This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-96SR18500 with the U. S. Department of Energy.

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 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. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

This report has been reproduced directly from the best available copy.

Available for sale to the public, in paper, from: U.S. Department of Commerce, National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161,
phone: (800) 553-6847,
fax: (703) 605-6900
email: orders@ntis.fedworld.gov
online ordering: http://www.ntis.gov/help/index.asp

Available electronically at http://www.osti.gov/bridge
Available for a processing fee to U.S. Department of Energy and its contractors, in paper, from: U.S. Department of Energy, Office of Scientific and Technical Information, P.O. Box 62, Oak Ridge, TN 37831-0062,
phone: (865)576-8401,
fax: (865)576-5728
email: reports@adonis.osti.gov

WSRC-TR-2003-00390, REVISION 0
SRT-RPP-2003-00185, REVISION 0

Key Words:
Rheology, Physical Properties

Retention: Permanent

Key WTP R&T References:
Test Specification:
24590-LAW-TSP-RT-01-024, Rev. 0
Test Plan: WSRC-TR-2002-00153
SRT-RPP-2002-00047, Rev. 0
Test Exceptions: 24590-WTP-TEF-RT-03-005
R&T Focus Area: Vitrification
Test Scoping Statement(s): S-62

RHEOLOGICAL AND PHYSICAL PROPERTIES OF HANFORD, RADIOACTIVE LAW AZ-102 PRETREATED WASTE AND MELTER FEED (U)

E. K. Hansen, 999-W
C. L. Crawford, 773-41A

NOVEMBER, 2003

Westinghouse Savannah River Company
Savannah River Site
Aiken, SC 29808

Prepared for the U.S. Department of Energy Under Contract Number DE-AC09-96SR18500

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

ACRONYMS

ITS	Immobilization Technology Section
LAW	Low Activity Waste
MFPV	Melter Feed Preparation Vessel
NIST	National Institute of Standards and Technology
N/A	Not Applicable
N/M	Not Measured
PSD	Particle Size Distribution
QA	Quality Assurance
QAPjP	Quality Assurance Project Plan
RPD	Relative Percent Difference
RPP	River Protection Project
R&T	Research and Technology
SRS	Savannah River Site
SRTC	Savannah River Technology Center
Stdev	Standard Deviation
VSL	Vitreous State Laboratory
WTP	Waste Treatment Plant

DEFINITIONS

Variable	Definition	Units
Apparent viscosity	The measured shear stress divided by the measured shear rate.	Pa·s cP
Density	Mass per unit volume.	g/mL
Flow curve/rheogram	Plot of shear stress versus shear rate.	N/A
Interstitial solution	Solution contained between suspended, settled, or centrifuged solids.	N/A
Newtonian Fluid	A fluid whose apparent viscosity is independent of shear rate.	N/A
Non-Newtonian Fluid	A fluid whose apparent viscosity varies with shear rate.	N/A
Settled solids shear strength	The maximum shear stress as determined by the vane method for a slurry sample that is allowed to settle for a specified amount of time.	Pa
Solution	A liquid phase that can contain soluble solids.	grams
Slurry	A mixture insoluble solids and solution	grams
Supernatant Liquid	A liquid phase overlying material deposited by settling, precipitation, or centrifugation.	grams
Solids Settling Rate	Rate at which solids in a homogenized sample settle. The change in the settled solids height as a function of time.	cm/sec
vol% centrifuged solids	The volume of the solids layer that separates from the bulk slurry after 1 hour of centrifugation at 1000 gravities divided by the total sample volume on a percentage basis. These centrifuged solids will contain interstitial solution.	vol%
vol% settled solids	The percentage of the volume of the slurry sample that the settled solids occupy after settling for 72 hours under one gravity. These settled solids will contain interstitial solution.	vol%
wt% centrifuged solids	The mass of the solids layer that separates from the bulk slurry after 1 hour of centrifugation at 1000 gravities divided by the total bulk slurry sample mass on a percentage basis. These centrifuged solids will contain interstitial solution.	wt%
wt% oven dried solids	The percent mass of the centrifuged solids remaining after removing volatiles including free water by drying at 105 + 5°C for 24 hours.	wt%
wt% settled solids	The percentage (mass basis) of settled solids present in the sample. Calculated by dividing the mass of the settled solids by the mass of sample.	wt%
wt% soluble solids in supernatant	Calculated by dividing the mass of the dried supernatant by the mass of the supernatant prior to drying.	wt%
wt% total oxides	The percentage of the mass of the bulk sample that remains after converting all non-volatile elements to oxides. Dried slurry calcined at 1050°C ± 50°C for 1 hour.	wt%
wt% total solids	The percentage of the mass of dried solids divided by the mass of the slurry.	wt%
wt% undissolved solids	Calculated by dividing the calculated mass of the undissolved solids by the mass of the bulk solids.	wt%
Yield Stress	The minimum stress required to initiate flow. Determined by fitting measured flow curve using non-Newtonian rheological models.	Pa

This page intentionally left blank.

ABSTRACT

The physical and rheological properties, pH, and particle size distribution (PSD) of a radioactive Hanford tank sample, 241-AZ-102 (Envelope B) pretreated by the reference River Protection Program (RPP) Waste Treatment Plant (WTP) pretreatment process at SRTC [Ref. 5] were measured. The pretreated waste was received at a concentration of 4.38M Na and then diluted and blended with glass former chemicals (GFCs) to make up the corresponding melter feeds. The low activity waste (LAW) pretreated wastes at two sodium molarities of 1.0M Na and 1.3M Na [Ref. 2] were characterized first and then their respective melter feeds were characterized after the addition of GFCs. As expected, the 1.0M Na and 1.3M Na pretreated wastes have similar properties due to the small change in sodium molarity. The 1.0M Na melter feed was characterized after 3 days and 7 days of mixing which showed no significant differences in properties. This indicates that the 1.0M Na melter feed is fairly stable at this sodium molarity.

The following technical issues were observed in the measured rheological properties of the melter feeds:

- Settling behavior of the melter feeds impacted the flow curves.
- Melter feeds are heterogeneous.
- Settled solids shear stress for the 1.3M Na melter feed exceeded the maximum operating shear strength limit (625Pa – Ref. 21).
- Settled solids shear strength increased as a function of measurement depth, indicating that a shear strength gradient was observed in the 1.0M Na melter feed.
- Settled solids in the 1.0M Na melter feed were observed to build strength within 30 minutes when not agitated.

This page intentionally left blank.

1.0 SUMMARY OF TESTING

1.1 OBJECTIVES

The objective of this task was to measure and report the physical and rheolgocical properties, pH, and PSD of a radioactive Hanford tank sample, 241-AZ-102 (Envelope B) pretreated by the reference RPP-WTP pretreatment process [Ref. 5]. The pretreated LAW waste and melter feed samples were characterized at two different sodium molarities in accordance with the RPP-WTP Research and Technology (R&T) guidelines for characterization [Ref. 1]. The initial LAW AZ-102 pretreated waste was received at a concentration of 4.38M Na. This pretreated waste was then diluted to two different sodium molarities of 1.0M Na and 1.3M Na. The LAW 1.0M Na and 1.3M Na pretreated wastes were characterized and then blended with RPP-WTP R&T- approved GFCs [Ref. 4] to make melter feeds. The LAW 1.0M Na melter feed was characterized after 3 days and 7 days of continuous mixing and the LAW 1.3M Na melter feed was characterized after 1 day of continuous mixing. All characterization work as specified by WTP R&T was performed [Ref. 2]. A more detailed description of the purpose and objectives of this task is provided in section 3.1.

1.2 CONDUCT OF TESTING

The radioactive1.0M Na and 1.3M Na LAW AZ-102 pretreated wastes were characterized for:

- density
- weight percent (wt%) total solids, and wt% oxide solids
- pH
- viscosity

The density was obtained using a graduated cone; the wt% data using an oven/furnace and weighing balances; the pH using pH probes; and the viscosity by curve fitting the resulting flow curve as a Newtonian fluid.

The LAW AZ-102 pretreated wastes were blended with RPP-WTP R&T approved GFCs [Ref. 4] to make melter feed. Laboratory scale mixing equipment was not determined nor scaled based on proposed LAW melter feed preparation vessel (MFPV) design/operating conditions. Mixing conditions were set by SRTC such that the condition of mixing (agitator speed) provided a well mixed product based on visual observation.

The LAW AZ-102 melter feeds were characterized for:

- settling rates
- total density, supernatant density, settled solids density, and centrifuged solids density
- wt% centrifuged solids, wt% settled solids, wt% soluble solids in supernatant, wt% total solids, wt% oven dried solids, wt% undissolved solids, and wt% total oxides
- volume percent (vol%) settled solids and vol% centrifuged solids
- pH
- rheology (flow curves and shear vane measurements)
- particle size distribution (PSD)

The settling data was obtained using graduated centrifuge cones. The density data was obtained using the graduated centrifuge cone and graduated cylinder; the wt% data using an oven/furnace and weighing balances; the pH using pH probes; viscosity of the supernatant by curve fitting the resulting flow curves as a Newtonian fluid; reporting the largest stress measurement using the vane as the settled shear strength; and the PSD using laser scatter method.

The composition of the LAW pretreated wastes and melter feeds are to be calculated based on the original composition of the LAW 4.38M Na pretreated waste [Ref. 5] and the composition of the individual GFCs [Ref. 4].

1.3 RESULTS AND PERFORMANCE AGAINST OBJECTIVES

All the objectives in this task were met and are described in detail in section 3.1. Any discrepancies with respect to the objectives stated in reference 2 are also stated in section 3.1. These discrepancies did not impact the deliverables requested in reference 2.

The physical properties (density and wt% solids) results for the LAW AZ-102 1.0M Na and 1.3M Na pretreated wastes are presented in Table 3-9. The pH is presented in Table 3-12 and the viscosity in Figure 3-6 and Figure 3-7. Due to the small change in sodium molarity, the viscosities of the resulting pretreated wastes are similar.

The physical properties (density, wt% solids and vol% solids) results for the LAW AZ-102 melter feeds are presented in Table 3-10. The melter feed flow curves are shown in Figure 3-8 through Figure 3-10 . These melter feeds are settling slurries, such that required flow properties could not be properly quantified using existing bench top rheological instrumentation at SRTC. The rheological responses of these slurries with respect to the flow curve measurements are explained in Table 3-17. The melter feed flow curves were not fit to any of the rheological models recommended in reference 1, because these rheological models assume the fluid is non-Newtonian. The melter feed supernatants were analyzed as Newtonian fluids and the results are shown in Table 3-18. There are measurable differences in the wt% soluble solids, densities, and viscosities of the pretreated wastes and melter feed supernatants, indicating some of the dry GFC solids had dissolved.

Even though the melter feed flow curves could not be analyzed using non-Newtonian rheological models, the viscosity and density of the melter feed supernatant were measured and the PSD and density of GFC particles are known such that these properties could potentially be used to estimate the required flow velocities and mixing parameters required to prevent settling using heterogeneous slurry correlations. These heterogeneous slurry correlations and their applicability to the RPP-WTP design/operations must be thoroughly understood prior to use.

The settled solids shear strengths of the AZ-102 melter feeds are shown in Figure 3-11 and observations reported in Table 3-20. The 1.3M Na melter feed settled solids shear strength exceeded the design basis value of 625 Pa. The particle size distribution is provided in Table 3-22 and shows slight differences between the 1.0M Na and 1.3M Na melter feed, with the 1.3M Na having slightly larger particles.

The 1.0M Na melter feeds has essentially the same physical, rheological, PSD, and pH properties after 3 days and 7 days of continuous mixing. The settled solids shear strengths were different between the two samples, but this could be due to the amount of time the samples were allowed to settle undisturbed prior to the vane measurements, how the solids settled out and the location of the measurement in the samples. There was also a settled solids shear strength gradient observed on one of the 1.0M Na samples where two different depths were measured and the settled solids shear strength increased as the depth increased. A more detailed description of the settled solids shear strength results are provided in section 3.6.6.

The calculated chemical composition of the LAW pretreated wastes and melter feeds are provided in section 3.8.

1.4 QUALITY REQUIREMENTS

This work was conducted in accordance with the RPP-WTP QA requirements specified for work conducted by SRTC as identified in DOE IWO M0SRLE60. Researchers followed the SRTC QA program, which has been approved by WTP, and the WSRC QA Management Plan (WSRC-RP-92-225). The program applied the appropriate QA requirements for this task, as indicated by the QA Plan Checklist in section IX of the Task Technical and Quality Assurance Plan [Ref. 3].

Analytical sample labeling and tracking complied with established procedures (WSRC Manual L1, Procedure 7.15). The Immobilization Technology Section (ITS) conducted all analyses using the routine level QA program. Calibrated measuring equipment was utilized. The Task Technical and Quality Assurance Plan [Ref. 3] provided the quality requirements for this work. NQA-1 1989, Basic and Supplementary Requirements and NQA-2a 1990, Part 2.7 were applied as appropriate,

1.5 ISSUES

The section discusses the potential impact that the measured properties will have on the known design bases, design or operating conditions of the RPP-WTP vitrification plant.

