit similar or better leaching behavior than the initial trial batch from 2007 (Test Exception 24590-WTP-TEF-RT-08-00013 Rev 0).	These results will be presented in a report that is yet to be released.
8) Determination of the mass loss and aluminum and chromium dissolution rates during caustic leaching under varying temperature-processing conditions without aeration in both UFP-1A/B and UFP-2A/B vessels as well as to measure the effect of aeration on chromium leaching in UFP-2A/B (Test Exception 24590-WTP-TEF-RT-08-00014 Rev 0).	These results will be presented in a report that is yet to be released.
9) Development of a rate equation for the dissolution of boehmite in concentrated caustic solutions containing aluminate ions approaching or exceeding the saturation solubility for boehmite (Test Exception 24590-WTP-TEF-RT-08-00015 Rev 0).	These results will be presented in a report that is yet to be released.
10) Verification of the effect of aluminate ion on the rate and the extent of dissolution of boehmite component B3 during caustic leach temperatures lower than 100°C (Test Exception 24590-WTP-TEF-RT-08-00015 Rev 0).	These results will be presented in a report that is yet to be released.
11) Determine the effect of temperature on the dissolution rate of boehmite component B7 (Test Exception 24590-WTP-TEF-RT-08-00015 Rev 0).	These results will be presented in a report that is yet to be released.
12) Determination of the extent of boehmite conversion one could expect under leaching conditions during the planned testing in PEP (Test Exception 24590-WTP-TEF-RT-08-00015 Rev 0).	These results will be presented in a report that is yet to be released.

## Test Exceptions

Four test exceptions were issued for Test Plan TP-RPP-WTP-509. These test exceptions are summarized in Table S.3 along with a brief description of how each exception impacted existing objectives and test plan scope.

**Table S.3.** Test Exceptions to Test Plan TP-RPP-WTP-509

Test Exception Number	Description of Test Exception
24590-WTP-TEF-RT-07-00016, Rev. 0	This test exception released PNNL from test objective 2 (see Table S.2).
24590-WTP-TEF-RT-08-00013, Rev. 0	This test exception did not affect any existing test plan objectives. It added test objectives concerned with 1) aging of the PEP simulants during storage and 2) leaching of the chromium simulant. These are objectives 6 and 7 in Table S.2. The results of objective 6 are reported here, and the results of objective 7 will be released at a later date.
24590-WTP-TEF-RT-08-00014, Rev. 0	This test exception both affected existing test objectives and added new test objectives. Tests associated with objective 4 were modified slightly in response to this test exception with the temperatures examined being changed slightly. The results of this testing are reported in WTP-RPT-168. In addition, a new objective concerned with the influence of temperature and aeration on caustic leaching processes was added. This is objective 8 in Table S.2, and the results will be released at a later date.
24590-WTP-TEF-RT-08-00015, Rev. 0	This test exception did not affect any existing objectives in the test plan. It added three new test objectives concerned with 1) in-depth assessment of the leaching kinetics with respect to dissolved aluminate concentration and 2) the extent of leaching under planned PEP operating conditions. The added objectives are 9, 10, and 11 in Table S.2, and the results will be released at a later date.

## Quality Requirements

Pacific Northwest National Laboratory (PNNL) is operated for the U.S. Department of Energy (DOE) by Battelle under Contract DE-AC05-76RL01830. PNNL implements a Quality Assurance Program that is based upon the requirements as defined in DOE Order 414.1C, “Quality Assurance,” and 10 CFR 830, “Energy/Nuclear Safety Management,” Subpart A—“Quality Assurance Requirements.” PNNL has chosen to implement the requirements of DOE Order 414.1C and 10 CFR 830, Subpart A by integrating them into the laboratory’s management systems and daily operating processes. The procedures necessary to implement the requirements are documented through the laboratory’s Standards-Based Management System (SBMS).

PNNL implemented the RPP-WTP quality requirements by performing work in accordance with the River Protection Project – Waste Treatment Plant Support Program (RPP-WTP) Quality Assurance Plan (RPP-WTP-QA-001, QAP). Work was performed to the quality requirements of NQA-1-1989 Part I, *Basic and Supplementary Requirements*, NQA-2a-1990, Part 2.7, and DOE/RW-0333P, Rev 13, *Quality Assurance Requirements and Descriptions (QARD)*. These quality requirements were implemented

through the River Protection Project–Waste Treatment Plant Support Program (RPP-WTP) Quality Assurance Manual (RPP-WTP-QA-003, QAM). All analytical services were provided by Southwest Research Institute (SWRI). The requirements of DOE/RW-0333P, Rev 13, *Quality Assurance Requirements and Descriptions (QARD)*, were not required for this work.

A matrix that cross-references the NQA-1 and NQA-2a requirements with RPP-WTP’s procedures for this work is given in TP-RPP-WTP-509. It includes justification for those requirements not implemented. Experiments that were not method-specific were performed in accordance with RPP-WTP’s procedures QA-RPP-WTP-1101 “Scientific Investigations” and QA-RPP-WTP-1201 “Calibration and Control of Measuring and Testing Equipment” so that sufficient data were taken with properly calibrated measuring and test equipment to obtain quality results.

RPP-WTP addressed internal verification and validation activities by conducting an independent technical review of the final data report in accordance with PNNL’s procedure QA-RPP-WTP-604. This review verifies that the reported results were traceable, inferences and conclusions were soundly based, and the reported work satisfied the Test Plan objectives. This review procedure is part of PNNL’s RPP-WTP QAM.

## R&T Test Conditions

The research and technology (R&T) test conditions, as defined in the Bechtel National Inc. (BNI) Test Specification 24590-WTP-TSP-RT-07-004, Rev. 0<sup>(a)</sup> associated with the test plan TP-RPP-WTP-509, Rev. 0 are summarized in Table S.4.

**Table S.4.** R&T Test Conditions from 24590-WTP-TSP-RT-07-004, Rev. 0

<b>List R&amp;T Test Conditions</b>	<b>Were Test Conditions Followed?</b>
1) Boehmite Dissolution Tests—examine the impact of aluminate, hydroxide, and other principal anions on boehmite dissolution kinetics. (Section 5.1 of TP-RPP-WTP-509, Rev. 0)	Not applicable to this report. Results discussed in WTP-RPT-184.
2) Boehmite Dissolution Tests—verify the effect of temperature on the dissolution of boehmite component B7 and verify the effect of aluminate ion on the performance of the boehmite component B3 during caustic leach at temperatures lower than 100°C. (Section 5.5 of TP-RPP-WTP-509, Rev. 0)	Not applicable to this report. It will be addressed in a future report.
3) Boehmite Dissolution Tests—provide greater discrimination on anion impact by performing tests under a greater range of anion concentrations. (Section 5.1 of TP-RPP-WTP-509, Rev. 0)	Not applicable to current testing. PNNL was released from this requirement by Test Exception 24590-WTP-TEF-RT-07-00016.
4) Filtration Tests—test a base simulant under identical process conditions with 2-ft and 8-ft filter elements. (Section 5.2 of TP-RPP-WTP-509, Rev. 0)	Not applicable to this report. Results discussed in WTP-RPT-168.

(a) PS Sundar. April 2007. *Simulant Testing in Support of Phase I Demonstration of the Ultrafiltration and Leaching Processes in the Integrated Test Facility*. 24590-WTP-TSP-RT-07-004, Rev. 0.

**Table S.4 (Contd)**

<b>List R&amp;T Test Conditions</b>	<b>Were Test Conditions Followed?</b>
5) Filtration Tests—increase the fines loading in filtration test base simulant to evaluate the impact of fouling on filtration performance. (Section 5.2 of TP-RPP-WTP-509, Rev. 0)	Not applicable to this report. Results discussed in WTP-RPT-183.
6) Filtration Tests—use an 8-ft filter element to measure the filtration rate as a function of temperature up to 45°C for the base filtration simulant. (Section 5.2 of TP-RPP-WTP-509, Rev. 0)	Not applicable to this report. Results discussed in WTP-RPT-168.
7) Aging Tests—will be performed in the 250-gal tote and a 1-gal container in the laboratory, a container in a heat-cycled oven, and a baffled 1-gal container that is mixed in the laboratory. Samples will be taken throughout the tests and characterized by particle-size distribution, settling, rheology, and centrifuged solids content to evaluate the effect of aging on the behavior of the simulant. (Section 5.3 of TP-RPP-WTP-509, Rev. 0)	Aging tests were performed in the 250-gal tote by storing it outside and in 1-gallon containers in the laboratory. One container was temperature cycled in an oven, one container was stored on the counter, and another was put into a baffled container and stirred continuously. Samples were taken throughout the tests and characterized by PSD, rheology at 5 wt% UDS (rheology was not measured at 20 wt% UDS due to the results obtained at 5 wt% UDS and the lack of resources), settling, and centrifuging of solids. It was found that there was no significant effect of aging on the physical characteristics of the simulant under the conditions tested in this time frame.
8) Chromium Simulant Leaching Tests—will be performed with both a caustic leach and an oxidative leach to evaluate the leaching performance of the various vendor batches of Cr-simulant. (Section 5.4 of TP-RPP-WTP-509, Rev. 0)	Not applicable to this report. It will be addressed in a future report.
9) PEP Leaching Support Tests—are to be carried out with the vendor-produced 250-gal batch of the PEP simulant and the vendor-produced CrOOH Test Batch 1 simulant slurry. The tests are directed to determine the mass loss and aluminum and chromium dissolution rates during caustic leaching under varying temperature processing conditions without aeration in both UFP-1A/B and UFP-2A/B vessels as well as to measure the effect of aeration on chromium leaching in UFP-2A/B. (Section 5.5 of TP-RPP-WTP-509, Rev. 0)	Not applicable to this report. It will be addressed in a future report.
10) PEP Leaching Support Tests—will be performed using a vendor-produced 250-gal batch of the PEP simulant. The tests are directed to measure the extent of boehmite conversion expected under leaching conditions during the planned testing in the PEP. (Section 5.5 of TP-RPP-WTP-509, Rev. 0)	Not applicable to this report. It will be addressed in a future report.

## **Simulant Use**

The simulant used for the aging tests described in this report was produced by simulant vendor Noah Technologies based on a recipe provided to them that was developed by PNNL. The recipe was based on specifications for the demonstration of the ultrafiltration and leaching processes in the PEP. The simulant does not mimic any actual tank waste or even any composite waste.

## **Discrepancies and Follow-on Tests**

None.

# 1.0 Introduction

The Hanford Tank Waste Treatment and Immobilization Plant (WTP) is procuring 3,500-gallon batches of waste simulant for Phase 1 testing in the Pretreatment Engineering Platform (PEP) through Pacific Northwest National Laboratory (PNNL). To make sure that the quality of the simulant is acceptable, the production method was scaled up starting from a laboratory-prepared 25-liter batch of simulant through 15-gallon vendor prepared simulant and 250-gallon vendor prepared simulant before embarking on the production of the 3500-gallon simulant batch by the vendor.

The 3,500-gallon PEP simulant batches were produced and packaged in 250-gallon high-molecular-weight polyethylene totes at NOAH Technologies. The simulant was stored at NOAH Technologies within their warehouse at about 70 to 75°F before blending or shipping. For the 15-gallon, the 250-gallon, and the 3,500-gallon Batch 0, the simulant was shipped in ambient temperature trucks with shipments requiring nominally 3 days. The 3,500-gallon Batch 1 traveled in a 70 to 75°F temperature-controlled truck. Typically, the simulant was uploaded into a PEP receiving tank within 24 hours of receipt. The first uploading required longer with the simulant stored outside from receipt until the transfer.

The stability of the physical and chemical characteristics of the simulant both in transit and in storage before its use in the PEP was paramount to accomplishing the process demonstration objectives in the PEP. Therefore, aging tests were conducted on the 250-gallon batch of the vendor-produced PEP blended feed simulant to identify and determine any changes to the physical characteristics of the simulant when in storage. The supernate was also chemically characterized. Four aging scenarios for the vendor-produced blended simulant were studied: 1) stored outside in a 250-gallon tote, 2) stored inside in a 1-gallon plastic bottle, 3) stored inside in a well mixed 5-L tank, and 4) subject to extended temperature cycling under summer temperature conditions in a 1-gallon plastic bottle. In particular, the following series of aging tests were conducted to accomplish these objectives.

1. The simulant was stored in the 250-gallon tote outside in a shaded area for 45 days. The maximum temperature was 108°F, and the minimum temperature was 43°F with an average of 72°F. The average high temperature was 88°F, and the average low temperature was 57°F. The supernatant composition was analyzed chemically every 7 days. The capability to re-suspend the solids in the simulant at the end of 45 days was also tested. Note that the tote was left undisturbed during the test duration, allowing extensive settling to occur. The tote was mixed using the method prescribed for PEP operational procedures using an air sparge and pumping out of the top of the tote and into the bottom of the same tote. This was performed at the beginning of the testing (0 days) and at the end of the 45 days with a homogenous slurry sample withdrawn for characterization.
2. About 1 gallon of simulant was stored in a plastic bottle in the laboratory (without continuous mixing) for 60 days at ambient temperature (64°F to 70°F). The simulant was allowed to settle between sampling. The simulant was well shaken to suspend the settled solids before sampling, and then an aliquot of the slurry was taken and physically characterized at 15, 30, and 60 days.

3. About 1 gallon of simulant was well mixed under turbulent conditions in a plastic, baffled container using a marine impeller-type stirrer in the laboratory for 30 days at ambient temperature (64°F to 70°F). It was physically characterized at 15 and 30 days.
4. Temperature cycling was performed on approximately 1 gallon of simulant in a 1-gallon plastic bottle in an oven. It was heated and held at 45°C (113°F) during the day (8 to 10 hours) and then allowed to cool and stand at ambient temperature overnight and on weekends and holidays. The sample was cycled for 45 calendar days (29 temperature cycles) and physically characterized at 7, 15, 30, and 45 days.

The sample characterizations performed for the above tests were:

- Particle size distribution (PSD)
- Solids-settling tests over a period of 72 hours to determine settled solids volume as a function of time
- Rheology determination—flow curves at 5 wt% (as received)
- Measurement of centrifuged solids content as a gel-point indicator. The slurry was centrifuged for 30 minutes at 4500 G.
- Measurement of supernatant composition of the 250-gallon tote every 7 days.

Based on the results obtained from the rheology measurements at 5 wt% undissolved solids and the lack of resources, it was decided not to perform the rheology curves at 20 wt% undissolved solids as stated in the Test Exception. It was also determined from previous work that centrifuging at 1,000 G does not separate the simulant efficiently or show the true gel point. Therefore, it was decided to centrifuge the samples at 4,500 G to obtain an efficient separation and accurate gel point of the simulants.

## 2.0 Aging Results

The various aging slurry samples were characterized in several ways as described below.

### 2.1 Particle-Size Distribution

PSD measurements were conducted on 11 samples collected during the aging experiments. The samples were analyzed with a S3000 Microtrac Analyzer according to procedure TPR-RPP-WTP-222, Rev. 3. Nominally 0.2 grams of each sample (run in duplicate) were placed into approximately 10 grams of de-ionized water (DIW), and 2 to 3 drops of Darvan<sup>®</sup> 821A Dispersing Agent were added. A transfer pipette was used to mix the slurry and transfer the required amount to the analyzer. The amount of the slurried sample aliquot required for analysis varied with the actual mass of the sample and the actual volume of DIW in the sample. Neither the amount of sample nor the amount of water was critical to the analytical process since the internal system software visually indicated to the analyst the amount needed for analysis. No sonication was performed with these samples. The 2 micron standard used in verifying the calibration of the instrument gave an error range of  $\pm 0.5$  microns. The 11 samples collected and analyzed are shown in Table 2.1.

**Table 2.1.** PSD Samples Collected During the Aging Experiment

<b>Aging Scenario</b>	<b>Sampling Time</b>
Stored Outside	0 days and 45 days
Well Stirred (Inside)	15 days and 30 days
Stored Inside	15 days, 30 days, and 60 days
Temperature Cycled	7 days, 15 days, 30 days, and 45 days

The PSD results are shown in Table 2.2 in terms of volume percent distribution and in Table 2.3 in terms of number percent distribution.

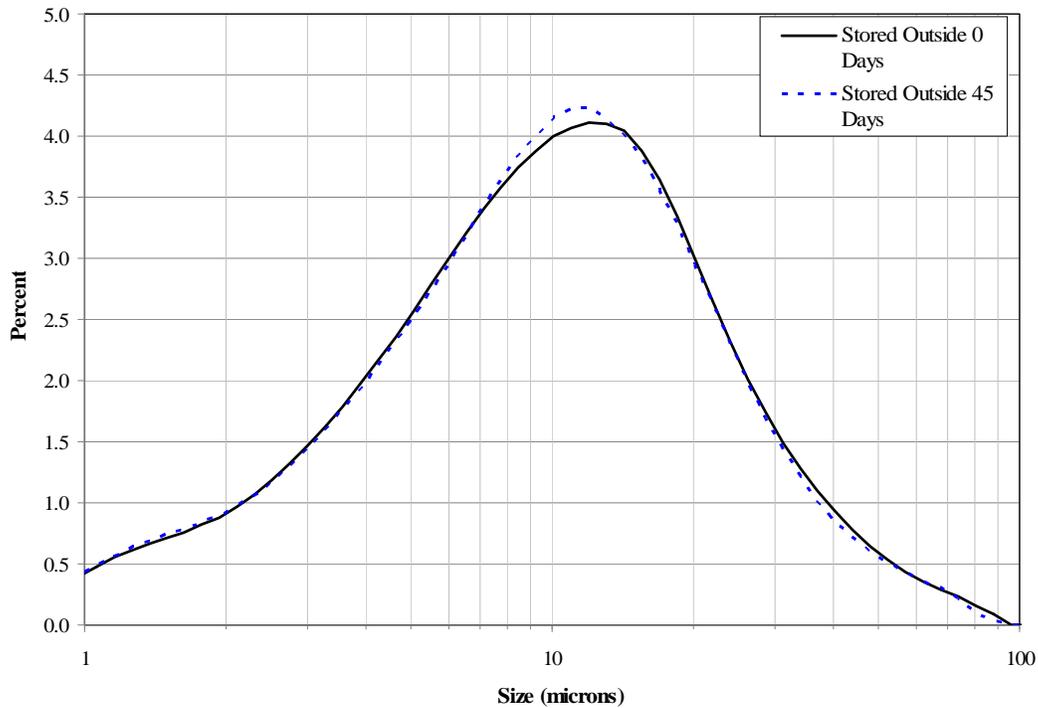
**Table 2.2.** Aging Test PSD Volume Percent Distribution (without sonication)

<b>Sample ID</b>	<b>5% (microns)</b>	<b>50% (microns)</b>	<b>90% (microns)</b>
Initial Feed	1.64	9.50	26.22
Stored Outside 45 days	1.62	9.43	25.63
Temp. Cycling 7 days	1.61	9.20	24.38
Temp. Cycling 15 days	1.22	8.67	23.00
Temp. Cycling 30 days	1.69	8.82	21.08
Temp. Cycling 45 days	1.67	8.98	22.77
Stored Inside 15 days	1.52	9.31	25.21
Stored Inside 30 days	1.61	8.78	20.57
Stored Inside 60 days	1.59	9.08	24.70
Well-Stirred 15 days	1.51	8.70	22.77
Well-Stirred 30 days	1.67	8.99	22.80

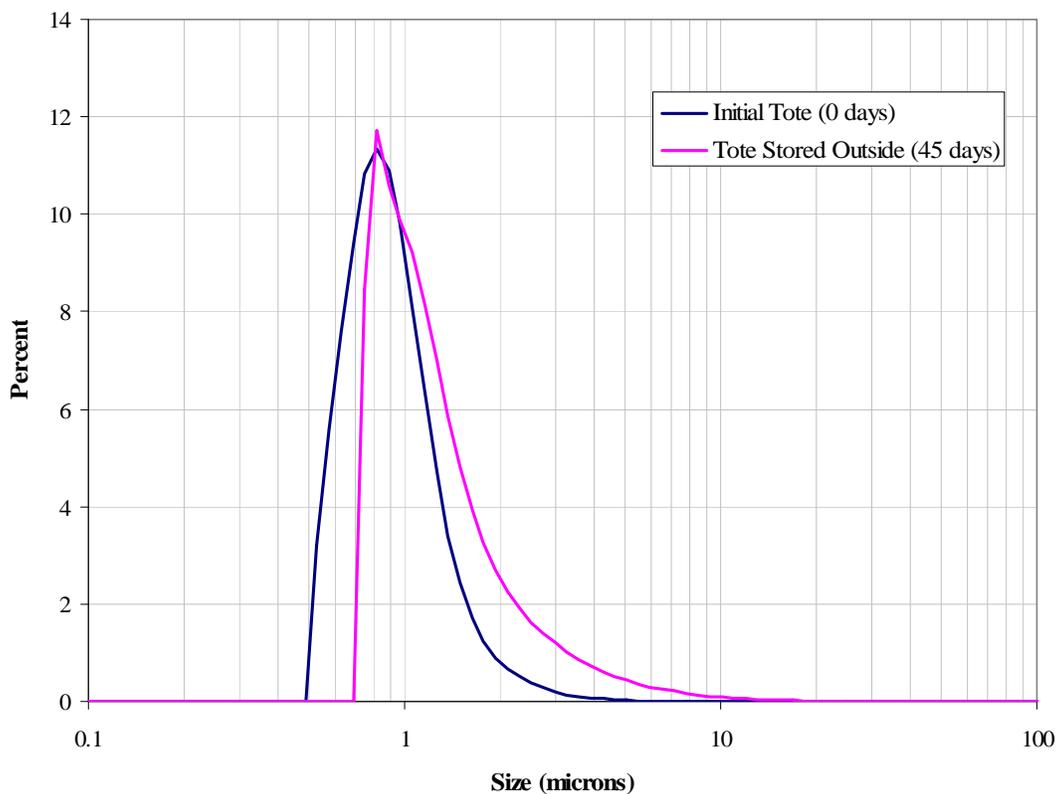
**Table 2.3.** Aging Test PSD Number Percent Distribution (without sonication)

Sample ID	5% (microns)	50% (microns)	90% (microns)
Initial Feed	0.73	1.06	2.38
Stored Outside 45 days	0.73	1.06	2.35
Temp. Cycling 7 days	0.73	1.07	2.38
Temp. Cycling 15 days	0.62	0.90	1.74
Temp. Cycling 30 days	0.73	1.09	2.52
Temp. Cycling 45 days	0.73	1.07	2.47
Stored Inside 15 days	0.73	1.05	2.24
Stored Inside 30 days	0.73	1.08	2.43
Stored Inside 60 days	0.73	1.08	2.39
Well-Stirred 15 days	0.73	1.08	2.32
Well-Stirred 30 days	0.73	1.08	2.49

The PSD result for the sample that was stored outside for 45 days is compared to the initial simulant sample (0 days in Table 2.1) in Figure 2.1 as volume percent and in Figure 2.2 as number percent. These results indicate that there was no discernable change in the average particle size during the 45 days of storage outside. There may have been some slight precipitate ripening based on the number distribution results but it is not definitive based on these results and therefore, it appears that storing the simulant in the tote outside undisturbed did not cause a definitive change in the average particle size over 45 days.