The 1.3M Na melter feed settled solids exceeded the maximum operating shear strength limit of 625 Pa [Ref. 21]. The agitator start up torque planned in the design is 1.5 times this maximum settled solids shear strength value [Ref. 25]. This maximum operating shear strength was calculated using the maximum horsepower and bottom blade design of the Defense Waste Processing Facility (DWPF) agitators in reference 21 and is being compared with to the project’s actual physical and rheological properties data as it becomes available. Additionally, no supporting calculation exists to determine if the agitator, given 625 Pa shear strength, can completely re-homogenize the mixing vessel. Additional issues related to the maximum shear strength are provided in section 3.9.1.

The lower consistency limit for LAW melter feed is 0.4 centipoises (cP) [Ref. 21]. The results of the melter feed settling tests indicate that there will be settling issues with these slurries. Calculations nor physical testing for determining the minimum deposition velocity or a velocity required to produce a homogenous mixture in the pipelines has been performed. Additional issues related to the lower viscosity limit are provided in section 3.9.2.

The issue of settled solids, when measuring the settled solids shear strength can be both a function of time and the level of settled solids. The present bench scale tests do not investigate these issues. Additionally, the viscosity of the carrier fluid (supernatant) of the LAW melter feed should be analyzed. This information may be useful in engineering calculations if the resulting melter feeds are considered as settling slurries. Additional information is provided in section 3.9.3.

2.0 CD-ROM ENCLOSURES

The following EXCEL worksheet is enclosed in the attached CD. The EXCEL worksheet contains the raw rheological data as required per Table 12, Sheet #4 [Ref. 1]. Table 12 sheets #1, #2, #3 and #5 are attached to this document in Appendix C. EXCEL worksheet provided in the CD is labeled LAW AZ-102 Radioactive Table 12, Sheet # 4.

This page intentionally left blank.

3.0 DISCUSSION

3.1 PURPOSE AND OBJECTIVES

The purpose and objective of this task was to measure and report the physical and rheological properties, pH, and PSD of a radioactive Hanford tank sample, 241-AZ-102 (Envelope B) pretreated by the reference RPP-WTP pretreatment process at SRTC [Ref. 5]. The LAW pretreated wastes and melter feeds were characterized at two different sodium molarities in accordance with RPP-WTP R&T guidelines for characterization [Ref. 1]. The initial AZ-102 pretreated waste was received at a concentration of 4.38M Na. This pretreated waste was then diluted to two different sodium molarities of 1.0M Na and 1.3M Na. After the 1.0M Na and 1.3M Na pretreated wastes were characterized, these waste streams were blended with the project approved GFCs [Ref. 4] to make melter feeds. The physical and rheological properties and pH of the resulting melter feeds were measured using the methods outlined in the RPP-WTP guidelines [Ref. 1], unless otherwise stated in this document.

The properties and conditions required for measurement of the LAW AZ-102 1.0M Na and 1.3M Na pretreated wastes and melter feeds are listed in Table 3-1. These properties and conditions are specified in the R&T test exception [Ref. 2]. The PSD was performed in accordance with reference 2.

Table 3-1. Objectives from Test Exception [Ref. 2]

Measurements and Conditions	1.3M Na		1.0 M Na		
	Pretreated waste	Melter Feed	Pretreated waste	Melter Feed (mixing times)	
				(24 hrs)**	(7 days)
Flow Curves @ 25°C	X	X	X	X	X
Vane @ room temp. – Settled at least 2 days	N/A	X	N/A	X	X
Flow Curve of supernatant @ 25°C*	N/A	X	N/A	X	X
pH @ room temperature	X	X	X	X	X
Total solids	X	N/A	X	N/A	N/A
Total oxide	X	N/A	X	N/A	N/A
Solids analysis via 24590-WTP-GPG-RTD-001	N/A	X	N/A	X	X
Settling Test @ room temperature	N/A	X	N/A	X	X
Particle size distribution- sample diluted with NaOH solution***	N/A	X	N/A	X	X
Approved GFCs Utilization	N/A	X	N/A	X	X
Approved GFC batch sheet	N/A	X	N/A	X	X
Mixing at room temperature	N/A	X	N/A	X	X
Reporting of Results in WTP format	X	X	X	X	X

* SRTC added this measurement

** Actual measurements were performed after 3 days of mixing. See discrepancies in this section.

*** An LAW AZ-102 pretreated waste simulant was used in place of NaOH solution. See discrepancies in this section.

N/A = Not Applicable

The composition of the LAW pretreated wastes and melter feeds were calculated based on the original composition of the LAW AZ-102 4.38M Na pretreated waste [Ref. 5] and the composition of the individual GFCs [Ref. 4].

The discrepancies in this task were:

- The 24-hr measurement after mixing the LAW 1.0M Na AZ-102 melter feed [Ref. 2] did not occur until after 3 days of continuous mixing. This was due to an unforeseen radiological issue that closed the access to the laboratories for 2 days. This radiological issue was not related to this sample. In this document, the measurement date will reflect 3 days of mixing.
- NaOH solution was specified in the test exception [Ref. 2] as the carrier fluid for PSD measurements. An LAW AZ-102 pretreated waste simulant was used in place of the NaOH solution. The LAW AZ-102 pretreated waste simulant was diluted to the calculated Na molarity of the melter feeds and then filtered through a 0.2 micron filter to remove particulates prior to use.
- The wt% solids and wt% total oxide for the 1.0M and 1.3M Na pretreated wastes were measured on the initial dilution of the 4.38M Na pretreated waste. Na analysis of these initial pretreated waste indicated they were low in Na concentration. The samples were evaporated and the wt% total solids and wt% total oxide for the 1.0M Na and 1.3M Na pretreated wastes were calculated based on the initial analyses of the dilute wastes. Additional details are provided in section 3.3.1.
- The density of the pretreated waste was not measured using a graduated cylinder [Ref.1]. Densities of the LAW AZ-102 pretreated wastes were determined as described in section 3.4.1.1.
- Sonication of the melter feed was not performed since the particle size analyzer does not have sonication capabilities. Details of the PSD technique used are provided in section 3.7.

All properties listed in Table 3-1 were obtained using the methods outlined in the RPP-WTP guidelines document [Ref. 1], hence meeting the objectives of this task. All work was performed in a warm radiological hood.

3.2 GLASS FORMER CHEMICALS AND AZ-102 BATCH SHEET

The GFCs utilized in this task are listed in Table 3-2 and are those approved by RPP-WTP R&T [Ref. 4].

Table 3-2. Glass Former Chemicals Utilized

Oxide Added	Mineral	Grade	Vendor
Al_2O_3	Kyanite - Al_2O_2-SiO_2	Raw –325 Mesh	Kyanite Mining Corp.
B_2O_3	Boric Acid – H_3BO_3	Technical Grade-Granular	U.S. Borax
Na_2O	Na_2CO_3 Anhydrous	Dense Soda Ash	Solvay Minerals
CaO	Wollastonite – $CaSiO_3$	NYADM325	NYCO
Fe_2O_3	Fe_2O_3	5001	Prince Mfg. Co.
Li_2O	Li_2CO_3	Technical Grade	Chemettal-Foote
MgO	Olivine – $MgSiO_3$	#180	Unimin Corp.
SiO_2	SiO_2	SCS-75	U.S. Silica
TiO_2	Rutile TiO_2/Fe_2O_3	Air Floated Rutile 94	Chemalloy Co.
ZnO	ZnO	Kadox 920	Zinc Corp America
ZrO_2	$ZrSiO_4$	Zircon Flour	American Mineral Inc.

The chemical composition of the LAW AZ-102 pre-treated waste was provided to Vitreous State Laboratories (VSL). In return, VSL then provided a GFC blend composition spreadsheet, LAWB96, as shown in Table 3-3. SRTC determined the mass of GFCs and the weight percent distribution of each of the GFCs for 1 mole of sodium. The results of the wt% GFCs in the blend are shown in Table 3-3. These calculated values were used to determine the GFCs necessary to support the melter feed mixing tasks in section 3.3.2.

Table 3-3. VSL GFC Batch Sheet LAWB98 for SRTC LAW AZ102 Pre-Treated Feed

Recipe using AZ102 SRTC 2001 Waste for glass: LAWB96
Formulation for feed rheology testing based on glass used for Duramelter testing of waste AZ-102

Envelope Constituents	SRTC AZ-102 mg/L concentrate email 5/24/02	Simulant Molarity	GLASS Oxides	AZ102 in glass @ 5% Na_2O	Glass Former Mix	**LAWB96** this target for AZ102	Additives this sample	Source in Additives	Assay	Ratio	Target Weight (g) Additives	other oxides present			wt. % in GFC
		M	Loading	3.55%	100%	3.55%	96.45%					% Al_2O_3	% Fe_2O_3	%SiO_2	
Al	798.0	0.0296	Al_2O_3	0.0333	6.41	6.2155	6.182	Kyanite (Al_2SiO_5) 325 Mesh	0.990	0.570	118.19	0.00%	0.00%	40.67%	9.481%
B	4.6	-----------	B_2O_3	0.0003	10.42	10.0500	10.050	H_3BO_3 (Technical Granular)	0.986	0.565	197.41	0.00%	0.00%	0.00%	15.835%
Ca	67.0	0.0017	CaO	0.0021	7.04	6.7919	6.790	Wollanstonite NYAD 325 Mesh	0.993	0.475	157.58	0.00%	0.40%	51.00%	12.640%
Cr	1018.1	0.0196	Cr_2O_3	0.0329	0.00	0.0329	0.000								
Cs	0.0	0.0000	Cs_2O	0.0000	0.00	0.0000	0.000								
Fe	5.5	0.0001	Fe_2O_3	0.0002	5.50	5.3047	5.305	Fe_2O_3 (5001-Prince)	0.970	1.000	53.73	1.50%	0.00%	0.00%	4.310%
K	4555.0	0.1165	K_2O	0.1212106	0.00	0.1212	0.000								
Li	2.0	-----------	Li_2O	0.0001	4.47	4.3112	4.311	Li2CO3 (Chemetall Foote Co. Tech. gr.)	0.994	0.401	118.34	0.00%	0.00%	0.00%	9.493%
Mg	3.0	-----------	MgO	0.0001	3.09	2.9803	2.980	Olivine (Mg2SiO4) 325 Mesh (#180)	0.990	0.480	68.64	0.00%	7.68%	42.52%	5.506%
Mn	0.9	0.0000	MnO_2	0.0000	0.00	0.0000	0.000								
Na	95038.5	4.1339	Na_2O	2.83	2.59	5.3280	2.498	Na_2CO_3 (anhydrous dense soda ash)	0.994	0.584	47.13	0.00%	0.00%	0.00%	3.781%
Ni	6.0	0.0001	NiO	0.0002	0.00	0.0002	0.000								
Si	56.0	0.0020	SiO_2	0.0026	50.68	48.8816	48.879	SiO_2 (Sil-co-Sil 75)	0.997	1.000	362.52	0.00%	0.00%	0.00%	29.079%
Ti	2.0	-----------	TiO_2	0.0001	1.45	1.3985	1.398	TiO_2 (Rutile Airfloated)	0.932	1.000	16.43	0.00%	0.71%	2.20%	1.318%
Zn	2.7	-----------	ZnO	0.0001	5.05	4.8706	4.871	ZnO (Kadox-920)	0.999	1.000	53.37	0.00%	0.00%	0.00%	4.281%
Zr	4.0	0.00004	ZrO2	0.0001	3.30	3.1828	3.183	Zircon $ZrSiO_4$ (Flour) Mesh 325	0.990	0.660	53.32	0.31%	0.00%	32.25%	4.277%
Cl	200.0	0.0056	Cl	0.0044	0.00	0.0044	0.000								
F	1370.0	0.0721	F	0.0303	0.00	0.0303	0.000								
PO_4	704.0	0.0074	P_2O_5	0.0116	0.00	0.0116	0.000	Total Sodium Moles			1	moles			
SO_4 total	26300.0	0.2738	SO_3	0.4842	0.00	0.4842	0.000	Expected Glass yield			1094.70	g			
NO_2	49440.0	1.0748	NO_2					Sum of Additives (g)			1246.6622	g			
NO_3	27305.0	0.4404	NO_3					Sugar as added reductant (decreased for TOC)			None - See note below				
CO_3	9396.0	0.1566	CO_3					Volume of Simulant AZ-102 SRTC Envelope B Recipe:			1000.0	ml			
NH_3	0.0	0.0000	NH_3					Water Addition required to dilute to recommended 1.3 Molar:			0.0	ml			
OH	0.0	0.0000	OH					Weight of AZ102 sample used			1161.0	g			
Org.Carbon	19914.0	1.6595	C					density			1.161				
SUM	-----------	-----------	SUM	3.55	100.00	100.00	96.446	Sodium Molarity of original sample			1.00				

VSL uses 12 moles Carbon (1 mole sucrose/342.3g) per 16 Moles NOx in order to mitigate foaming.
13.637 g/L Carbon. This sample already has sufficient TOC

3.3 LAW AZ-102 SAMPLE PREPARATION

3.3.1 Pretreated Waste

A detailed description of the preparation of the radioactive LAW AZ-102 tank sample through the RPP-WTP pretreatment process is part of another test program at SRTC and is provided in another report [Ref. 5]. This report also provides the chemical composition and density of the LAW AZ-102 pretreated waste. The Na molarity, physical properties, and quantity of the original pretreated waste provided for this task is shown in Table 3-4. This 4.38M Na pretreated waste contained precipitated white solids and the solution was yellow in color. The precipitated white solids were easily suspended and homogenized upon slight agitation of the sample. The presence of trace amounts of fine white solids particles in the LAW AZ-102 pretreated waste could have been due to the fact that this concentrated pretreated waste was originally concentrated back in May of 2000 and was stored in a sealed poly bottle at ambient laboratory temperatures for over 3 years prior to the current physical and rheological properties and pH testing. It should be noted that these trace white solids particles were not observed in the original LAW AZ-102 evaporator waste concentrate [Ref. 5]. Further identification and analysis of these precipitated white solids was not within the scope of this task.