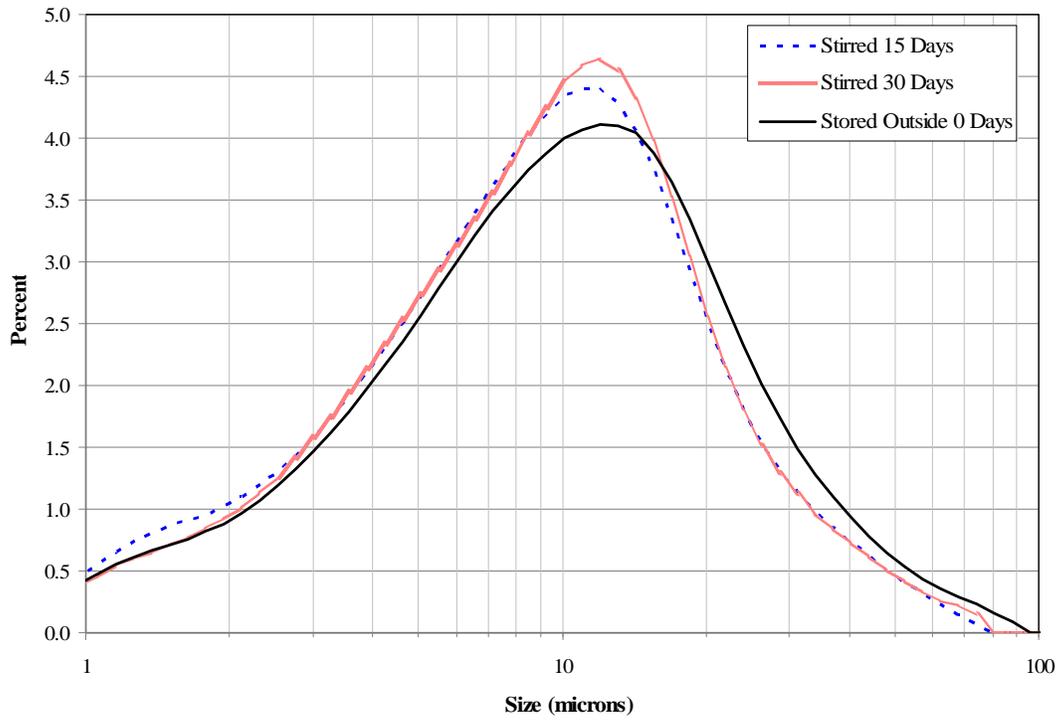


**Figure 2.1.** PSD (Volume Percent) for the Stored-Outside Samples

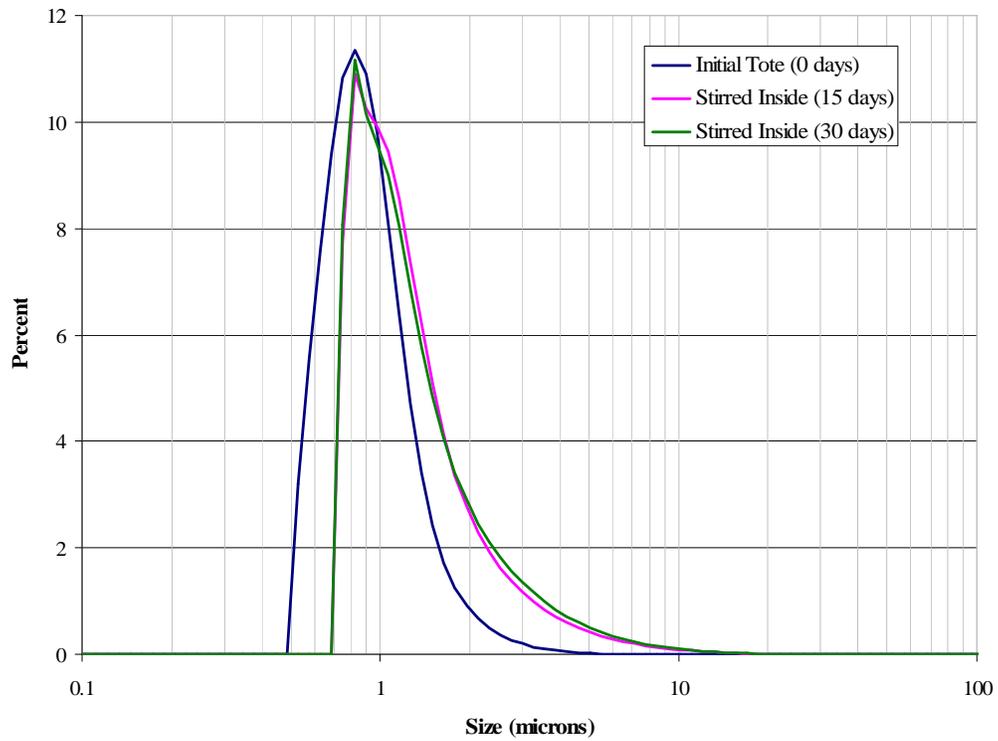


**Figure 2.2.** PSD (Number Percent) for the Stored-Outside Samples

The PSD of the two well-stirred samples and the initial simulant sample (0 days in Table 2.1) are compared in Figure 2.3 as volume percent distribution and in Figure 2.4 as number size distribution. These results do not show any definitive differences in the average particle size, indicating that stirring the simulant over 30 days did not change the particle size. There may have been some slight precipitate ripening based on the number distribution results but it is not definitive based on these results and no definitive conclusion can be drawn.

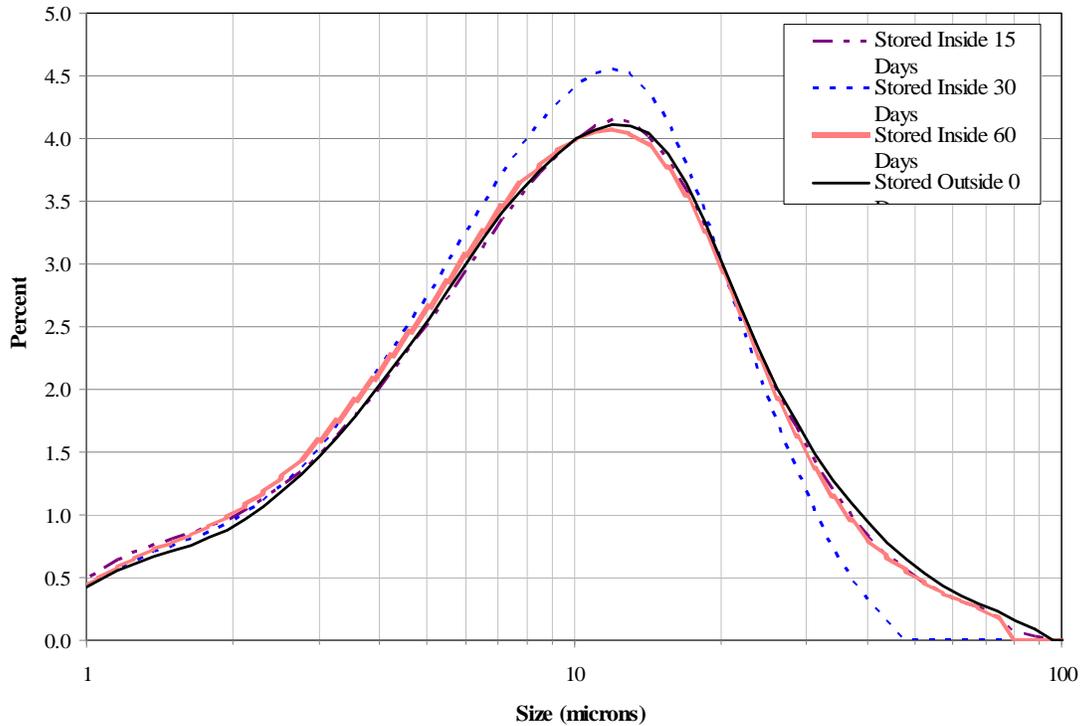


**Figure 2.3.** PSD (Volume Percent) for the Two Well Stirred Samples

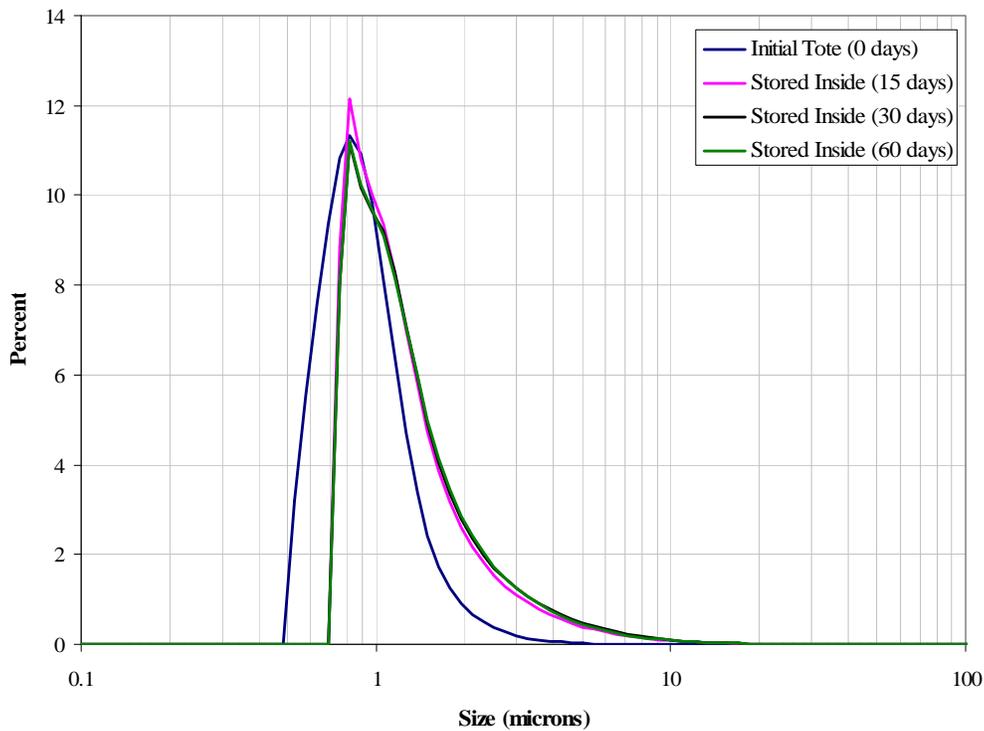


**Figure 2.4.** PSD (Number Percent) for the Two Well Stirred Samples

The PSD results for the samples stored inside are compared with the initial simulant sample (0 days in Table 2.1) in Figure 2.5 as volume percent distribution and Figure 2.6 as number percent distribution. The 15-day and 60-day volume distribution samples are nearly identical to the initial sample, indicating that storage at ambient conditions does not affect the average particle size of the simulant. There may have been some slight precipitate ripening based on the number distribution results but it is not definitive based on these results and no definitive conclusion can be drawn. The sample collected after 30 days of storage exhibits a difference in the volume percent distribution from the other samples. This result is not consistent with the other results, and the reason for this disparity has not yet been identified.

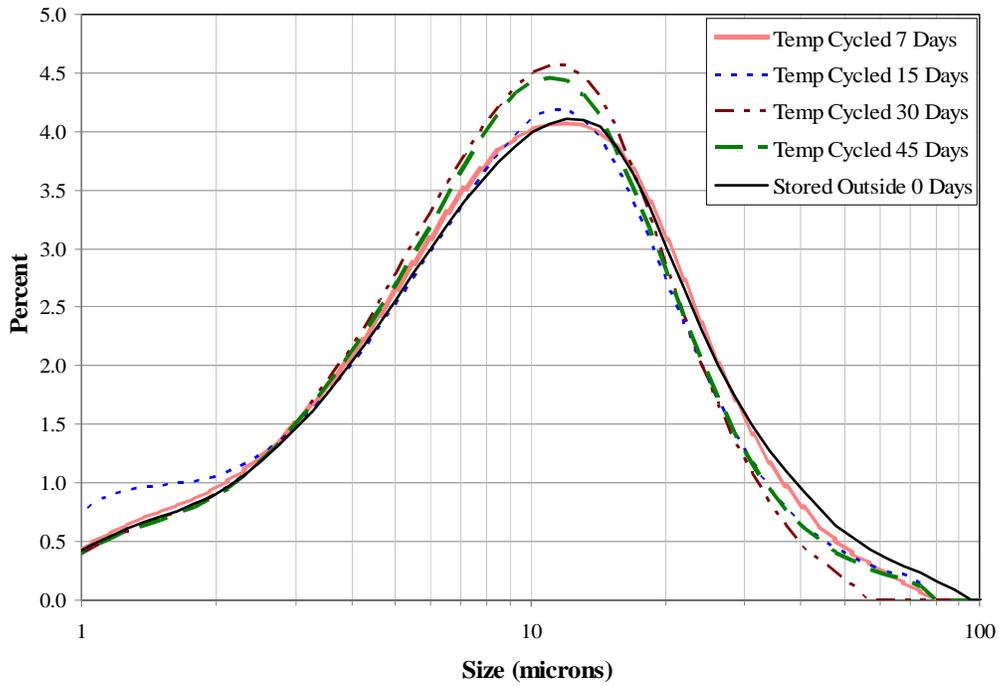


**Figure 2.5.** PSD (Volume Percent) for the Stored Inside Samples

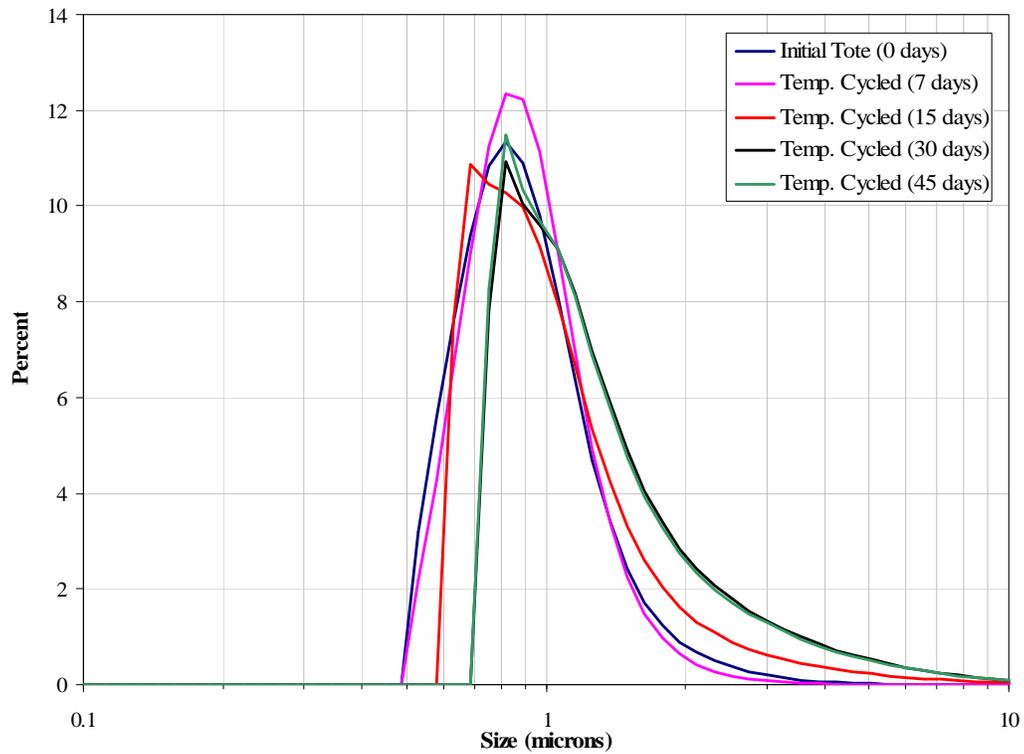


**Figure 2.6.** PSD (Number Percent) for the Stored Inside Samples

Results of the PSD analysis for the temperature-cycled samples are compared with the initial simulant sample (0 days in Table 2.1) in Figure 2.7 for the volume percent distribution and Figure 2.8 for the number percent distribution. All of the temperature-cycled samples except the 15-day sample are nearly identical to the initial volume distribution sample, indicating that temperature cycling did not significantly affect the average particle size. The 15-day temperature-cycled sample has an anomalous fractional increase of particles in the size range of 1  $\mu\text{m}$  to 2  $\mu\text{m}$ , and the reason for this is not known at this time. There may have been some slight precipitate ripening based on the number distribution results but it is not definitive based on these results and no definitive conclusion can be drawn.

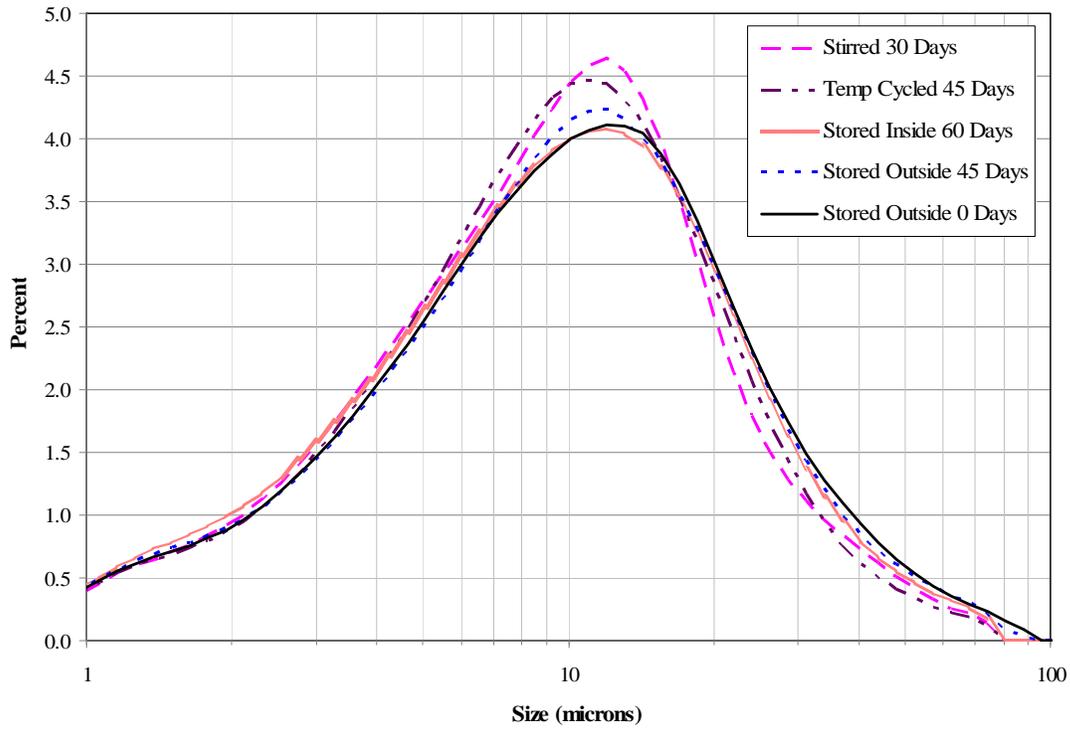


**Figure 2.7.** PSD (Volume Percent) for the Temperature Cycled Samples

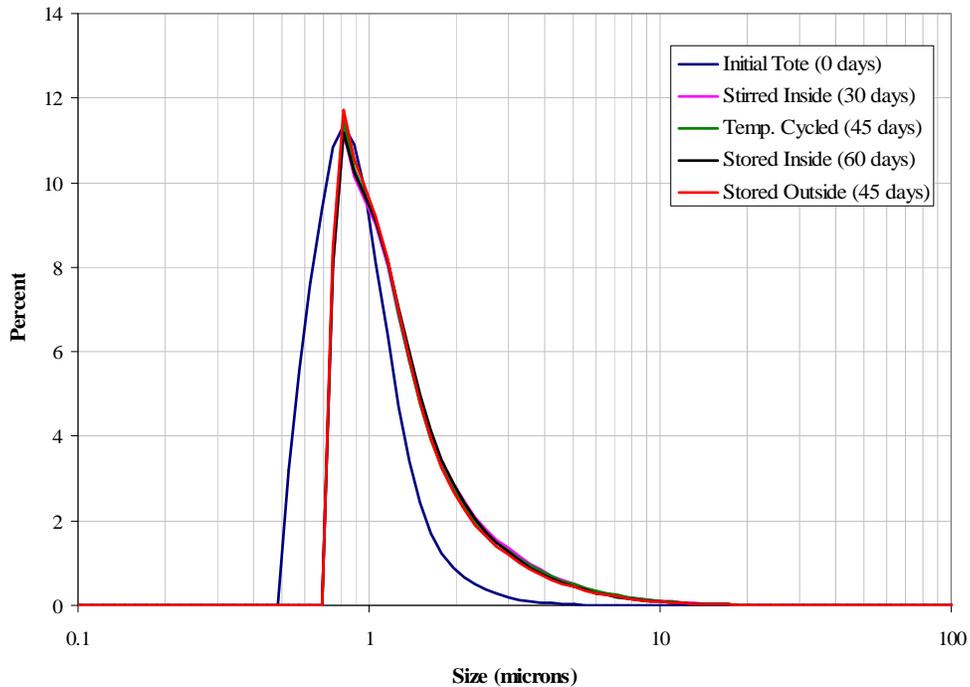


**Figure 2.8.** PSD (Number Percent) for the Temperature Cycled Samples

The PSD analysis results for the longest time period for each aging scenario are compared with that of the initial simulant sample (0 days in Table 2.1) in Figure 2.9 as volume distribution and Figure 2.10 as number distribution. All of the curves showed the same pattern with no significant differences indicating that aging method appears to have little effect on the steady state ~~PSD~~ PSD. This indicates that storing, stirring, or temperature cycling the simulant should not affect the average particle size of the simulant.



**Figure 2.9.** PSD (Volume Percent) for the Last Sample Collected for Each Aging Scenario

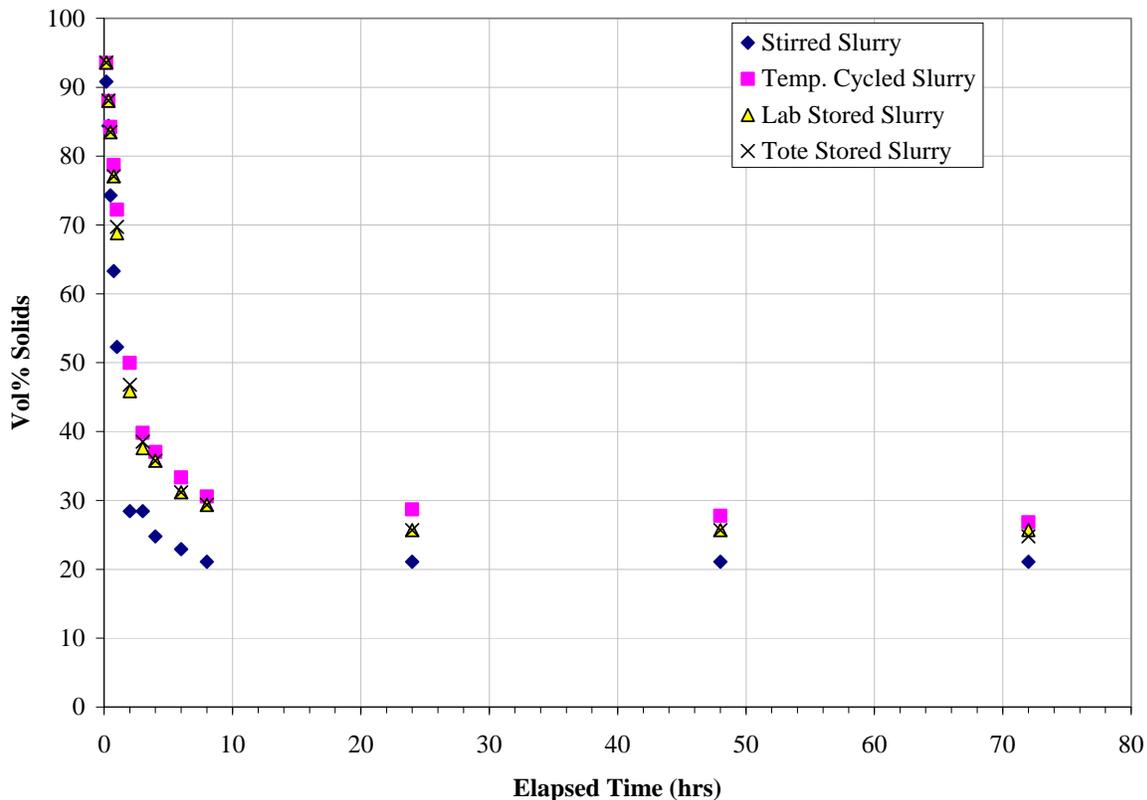


**Figure 2.10.** PSD (Number Percent) for the Last Sample Collected for Each Aging Scenario

## 2.2 Settling Tests

These tests were performed by placing a homogenous sample of the slurry in a 50-mL centrifuge tube and then measuring the height of the solids in the tube in terms of inches over time. Results of these settling tests are shown in Figure 2.11. The volume percent of solids is plotted as a function of time. The highest degree of settling occurred in the stirred simulant sample. The temperature-cycled sample and the two samples that were stored inside and outside were nearly identical in their settling. The stirred sample was essentially finished settling after 8 hours, and the stored samples did not finish settling until ~24 hours whereas the temperature-cycled sample was still settling at the end of the 72-hour test. They all settled to between 20 vol% and 30 vol% solids after 72 hours. However, the continuously stirred sample resulted in the most significant simulant change settling to a solids volume of ~21% in 8 hours while the others took 24 hours or longer to settle to a solids volume of ~26%. These were not compared to the settling rate of the initial slurry because its settling rate was not measured.

The 250-gallon tote contents were mixed by air sparging and diaphragm pumping for approximately 30 minutes, and then a 2-liter sample was taken mid-stream during recirculation. The density of the slurry was then measured to make sure that homogenization was complete. After settling for 45 days in the tote outside, the slurry was mixed again with the air sparger and diaphragm pump in the exact same way as initially to determine how well the solids were re-suspended. The solids were easily mixed and re-suspended with no difference from the initial mixing. The initial density was measured as 1.262 g/mL, and the final density was measured as 1.257 g/mL, indicating the same degree of homogenization.



**Figure 2.11.** Volume-Percent Solids as a Function of Time

## 2.3 Rheology

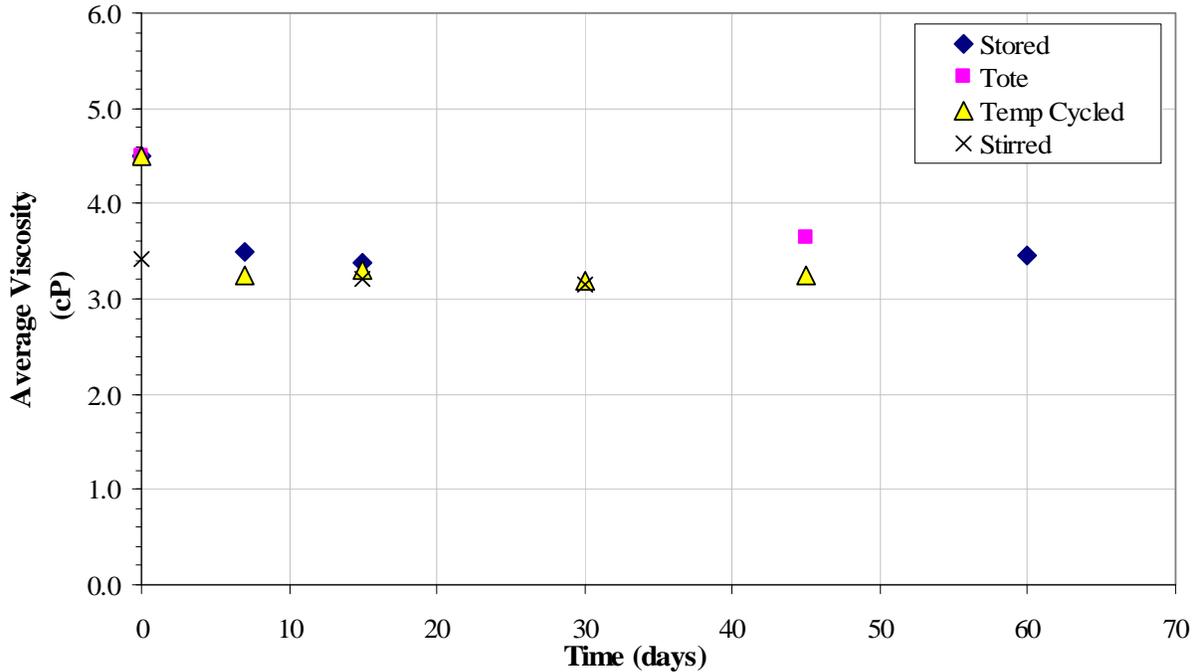
Rheology data were obtained on an Anton Paar Physica MCR 301 rheometer (Anton Paar Ostfildern Germany) with a Peltier-controlled cylinder measuring system where both speed and torque are measured at the rotating shaft. The rheometer was operated in the controlled-rate mode. This geometry required approximately 37 mL of sample and had a gap of 3.46 mm. The samples were shaken before being measured to produce a fully suspended material. Shear stress was measured as a function of shear rate over a range of 0 to 1000  $s^{-1}$ . Measurements were made with both increasing and decreasing shear rate. Rheometer control and data acquisition were accomplished with a computer connection using the RheoPlus Software, Version 3.21.

The results of the rheology tests are presented in Table 2.4 and Figure 2.12. The flow curves are shown in Appendix B. All the aging study samples exhibited Newtonian flow behavior. The overall average simulant viscosity was 3.43 cP with a standard deviation of 0.37 cP. All of the measurements were within this range except for the initial slurry, which indicates that the slurry may have become slightly less viscous by <1cP over this time frame. This indicates that aging did not have a significant effect on the rheology of the PEP simulant under these various conditions, and its flow behavior remained essentially the same.

**Table 2.4.** Rheology Results for the Aging Test Samples

Days	Stored Outside	Stored Inside	Temperature Cycled	Well-Stirred
	Viscosity cP			
0	4.50	No sample <sup>(a)</sup>	No sample <sup>(a)</sup>	3.41
7	No sample	No sample	3.24	No sample
15	No sample	3.49	3.30	3.21
30	No sample	3.38	3.19	3.15
45	3.64	No sample	3.24	No sample
60	No sample	3.45	No sample	No sample

(a) The initial value may be assumed to be 4.50 cP since this was a well mixed sample withdrawn from the tote at the beginning of the testing.



**Figure 2.12.** Average Viscosity as a Function of Time

## 2.4 Solids Content

The solids content for the 45-day temperature-cycled sample and the 45-day stored-outside sample are shown in Table 2.5. The total solids content and the dissolved solids content are essentially the same for both samples with a difference of approximately 1.02 wt% and 0.58 wt%, respectively, which is well within analytical uncertainty of  $\pm 15\%$  variance. The undissolved solids content shows a difference of 0.44 wt%.

The samples were also centrifuged at 4500 G for 30 minutes to determine the solids content at this point. This point represents the gel point of the slurry in which the column height is self-supporting at whatever G force is applied. The results of the tests for centrifuged solids content are shown in Table 2.6. The initial slurry was not centrifuged and measured. The temperature-cycled slurry had slightly higher vol% solids than the other samples. The other samples were all about the same, indicating that they all centrifuge to essentially the same amount, and different aging scenarios do not alter the final results significantly.

**Table 2.5.** Percent Total, Dissolved, and Undissolved Solids Content

<b>Sample</b>	<b>% Total Solids</b>	<b>% Dissolved Solids</b>	<b>% Undissolved Solids</b>
Stored Outside 45 days	31.3	27.0	4.3
Temperature Cycled 45 days	30.3	26.4	3.9

**Table 2.6.** Centrifuged Solids Content

<b>Aging Scenario</b>	<b>Approximate Percent Centrifuged Solids</b>
Stored Outside	10.0
Well Stirred (Inside)	10.0
Stored Inside	11.3
Temperature Cycled	12.5

## 2.5 Supernatant Composition

Supernatant samples were collected at 14, 21, 28, 35, 42, and 45 days from the tote that was stored outside. The samples were analyzed for metals by inductively coupled plasma-atomic emission spectroscopy (ICP-AES) and anions by ion chromatography (IC). Supernatant samples were also collected at 15, 22, and 30 days from the well-stirred aging test. These samples were analyzed for Al, Cr, Fe, and Na by ICP-AES.

Analytical results for the supernatant samples collected from the stored-outside aging-scenario test for components that had concentrations above their respective detection limits are compiled in Table 2.7, and the entire analytical results are shown in Appendix C. The composition is essentially the same with all results well within the range of experimental error ( $\pm 15\%$ ) with no significant trends occurring for any component between days 14 and 45. Analytical results for the supernatant samples collected from the well-stirred aging-scenario test for Al, Cr, Fe, and Na are compiled in Table 2.8. The results for these components are very similar to those of the outside aging scenario with no aging trends apparent and all results well within the range of experimental error ( $\pm 15\%$ ). Both of these sets of results indicate that the supernatant reached equilibrium by the time the first sample was collected, and aging did not affect it.

**Table 2.7.** Supernatant Composition Results for Stored-Outside Aging Scenario (mg/kg solution)

Component	Sample ID					
	TST-14	TST-21	TST-28	TST-35	TST-42	TST-45
Al	2,820	2,930	3,030	2,960	2,900	2,820
B	4.96	5.15	5.24	5.18	5.17	4.98
Cr	0.979	1.03	1.09	1.03	1.11	1.02
Fe	<1.04	<0.860	1.09	<0.865	2.40	<1.11
P	1,640	1,740	1,790	1,760	1,730	1,680
K	643	693	695	693	675	651
Si	1.69	1.51	1.76	1.57	2.01	1.59
Na	85,100	81,200	87,000	88,400	89,400	85,600
S	4,360	4,570	4,700	4,560	4,570	4,410
Zr	0.786	0.811	0.882	0.804	0.932	0.813
Nitrate-N	19,400	19,600	19,800	19,900	18,900	19,200
Nitrite-N	5,690	5,750	5,790	5,830	5,590	5,660
Oxalate	701	764	751	733	727	725
Phosphate-P	1,860	1,890	1,880	1,880	1,810	1,830
Sulfate	15,600	15,700	15,800	15,900	15,400	15,600

**Table 2.8.** Supernatant Composition Results for Stirred Aging Scenario (mg/kg solution)

Component	Sample ID		
	SAT-15	SAT-22	SAT-30
Al	2,780	2,910	2,930
Cr	0.965	1.03	1.24
Fe	<5.40	<10.5	<8.03
Na	83,300	85,600	85,000

## 3.0 Conclusions

These tests indicate that there are only minor changes in the characteristics of the simulant with aging under the time and conditions tested as shown in Table 3.1.

These results indicate that storing, stirring, or temperature cycling the simulant should not affect the behavior of the simulant over the time frame tested.

The stirred sample was essentially finished settling after 8 hours, and the stored samples finished settling at ~24 hours whereas the temperature-cycled sample continued to settle throughout the test until 72 hours. They all settled to between 20 vol% and 30 vol% solids after 72 hours. However, the continuously stirred sample resulted in the most significant simulant change, settling to a solids volume of ~21% in 8 hours while the others took 24 hours or longer to settle to a solids volume of ~26%. After settling for 45 days in the tote outside, the solids were easily mixed and re-suspended with no difference in the effort required to re-suspend the solids in the tote at the beginning of the test.

All the aging study samples exhibited Newtonian flow behavior. The overall average simulant viscosity was 3.43 cP with a standard deviation of 0.37 cP. All of the measurements were within this range except for the initial slurry, which indicates that the slurry may have become slightly less viscous by <1cP over this time frame. This indicates that aging did not have a significant effect on the rheology of the PEP simulant under these various conditions, and its flow behavior remained essentially the same.

Measurement of solids content of the simulants did not allow one to discern the effect of the aging conditions on the initial simulant slurry. This is not considered a sufficiently accurate metric for determining aging effects.

The supernatant composition is essentially the same with no significant trends occurring for any component between days 14 and 45. These results indicate that the supernatant reached equilibrium by the time the first sample was collected, and aging did not affect it for both the stored-outside and the stirred aging scenarios.

**Table 3.1.** Summary Table of Aging Effects

<b>Aging Test</b>	<b>Volume Dist. PSD Change at 50%</b>	<b>Number Dist. PSD Change at 50%</b>	<b>Rheological Change</b>	<b>Chemical Change</b>
Stored Outside	-0.07 microns	0.00 microns	-0.86 cP	Al-0 mg/kg Cr-0.041 mg/kg Na-500 mg/kg
Stirred 30 Days (Inside)	-0.51 microns	+0.02 microns	-0.26 cP	Al-150 mg/kg Cr-0.275 mg/kg Na-1,700 mg/kg
Stored Inside 60 Days	-0.42 microns	+0.02 microns	-1.05 cP	Not Measured
Temperature Cycled 29 Times Over 45 Days	-0.52 microns	+0.01 microns	-1.26 cP	Not Measured

## 4.0 References

Daniel RC, and RW Shimskey. 2007. "Test Plan for Simulant Testing in Support of Phase I Demonstration of the Ultrafiltration and Leaching Processes in the Integrated Test Facility." TP-RPP-WTP-509, Rev. 0. Pacific Northwest National Laboratory, Richland, Washington.

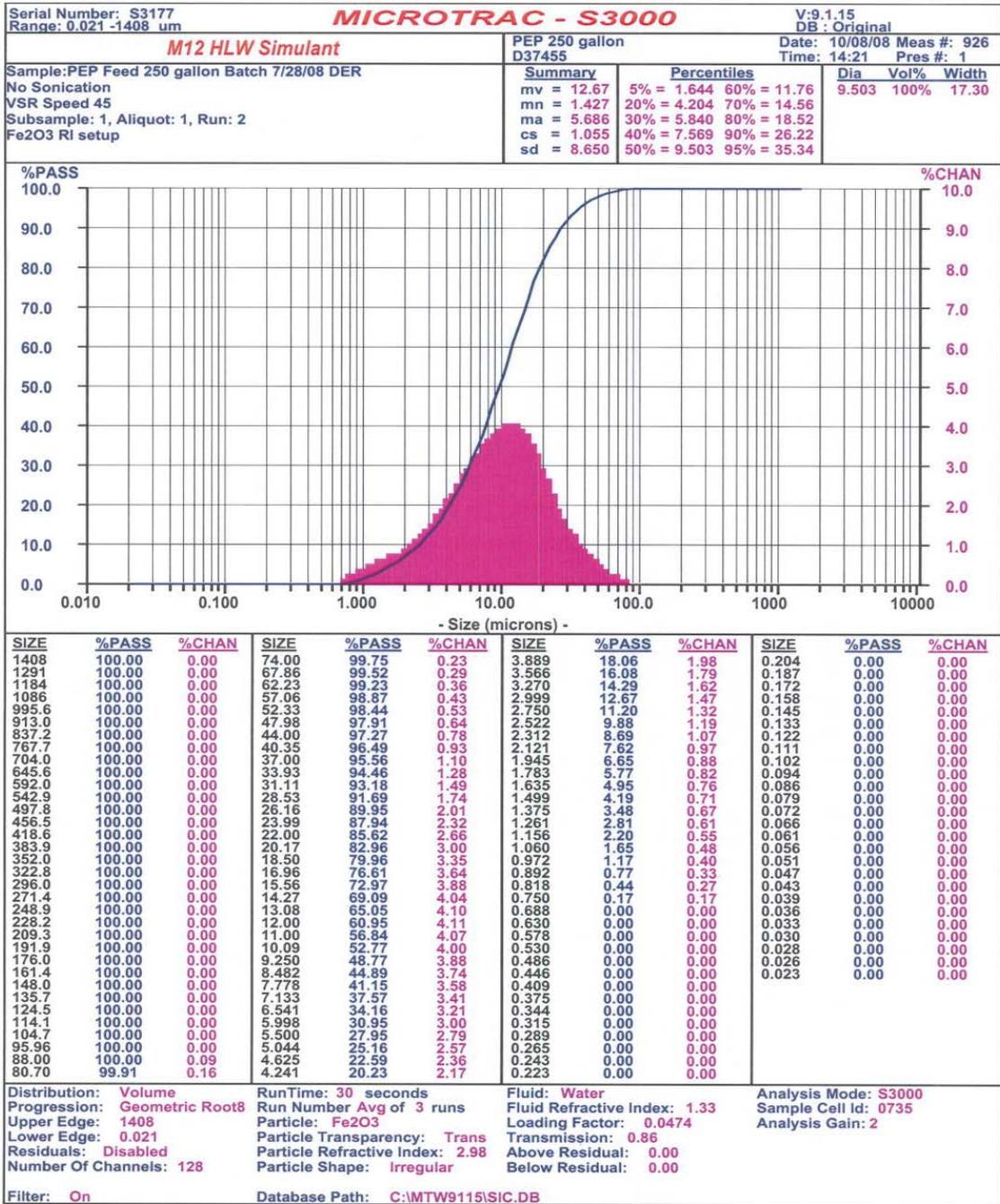
Sundar PS. 2007. *Simulant Testing in Support of Phase I Demonstration of the Ultrafiltration and Leaching Processes in the Integrated Test Facility*. 24590-WTP-TSP-RT-07-004, Rev. 0, Bechtel National, Inc., Richland, Washington.

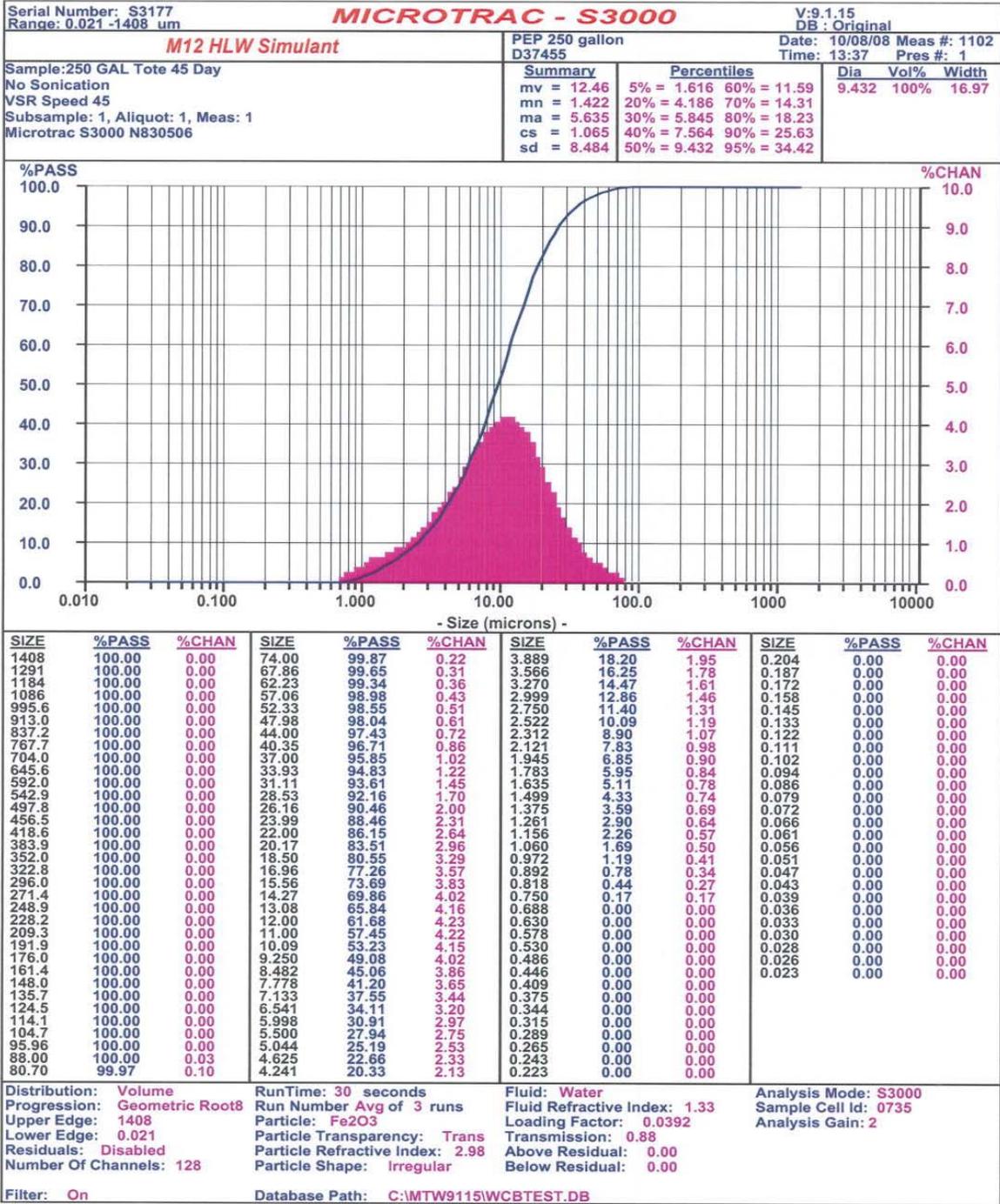
Sundar PS. 2008. *Test Exception to 24590-101-TSA-0004-72-00019 Rev 00A: Test Plan for Simulant Testing in Support of Phase I Demonstration of the Ultrafiltration and Leaching Processes in the Integrated Test Facility*. 24590-WTP-TEF-RT-08-00013, Rev. 0, Bechtel National, Inc., Richland, Washington.