Table 3-4 Original Undiluted LAW AZ-102 Pretreated Waste Properties and Quantity

Na Molarity	Density (g/mL)	wt% total solids	Mass of sample (g)
4.38	1.16	25	~104

Targeted Na molarity concentrations of 1.0M Na and 1.3M Na [Ref. 2], mixing conditions, and the type of analyses required are stated in Table 3-1. The amounts of 4.38M Na pretreated waste required for making up the 1.0M Na and 1.3M Na pretreated wastes are shown in Table 3-5. The amount of DI water used to dilute the original waste to the targeted Na molarity values was calculated using the density, Na molarity, and mass of the 4.38M Na pretreated waste used. The pretreated waste was homogenized and analyzed for Na molarity, density, wt% total solids, and wt% total oxide. The initial Na analysis indicated that both the 1.0M Na and 1.3M Na pretreated wastes were below the target sodium concentrations. This could have been due to the uncertainty of the Na analysis, the density of the original 4.38M Na pretreated waste, or the method used to calculate the targeted molarities. A calculated amount of water was evaporated from the samples to obtain the targeted sodium molarities and the results are shown in Table 3-5. Confirmatory samples were analyzed for sodium concentration and the results are shown in Table 3-6. Rheology (flow curves), density, and pH measurements were performed on the final pretreated wastes, with the intent to recover as much of the pretreated waste material as possible to accommodate for the melter feed tests. The wt% total solids and wt% total oxide solids were calculated from the original diluted measurements and are described in section 3.4.1.1. The mass and density of the final LAW AZ-102 1.0M Na and 1.3M Na pretreated wastes available for melter feed work are shown in Table 3-6 . Both the 1.0M Na and 1.3M Na LAW AZ-102 pretreated wastes were a clear yellow fluid with a small quantity (that could not be quantified) of white precipitated solids.

Table 3-5. Diluted LAW Pretreated Waste Makeup

Diluted Pretreated Waste	Mass (grams) of		
	4.38M Na Feed Used	DI water added	DI water evaporated
1.0M Na	62.347	170.893	13.259
1.3M Na	41.574	79.35	21.432

3.3.2 Melter Feed

Given the Na molarity, density, and mass of the pretreated feed, the quantity of each individual GFC was determined using Equation 3-1:

Equation 3-1

$$m_{GFC,i,j} = \frac{\widetilde{M} \cdot M_j \cdot m_j \cdot wt_{GFC,i}}{1000 \cdot \rho_j \cdot 100\%}$$

where: $m_{GFC,i,j}$ = mass of a specific glass former chemical (grams)
$\widetilde{M}$ = Total mass of GFCs per mole of Na (from Table 3-3)
M_j = Na molarity of pretreated waste j (moles Na/Liter)
ρ_j = density of pretreated waste j (g/mL)
m_j = mass of pretreated waste j (g)
$wt_{GFC,i}$ = weight percent of a specific GFC in the GFC batch (from Table 3-3)

The actual quantities of each GFC used for the LAW AZ-102 1.0M and 1.3M Na melter feeds are shown in Table 3-6. For a given melter feed composition, the individual GFCs were weighed and placed into a single large wide mouth plastic bottle. Upon completion of weighing out the GFCs, the bottle was capped and vigorously shaken to provide a homogenous mixture of blended solids.

Laboratory mixing equipment was selected based on the calculated volume of melter feed that would result from the blending of the pretreated waste with the blended GFCs. The mixing equipment used for the LAW AZ-102 Na 1.0M Na and 1.3M Na melter feeds is described in Table 3-8. The plastic caps used for the mixing vessels were predrilled with holes slightly off-center and slightly larger than the diameter of the agitator shaft. The holes were placed slightly off-center to allow for better mixing, since there were no baffles present in any of the mixing vessels.

Table 3-6. Glass Former Used For LAW AZ-102 Melter Feeds

LAW AZ-102 Pretreated Waste Information	Units	1.0M Na	1.3M Na
Batching Sheet Used	N/A	LAWB96	LAWB96
Grams of GFCs per Mole of Na	Grams/mole	1246.3317	1246.3317
Sodium Concentration of Pretreated Waste	Na Molarity	1.02	1.33
Mass of Pretreated Waste	Grams	193.508	88.711
Density of Pretreated Waste	g/mL	1.049	1.073
Volume of Pretreated Waste	mL	184.53	82.68
Moles of sodium of Pretreated Waste	Moles Na	0.1878	0.1100

GFC Distribution					
GFCs		1.0M Na Melter Feed		1.3M Na Melter Feed	
Mineral	Target wt %	Target Mass (g)	Actual Mass Added (g)	Target Mass (g)	Actual Mass Added (g)
Kyanite - Al_2O_2-SiO_2	9.439	22.10	22.102	12.95	12.953
Boric Acid - H_3BO_3	15.839	37.08	37.083	21.72	21.720
Na_2CO_3 Anhydrous	3.782	8.85	8.850	5.19	5.190
Wollastonite – $CaSiO_3$	12.644	29.60	29.602	17.34	17.340
Fe_2O_3	4.311	10.09	10.091	5.91	5.912
Li_2CO_3	9.495	22.23	22.233	13.02	13.020
Olivine – $MgSiO_3$	5.507	12.89	12.890	7.55	7.550
SiO_2	29.105	68.13	68.181	39.92	39.920
Rutile TiO_2/Fe_2O_3	1.318	3.09	3.092	1.81	1.812
ZnO	4.282	10.02	10.022	5.87	5.872
$ZrSiO_4$	4.278	10.01	10.010	5.87	5.870
Total:		**234.09**	**234.156**	**137.15**	**137.159**

First, the pretreated waste was added to the mixing vessel. The agitator impeller was then installed off-center and as close to the bottom of the mixing vessel as possible. The mixer was then started and the initial agitator speed was determined by visually observing for the presence of a vortex, such that the GFC solids could be easily entrained and dispersed by agitation. The GFCs were added manually with a spatula at a rate that could easily be blended into the mixture (based on visual observation). It took approximately 20 to 30 seconds for the mass of GFCs added to become entrained, during which time GFC addition was stopped and the mass of GFCs remaining to be added was recorded. The agitator speed was increased until an adequate vortex was again obtained and this cycle was repeated until all the GFCs were added.

Figure 3-1 shows the LAW AZ-102 1.3M Na melter feed after all of the GFCs were added. The bubbles present were most likely due to air that was entrained during the GFC addition, and not by the vortex itself. The next day all the bubbles were gone. The effect of bubbles generated due to air entrainment during the addition of GFCs could be a factor within the WTP. The bubbles were common to both the 1.0M Na and 1.3M Na melter feeds during GFC addition. After all the GFCs were added, the slurry, agitator shaft/blade, and vessel cap were weighed. This was considered to be the baseline weight of the blended slurry. The addition rate of the GFCs and agitator speeds are shown in Table 3-7.

Bubbles are due to air entrainment encountered during GFC addition.

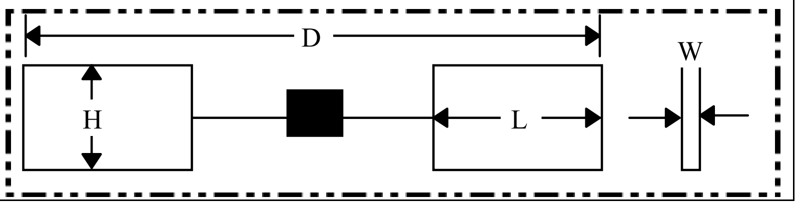
Figure 3-1. GFCs Added to LAW AZ-102 1.3M Na Pre-Treated Feed

The mixing system was reconfigured with the cap in place and the impeller located just off-bottom. The final speed was then adjusted to provide an adequate vortex that resulted in a system where visual motion was observed everywhere and was maintained until the sample was analyzed. For the LAW AZ-102 1.0M Na melter feed, a sample was pulled after 3 days of mixing and a new agitator speed was set based on the new volume of material in the mixing vessel. The agitator speeds used for both the LAW AZ-102 1.0M Na and 1.3M Na melter feeds are shown in Table 3-7. One hour prior to pulling samples for analyses, the slurry, agitator shaft/blade, and tank cap were weighed and any mass difference against the baselines were compensated for with DI water since it was assumed that the mass lost was due to water evaporation only. The largest quantity of water loss was 5 grams after the initial 3 days of mixing the LAW AZ-102 1.0M Na melter feed.

Table 3-7. Agitator Speeds During GFCs Addition and Extended Mixing

1.0 M Na Melter Feed			1.3 M Na Melter Feed		
Time Minutes	Agitator Speed RPM	Cumulative Mass of GFC added (g)	Time Minutes	Agitator Speed RPM	Cumulative Mass of GFC added (g)
Initial	147	0	Initial	245	0
2	193	26.52	3	285	46.70
4	231	85.52	5	330	72.60
7	265	216.89	7	400	103.88
10	265	233.92	10	500	123.30
First 3 days	325	N/A	13	550	137.12
Next 4 days	213	N/A	First day	600	N/A

Table 3-8. Description of Mixing Tanks and Agitators

Description	1.0 M Na Melter Feed	1.3M Na Melter Feed
Order of Chemical Additions	GFCs are blended	GFC are blended
Mixing Time	3 days + 4 days	24 hours total
Impeller Diameter D (mm)	50.69	38.30
Impeller Height H (mm)	10.30	10.22
Impeller Width W (mm)	2.21	2.33
Blade Length L (mm)	12.40	6.10
Type of Impeller	Rushton	Rushton
Tank Type	Right Cylinder Plastic Cup	Right Cylinder Plastic Cup
Tank Inside Diameter (mm)	83.4	63.25
Tank Height (mm)	99.9	106.4
Number of Baffles	None	None
Size of Baffles	None	None
Depth of impeller	Just off bottom	Just off bottom
Location of impeller (mm)	Slightly off-centered	Slightly off-centered
Comments	Rushton Blade  	

When the LAW AZ-102 1.0M Na melter feed was sampled after 3 days of mixing and then weighed, it remained undisturbed for approximately 15 minutes and it was noted that a hard thin layer (not quantified) had already settled to the bottom of the mixing vessel. This indicated that some of the solids had settled and quickly formed a strong structure. Prior to placing the 1.0M Na melter feed back onto the mixing stand for 4 more days of mixing, the settled slurry was re-suspended using the Rushton impeller by scraping the bottom of the mixing vessel.

3.4 PHYSICAL PROPERTIES TESTING

The physical properties of the LAW AZ-102 pretreated wastes were analyzed for density, wt% total solids, and wt% total oxides.

The physical properties of the LAW AZ-102 melter feeds were measured in accordance with the project Guidelines [Ref. 1]. The following properties were either measured or calculated:

- density of slurry, density of supernatant, density of settled solids, and density of centrifuged solids (g/mL)
- wt% and vol% of settled solids and vol% centrifuged solids
- wt% total solids, wt% dried solids, wt% oven dried solids, wt% undissolved solids (UDS), and wt% total oxides
- settling rate (interfacial volume level versus time)

All the physical properties measurements were performed at room temperature (approximately 18 to 23°C) unless otherwise specified. Rheological properties are physical properties and are discussed separately in section 3.6.

3.4.1 Methodology for Measuring Physical Properties

3.4.1.1 LAW AZ-102 Pretreated Waste Physical Properties

The density of the pretreated waste was determined using a 3.0 mL volume graduated cone. Triplicate samples of pretreated feed were placed into three graduated cones, filled to the 3.0 mL mark and weighed (M_L) at room temperature. The density was calculated by dividing M_L by the volume. The average density and standard deviation (Stdev) of the three readings are provided in Table 3-9. The method in which the wt% total solids and wt% total oxides were determined is described in section 3.4.1.2. The initial diluted AZ-102 pretreated wastes were characterized for wt% total solids and wt% total oxides. Due to limited amount of waste available for testing, the final 1.0M Na and 1.3M Na pretreated wastes wt% total solids and wt% total oxides were calculated from the initial 1.0M Na and 1.3M Na pretreated wastes that were analyzed for these properties. Note: There was not adequate sample volume to permit additional solids analysis without impacting the melter feed testing. The wt% total solids for the final pretreated wastes were re-calculated using Equation 3-2.