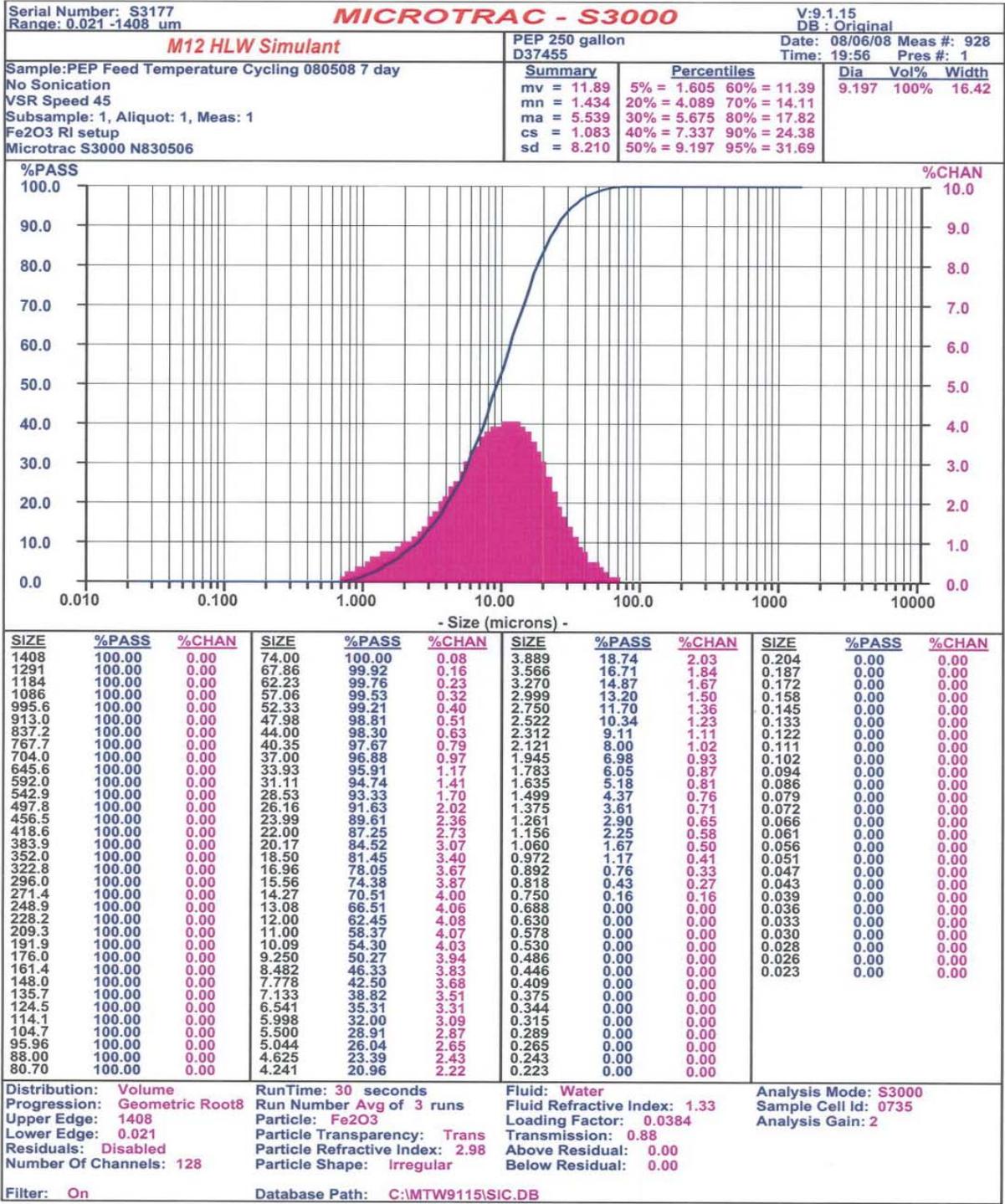
## **Appendix A**

### **PSD Plots**

# Appendix A: PSD Plots



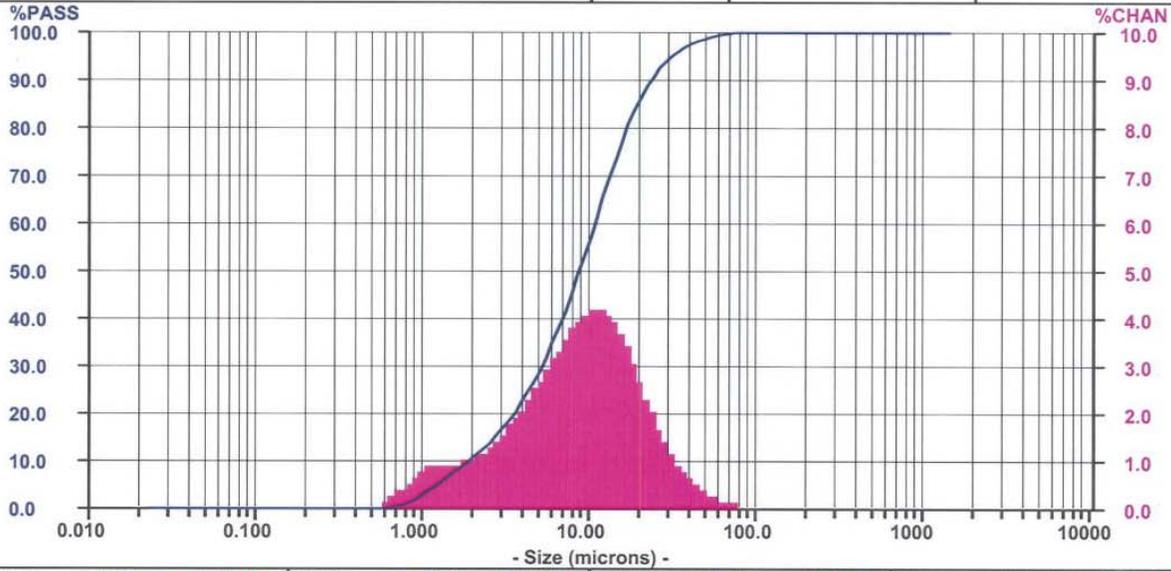




Serial Number: S3177 **MICROTRAC - S3000** V:9.1.15  
 Range: 0.021 -1408 um DB: Original

**M12 HLW Simulant** PEP Temp Cycl 15 d Date: 08/13/08 Meas #: 1044  
 D37455 Time: 23:45 Pres #: 1

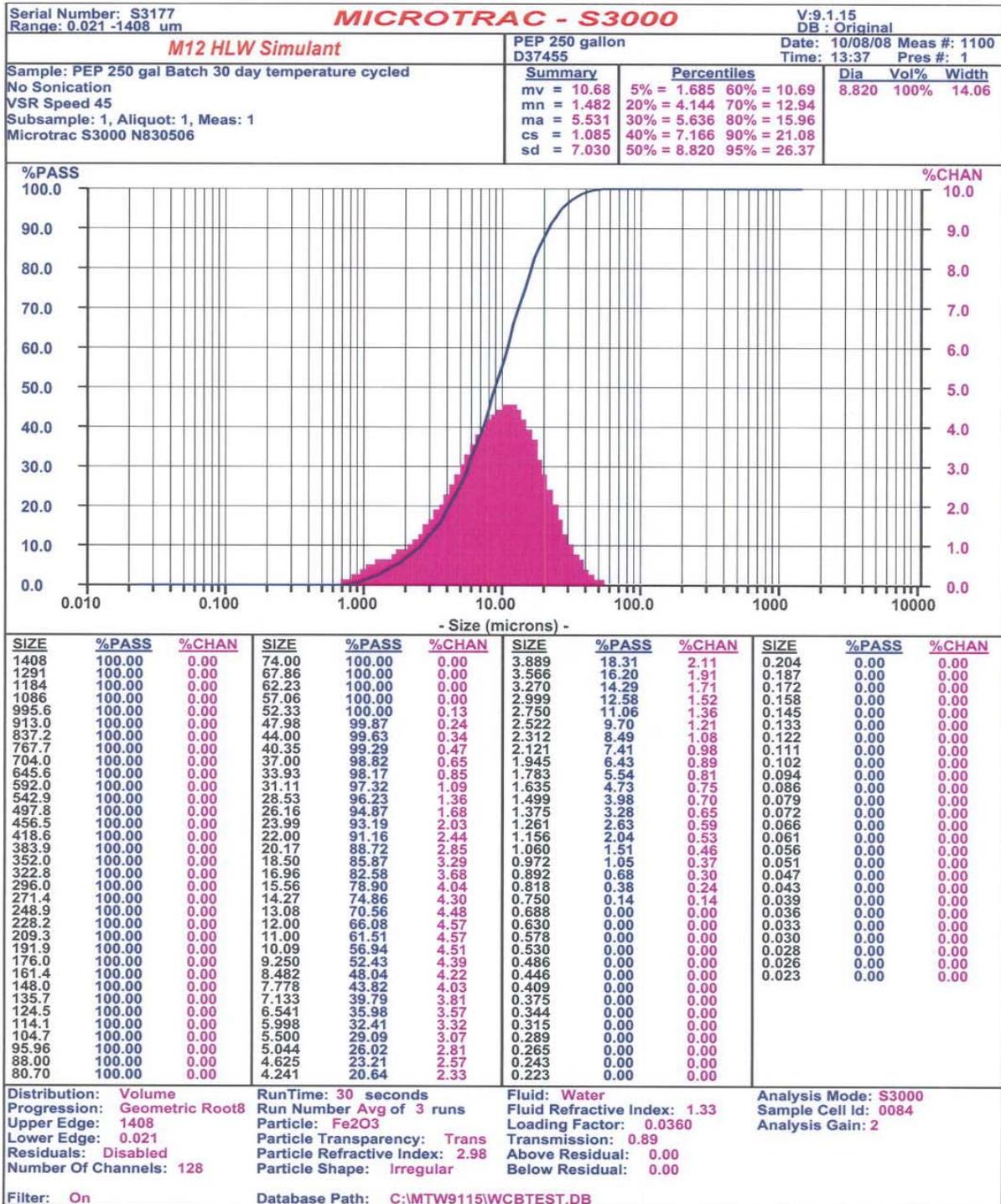
Sample: PEP 250 Gallon Feed Batch Temp Cycl 15 day Test No Sonication VSR Speed 45 Subsample: 1, Aliquot: 1, Meas: 1 Microtrac S3000 N830506	<b>Summary</b>		<b>Percentiles</b>			<b>Dia</b>	<b>Vol%</b>	<b>Width</b>
	mv = 11.21	5% = 1.221	60% = 10.70	8.669	100%	15.73		
	mn = 1.144	20% = 3.533	70% = 13.19					
	ma = 4.688	30% = 5.173	80% = 16.64					
	cs = 1.280	40% = 6.860	90% = 23.00					
	sd = 7.863	50% = 8.669	95% = 30.41					

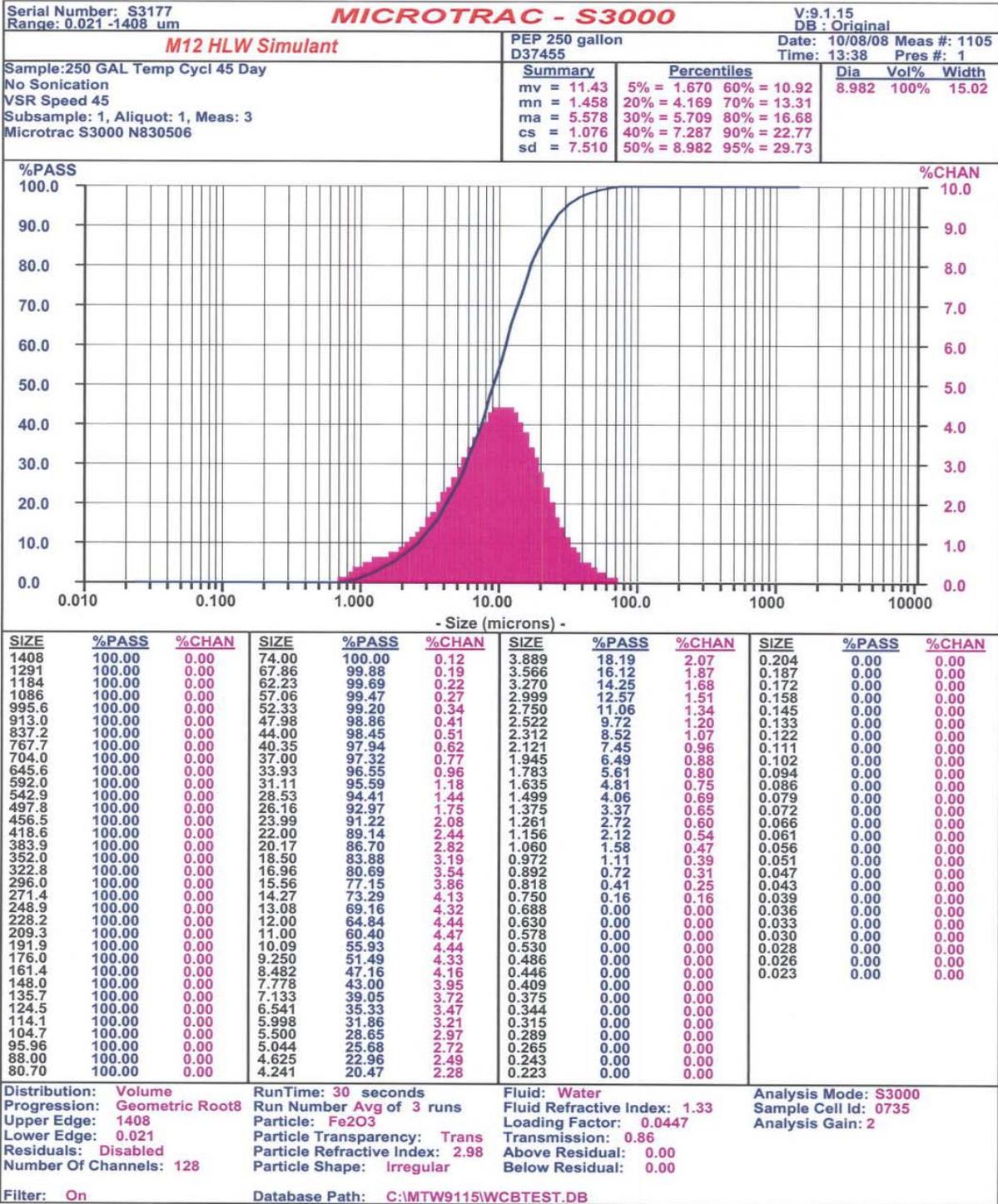


SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1408	100.00	0.00	74.00	100.00	0.15	3.889	22.17	1.97	0.204	0.00	0.00
1291	100.00	0.00	67.86	99.85	0.22	3.566	20.20	1.80	0.187	0.00	0.00
1184	100.00	0.00	62.23	99.63	0.25	3.270	18.40	1.64	0.172	0.00	0.00
1086	100.00	0.00	57.06	99.38	0.30	2.999	16.76	1.50	0.158	0.00	0.00
995.6	100.00	0.00	52.33	99.08	0.37	2.750	15.26	1.37	0.145	0.00	0.00
913.0	100.00	0.00	47.98	98.71	0.44	2.522	13.89	1.26	0.133	0.00	0.00
837.2	100.00	0.00	44.00	98.27	0.54	2.312	12.63	1.17	0.122	0.00	0.00
767.7	100.00	0.00	40.35	97.73	0.65	2.121	11.46	1.10	0.111	0.00	0.00
704.0	100.00	0.00	37.00	97.08	0.80	1.945	10.36	1.05	0.102	0.00	0.00
645.6	100.00	0.00	33.93	96.28	0.98	1.783	9.31	1.01	0.094	0.00	0.00
592.0	100.00	0.00	31.11	95.30	1.19	1.635	8.30	1.00	0.086	0.00	0.00
542.9	100.00	0.00	28.53	94.11	1.44	1.499	7.30	0.98	0.079	0.00	0.00
497.8	100.00	0.00	26.16	92.67	1.72	1.375	6.32	0.97	0.072	0.00	0.00
456.5	100.00	0.00	23.99	90.95	2.03	1.261	5.35	0.94	0.066	0.00	0.00
418.6	100.00	0.00	22.00	88.92	2.37	1.156	4.41	0.90	0.061	0.00	0.00
383.9	100.00	0.00	20.17	86.55	2.72	1.060	3.51	0.83	0.056	0.00	0.00
352.0	100.00	0.00	18.50	83.83	3.09	0.972	2.68	0.73	0.051	0.00	0.00
322.8	100.00	0.00	16.96	80.74	3.42	0.892	1.95	0.62	0.047	0.00	0.00
296.0	100.00	0.00	15.56	77.32	3.73	0.818	1.33	0.49	0.043	0.00	0.00
271.4	100.00	0.00	14.27	73.59	3.96	0.750	0.84	0.38	0.039	0.00	0.00
248.9	100.00	0.00	13.08	69.63	4.11	0.688	0.46	0.29	0.036	0.00	0.00
228.2	100.00	0.00	12.00	65.52	4.19	0.630	0.17	0.17	0.033	0.00	0.00
209.3	100.00	0.00	11.00	61.33	4.19	0.578	0.00	0.00	0.030	0.00	0.00
191.9	100.00	0.00	10.09	57.14	4.13	0.530	0.00	0.00	0.028	0.00	0.00
176.0	100.00	0.00	9.250	53.01	3.99	0.486	0.00	0.00	0.026	0.00	0.00
161.4	100.00	0.00	8.482	49.02	3.83	0.446	0.00	0.00	0.023	0.00	0.00
148.0	100.00	0.00	7.778	45.19	3.63	0.409	0.00	0.00			
135.7	100.00	0.00	7.133	41.56	3.41	0.375	0.00	0.00			
124.5	100.00	0.00	6.541	38.15	3.19	0.344	0.00	0.00			
114.1	100.00	0.00	5.998	34.96	2.98	0.315	0.00	0.00			
104.7	100.00	0.00	5.500	31.98	2.76	0.289	0.00	0.00			
95.96	100.00	0.00	5.044	29.22	2.55	0.265	0.00	0.00			
88.00	100.00	0.00	4.625	26.67	2.35	0.243	0.00	0.00			
80.70	100.00	0.00	4.241	24.32	2.15	0.223	0.00	0.00			

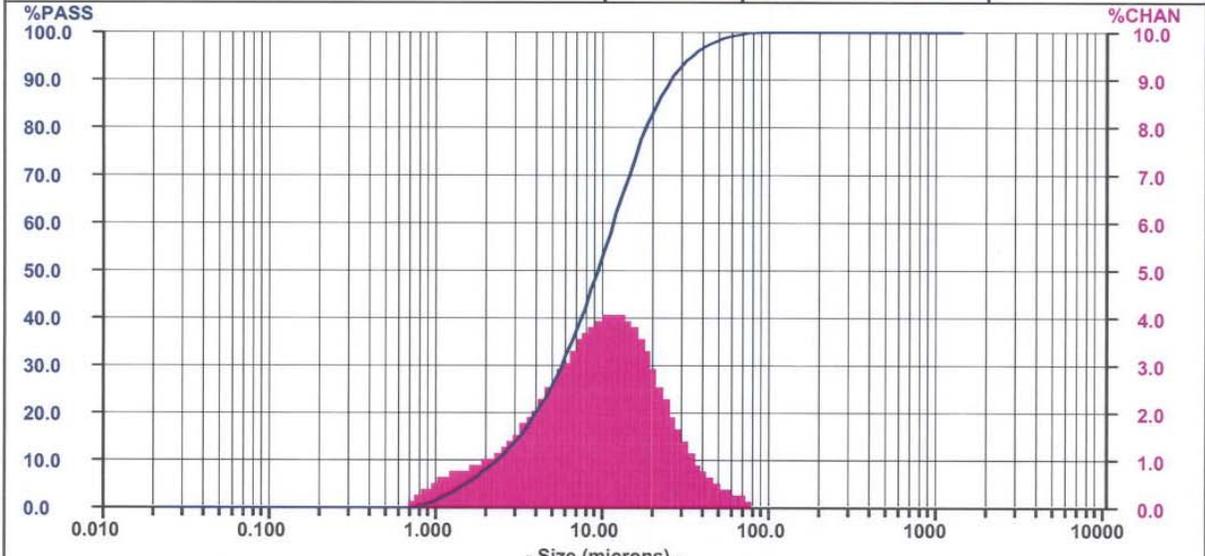
Distribution: Volume RunTime: 30 seconds Fluid: Water Analysis Mode: S3000  
 Progression: Geometric Root8 Run Number Avg of 3 runs Fluid Refractive Index: 1.33 Sample Cell Id: 0735  
 Upper Edge: 1408 Particle: Fe2O3 Loading Factor: 0.0433 Analysis Gain: 2  
 Lower Edge: 0.021 Particle Transparency: Trans Transmission: 0.85  
 Residuals: Disabled Particle Refractive Index: 2.98 Above Residual: 0.00  
 Number Of Channels: 128 Particle Shape: Irregular Below Residual: 0.00