Equation 3-2 $$P_{MDS,2} = \frac{P_{MDS,1} \cdot M_{T1}}{M_{T1} - M_{Evap}}$$

where:

$P_{MDS,1}$ = wt% total solids of initial diluted pre-treated waste (%)
M_{T1} = mass of initial diluted pretreated waste prior to evaporation (g)
M_{evap} = mass of water loss due to evaporation (g)
$P_{MDS,2}$ = wt% total solids of final diluted pretreated waste (%)

The same method was used to calculate the wt% total oxides. The standard deviations are those of the errors from the initial diluted pretreated feed samples.

Table 3-9. Physical Properties of AZ-102 Pretreated Waste

Physical Property	Units	1.0M Na pretreated waste		1.3M Na pretreated waste	
		Average	Stdev	Average	Stdev
Density – supernatant liquid	g/mL	1.049	0.005	1.072	0.006
wt% total solids - P_{MDS}	%	6.34	0.03	8.14	0.05
wt% total oxides - P_{MOX}	%	0.87	0.08	1.07	0.17
total oxides/total solids ratio	g_{oxide}/g_{solids}	0.137	N/A	0.132	N/A

3.4.1.2 LAW AZ-102 Melter Feed Physical Properties

Below is a description of how a melter feed sample was handled and the variables calculated, in accordance with the project Guidelines procedure [Ref. 1]. Triplicate samples were run for each of LAW AZ-102 melter feeds. The samples for the settling tests, rheology, total oxides, and PSD were pulled using a 5 mL slurry pipette from the mixing vessel, while maintaining a slight vortex and good mixing.

Approximately 7 to 10 mL of the LAW AZ-102 melter feed (denoted slurry) was placed into a 10 mL volumetric graduated centrifuge cone. The mass (M_B) and volume of the slurry were recorded. The sample was then allowed to settle for at least 3 days. A detailed description of the settling test is provided in section 3.4.2. After the settling test was completed, the total volume (V_{SB}) of the sample and the volume of settled solids (V_{SS}) were recorded. The error associated with this measurement is the total volume, which may be under or over estimated, based on how the meniscus was read (Figure 3-2). Additionally, the materials hanging onto the sides of the tube would slightly decrease the level of settled slurry and the total volume (Figure 3-2).

Figure 3-2. Settling of LAW AZ-102 1.3M Na Melter Feed

The settled slurry was then centrifuged at approximately one thousand times the force of gravity for 60 minutes. The total volume after centrifuging (V_B) and the solids volume after centrifuging (V_{CS}) were recorded. The bulk density ($\rho_B = M_B/V_B$), vol% settled solids ($P_{VSS} = V_{SS}/V_B$ x 100%) and vol% centrifuged solids ($P_{VCS} = V_{CS}/V_B$ x 100%) were then calculated. The issue concerning the reading of the meniscus will impact the calculated results.

The transparent (yellow) supernatant was then transferred from the centrifuged cone to a graduated cylinder. The mass ($M_S = M_{VL}$ in this case) and volume (V_S) of the supernatant and the mass (M_{CS}) of centrifuged solids were recorded. The density of the supernatant ($\rho_S = M_S/V_S$), the density of centrifuged solids ($\rho_{CS} = M_{CS}/V_{CS}$), the wt% centrifuged solids ($P_{MCS} = M_{CS}/M_B$ x 100%), the mass of settled solids ($M_{SS} = M_B - \rho_S$ x (V_{SB}-V_{SS})), density of settled solids ($\rho_{SS} = M_{SS}/V_{SS}$) and wt% of settled solids ($P_{SS} = M_{SS}/M_B$ x 100%) were then calculated. Variables that could potentially impact these results are:

- Air entrainment. Air could potentially be released during centrifuging. Differences in the final volume between the settling and centrifuged total volume would indicate that such a condition exists. It is also possible that the bubbles would not be released by centrifuging. Correction to the data set requires making assumptions that could bias the results. Correction to this data set will not be performed because the total volumes before/after centrifuging are the same and air entrainment did not seem to be an issue for the LAW AZ-102 melter feeds processed. These slurries were very thin.
- The volume of supernatant transferred from the cone to the graduated cylinder is not 100% (i.e. not all the free standing supernatant was transferred). This would bias the wt% of undissolved solids high. The opposite would be true if insoluble solids were transferred for supernatant analysis.

- Volume of supernatant, as read on the graduated cylinder, could bias the density result high or low. This would impact the settled solids results.

The graduated cylinder containing the supernatant and the centrifuged cone containing the centrifuged solids were then placed overnight into a Thermolyne Model 19200 drying oven at 90°C The oven temperature was then increased to 105°C and the sample was maintained in the oven until the dried weights stabilized (approximately 2 days). The mass of the dried supernatant solids (M_{DCL}) and mass of the dried centrifuged solids (M_{DCS}) were recorded. Assuming that the mass lost is only water, the wt% soluble solids in the supernatant ($P_{SSS} = M_{DCL}/M_{VL}$ x 100%), wt% total dried solids (Equation 3-3), wt. % oven dried solids ($P_{ODS} = M_{DCS}/M_{CS}$ x 100%) and wt% UDS (Equation 3-4) were then calculated. Variables that could potentially impact these results are:

- Volatiles (organics) lost during the oven drying process. Organics would be lost in both the supernatant and centrifuged solids and would result in a lower solids measurement.
- Formation of a hard solid surface over the top of the samples during the evaporation process, which does not allow for the releasing of water (observed with high salt solutions and melter feeds). This condition would yield higher solids measurements. One way to prevent this would be to increase the surface area of the sample.

Generally, these methods are not designed for a production plant and are not recommended for implementation at WTP. The DWPF uses microwave ovens to obtain solids analysis. Additionally, infrared red (heating element) drying ovens have also been tested on WTP slurries and provide equally as reliable results as those obtained using the analysis method applied during this study.

Equation 3-3

$$P_{MTS} = \left[\frac{Psss}{100\%} \cdot \frac{M_S}{M_B} + \frac{M_{DCS}}{M_B} \right] \times 100\%$$

Equation 3-4

$$P_{MUS} = \left[1 - \frac{1 - \dfrac{M_{DCL}}{M_{CS}}}{1 - \dfrac{M_{DCL}}{M_{VL}}} \right] \times \frac{M_{CS}}{M_B} \times 100\%$$

A homogenized sample of the slurry was also transferred to a 50-mL pre-fired high-purity alumina crucible (CoorsTek, Golden, CO). The mass of the sample transferred was recorded (M_{WCS}) and the crucible placed overnight into an oven at 90°C. The oven temperature was increased to 105°C and the sample was maintained in the oven until the dried weight stabilized (about 1 day). The oven dried mass (M_{OSC}) was recorded and the wt% dried sample ($P_{MDS} = M_{OSC}/M_{WCS} * 100\%$) calculated. The sample was then placed into a Thermolyne Model 1400 resistance-heated furnace at room temperature, the temperature was raised (200°C/hr) to 1050°C and maintained at that temperature for 1 hour. The sample was then allowed to cool in the furnace. The weight of the oven fired sample (M_{FSC}) was recorded and the wt% total oxides ($P_{MOX} = M_{OSC}/M_{WCS} * 100\%$) calculated. The wt% UDS (Equation 3-5), was then calculated using the method employed by SRTC using the wt% Total Solids (TS) determined by the crucible method and the wt% soluble solids determined in the supernatant (as calculated above). The same issues as described in section 3.4.1.2 above could also impact the wt% dried solids in the sample. Impacts to the wt% total oxides could also occur due to volatilization of specific oxides, resulting in a lower wt%.

Equation 3-5

$$P_{MUS,SRTC} = \left[\frac{P_{MDS} - P_{SSS}}{100 - P_{SSS}}\right] \times 100\%$$

The standard deviations of the measured and calculated values are reported. Calculations, to take into consideration errors with the mass and volume measurements, were not included.

Table 3-10 lists the results for the physical properties of the LAW AZ-102 1.0M Na and 1.3M Na melter feeds. Results from the 3 days and 7 days of continuous mixing of the LAW AZ-102 1.0M Na melter feed show little physical difference between the two. The results clearly indicate that any soluble solids from the GFCs that were added had dissolved into the supernatant within the first 3 days of mixing, since there was little difference in the density and soluble solids in the supernatant between the 3 and 7 day measurements.

The total solids from the graduated cone/cylinder method and the crucible method are in agreement. This indicates that there was not a drying issue related to the cone/cylinder in the cone/cylinder method. The wt% UDS between the SRTC method and WTP project procedure are also in agreement and would have been exact if the same wt% total solids and wt% UDS data were used.

Table 3-10. Physical Properties of LAW AZ-102 1.0M Na and 1.3M Na Melter Feed

			1.0M Na 3-day		1.0M Na 7-day		1.3M Na 1-day	
	Description	Units	Average	STD	Average	STD	Average	STD
Cone/Graduated Cylinder	Bulk Density - ρ_B	g/mL	1.55*	0.00	1.53	0.00	1.67	0.02
	Vol % Settled Solids - P_{VSS}	%	69.00*	0.46	67.39	0.00	78.65	0.82
	Density of Centrifuged Solids - ρ_{CS}	g/mL	1.86*	0.01	1.89	0.01	1.90	0.01
	Vol % Centrifuged Solids - P_{VCS}	%	61.12*	0.46	59.42	0.00	72.40	0.62
	Wt. % Centrifuged Solids - P_{MCS}	%	73.37*	0.05	73.21	0.35	82.67	0.14
	Supernatant Liquid Density - ρ_S	g/mL	1.09*	0.01	1.08	0.01	1.10	0.01
	Density of Settled Solids - ρ_{SS}	g/mL	1.76*	0.01	1.75	0.01	1.82	0.02
	Wt. % Settled Solids - P_{MSS}	%	78.26*	0.11	77.12	0.19	85.94	0.77
	Wt% soluble solids in supernatant - P_{SSS}	%	8.96*	0.11	9.46	0.78	9.68	0.40
	Wt. % Total Solids - P_{MTS}	%	53.22*	0.05	53.41	0.34	59.61	0.11
	Wt. % Oven Dried Solids - P_{ODS}	%	69.25*	0.02	69.46	0.06	70.06	0.12
	Wt. % UDS - P_{MUS}	%	48.59*	0.01	48.52	0.16	55.26	0.10
**	Wt% dried total sample - P_{MDS}	%	53.89	0.42	53.49	0.56	59.90	0.07
	Wt% total oxides P_{MOX}	%	48.66	0.41	48.28	0.35	54.31	0.15
	Wt. % UDS $P_{MUS\text{-}SRTC}$	%	49.15*	0.79	48.62	0.94	55.60	0.21

*Average of two results. One melter feed sample overflowed during drying.
** Crucible results

3.4.2 Settling Results of LAW AZ-102 Melter Feed

Settling tests were performed on the both LAW AZ-102 melter feeds as described in the project Guidelines method [Ref. 1]. First, the slurry sample was placed into a graduated centrifuging cone that was used for the physical property determinations, after completion of the settling test. The tube was shaken to homogenize the slurry sample and then it was allowed to settle for at least three days. The three samples of slurry were homogenized within 10 seconds of each other and placed into a centrifuge tube holder. The time after homogenization was recorded and volume measurements were taken of the interface layer, which is the layer between the clear supernatant and settling slurry at specified time intervals [Ref. 1]. Figure 3-2 shows the graduated centrifuge tubes used for the settling test. The recorded settling data was normalized and the average normalized interface volume data for the first 60 minutes is shown in Figure 3-3. After 30 minutes, there is little change in the normalized interface volume for all the LAW AZ-102 melter feeds as shown in Table 3-11. The LAW AZ-102 1.0M Na melter feed 3 day and 7 day results are very similar, but the 3-day results indicate that it takes a little longer for the solids to settle.

The settling data has not been corrected for wall or hindered settling effects. This data is limited only to decanting operations or to determine the amount of settled solids. The data can also be used to determine if rheological measurements using standardized rotational bench scale rheometers can be utilized to measure the rheological properties of the sample based on how quickly the interface is produced relative to the time it takes to perform flow curve measurement. There are established standards for settling tests that are used for decanting, but the measuring vessel and sample size are much larger than what is used here. The larger vessel size is used to minimize wall effects.