Filter: On Database Path: C:\MTW9115\WCBTEST.DB



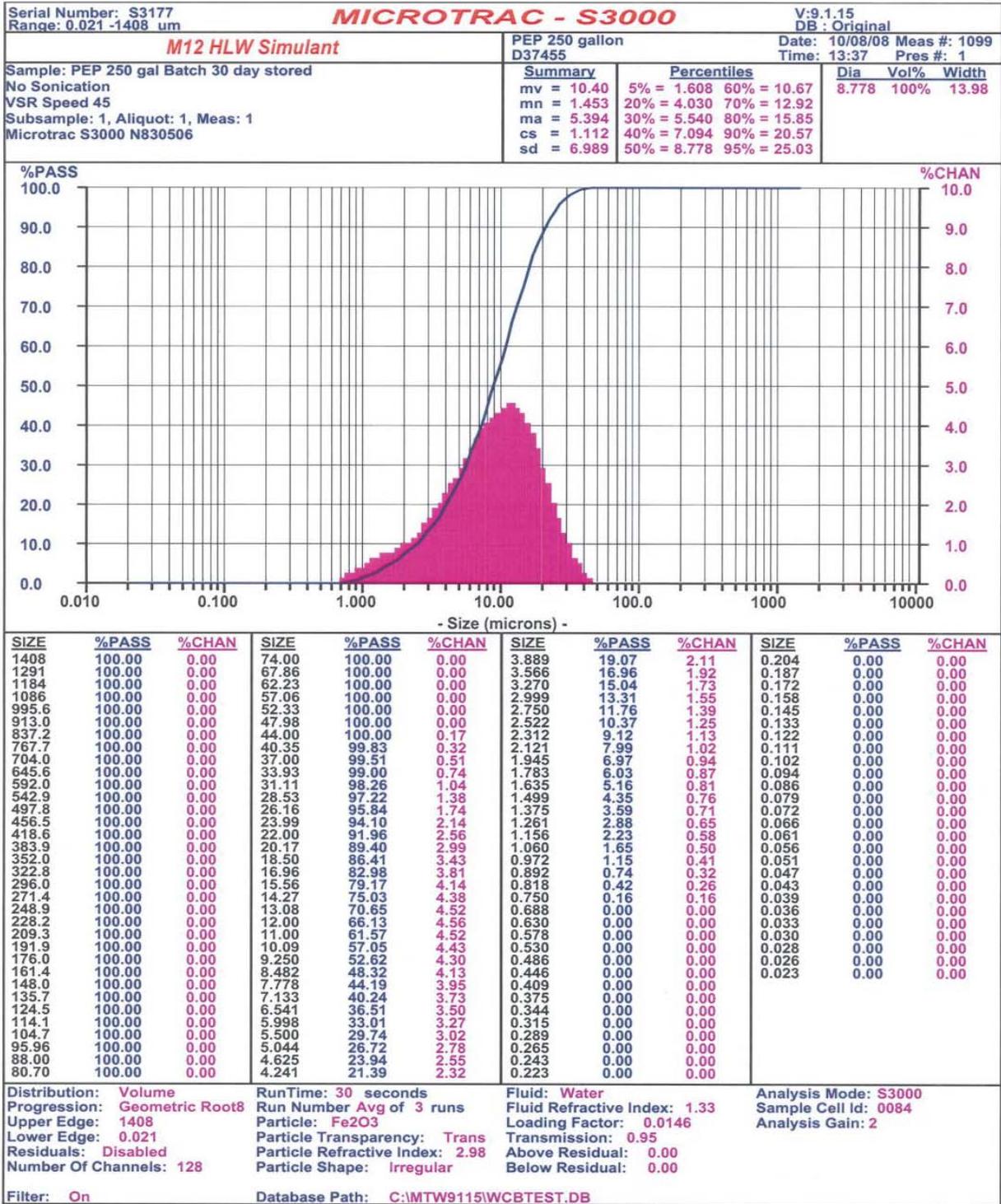


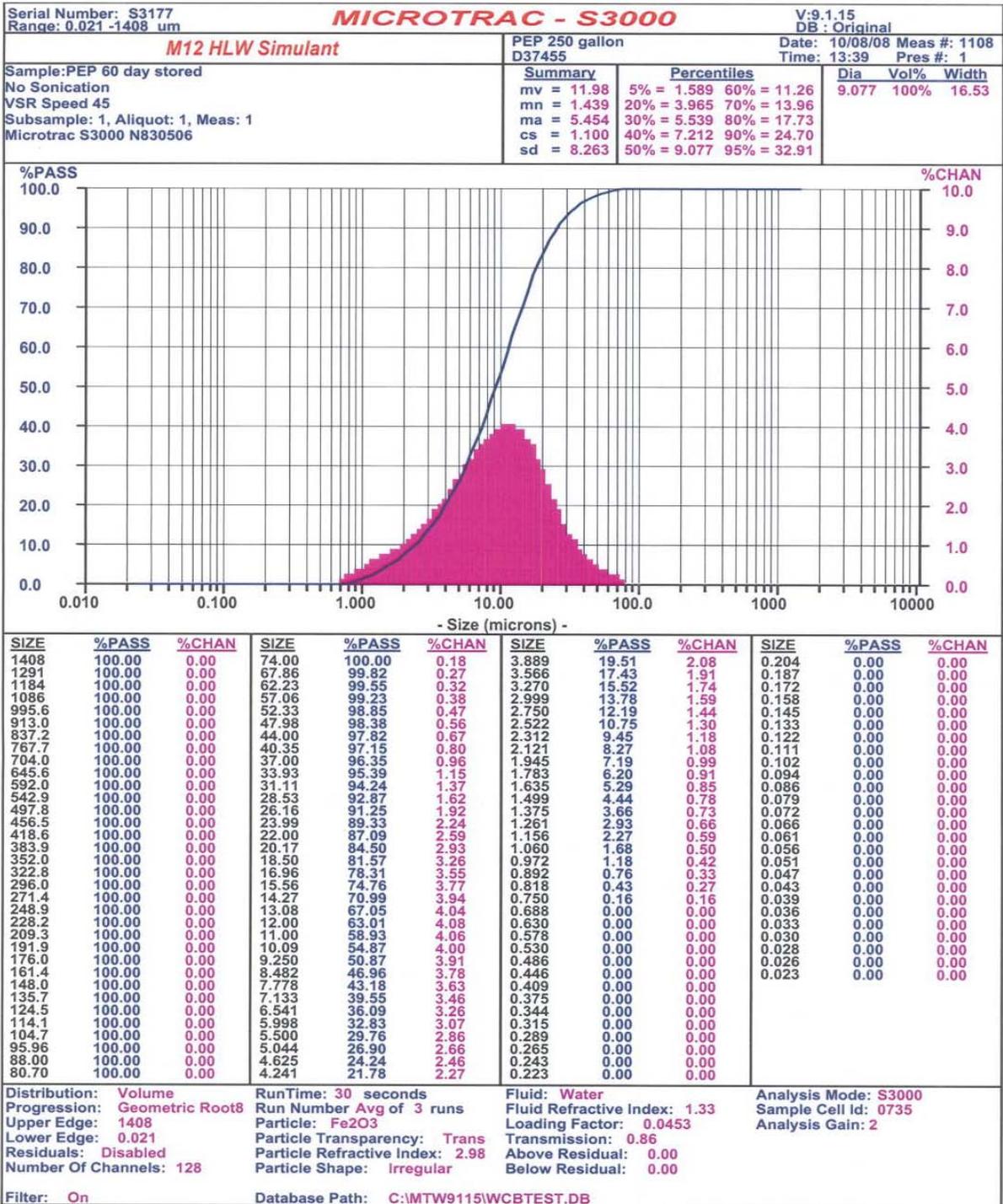
<b>M12 HLW Simulant</b>		PEP Temp Cycl 15 d D37455	Date: 08/13/08 Meas #: 1047 Time: 23:59 Pres #: 1
Sample: PEP 250 Gallon Feed Batch Stored 15 day Test	<b>Summary</b>	<b>Percentiles</b>	<b>Dia Vol% Width</b>
No Sonication	mv = 12.22	5% = 1.521 60% = 11.51	9.306 100% 16.90
VSR Speed 45	mn = 1.385	20% = 4.005 70% = 14.23	
Subsample: 1, Aliquot: 1, Meas: 1	ma = 5.427	30% = 5.655 80% = 18.07	
Microtrac S3000 N830506	cs = 1.106	40% = 7.391 90% = 25.21	
	sd = 8.452	50% = 9.306 95% = 33.48	

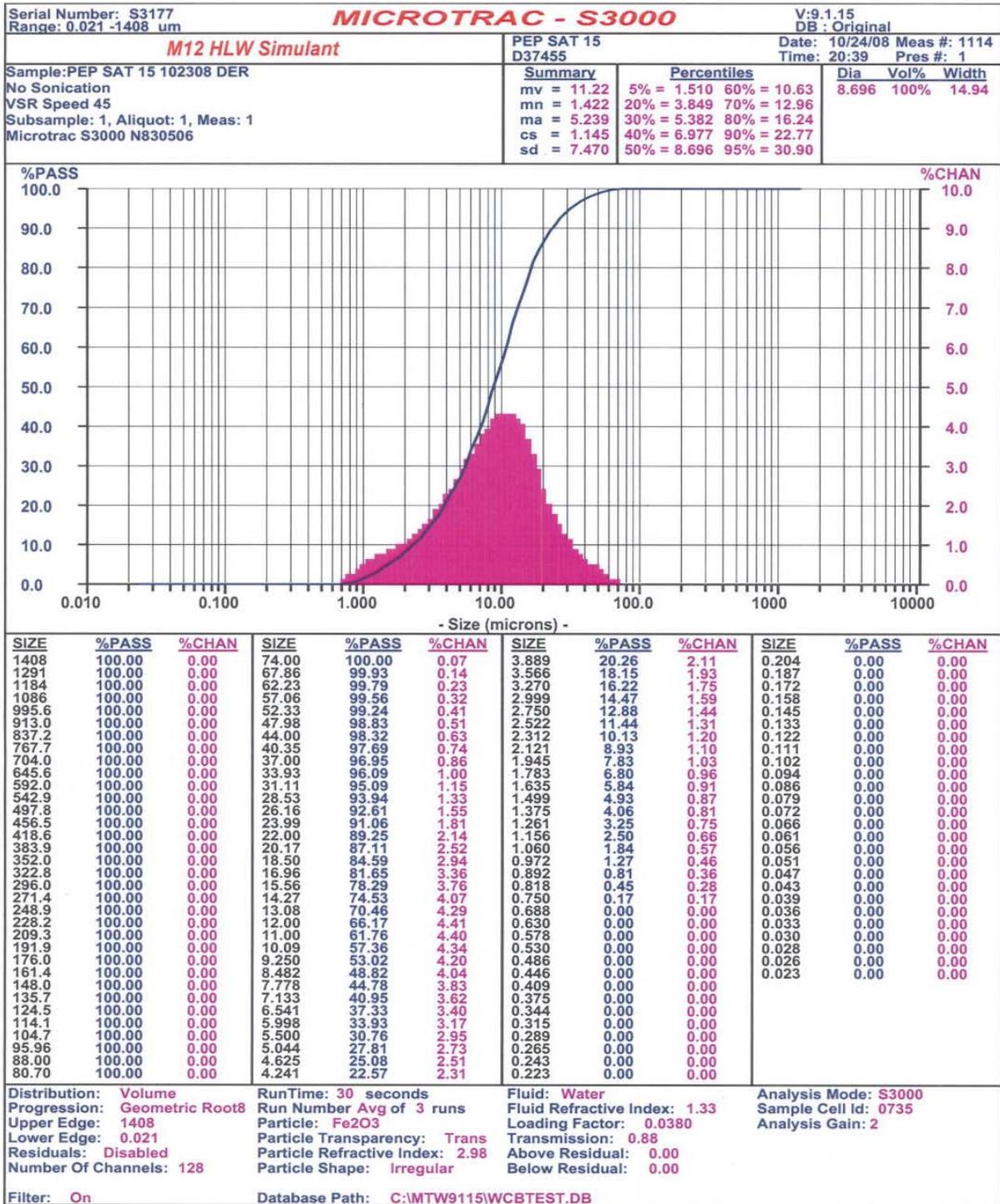


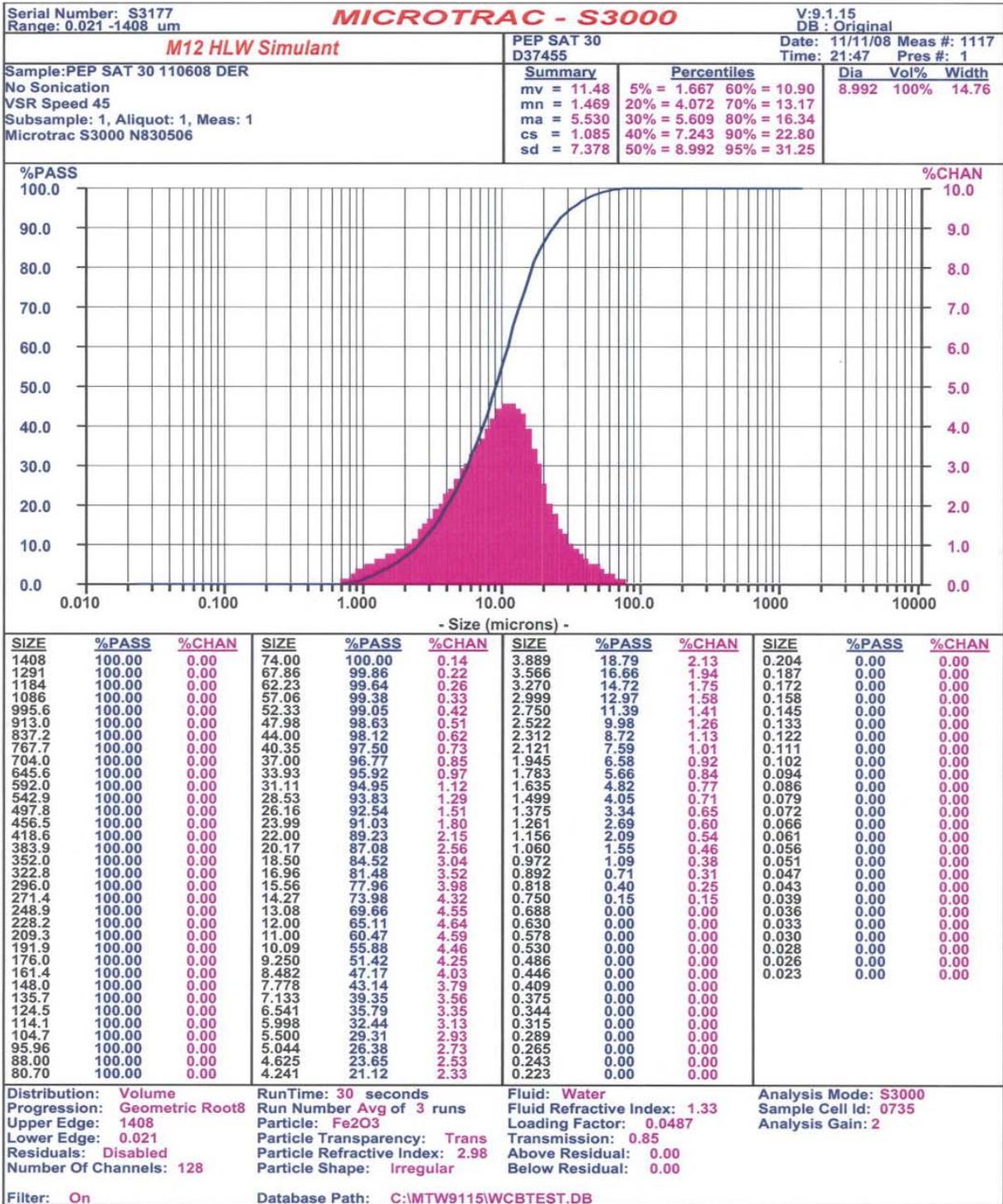
SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1408	100.00	0.00	74.00	99.89	0.19	3.889	19.29	1.97	0.204	0.00	0.00
1291	100.00	0.00	67.86	99.70	0.27	3.566	17.32	1.80	0.187	0.00	0.00
1184	100.00	0.00	62.23	99.43	0.31	3.270	15.52	1.64	0.172	0.00	0.00
1086	100.00	0.00	57.06	99.12	0.38	2.999	13.88	1.50	0.158	0.00	0.00
995.6	100.00	0.00	52.33	98.74	0.46	2.750	12.38	1.36	0.145	0.00	0.00
913.0	100.00	0.00	47.98	98.28	0.57	2.522	11.02	1.24	0.133	0.00	0.00
837.2	100.00	0.00	44.00	97.71	0.69	2.312	9.78	1.13	0.122	0.00	0.00
767.7	100.00	0.00	40.35	97.02	0.83	2.121	8.65	1.05	0.111	0.00	0.00
704.0	100.00	0.00	37.00	96.19	1.01	1.945	7.60	0.97	0.102	0.00	0.00
645.6	100.00	0.00	33.93	95.18	1.21	1.783	6.63	0.91	0.094	0.00	0.00
592.0	100.00	0.00	31.11	93.97	1.44	1.635	5.72	0.86	0.086	0.00	0.00
542.9	100.00	0.00	28.53	92.53	1.71	1.499	4.86	0.81	0.079	0.00	0.00
497.8	100.00	0.00	26.16	90.82	1.99	1.375	4.05	0.77	0.072	0.00	0.00
456.5	100.00	0.00	23.99	88.83	2.31	1.261	3.28	0.71	0.066	0.00	0.00
418.6	100.00	0.00	22.00	86.52	2.65	1.156	2.57	0.65	0.061	0.00	0.00
383.9	100.00	0.00	20.17	83.87	2.98	1.060	1.92	0.56	0.056	0.00	0.00
352.0	100.00	0.00	18.50	80.89	3.32	0.972	1.36	0.47	0.051	0.00	0.00
322.8	100.00	0.00	16.96	77.57	3.60	0.892	0.89	0.38	0.047	0.00	0.00
296.0	100.00	0.00	15.56	73.97	3.84	0.818	0.51	0.31	0.043	0.00	0.00
271.4	100.00	0.00	14.27	70.13	4.02	0.750	0.20	0.20	0.039	0.00	0.00
248.9	100.00	0.00	13.08	66.11	4.13	0.688	0.00	0.00	0.036	0.00	0.00
228.2	100.00	0.00	12.00	61.98	4.15	0.630	0.00	0.00	0.033	0.00	0.00
209.3	100.00	0.00	11.00	57.83	4.10	0.578	0.00	0.00	0.030	0.00	0.00
191.9	100.00	0.00	10.09	53.73	4.00	0.530	0.00	0.00	0.028	0.00	0.00
176.0	100.00	0.00	9.250	49.73	3.88	0.486	0.00	0.00	0.026	0.00	0.00
161.4	100.00	0.00	8.482	45.85	3.73	0.446	0.00	0.00	0.023	0.00	0.00
148.0	100.00	0.00	7.778	42.12	3.55	0.409	0.00	0.00			
135.7	100.00	0.00	7.133	38.57	3.37	0.375	0.00	0.00			
124.5	100.00	0.00	6.541	35.20	3.16	0.344	0.00	0.00			
114.1	100.00	0.00	5.998	32.04	2.96	0.315	0.00	0.00			
104.7	100.00	0.00	5.500	29.08	2.75	0.289	0.00	0.00			
95.96	100.00	0.00	5.044	26.33	2.54	0.265	0.00	0.00			
88.00	100.00	0.03	4.625	23.79	2.35	0.243	0.00	0.00			
80.70	99.97	0.08	4.241	21.44	2.15	0.223	0.00	0.00			

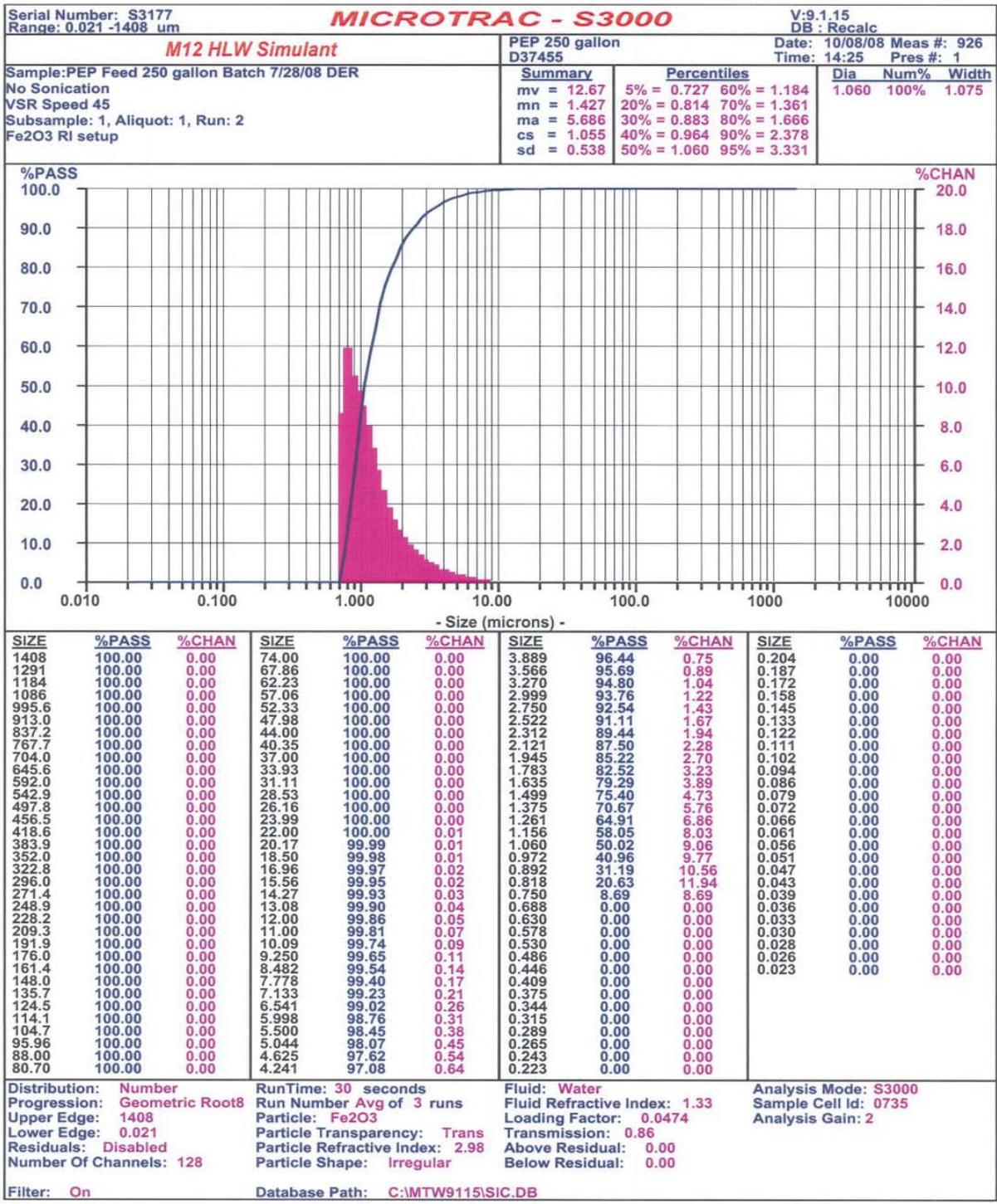
Distribution: Volume	RunTime: 30 seconds	Fluid: Water	Analysis Mode: S3000
Progression: Geometric Root8	Run Number Avg of 3 runs	Fluid Refractive Index: 1.33	Sample Cell Id: 0735
Upper Edge: 1408	Particle: Fe2O3	Loading Factor: 0.0609	Analysis Gain: 2
Lower Edge: 0.021	Particle Transparency: Trans	Transmission: 0.82	
Residuals: Disabled	Particle Refractive Index: 2.98	Above Residual: 0.00	
Number Of Channels: 128	Particle Shape: Irregular	Below Residual: 0.00	
Filter: On	Database Path: C:\MTW9115\WCBTEST.DB		