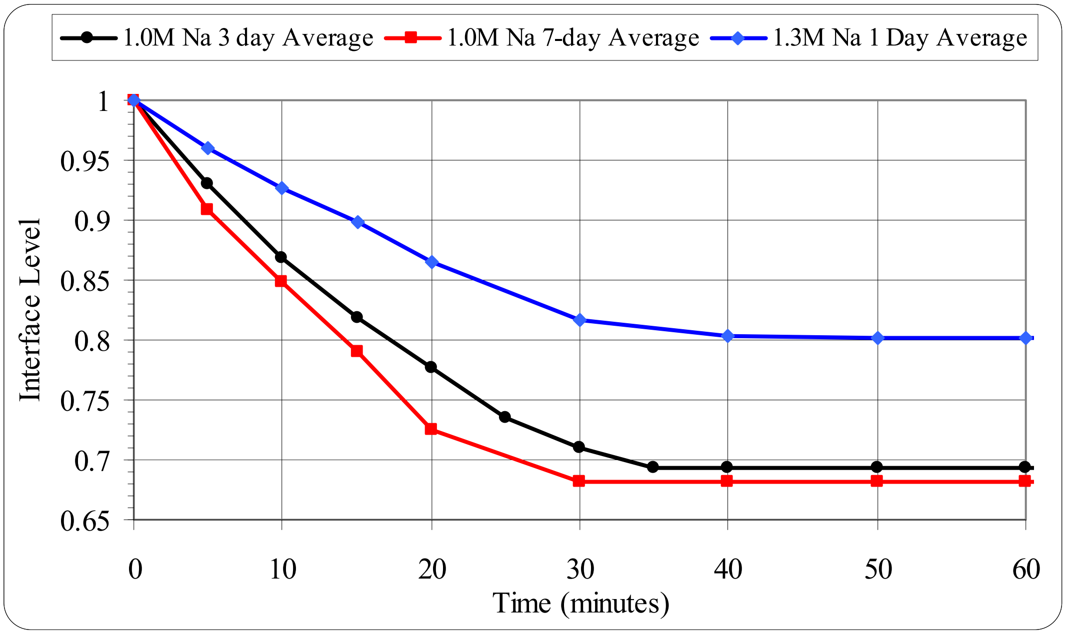


Figure 3-3. Normalized Settling Curves for LAW AZ-102 Melter Feed

Table 3-11. Normalized Interface Volume for LAW AZ-102 Melter Feed

1.0M Na – 3 days mixing		1.0M Na – 7 days mixing		1.3M Na - 1 day Mixing	
Time (min)	Normalized Interface Volume	Time (min)	Normalized Interface Volume	Time (min)	Normalized Interface Volume
0	1	0	1.000	0	1.000
5	0.930	5	0.908	5	0.960
10	0.869	10	0.848	10	0.927
15	0.818	15	0.790	15	0.899
20	0.777	20	0.725	20	0.864
25	0.735	30	0.681	30	0.816
30	0.710	40	0.681	40	0.804
35	0.694	50	0.681	50	0.802
40	0.694	60	0.681	60	0.802
50	0.694	1440	0.676	120	0.802
60	0.694	1920	0.676	180	0.802
240	0.694	2880	0.676	360	0.802
360	0.694	4320	0.676	1440	0.792
1440	0.694	N/A	N/A	2880	0.792
4320	0.694	N/A	N/A	4320	0.788

3.5 pH MEASUREMENT

The pHs of the LAW AZ-102 pretreated waste and melter feeds was measured using a Fisher Scientific 'Accumet AB15' pH meter fitted with a Fisher Scientific gel-filled, epoxy body combination electrode with Ag/Ag/Cl reference pH probe at ambient conditions. The results are shown in Table 3-12. The functionality of the pH meter and probe were confirmed by measuring three different pH buffers at pHs of 4, 7, and 10 before sample measurements were performed. These Fisher Scientific pH buffers are certified to within +/- 0.02 pH units and were used within the designated expiration date listed on each buffer. The pH of the pretreated feeds decreased when GFCs were added. This is due to the addition of acidic species in the GFCs, such as boric acid. There is not much change in the pH of the LAW AZ-102 1.0M Na melter feed after 3 days and 7 days of mixing.

The measured pH values were outside the calibrated range of the pH instrument for the pretreated feeds. The error in the measurement is unknown, since no sample exists where pH paper can be used to verify the measurement.

Table 3-12. pH of the LAW AZ-102 Pretreated Wastes and Melter Feeds

Pretreated Waste		Melter Feed	
Na Molarity	pH	Na Molarity	pH
1.0	12.01	**1.0** – 3 days of mixing	9.04
1.3	12.15	**1.0** – 7 days of mixing	9.22
N/A	N/A	**1.3** – 1 day of mixing	8.72

3.6 RHEOLOGICAL MEASUREMENTS

Rheological properties of the LAW AZ-102 pretreated wastes and melter feeds were analyzed per the project Guidelines methods [Ref. 1]. The rheological properties of the pretreated waste and melter feeds were analyzed by measuring the shear stress-shear rate flow curves. The melter feeds settled solids shear strength was determined using the vane technique.

3.6.1 Instrumentation

The Haake M5/RV20 rheometer was used for all rheological measurements performed in this task. The M5 measuring head specifications are shown in Table 3-13.

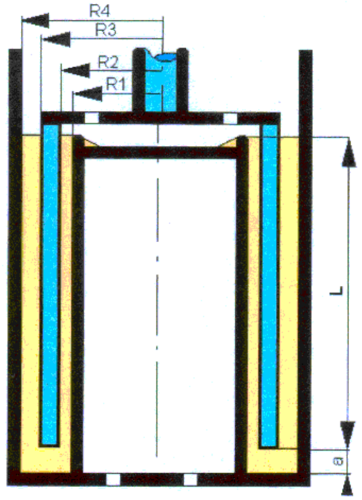
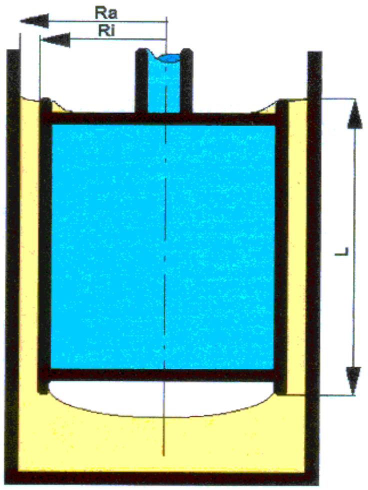
Table 3-13. M5 Measuring Head Specifications

Specification	Units	Value
Maximum Torque	N-cm	4.9
Minimum Torque (recommended)	N-cm	0.049
Deviation in Torque Measurement	% of maximum	+/- 0.5
Maximum Speed	RPM	500
Minimum Speed	RPM	0.05
Error in Speed Measurement	% of reading	+/- 0.5

3.6.2 Flow Curve Measurements Using Concentric Geometry

Flow curve measurements were obtained using concentric (MV1) and double concentric (NV) cylindrical rotors. Their designs are shown in Table 3-14. The MV1 or NV rotor was initially installed onto the M5 measuring head. A sample was then placed into the appropriate cup (cup dimension provided in Table 3-14), given which rotor is being utilized, and raised into a temperature- controlled cup holder, which controlled the temperature at 25°C. The RV20 rheometer controls the rate at which the M5 measuring head spins the rotor and measures both the rotational speed and the torque (the resistance to shear). The shear stress at the wall of the rotating rotor is then calculated (internally by the Haake software) based on the product of the measured torque and geometry (A-factor) of the rotor. The shear rate of the rotating rotor is calculated as the product of the measured speed and geometry (M-factor, assumes fluid is Newtonian) of the rotor. The A-factor, M-factor, shear rate range and the ramp up time, hold time at maximum shear rate, and ramp down time are provided in Table 3-14. The NV rotor was used to measure the pretreated feed and supernatant. The MV1 rotor was used to measure the melter feed.

Table 3-14. NV and MV1 Rotor Specifications and Ramp Rates

	NV Rotor	MV1 Rotor
Design of Rotor		
Rotor radius (mm)	$R_2 = 17.85$, $R_3 = 20.1$	$R_i = 20.04$
Cup Radius (mm)	$R_1 = 17.5$, $R_4 = 20.5$	$R_a = 21.0$
Height of rotor (mm)	L = 60	L = 60
Sample Volume (cm^3)	V = 9	V = 34 to 50
A factor (Pa/%torque)	1.78	3.22
M factor (s^{-1}/%RPM)	27.0	11.7
Measuring Range (s^{-1})	0 – 2000	0 – 1000
Ramp up time (min)	5	5
Hold time (min)	1	1
Ramp down time (min)	5	5

Prior to performing any flow curve measurement, the rotor and cup are inspected for visual damage that could potentially impact the flow measurement. National Institute of Standards and Technology (NIST) traceable Newtonian oil standards were used to verify the operability of the M5 head at a measurement temperature of 25°C. The viscosities of the NIST traceable Newtonian oil standards at 25°C are shown in Table 3-15. The resulting flow curves were analyzed as a Newtonian fluid and the calculated viscosity was compared to the NIST traceable Newtonian oil standard. The rheometer was considered operable if the calculated viscosity was within ± 10% of the NIST traceable Newtonian oil standard viscosity as stated in Table 3-15. The measuring range of the NV/M5 as specified by Haake shows a lower limit around approximately 5 cP at very high shear rates (+ 2000 sec^{-1}), but does not provide any error associated with the measurement. Using the uncertainties provided by Haake in the measured torque and speed of the M5 measuring head (Table 3-13), the resulting viscosity, when using the S3 NIST traceable Newtonian oil standard used in this study, may vary as much as ± 13.6% (given a 95% or 2σ confidence [Ref. 6] level at a shear rate of 2000 sec^{-1} and assuming the uncertainties provided by Haake in the torque and speed has 2σ confidence level), but the rheometer was not considered inoperable. Measurements below 5 cP will have to be used with a higher level of uncertainty and will be based on the actual measurement results.

Table 3-15. Cole-Parmer NIST Traceable Newtonian Oil Standard

Standard Type	Viscosity (cP) at 25°C			Lot Number	Expiration Date
	-10%	Reported	+10%		
S3	2.939	3.266	3.593	F102609	Oct. 10, 2004
N35	45.44	50.49	55.54	F102205	Nov. 27, 2004
DI H_2O	0.801	0.89011	0.979	N/A	N/A

The Newtonian results of the NIST traceable Newtonian oil standard are shown in Table 3-16. The NV rotor was run with the S3 oil standard, with 2 of the 3 measurements being within 10% of the standard. Table 3-16 shows the result for DI water using the NV rotor, resulting in a slightly higher viscosity than predicted. The results obtained in this study using the NV rotor for fluids with low viscosity (less than 3.266 cP) had a 16% error in the measured viscosity. The NV rotor is not recommended for measuring fluids that are less viscous than water, since there is no reference to compare the results. The results of using such low viscosity fluids will not impact the normal operation of the M5 measuring head. Another issue when characterizing low viscosity fluids is the inertia effect of the rotor, which becomes much more predominate for low viscosity fluids as compared to more viscous fluids.

Table 3-16. M5 Operability Checks Using NIST Traceable Viscosity Oil Standards

Rotor	NIST Standard	Run Date	Measured Viscosity (cP)	Viscosity within +/- 10%
NV	S3	6/18/03	3.08	Yes (-5.7%)
MV1	N35	6/25/03	53.2	Yes (5.4%)
NV	S3	6/27/03	3.15	Yes (-3.4%)
MV1	N35	7/3/03	52.8	Yes (-4.6%)
MV1	N35	7/7/03	52.6	Yes (-4.2%)
NV	S3	7/9/03	2.80	No (-14.3%)
NV	DI water	6/18/03	1.03	No (+15.7%)

3.6.3 Yield Stress Measurement Using Vane Geometry

Vanes have been used [Ref. 7 through 20] to measure the yield stress of non-Newtonian fluids as is shown in Figure 3-4. The vane is inserted into the fluid and rotated at a very slow speed. The surface area used to determine the shear stress is the surface area produced by the vane, in other words, a cylinder. It has been shown that this is a good assumption [Ref. 8, 9, 12, and 13] for determining the stress of the fluid as the vane rotates through it. The derived equation ( ) assumes the stress is constant on all surfaces. The shearing due to the immersed section of the vane shaft, stress contribution of the immersed section of the shaft, and the wall effects are negligible by meeting the criteria as shown in Figure 3-4.

Equation 3-6

$$\tau_{vane} = \frac{\Gamma}{\frac{\pi \cdot D^3}{2}\left(\frac{H}{D} + \frac{1}{3}\right)} = A \cdot \Gamma$$

where Γ = measured torque (N-m or % torque)
D = diameter of vane (m)
H = height of vane (m)
A = geometric constant (m^3 or Pa/%Γ)
τ_{vane} = shear stress (Pa)

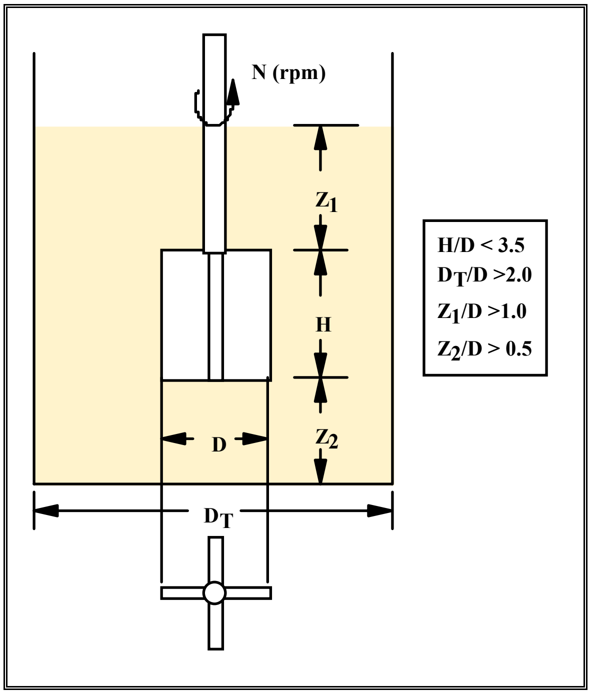


Figure 3-4. Vane Geometric Requirements

A typical stress versus time (or displacement) curve is shown in Figure 3-5. The initial response for a non-Newtonian fluid having a yield stress is typically linear and this slope is called the Hookean elastic modulus (G). The point of departure from this linear region is called the static yield stress [Ref. 7] when the fluid starts to transition from a fully elastic to viscoelastic behavior. At the maximum stress, the behavior of the material transitions between viscoelastic and fully viscous and is called the yield stress (known also as the dynamic yield stress).