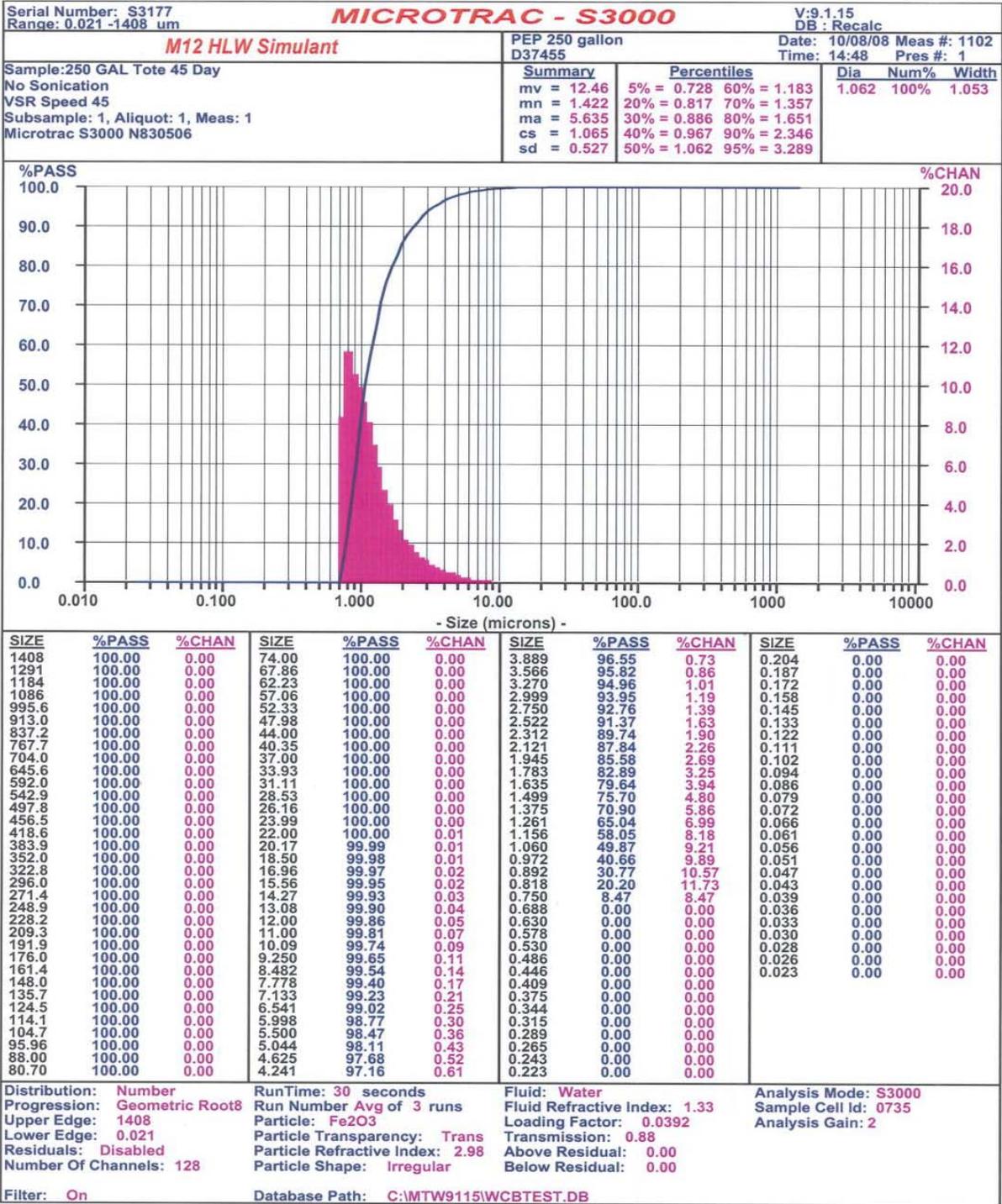


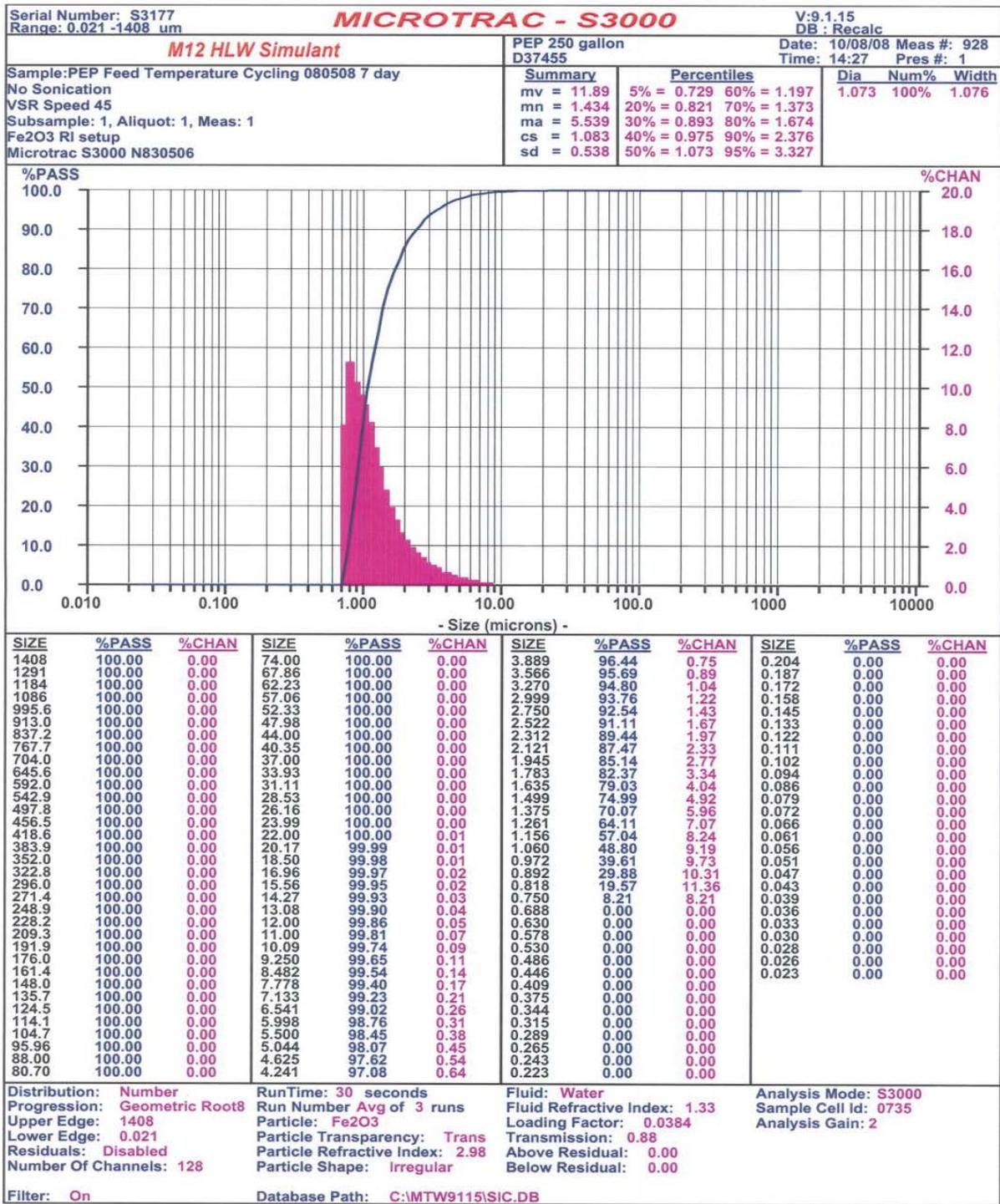


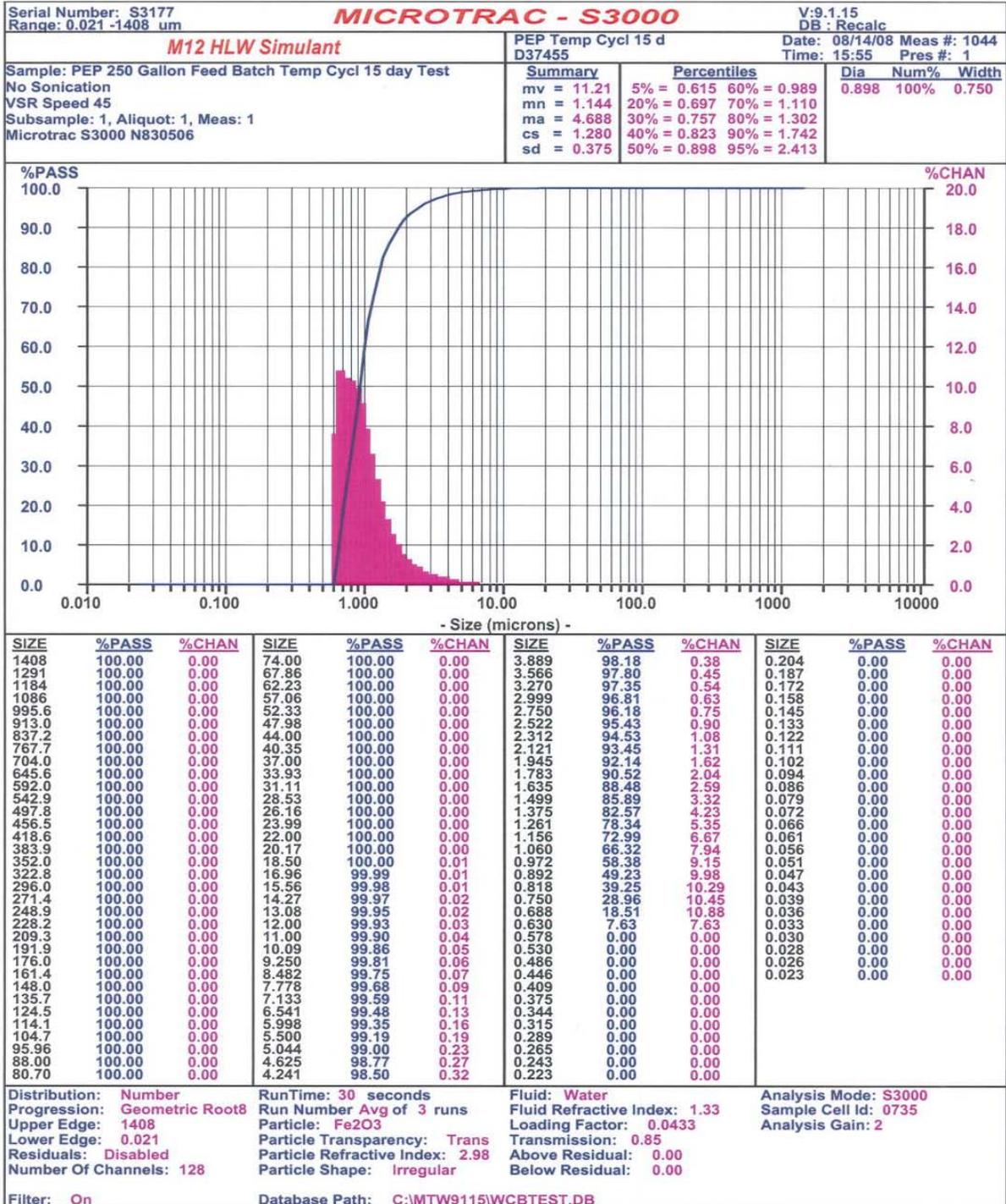


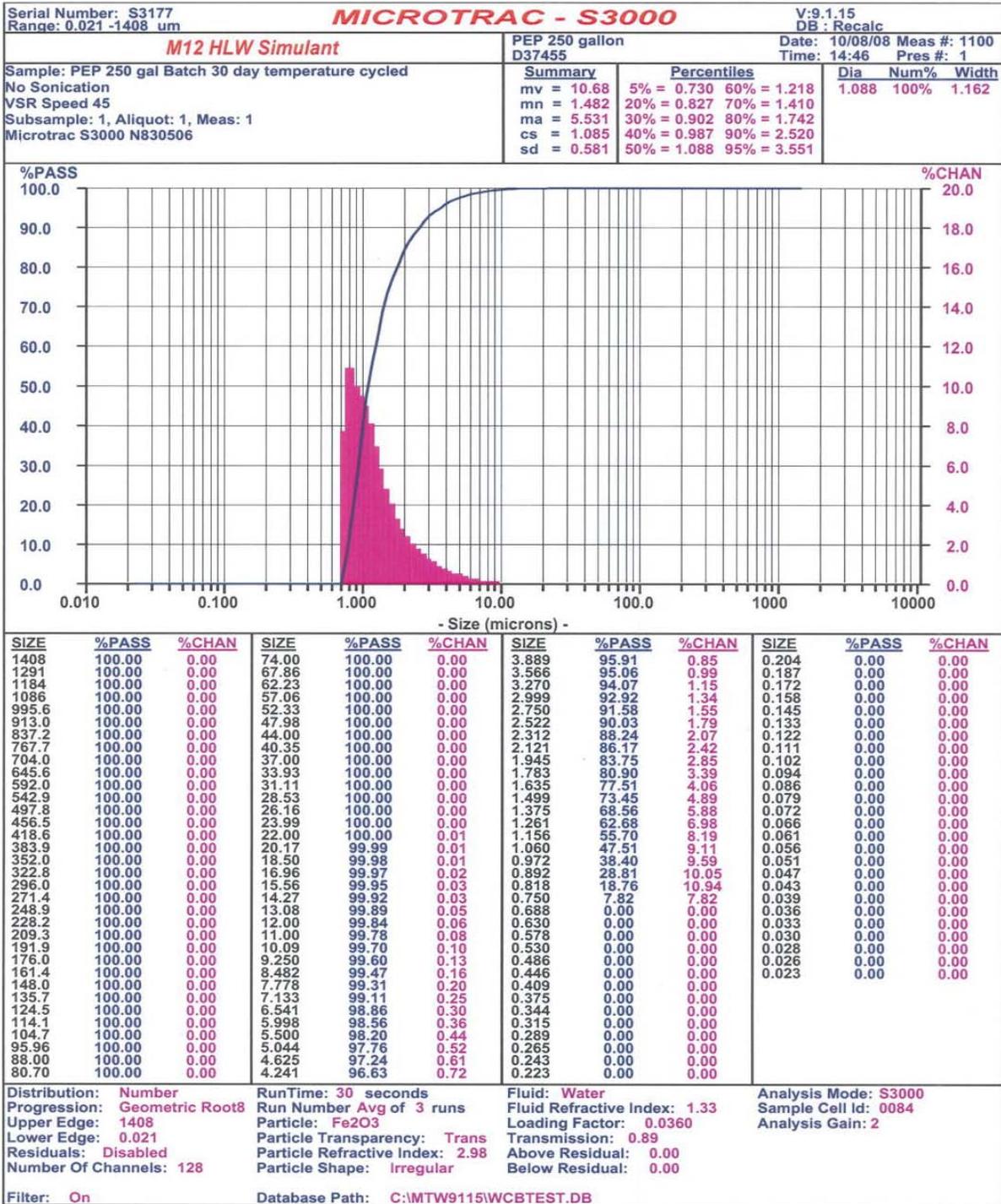


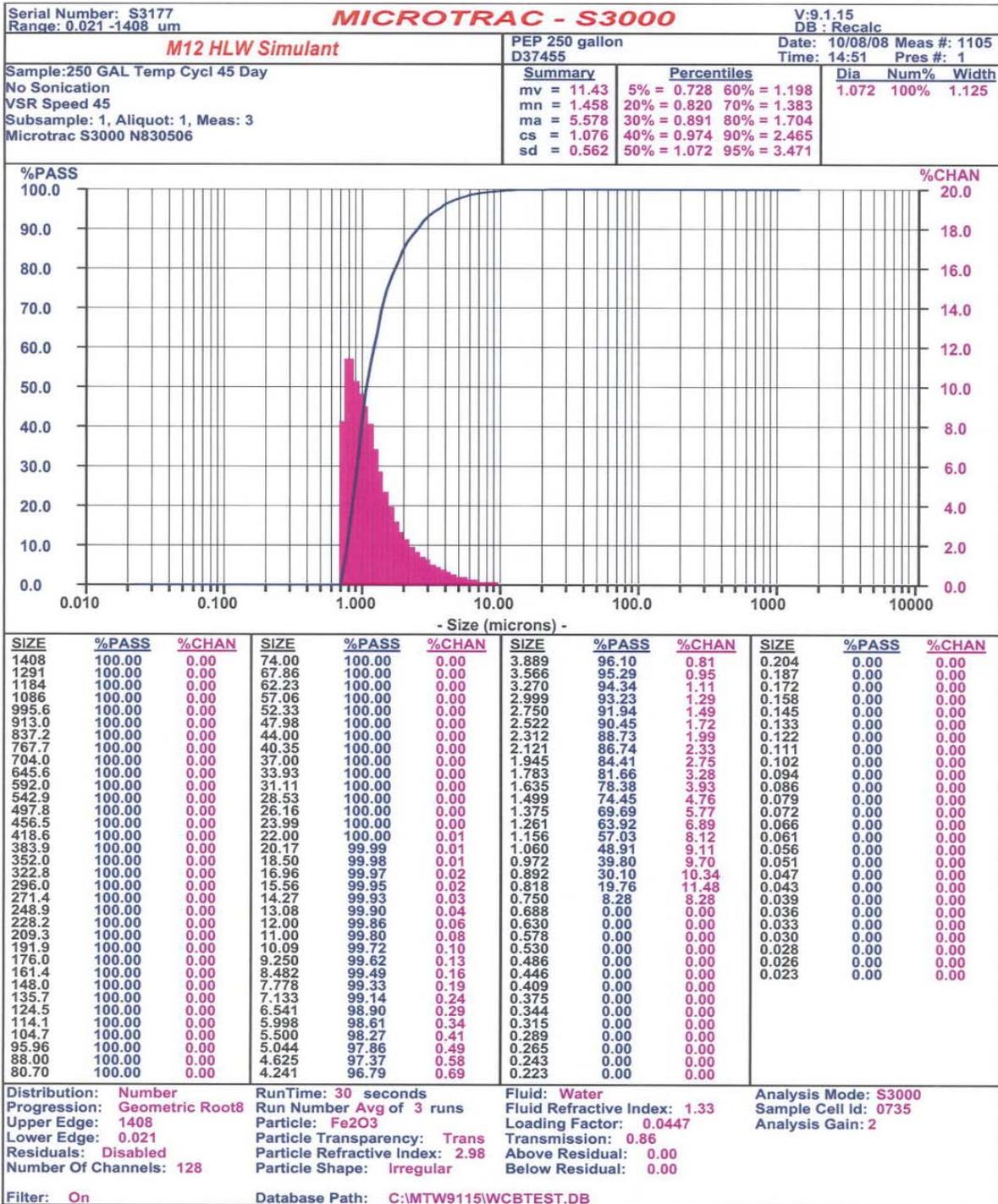


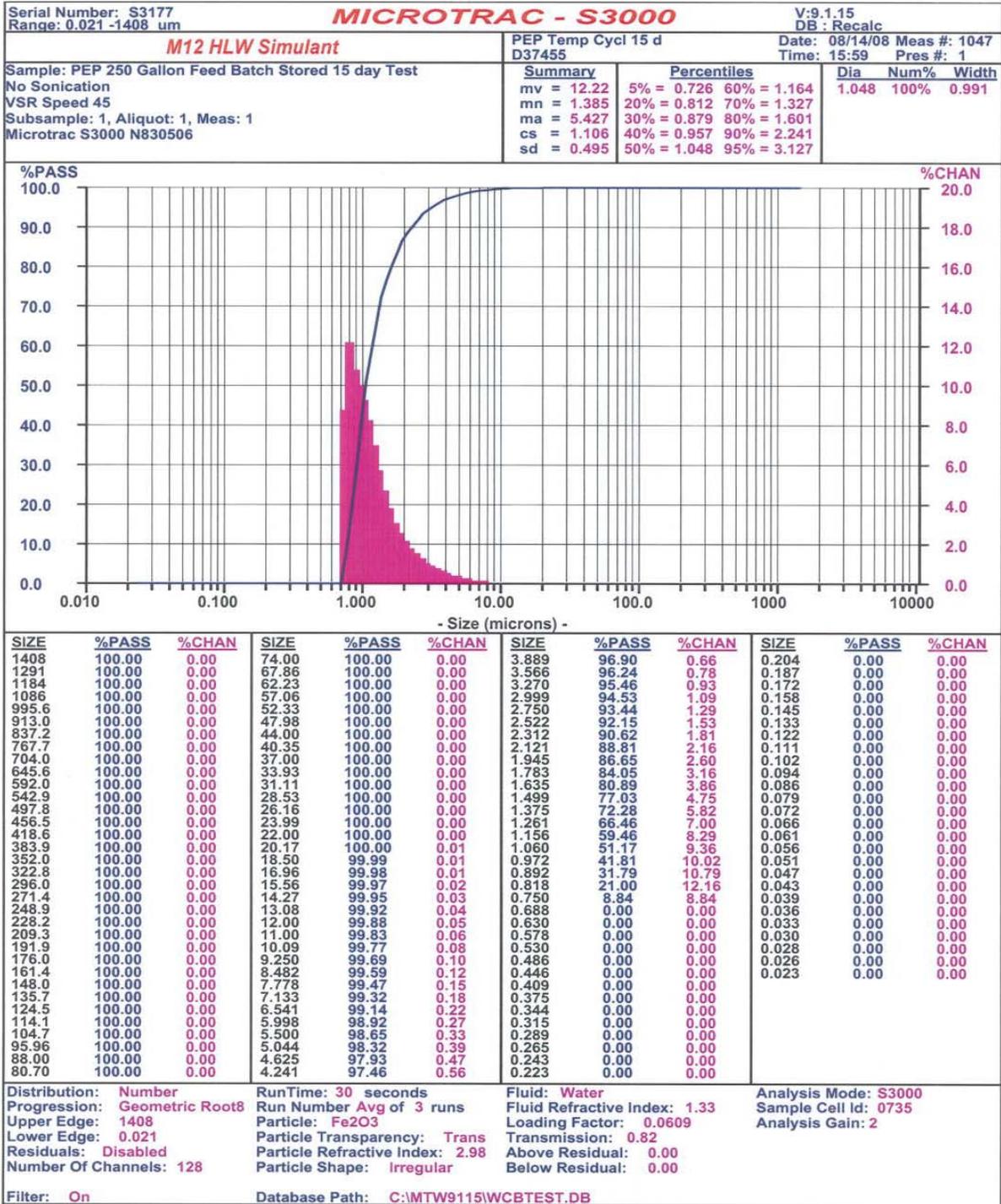


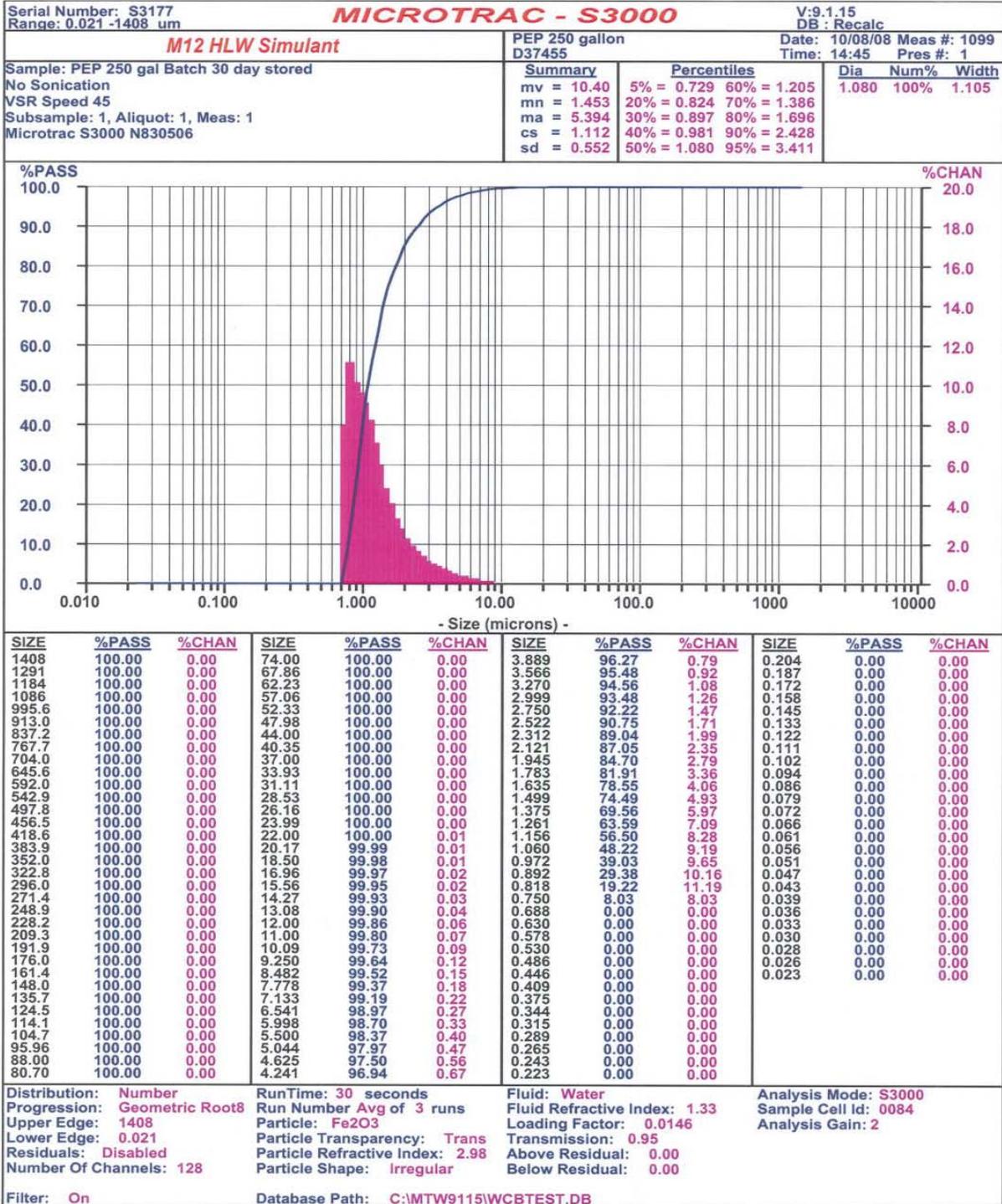


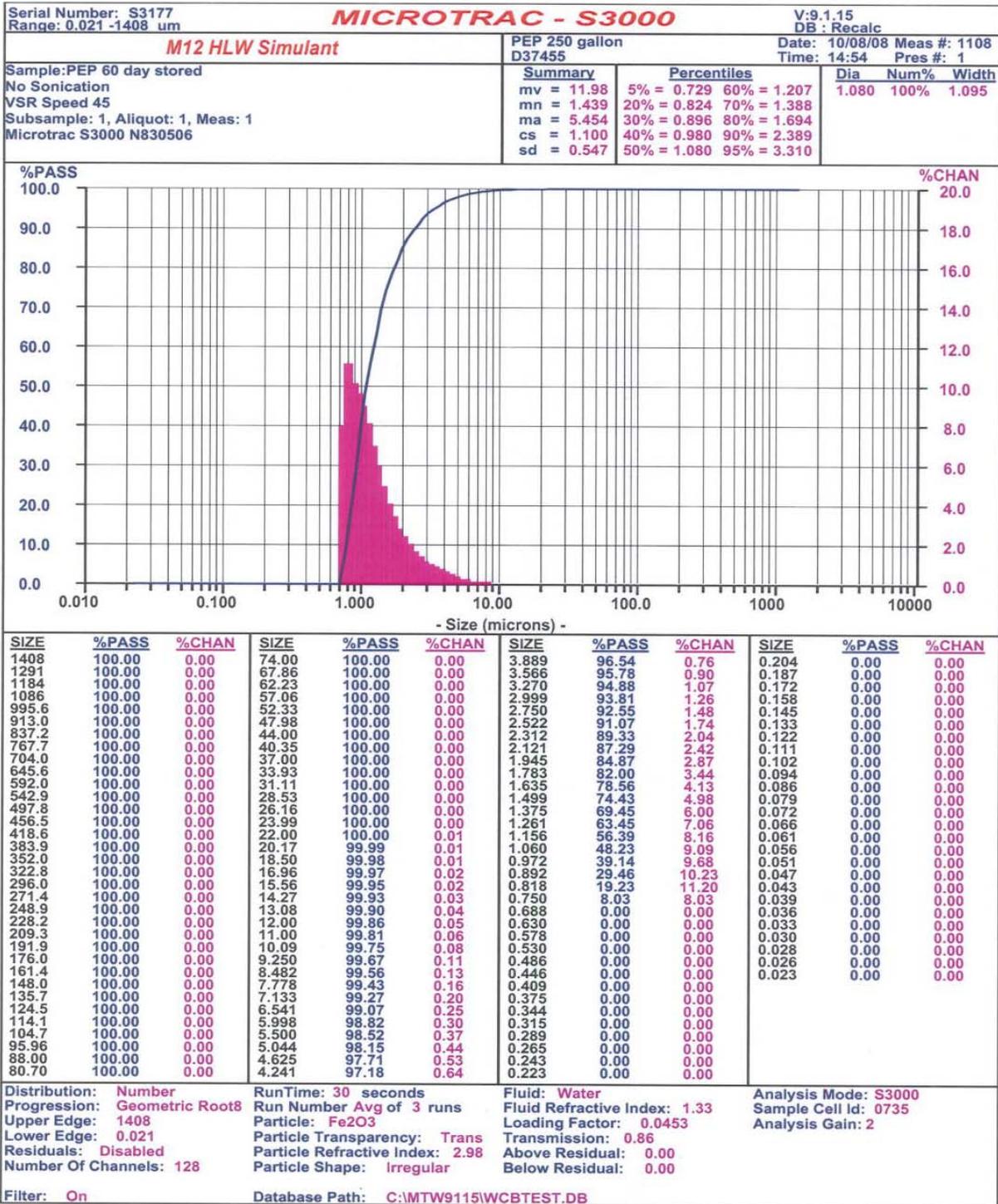


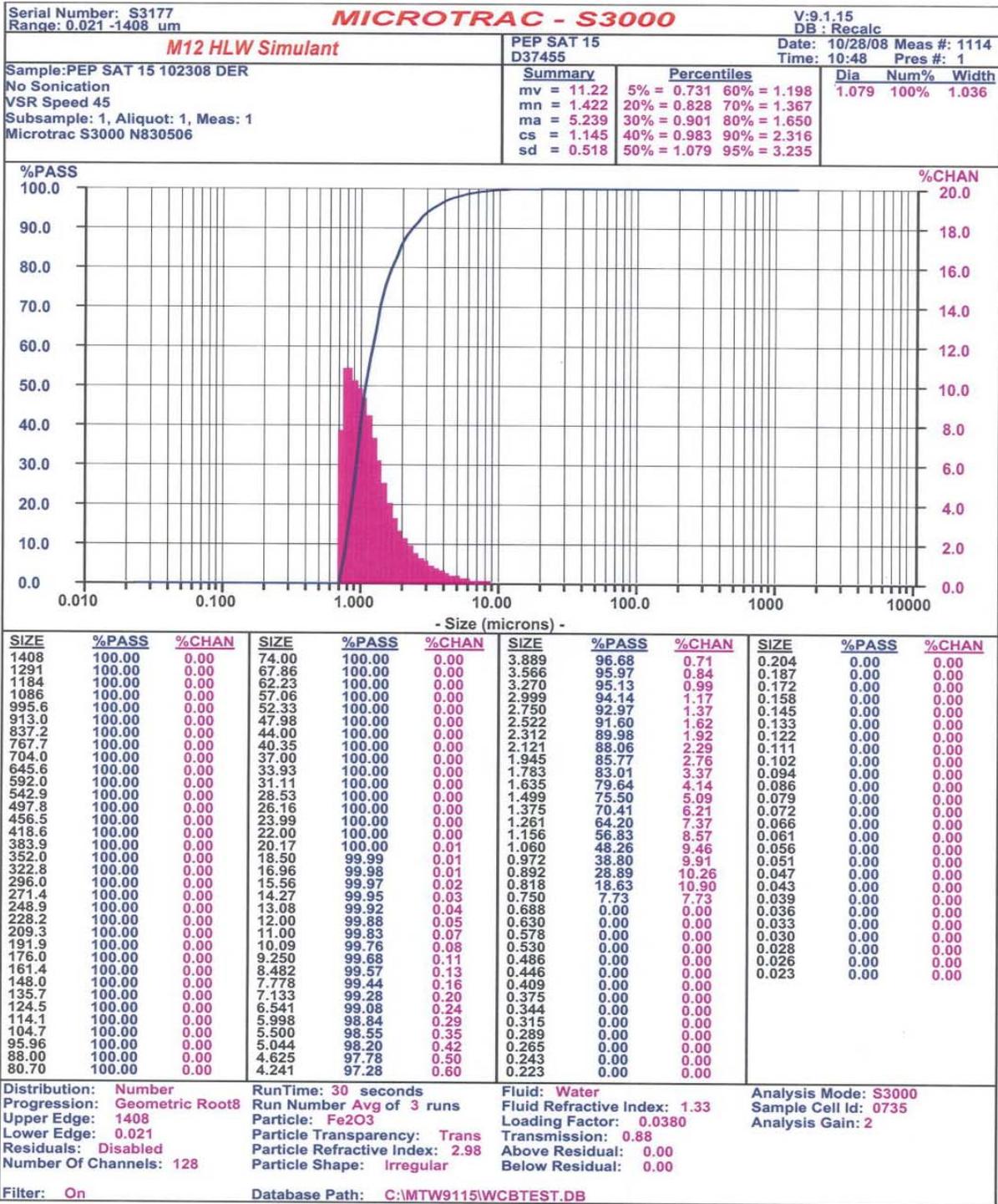




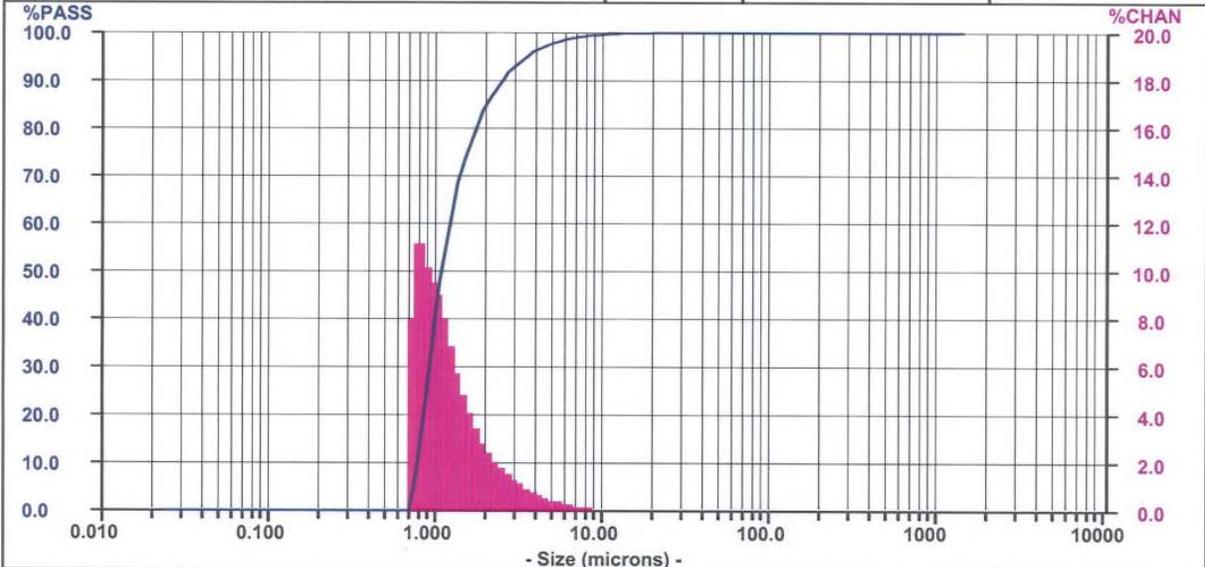








<b>M12 HLW Simulant</b>		PEP SAT 30 D37455	Date: 11/11/08 Meas #: 1117 Time: 21:21 Pres #: 1
Sample: PEP SAT 30 110608 DER No Sonication VSR Speed 45 Subsample: 1, Aliquot: 1, Meas: 1 Microtrac S3000 N830506		Summary mv = 11.48 mn = 1.469 ma = 5.530 cs = 1.085 sd = 0.578	Percentiles 5% = 0.729 20% = 0.823 30% = 0.897 40% = 0.982 50% = 1.083 60% = 1.214 70% = 1.407 80% = 1.738 90% = 2.493 95% = 3.474
		Dia	Num% Width
		1.083	100% 1.156



SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1408	100.00	0.00	74.00	100.00	0.00	3.889	96.13	0.84	0.204	0.00	0.00
1291	100.00	0.00	67.86	100.00	0.00	3.566	95.29	0.99	0.187	0.00	0.00
1184	100.00	0.00	62.23	100.00	0.00	3.270	94.30	1.16	0.172	0.00	0.00
1086	100.00	0.00	57.06	100.00	0.00	2.999	93.14	1.35	0.158	0.00	0.00
995.6	100.00	0.00	52.33	100.00	0.00	2.750	91.79	1.56	0.145	0.00	0.00
913.0	100.00	0.00	47.98	100.00	0.00	2.522	90.23	1.81	0.133	0.00	0.00
837.2	100.00	0.00	44.00	100.00	0.00	2.312	88.42	2.10	0.122	0.00	0.00
767.7	100.00	0.00	40.35	100.00	0.00	2.121	86.32	2.45	0.111	0.00	0.00
704.0	100.00	0.00	37.00	100.00	0.00	1.945	83.87	2.88	0.102	0.00	0.00
645.6	100.00	0.00	33.93	100.00	0.00	1.783	80.99	3.42	0.094	0.00	0.00
592.0	100.00	0.00	31.11	100.00	0.00	1.635	77.57	4.06	0.086	0.00	0.00
542.9	100.00	0.00	28.53	100.00	0.00	1.499	73.51	4.85	0.079	0.00	0.00
497.8	100.00	0.00	26.16	100.00	0.00	1.375	68.86	5.79	0.072	0.00	0.00
456.5	100.00	0.00	23.99	100.00	0.00	1.261	62.87	6.86	0.066	0.00	0.00
418.6	100.00	0.00	22.00	100.00	0.00	1.156	56.01	8.04	0.061	0.00	0.00
383.9	100.00	0.00	20.17	100.00	0.01	1.060	47.97	9.00	0.056	0.00	0.00
352.0	100.00	0.00	18.50	99.99	0.01	0.972	38.97	9.57	0.051	0.00	0.00
322.8	100.00	0.00	16.96	99.98	0.02	0.892	29.40	10.16	0.047	0.00	0.00
296.0	100.00	0.00	15.56	99.96	0.02	0.818	19.24	11.18	0.043	0.00	0.00
271.4	100.00	0.00	14.27	99.94	0.03	0.750	8.06	8.06	0.039	0.00	0.00
248.9	100.00	0.00	13.08	99.91	0.05	0.688	0.00	0.00	0.036	0.00	0.00
228.2	100.00	0.00	12.00	99.86	0.06	0.630	0.00	0.00	0.033	0.00	0.00
209.3	100.00	0.00	11.00	99.80	0.08	0.578	0.00	0.00	0.030	0.00	0.00
191.9	100.00	0.00	10.09	99.72	0.10	0.530	0.00	0.00	0.028	0.00	0.00
176.0	100.00	0.00	9.250	99.62	0.12	0.486	0.00	0.00	0.026	0.00	0.00
161.4	100.00	0.00	8.482	99.50	0.15	0.446	0.00	0.00	0.023	0.00	0.00
148.0	100.00	0.00	7.778	99.35	0.19	0.409	0.00	0.00			
135.7	100.00	0.00	7.133	99.16	0.23	0.375	0.00	0.00			
124.5	100.00	0.00	6.541	98.93	0.28	0.344	0.00	0.00			
114.1	100.00	0.00	5.998	98.65	0.33	0.315	0.00	0.00			
104.7	100.00	0.00	5.500	98.32	0.41	0.289	0.00	0.00			
95.96	100.00	0.00	5.044	97.91	0.49	0.265	0.00	0.00			
88.00	100.00	0.00	4.625	97.42	0.59	0.243	0.00	0.00			
80.70	100.00	0.00	4.241	96.83	0.70	0.223	0.00	0.00			

Distribution: Number  
 Progression: Geometric Root8  
 Upper Edge: 1408  
 Lower Edge: 0.021  
 Residuals: Disabled  
 Number Of Channels: 128  
 Filter: On

RunTime: 30 seconds  
 Run Number Avg of 3 runs  
 Particle: Fe2O3  
 Particle Transparency: Trans  
 Particle Refractive Index: 2.98  
 Particle Shape: Irregular  
 Database Path: C:\MTW9115\WCBTEST.DB

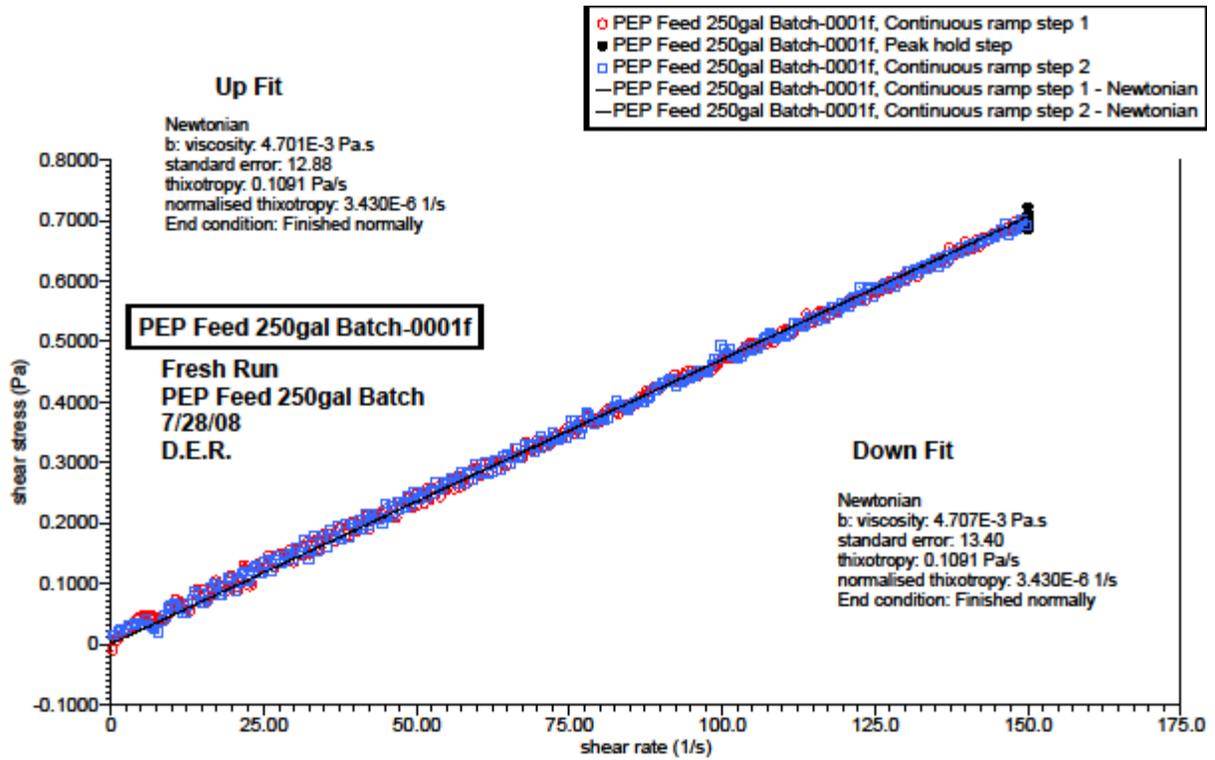
Fluid: Water  
 Fluid Refractive Index: 1.33  
 Loading Factor: 0.0487  
 Transmission: 0.85  
 Above Residual: 0.00  
 Below Residual: 0.00

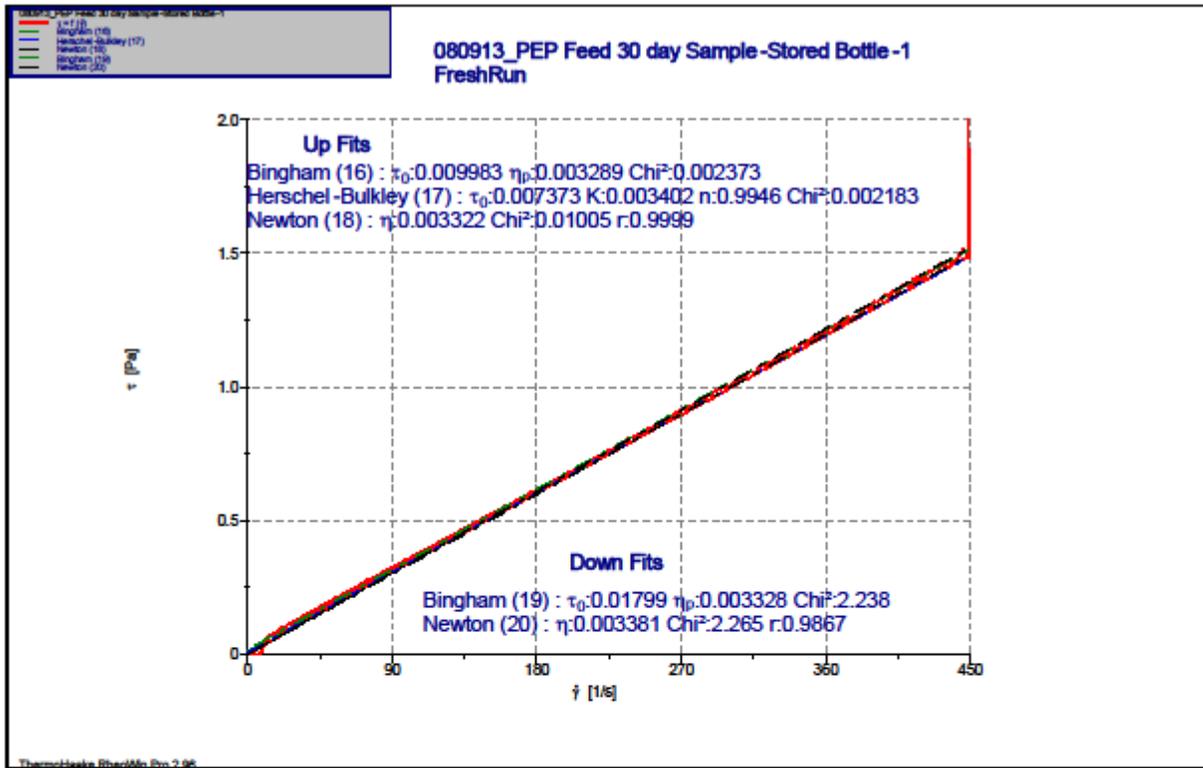
Analysis Mode: S3000  
 Sample Cell Id: 0735  
 Analysis Gain: 2