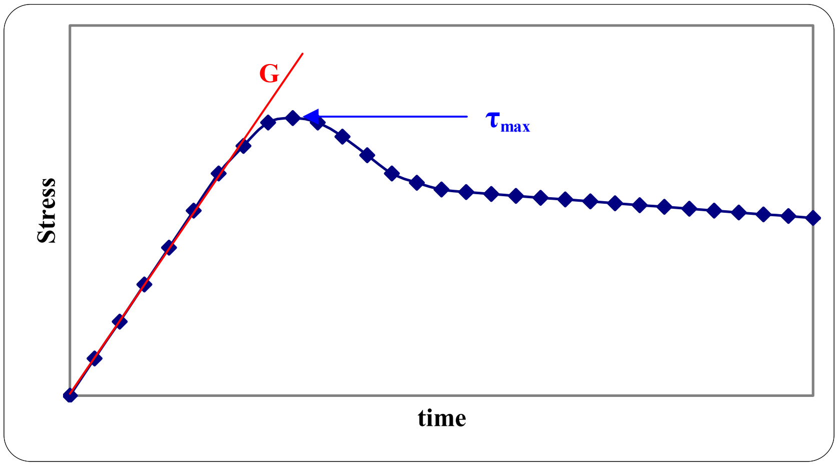


Figure 3-5. Typical Torque – Time/Displacement Curve

The vane dimensions used in this task were D = H = 16 mm. The A factor for the M5 measuring head was calculated and used in the RV20 to calculate the stress from the measured torque, given the measured %torque. The A factor used is shown in Equation 3-7.

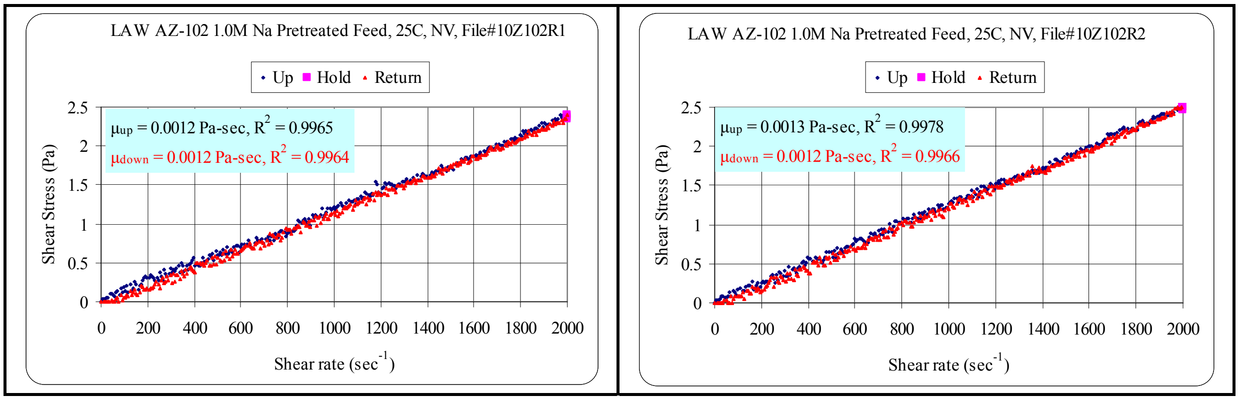
Equation 3-7 $$A = \frac{A' \cdot \Gamma_{\max}}{100\%} = \frac{2}{\pi \cdot (0.016m)^3 \cdot \left(\left(\frac{16}{16} \right) + \frac{1}{3} \right)} \cdot \frac{4.9N \cdot cm}{100\%\Gamma} \cdot \frac{m}{100cm} = 57.12 \frac{Pa}{\%\Gamma}$$

For the vane measurements, the M factor was set at 1.0 $\sec^{-1}$/%RPM. Going through the same exercise as that shown in Equation 3-7, for a rotational speed of 0.3 RPM, the controlled shear rate was 0.06 $\sec^{-1}$ and used in the RV20. The rotational speed was also visually verified at approximately 0.3 revolutions per minute (RPM). This rotational speed is specified in project approved Guidelines [Ref. 1]. The sample cup used for vane measurements had an inside diameter of 40 mm. The samples were allowed to settle for at least 2 days prior to performing the yield stress measurement. There is no NIST standard available in which the vane can be used to verify the operability of a rheometer. The M5/RV20 rheometer is verified operational via the functional check using a NIST Newtonian oil standard, using concentric geometry. The M5 head has an error of 0.5% of full torque (Table 3-13), resulting in an error of ± 28.56 Pa or ± 285.6 dynes/cm^2 in the measured stress.

The yield stress after settling at various times will be reported as the “settled solids shear strength” which is consistent with WTP terminology.

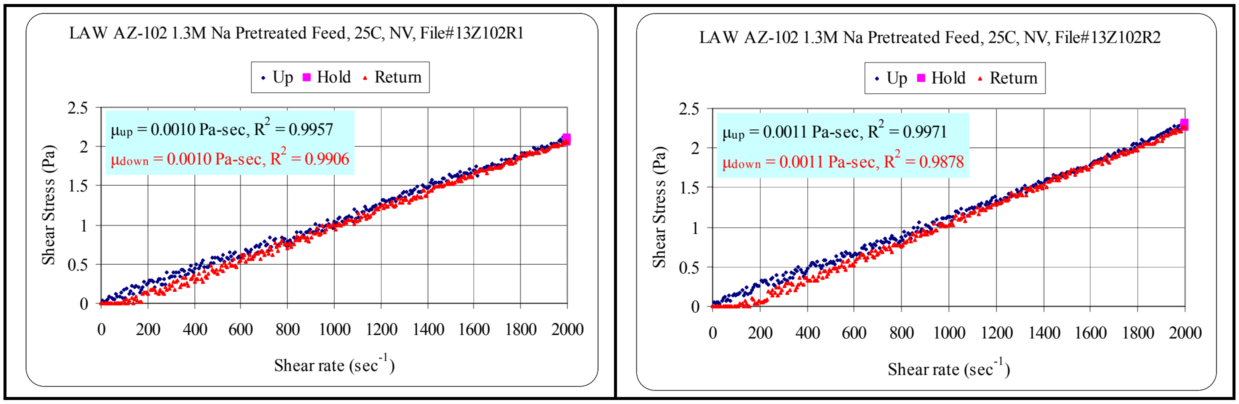
3.6.4 LAW AZ-102 Pretreated Waste Rheology Results

Duplicate flow curves for each LAW AZ-102 pretreated waste were measured at 25°C. The flow curves are shown in Figure 3-6 for the 1.0M Na pretreated waste and Figure 3-7 for the 1.3M Na pretreated waste. The averaged viscosities of the 1.0M Na and 1.3M Na pretreated wastes are 1.2 cP (or 1.2 mPa-sec) and 1.1cP. This indicates that the 1.3M Na pretreated waste is less viscous than the 1.0M Na pretreated waste; but considering only instrument error, the results easily overlap each other. The instrument error, at the maximum shear rate of 2000 $\sec^{-1}$, as measured in this report, is at least ± 37% [Ref. 6] given a 95% confidence level. In either case, the results clearly show that these pretreated wastes are not much different from each other and are very close to the viscosity of water.


LAW AZ-102 1.0M Na Pretreated Feed, 25C, NV, File#10Z102R1

LAW AZ-102 1.0M Na Pretreated Feed, 25C, NV, File#10Z102R2

Figure 3-6. LAW AZ-102 1.0M Na Pretreated Waste Flow Curve


LAW AZ-102 1.3M Na Pretreated Feed, 25C, NV, File#13Z102R1

LAW AZ-102 1.3M Na Pretreated Feed, 25C, NV, File#13Z102R2

Figure 3-7. LAW AZ-102 1.3M Na Pretreated Waste Flow Curve

3.6.5 LAW AZ-102 Melter Feed Rheology Results

Duplicate flow curves of each LAW AZ-102 melter feed were measured at 25°C. It was observed that a hard settled layer (thickness never quantified) had developed within 30 minutes after the sample was removed from the agitated mixing vessel in preparation of performing the rheology measurements. Based on the settling curve results (section 3.4.2), the melter feed samples were homogenized, placed into the MV cup, loaded into the cooling/heating jacket, and trimmed. The program started within 30 seconds from the time the sample was loaded into the cup and the flow curves for the 1.0M Na melter feed, after 3 and 7 days of mixing, are shown in Figure 3-8 and Figure 3-9. Both exhibit essentially the same behavior and maximum measured stress. The 1.3M Na melter feed is shown in Figure 3-10. This feed is more viscous than the 1.0M Na melter feeds, but it also has the same shape flow curves as that of the 1.0M Na melter feeds. The resulting flow curves can not be analyzed, using the rheological models as specified in project approved Guidelines [Ref. 1] or any rheological model, since the results would be meaningless. All the melter feed flow curves were impacted by the settling nature of the melter feed material. An explanation of the behavior of each section of the flow curve is provided in Table 3-17.

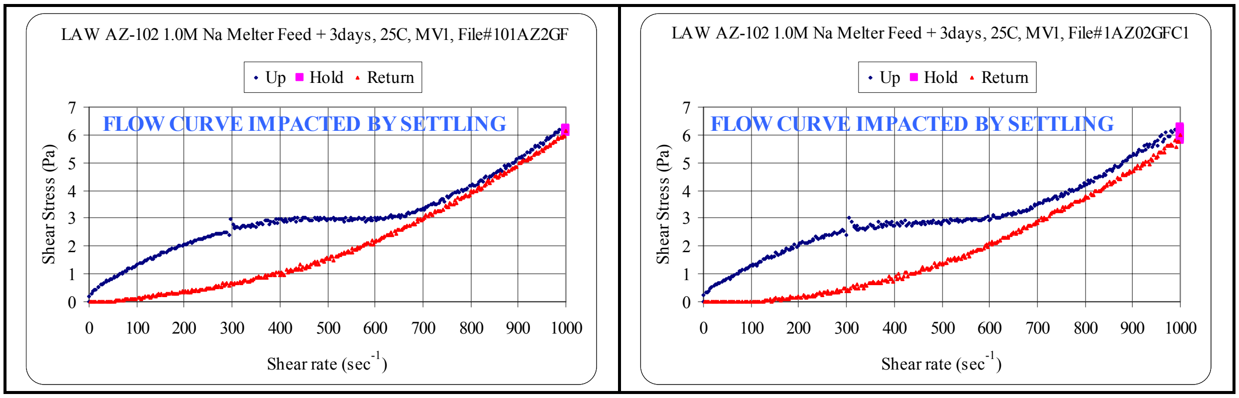


Figure 3-8. LAW AZ-102 1.0M Na Melter Feed, After 3 Days of Mixing

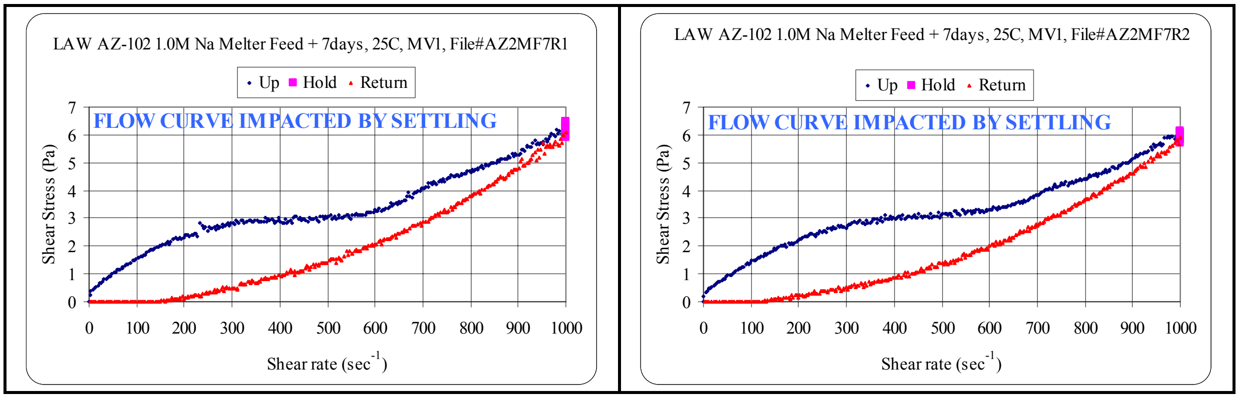


Figure 3-9. LAW AZ-102 1.0M Na Melter Feed, After 7 Days of Mixing

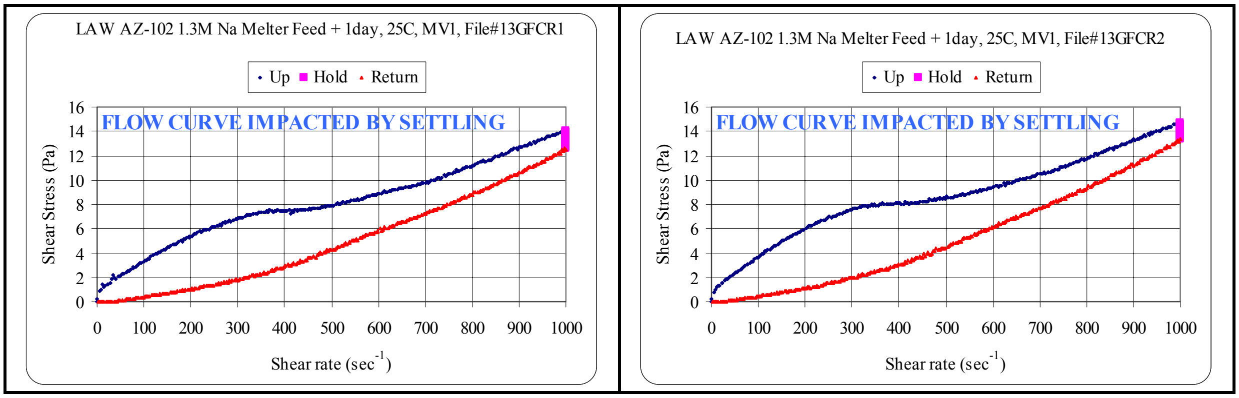


Figure 3-10. LAW AZ-102 1.3M Na Melter Feed, After 1 Day of Mixing

Table 3-17. Settling Impact on LAW AZ-102 Melter Feed Flow Curves

Curve Location	Shear rate range (sec^{-1})	Explanation
Up Section	0 – 300 (1.0M) 0 – 325 (1.3M)	Curve fit of flow curves would result in a power law index of less than one and a yield stress. From settling data and from visual observation, both the 1.0 and 1.3M Na melter feeds do not have any yield stress. Power law index less than one due to settling of solids out of shearing gap.
	300 – 625 (1.0M) 325 – 500 (1.3M)	Increase in shear rate causes essentially no increase in shear stress, indicating material is settling through and out of the shearing gap. Or massive slip is occurring.
	up to 1000	Curves are somewhat shear thickening (n>1). Could be due to the clear supernatant generated by settling is producing Taylor vortices.
Hold	1000	Shear stress going down, due to solids settling out of the shearing zone. More material settling out of the 1.3M as compared to the 1.0M when comparing the drop in shear stress.
Down Section	1000 to 0	If curve fitted, would be shear thickening (n>1). Reduction in shear stress is most likely due to settling or Taylor Vortices. Other issues could be due to inertia effects of the rotor slowing down in a thin solution.

A clear supernatant level developed between the gap at the top of the concentric cylinder and cup was clearly evident within 30 seconds after starting the flow curve measurements. Because these slurries are not thixotropic (in the rheological sense), a second flow curve was not performed while the settled slurry was in the MV1 geometry. If such a measurement were performed, it would be too difficult to distinguish between the shear stress contribution of the clear supernatant (and potential Taylor vortices that could be generated) and that of the settled solids (with the amount of settled solids concentration unknown). At this time, the settled solids would not be representative of wt% solids concentration in the original slurry. Slurries with such behavior must be tested in a slurry test loop to determine the critical velocity at which all the solids are just suspended. The velocity may also be determined for other conditions, such as complete homogeneity in the pipeline.

The slurry used for the flow curves was placed into a settling cup (for the yield stress – settled solids shear strength measurements) and allowed to settle for the allotted time (see Table 3-19 for settling times). After the settled solids shear strength was measured, the clear supernatant was analyzed at 25°C for rheology using the NV geometry. All the flow curves obtained indicate that the supernatant was Newtonian as fitted from a shear rate range of 0 to 2000 sec^{-1} with the results presented in Table 3-18. The viscosity of the 1.0M Na melter feed supernatant did not change after 3 days and 7 days of mixing. The viscosity of the 1.3M Na melter feed supernatant is more viscous than the 1.0M Na melter feed supernatant. However, considering only instrument error, the results could easily overlap each other. The instrument error, at the maximum shear rate of 2000 sec^{-1}, as measured in this report, is at least ± 25% [Ref. 6] given a 95% confidence level.

Table 3-18. LAW AZ-102 Melter Feed Supernatant Viscosity

Supernatant	Run	File	Fitted Region	R^2	Viscosity (cP)
1.0M Na +3 Day	1	1AZSUP34.ROT	Up	0.9938	1.83
			Down	0.9973	1.78
	2	1AZSUP36.ROT	Up	0.9968	1.82
			Down	0.9978	1.81
				Average	**1.81**
1.0M Na + 7 Day	1	1AZSUP71.ROT	Up	0.9982	1.79
			Down	0.9981	1.78
	2	1AZSUP72.ROT	Up	0.9974	1.74
			Down	0.9970	1.71
				Average	**1.76**
1.3M Na + 7 Day	1	13MFSUP1.ROT	Up	0.9987	1.97
			Down	0.9989	1.98
	2	13MFSUP2.ROT	Up	0.9985	1.97
			Down	0.9988	1.92
				Average	**1.96**

3.6.6 Settled Solids Vane Measurement Results

Upon completion of the flow curve measurements, the slurry samples in the MV cup were placed into the cup used for the vane measurement and additional slurry was added to provide a sufficient zone of settled solids. The resulting settled solids met the criteria stated in Figure 3-4. The inside diameter of the cup was 40 mm. The total height, settled solids height, the supernatant height, and undisturbed settling time are provided in Table 3-19.

Table 3-19. Settled Solids Height and Times for Vane Measurements

Settled LAW AZ-102 Melter Feed Sample Description	Height (mm)			Settling Time (Hrs)
	Total	Settled	Supernatant	
1.0M Na, 3 days of mixing	77	49	28	72
1.0M Na, 7 days of mixing	80	53	27	48
1.3M Na, 1 day of mixing	64	51	13	50

For each sample, the vane was inserted 16mm down into the settled solids at the center of the cross sectional area of the cup. A second measurement of the 1.0M Na sample, after 7 days of mixing, was made immediately after the initial vane measurement by inserting the vane an additional 13 mm into the sample (leaving the bottom of the vane about 8 mm from the bottom of the cup). For this second measurement, the sample did not meet the bottom clearance criteria stated in Figure 3-4 and was partially extended into the sheared volume of the previous sample. The vane results are shown in Figure 3-11. After 7 days of mixing, the 1.0M Na sample showed a much greater shear strength measurement than the after 3 days of mixing measurement. The settled solids shear strength and other observations of the settled solids are described in Table 3-20.

Table 3-20. Shear Strength and Observations of Settled LAW AZ-102 Melter Feed

LAW-102 Settled Solids	Shear Strength (Pa)	Observations
1.0M Na, 3 days of mixing	218	• After the measurement, the vane was manually placed into settled solids and rotated manually (using researcher's hand) to inspect the settled solids. Based on this observation, the material at the top yielded much easier than the material at bottom. • The sample was homogenized and allowed to settle for 5 additional days. After the supernatant was analyzed, the settled solids were inspected using the vane and these settled solids strength was consistent throughout the sample and comparable (manually) with the resistance felt in the 1.3M Na sample.
1.0M Na, 7 days of mixing	122 (1st) 494 (2nd)	• Same observation as above relative to how the material yielded from top to bottom by manually using the vane to inspect the settled solids. • 2nd measurement confirms how material felt via the vane. • 2nd measurement results could be impacted by bottom of wall and from sheared material on upper section and above the vane. Results indicate a shear strength gradient exists in the settled solids.
1.3M Na, 1 day of mixing	2349	• After measurement, the vane was used to manually inspect settled solids. Unlike the 1.0M Na settled solids, the material felt consistent throughout the sample.
General		• After the settled solids were homogenized, the slurry was very fluid and behaved like the original slurry and settled fairly quickly. This was true for all the slurries. • Homogenization of settled solids required that the vane make physical contact with undistributed settled solids.

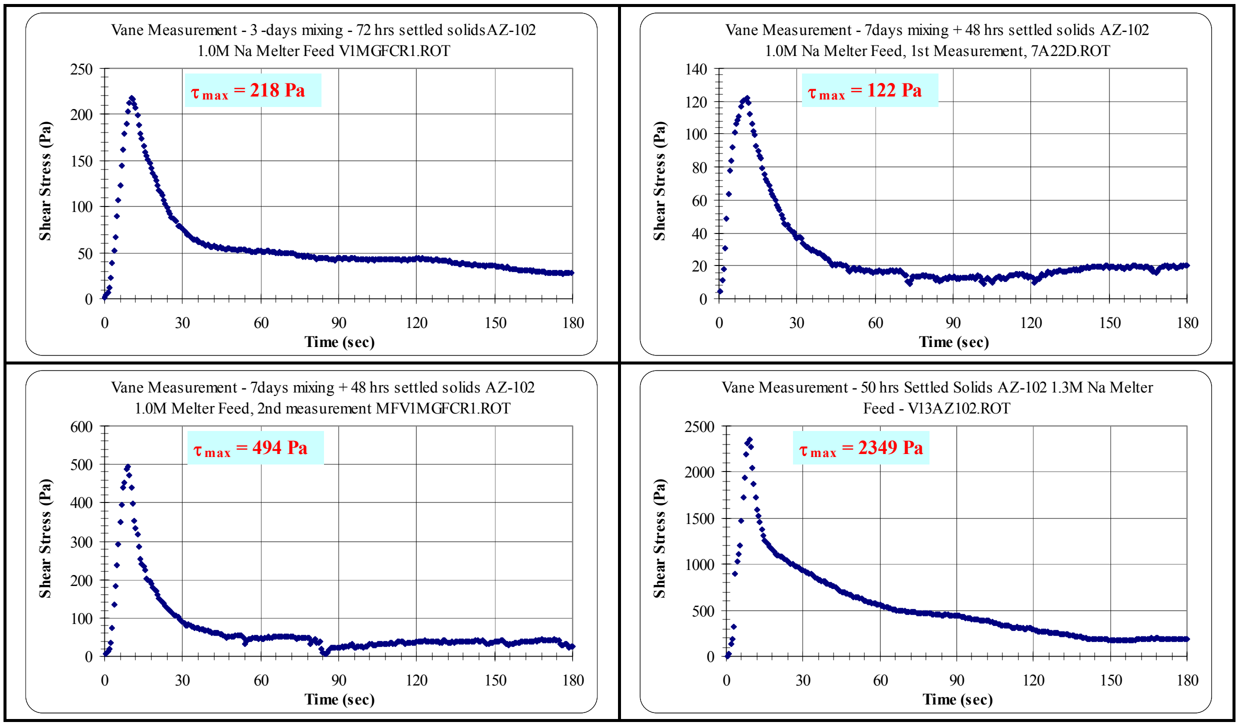


Figure 3-11. Vane Results of the LAW AZ-102 Settled Melter Feed

3.7 PARTICLE SIZE DISTRIBUTION

A Microtrac X-100 particle size analyzer was used to measure the particle size distribution (PSD) of these samples. Prior to analyzing the sample, the melter feed sample was diluted using a filtered simulant supernatant with approximately the same molarity as the feed sample. The Microtrac X-100 particle size analyzer measures the particle diameters by measuring the scattered light from a laser beam projected through a stream of the fluid carrying the diluted sample particles. The amount and direction of the light scattered by the particles is measured by an optical detector array and then analyzed to determine the size distribution of the particles. The Microtrac X-100 range is nominally 0.12 to 704 µm. It should be noted that this instrument does not have sonication (ultrasonic energy that breaks up agglomerations) capabilities; hence, no PSD measurements were taken with sonication. The Microtrac X-100 has software that was utilized to calculate the mean number, mean area, mean volume and number and volume distribution data for standards and melter feed.

The X-100 was functionally checked at the beginning and at the end of the melter feed PSD measurements using NIST traceable particle size standards from Duke Scientific Corporation. These particle size standards are polystyrene micro spheres dispersed in a 1 mM potassium chloride solution. The particle size standards were diluted using de-ionized water as the suspending medium (refractive index of mixture was set to 1.33) and a nominal flow rate of 40 mL/s. The X-100 instrument was set to 'transparent' mode with the particle refractive index equal to 1.59 and particle shape as spherical. A small size, 39.6µm ± 0.8µm, and a larger size, 496µm ± 10µm, particle size standard were used to verify the functionality of the Microtrac X-100. Each standard was run in triplicate with a measuring time of 30 seconds per measurement. The average of three measurements is reported. The standards were run before (initial) and after (final) the LAW AZ-102 melter feeds were analyzed. The Microtrac X-100 calculated the mean volume diameter and the mean number diameter and the results for the standards are presented in Table 3-21. Additional information for the Duke standards, including the volume distribution, the cumulative volume distribution, number distribution and the cumulative number distribution plots, and numerical data is presented in Figure A- 1 through Figure A- 4, Figure A- 11 and Figure A- 12 in Appendix A. Unfortunately, the larger particle size standard was consumed in the initial measurements, so only the smaller particle size standard was measured after the AZ-102 slurry was analyzed. The mean diameter of a distribution represents the centroid of the distribution. Instrument functionality is typically verified by comparing the calculated mean number diameter from measuring the PSD of the certified standard to the certified values of the standards. The calculated value is recommended to be within ± 10% of the standard for ideal instrumentation functionality verification as stated in reference 2.