## **Appendix B**

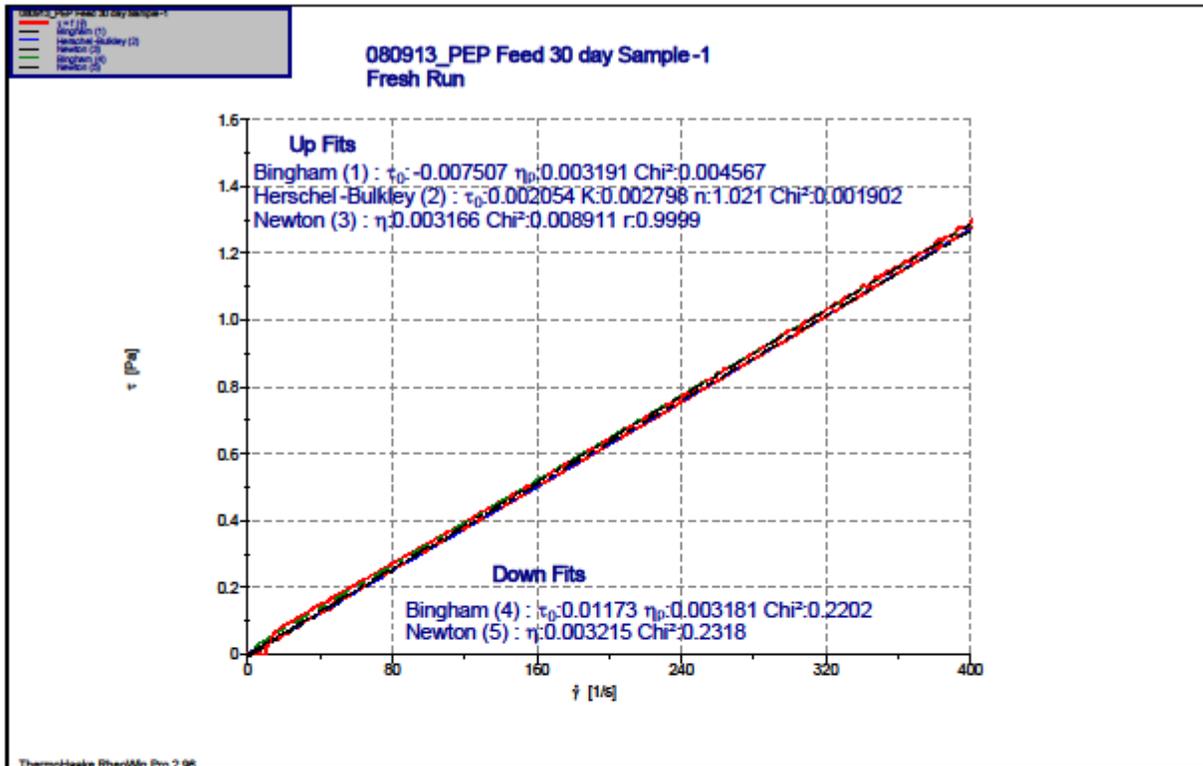
### **Rheology Flow Curves**

# Appendix B: Rheology Flow Curves



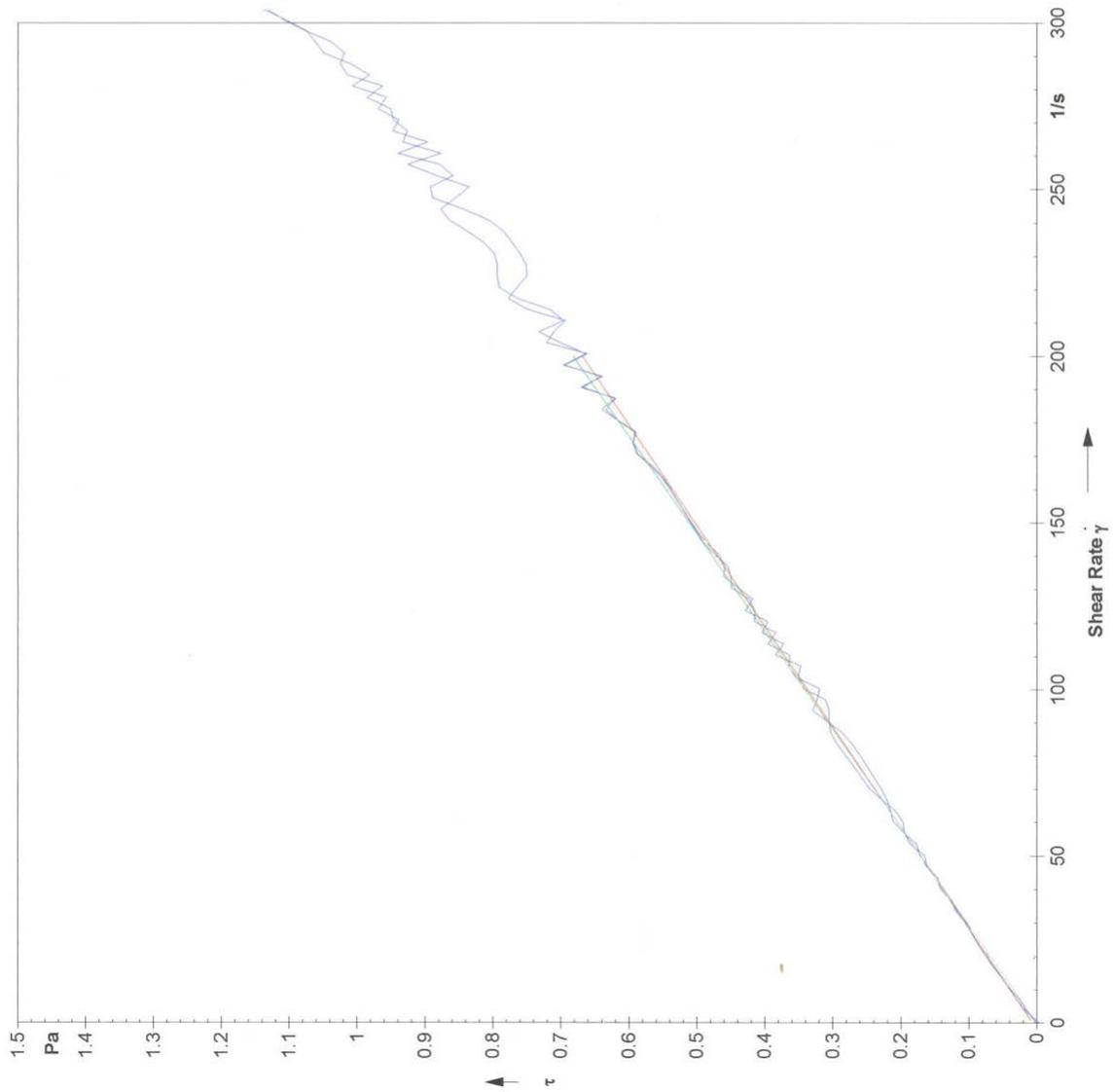


1: C:\Rheology Results\53019-M12\080913\080913\_PEP Feed 30 day Sample-Stored Bottle-1.rwd  
 Company / Operator: PNNL / Xiao-Ying Yu  
 Date / Time / Version: 13.09.2008 / 9:25:48 AM / RheoWin Pro 296  
 Substance / Sample no: PEP Feed 30 day Sample-Stored Bottle / 080913 PEP Feed 30 day Sample-Stored Bott  
 Bingham (16) :  $\tau_0:0.009983$   $\eta_p:0.003289$   $\text{Chi}^2:0.002373$   $r:1$   $\square\square$   $x = \dot{\gamma} [1/s]$ ,  $y = \tau [Pa]$   
 Herschel-Bulkley (17) :  $\tau_0:0.007373$   $K:0.003402$   $n:0.9946$   $\text{Chi}^2:0.002183$   $r:1$   $\square\square$   $x = \dot{\gamma} [1/s]$ ,  $y = \tau [Pa]$   
 Newton (18) :  $\eta:0.003322$   $\text{Chi}^2:0.01005$   $r:0.9999$   $\square\square$   $x = \dot{\gamma} [1/s]$ ,  $y = \tau [Pa]$   
 Bingham (19) :  $\tau_0:0.01799$   $\eta_p:0.003328$   $\text{Chi}^2:2.238$   $r:0.9869$   $\square\square$   $x = \dot{\gamma} [1/s]$ ,  $y = \tau [Pa]$   
 Newton (20) :  $\eta:0.003381$   $\text{Chi}^2:2.265$   $r:0.9867$   $\square\square$   $x = \dot{\gamma} [1/s]$ ,  $y = \tau [Pa]$

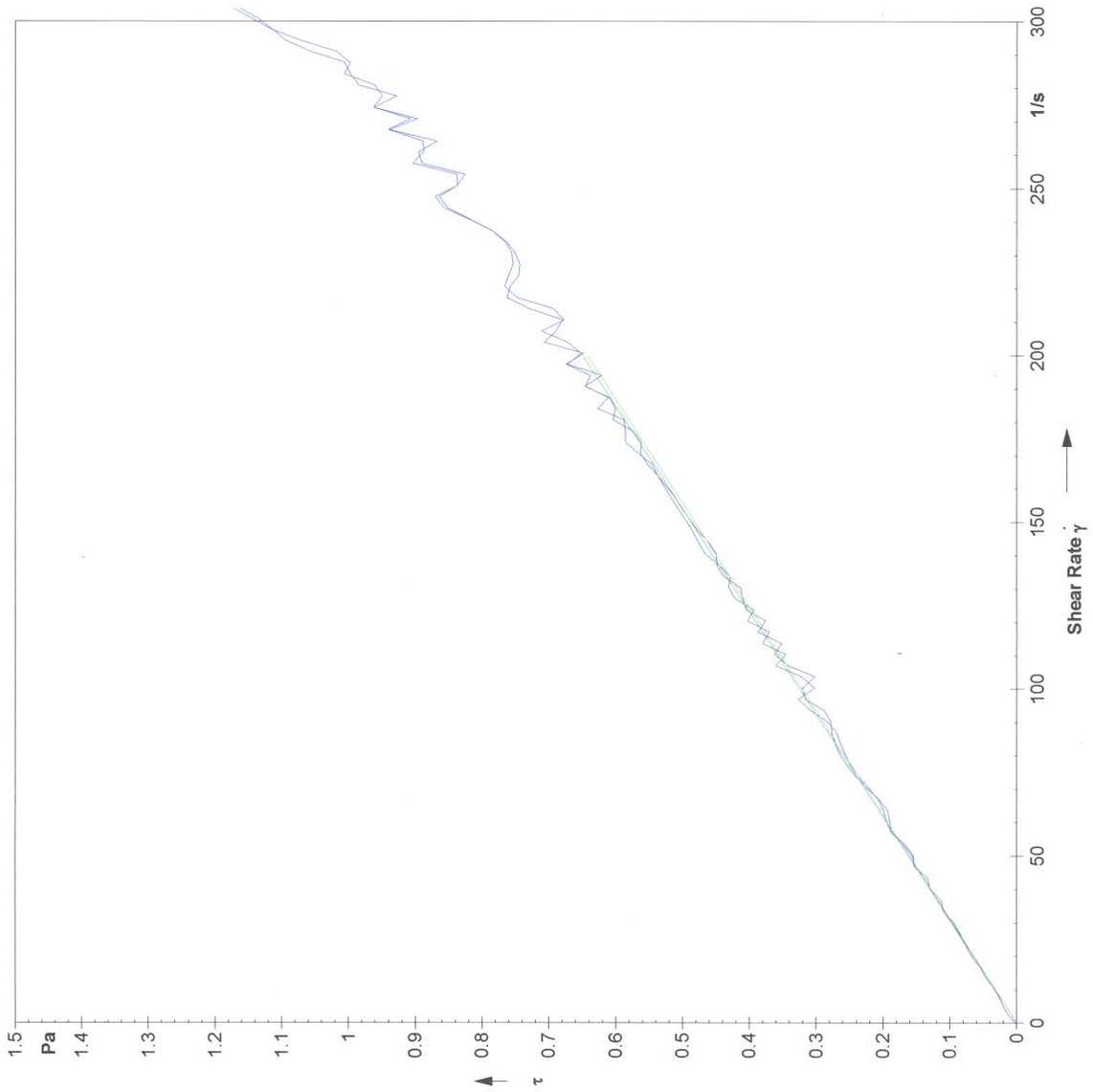


1: C:\Rheology Results\53019-M12\080913\080913\_PEP Feed 30 day Sample-1.rwd  
 Company / Operator: PNNL / Xiao-Ying Yu  
 Date / Time / Version: 13.09.2008 / 8:51:41 AM / RheoWin Pro 296  
 Substance / Sample no: PEP Feed 30 day Sample / 080913 PEP Feed 30 day Sample-1  
 Bingham (1) :  $\tau_0: -0.007507$   $\eta_0: 0.003191$   $\text{Chi}^2: 0.004567$   $r: 1$   $\square\square$   $x = \dot{\gamma}$  [ 1/s ] ,  $y = \tau$  [ Pa ]  
 Herschel-Bulkley (2) :  $\tau_0: 0.002054$   $K: 0.002798$   $n: 1.021$   $\text{Chi}^2: 0.001902$   $r: 1$   $\square\square$   $x = \dot{\gamma}$  [ 1/s ] ,  $y = \tau$  [ Pa ]  
 Newton (3) :  $\eta: 0.003166$   $\text{Chi}^2: 0.008911$   $r: 0.9999$   $\square\square$   $x = \dot{\gamma}$  [ 1/s ] ,  $y = \tau$  [ Pa ]  
 Bingham (4) :  $\tau_0: 0.011173$   $\eta_0: 0.003181$   $\text{Chi}^2: 0.2202$   $r: 0.9986$   $\square\square$   $x = \dot{\gamma}$  [ 1/s ] ,  $y = \tau$  [ Pa ]  
 Newton (5) :  $\eta: 0.003215$   $\text{Chi}^2: 0.2318$   $r: 0.9985$   $\square\square$   $x = \dot{\gamma}$  [ 1/s ] ,  $y = \tau$  [ Pa ]

Shear Stress vs Shear Rate



Shear Stress vs Shear Rate



090203\_SAT\_15\_Fresh 1  
 CC27-SN13634; d=0 mm  
 $\tau$  Shear Stress

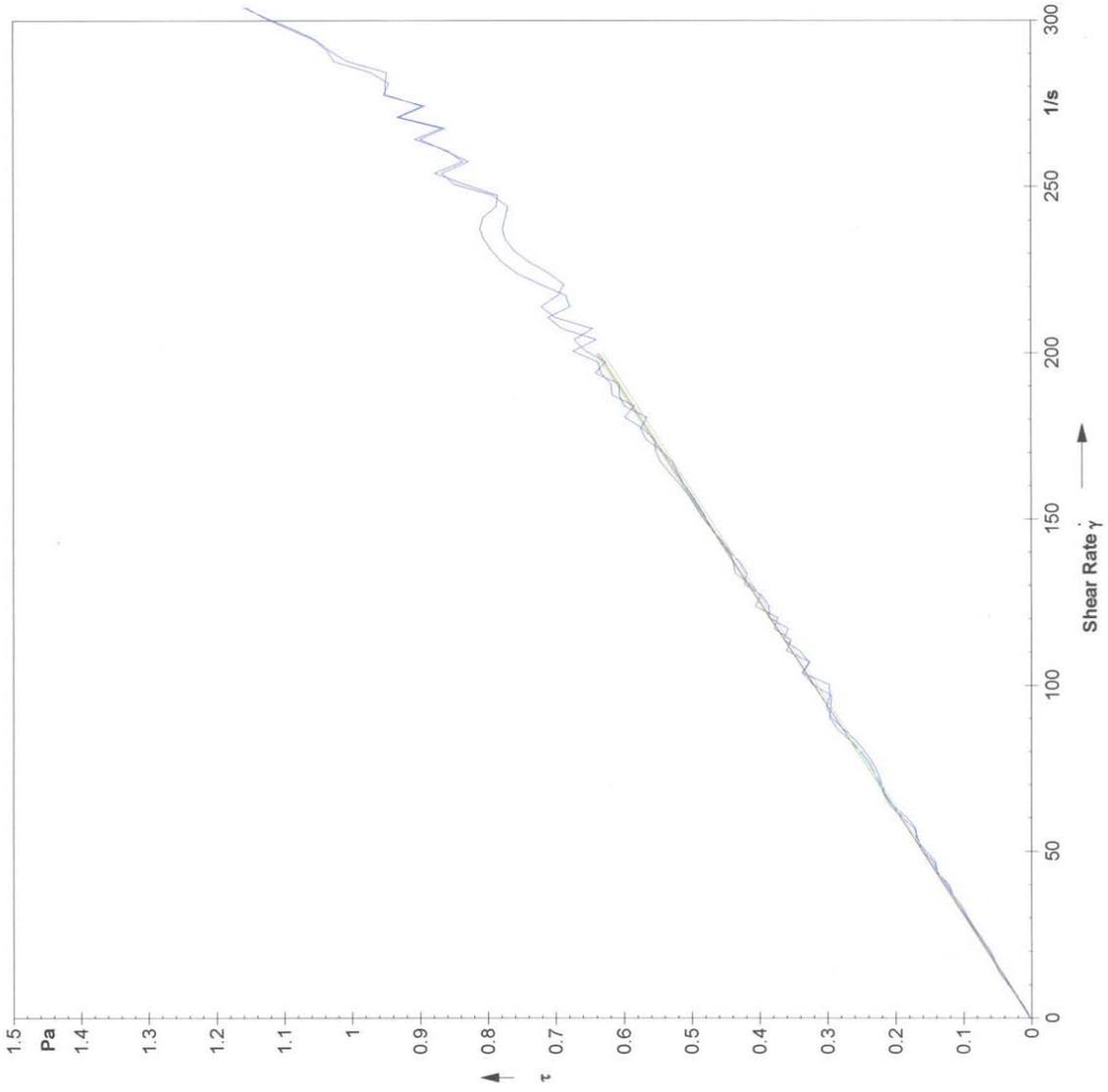
090203\_SAT\_15\_Fresh 1 Bingham | UP 1  
 $\tau_0 = ---$ ;  $\eta_{inf} = 0.0032489$  Pa s  
 $\tau$  Shear Stress

090203\_SAT\_15\_Fresh 1 Newton | UP 1  
 $\eta = 0.0032471$  Pa s  
 $\tau$  Shear Stress

090203\_SAT\_15\_Fresh 1 Bingham | Down 1  
 $\tau_0 = ---$ ;  $\eta_{inf} = 0.0032173$  Pa s  
 $\tau$  Shear Stress

090203\_SAT\_15\_Fresh 1 Newton | Down 1  
 $\eta = 0.0032081$  Pa s  
 $\tau$  Shear Stress

Shear Stress vs Shear Rate



090203\_SAT\_30\_Fresh 1  
 CC27-SN13634; d=0 mm  
 —  $\tau$  Shear Stress  
 090203\_SAT\_30\_Fresh 1 Bingham | UP 1  
 $\tau_{u0} = 0.0018696 \text{ Pa}$ ;  $\eta_{inf} = 0.0031722 \text{ Pa s}$   
 —  $\tau$  Shear Stress  
 090203\_SAT\_30\_Fresh 1 Newton | UP 1  
 $\eta = 0.0032022 \text{ Pa s}$   
 —  $\tau$  Shear Stress  
 090203\_SAT\_30\_Fresh 1 Bingham | Down 1  
 $\tau_{u0} = -$ ;  $\eta_{inf} = 0.0031513 \text{ Pa s}$   
 —  $\tau$  Shear Stress  
 090203\_SAT\_30\_Fresh 1 Newton | Down 1  
 $\eta = 0.0031496 \text{ Pa s}$   
 —  $\tau$  Shear Stress

## **Appendix C**

### **Chemical Analysis**

## Appendix C: Chemical Analysis

**Table C.1.** ICP-AES Analysis of 250-Gallon Tote Stored Supernate (mg/kg)

Element	TST-14	TST-21	TST-28	TST-35	TST-42	TST-45
Al	2,820	2,930	3,030	2,960	2,900	2,820
Sb	<0.417	<0.344	<0.359	<0.346	<0.497	<0.445
As	0.321	0.396	0.275	0.359	0.180	0.121
Ba	<0.104	<0.0860	<0.0899	<0.0865	<0.124	<0.111
Be	<0.104	<0.0860	<0.0899	<0.0865	<0.124	<0.111
Bi	<0.417	<0.344	<0.359	<0.346	<0.497	<0.445
B	4.96	5.15	5.24	5.18	5.17	4.98
Cd	<0.104	<0.0860	<0.0899	<0.0865	<0.124	<0.111
Ca	<5.21	<4.30	<4.49	<4.33	<6.21	<5.56
Cr	0.979	1.03	1.09	1.03	1.11	1.02
Co	<0.104	<0.0860	<0.0899	<0.0865	<0.124	<0.111
Cu	<0.104	<0.0860	<0.0899	<0.0865	<0.124	<0.111
Fe	<1.04	<0.860	1.09	<0.865	2.40	<1.11
La	<0.104	<0.0860	<0.0899	<0.0865	0.131	<0.111
Pb	<0.104	<0.0860	<0.0899	<0.0865	<0.124	<0.111
Li	<0.209	<0.172	<0.180	<0.173	<0.248	<0.223
Mg	<1.04	<0.860	<0.899	<0.865	<1.24	<1.11
Mn	<0.104	<0.0860	<0.0899	<0.0865	<0.124	<0.111
Mo	<0.104	<0.0860	<0.0899	<0.0865	0.136	<0.111
Ni	<0.104	<0.0860	<0.0899	<0.0865	<0.124	<0.111
Pd	<0.209	<0.172	<0.180	<0.173	<0.248	<0.223
P	1,640	1,740	1,790	1,760	1,730	1,680
K	643	693	695	693	675	651
Se	<0.209	<0.172	<0.180	<0.173	<0.248	<0.223
Si	1.69	1.51	1.76	1.57	2.01	1.59
Ag	<0.104	<0.0860	<0.0899	<0.0865	0.132	<0.111
Na	85,100	81,200	87,000	88,400	89,400	85,600
Sr	<0.104	<0.0860	<0.0899	<0.0865	<0.124	<0.111
S	4,360	4,570	4,700	4,560	4,570	4,410
Tl	<0.417	<0.344	<0.359	<0.346	<0.497	<0.445
Th	<0.417	<0.344	<0.359	<0.346	<0.497	<0.445
Sn	<0.104	<0.0860	<0.0899	<0.0865	<0.124	<0.111
Ti	<0.104	<0.0860	<0.0899	<0.0865	<0.124	<0.111
W	<0.209	<0.172	<0.180	<0.173	<0.248	<0.223
U	<5.21	<4.30	<4.49	<4.33	<6.21	<5.56
V	<0.104	<0.0860	<0.0899	<0.0865	<0.124	<0.111
Y	<0.104	<0.0860	<0.0899	<0.0865	<0.124	<0.111
Zn	<0.104	<0.0860	<0.0899	<0.0865	0.221	<0.111
Zr	0.786	0.811	0.882	0.804	0.932	0.813

**Table C.2.** IC Analysis of 250-Gallon Tote Stored Supernate (mg/kg)

<b>Element</b>	<b>TST-14</b>	<b>TST-21</b>	<b>TST-28</b>	<b>TST-35</b>	<b>TST-42</b>	<b>TST-45</b>
Nitrate-N	19,400	19,600	19,800	19,900	18,900	19,200
Nitrite-N	5,690	5,750	5,790	5,830	5,590	5,660
Oxalate	701	764	751	733	727	725
Phosphate-P	1,860	1,890	1,880	1,880	1,810	1,830
Sulfate	15,600	15,700	15,800	15,900	15,400	15,600

## Distribution

**No. of  
Copies**

**No. of  
Copies**

**OFFSITE**

**ONSITE**

7	<u>Pacific Northwest National Laboratory</u>	
	W. C. Buchmiller	K6-24
	K. J. Cantrell	K6-81
	D. E. Kurath	P7-28
	R. A. Peterson	P7-22
	D. E. Rinehart	K6-24
	R. L. Russell	K6-24
	Project File (1)	P7-28
2	<u>Bechtel National, Inc.</u>	
	WTP R&T Docs (2)	H4-02