Table 3-21 shows the calculated mean diameters on a number and volume basis for both the smaller and larger standards. The calculated mean number and volume diameter are within ± 10% of the standard mean diameter, though only the number basis is required for instrument functionality.

Table 3-21. Summary of Particle Size Data for Standards

NIST Traceable Particle Size Standard		Measured Mean and Medium Diameters on a Number and Volume Basis (µm)			
Description	Mean Diameter (µm)	Initial Measurements		Final Measurements	
		Number	Volume	Number	Volume
Duke Scientific Standard 2040A	39.6 ± 0.8	37.67	40.45	38.23	39.89
Duke Scientific Standard 4350A	497 ± 10	495.9	535.1	N/M	N/M

N/M = not measured

After the initial particle size standards were measured on the Microtrac X-100, the 1.0M Na and 1.3M Na LAW AZ-102 melter feeds PSD measurements were performed. These melter feeds were diluted using an LAW AZ-102 pretreated waste simulant as the suspending medium with a nominal flow rate of 40 mL/s. The LAW AZ-102 pretreated waste simulant is described in detail in an SRTC interim report on the AZ-102 LAW vitrification testing [Ref. 5]. The LAW AZ-102 pretreated waste simulant was diluted to the appropriate Na molarity of the melter feed and filtered through a 0.2μm filter prior to being used to dilute the slurry sample. The Microtrac X-100 was set to 'Absorb' mode with particle shape, particle refractive index and fluid refractive index unspecified. For each melter feed sample, a homogenized subset sample was taken and diluted with the LAW AZ-102 pretreated waste simulant. Five homogenized subsets of this sample were then analyzed, using a 60 second cycle time, through the Microtrac X-100. The average of the five measurements is reported in Table 3-22.

Table 3-22 summarizes the mean and medium data for the melter feed PSD for both volume and number basis. Additional information for the three different melter feeds, including volume distribution, cumulative volume distribution, number distribution, cumulative number distribution plots, numerical data, and Microtrac X-100 operating conditions is presented in Appendix A (Figure A- 5 through Figure A- 10). The plots of the particle size of the melter feed slurry particles, in Appendix A, show the percentage of particles that pass (right Y-axis, histogram) and percent cumulative of particles that have passed (left Y-axis, solid line) for a given micron size range (X-axis). This data is also shown in tabular form, located below the plots.

The volume basis data for the melter feeds show that they consist of particles in the 0.122μm to 352μm range. Similar data for the number basis data indicates particle sizes between 0.122μm to 10μm range. Both the volume basis and the number basis plots show that the distribution of particles in these melter feed slurries is contained in two different groups (bimodal distribution). The volume basis data indicates the smaller group centered around 1.5μm and the larger group is centered around 30μm.

The number basis data, which is weighed towards smaller particles, indicates that the smaller group is centered around 0.15μm and the larger group is centered around 0.3μm. The number distribution typically yields smaller mean and median values as compared to volume distribution for a wide distribution of diameters. The number distribution is based on the volume distribution, with the cube of the diameter divided into the volume distribution, hence yielding a higher number distribution for smaller diameter particles. Note: The carrier fluid used was an AZ-102 pretreated waste simulant that was filtered using a 0.2μm filter.

No baseline PSD measurements were made of the filtered LAW AZ-102 pretreated waste simulant. The impact of solids smaller than 0.2μm in the AZ-102 pretreated waste simulant would bias the number results in the direction of smaller particle size data. From a numbers perspective, many of the individual GFCs have number distributions that contain particles that are also in this range [Ref. 4]. This would have little impact on the volume distribution, which is typically the data used for performing engineering calculations relative to settling.

Table 3-22 shows the mean and median values for the volume and number for the melter feeds. The mean number indicates that there are a lot of small particles in all the melter feeds. The mean volume indicates the large particles, though not as numerous as the small particles, have a much larger impact on the volume. This is expected since volume is a cubic function of the diameter. There was little difference between the data sets. After 3 days and 7 days of mixing, there was little change in the LAW AZ-102 1.0M Na melter feed. This was also reflected in the physical and rheological results. The LAW AZ-102 1.3M Na melter feed had slightly larger particles than the LAW AZ-102 1.0M Na melter feed.

Table 3-22. Summary of Particle Size Distribution Data for Melter Feeds

LAW AZ-102 Melter Feed Description	Volume			Number		
	Mean		Median	Mean		Median
	µm	% Pass	µm	µm	% Pass	µm
1.0M Na, 3 days mixing	28.86	65.6	17.62	0.353	69.0	0.283
1.0M Na, 7 days mixing	27.32	64.2	16.89	0.361	68.6	0.287
1.3M Na, 1 day mixing	30.53	67.4	17.08	0.354	68.6	0.284

3.8 COMPOSITIONAL DATA

3.8.1 Pretreated Waste Composition

The LAW AZ-102 pretreated waste compositions were not analytically measured, but calculated. To calculate the pretreated waste compositions, the AZ-102 concentrate analysis of the original LAW AZ-102 4.38M Na pretreated waste reported in Table 5 of WSCR-TR-20001-00395 [Ref. 5] was used. The data reported in Table 5 [Ref. 5] are in units of radioactivity per unit volume, mass per unit volume or molarity. The average analyte values in Table 5 [Ref. 5] which have a reportable level above the minimum reportable quantity levels were used. The volumes of the 1.0M Na and 1.3M Na and that of the 4.38M Na pretreated waste were determined after the diluted feeds were corrected (via evaporation) for their targeted molarities and the volumes are shown in Table 3-23.

Table 3-23. Volume of Pretreated Waste

Pretreated waste	Volume (mL)	
	4.38M Na Solution Used	Final waste
LAW AZ-102 1.0M Na	50.20	187.45
LAW AZ-102 1.3M Na	31.55	86.95

The LAW AZ-102 1.0M Na and 1.3M Na pretreated waste analytes were determined using Equation 3-8 and the results are shown in Table 3-24.

Equation 3-8 $$\rho_{i,j} = \rho_{i,4.38M}\frac{V_{4.38M,j}}{V_j},\ \hat{\alpha}_{i,j} = \hat{\alpha}_{i,4.38M}\frac{V_{4.38M,j}}{V_j},\ M_{i,j} = M_{i,4.38M}\frac{V_{4.38M,j}}{V_j}$$

where: $\rho_{i,j}$ = density of analyte i for j M Na pretreated waste (mg/L or µg/L)
$\rho_{i,4.38M}$ = density of analyte i in the 4.38M Na pretreated waste (mg/L or µg/L)
$\hat{\alpha}_{i,j}$ = activity density of analyte i for j M Na pretreated waste (µCi/L)
$\hat{\alpha}_{i,4.38M}$ = activity density of analyte i in the 4.38M Na waste (µCi/L)
$M_{i,j}$ = molarity of analyte i for j M Na pretreated waste (moles/L)
$M_{i,4.38M}$ = molarity of analyte i in the 4.38M Na waste(moles/L)
V_j = volume of the j M Na pretreated waste (L)
$V_{4.38M,j}$ = volume of 4.38M Na used to make the j M Na pretreated waste (L)

Table 3-24. Calculated 1.0M Na and 1.3M Na LAW Pretreated Waste Compositional Data

Analytes	Units	LAW AZ-102 Pretreated Waste		Analytes	Units	LAW AZ-102 Pretreated Waste	
		1.0M Na	1.3M Na			1.0M Na	1.3M Na
Cs-137	µCi/L	329	446	NO_2	mg/L	13239	17937
Sr-90	µCi/L	332	450	SO_4	mg/L	7043	9542
Tc-99	µg/L	1.6	2.2	Oxalate	mg/L	1238	1678
235	µg/L	9.3	12.7	F	mg/L	367	497
237	µg/L	53.5	72.5	Al	mg/L	213	289
238	µg/L	1024.3	1387.8	Ca	mg/L	17.9	24.3
239	µg/L	20.1	27.3	Cr	mg/L	272	369
K (AA)	mg/L	1220	1653	Mg	mg/L	0.8	1.1
Na (AA)**	mg/L	23450	30576	Mo	mg/L	21.2	28.7
Inorganic (TIC)	mg/L	2516	3409	Na	mg/L	23449	30576
Organic (TOC)	mg/L	5333	7225	P	mg/L	61.5	83.3
Free OH (M)	M	0.29	0.40	Pb	mg/L	6.9	9.3
CO_3(M)	M	0.13	0.18	Si	mg/L	15.0	20.4
NO_3	mg/L	7312	9907	Sn	mg/L	4.6	6.2

** Measured values as reported

3.8.2 Melter Feed Composition

The LAW AZ-102 1.0M Na and 1.3M Na melter feed compositions were also calculated. The mass of the analytes in Table 3-25 was determined for each waste stream using the volume of the pretreated waste compositions stated in Table 3-6. The elemental/compound mass contribution from the GFCs was determined by taking the mass of GFC used (Table 3-6), multiplying this number by the wt% of the typical chemical makeup as stated in the summary data sheet of the specified GFC [Ref. 4] and then correcting it for elemental/ compound, if required. CO_2 will be reported as a compound (not carbon). Th/U in the Zircon sand will be reported as µg/L of Th/U, since there is no breakdown of the individual elements. The composition of the individual GFCs taken from reference 4 and used are provided in Appendix B. The volume of the melter feed was then determined by dividing the sum of the masses of the pretreated feed and GFCs (Table 3-6) by the density of the melter feed (Table 3-10). For the 1.0M Na melter feed, the 3 day density was used. The masses of each analyte from the pretreated feeds and GFCs were added and then divided by the calculated volume. The calculated melter feed compositions are shown in Table 3-26. Note that due to the addition of sodium carbonate, the Na molarity of the melter feed is higher than that of the pretreated feed.

Table 3-25. Calculated 1.0M Na and 1.3M Na LAW Melter Feed Compositional Data

Analytes	Units	LAW AZ-102 Melter Feed		Analytes	Units	LAW AZ-102 Melter Feed	
		1.0M Na	1.3M Na			1.0M Na	1.3M Na
Cs-137	µCi/L	220	273	Al	mg/L	12931	15465
Sr-90	µCi/L	222	275	B	mg/L	23596	28194
Tc-99	µg/L	1.1	1.33	Ca	mg/L	36434	43538
235	µg/L	6.3	7.7	Cr	mg/L	236	290
237	µg/L	35.8	44.3	Fe	mg/L	27877	33316
238	µg/L	685.0	848.3	Li	mg/L	5773	6896
239	µg/L	13.5	16.7	Mg	mg/L	13672	16336
U+Th	µg/L	16.3	19.5	Mn	mg/L	107	128
K (AA)	mg/L	816	1010	Mo	mg/L	14.2	17.5
Na (AA)	mg/L	29572	35308	Na	mg/L	29572	35308
Inorganic (TIC)	mg/L	1683	2084	Nb	mg/L	15.7	18.7
Organic (TOC)	mg/L	3566	4417	Ni	mg/L	136	162
Free OH (M)	M	0.20	0.24	P	mg/L	83	101
CO_3(M)	M	0.09	0.11	Pb	mg/L	4.6	5.7
CO_2	mg/L	60992	72887	Si	mg/L	171015	204310
NO_3	mg/L	4890	6056	Sn	mg/L	3.0	3.8
NO_2	mg/L	8854	10965	Ti	mg/L	6789	8116
SO_4	mg/L	4710	5833	V	mg/L	28.2	33.8
Oxalate	mg/L	828	1026	Zn	mg/L	29123	34809
F	mg/L	245	304	Zr	mg/L	17750	21234

3.9 DISCUSSION ON BOUNDING PHYSICAL LIMITS AND DESIGN ISSUES

The documents listed in Table 3-26 have been reviewed for bounding physical limits and design limits. Issues related to these documents, such as the shear strength are discussed in section 3.9.1 and pipeline transport issues and agitator speed for homogeneity are discussed in section 3.9.2. Rheological measurements with respect to the project-approved Guidelines [Ref. 1] are discussed in section 3.9.3.

Table 3-26. Bounding and Design Limit Documents

Document Number	Ref.	Document Provides
WTP-RPT-075	21	• Basis for shear strength, using DWPF design • Basis for lower rheological limit, using settling and general statement about mixing. • Maximum transport velocity. Uses DWPF pump design.
24590-WTP-3PS-MPCO-T0006	22	• RPP-WTP engineering LAW vitrification pumps specifications.
24590-LAW-MPC-LFP-00001	23	• RPP-WTP engineering calculation for LAW vitrification pump sizing.
24590-LAW-M6C-LFP-00001	24	• RPP-WTP engineering calculation for LAW melter feed preparation vessel volume levels.
24590-WTP-3PS-MACS-T0003	25	• RPP-WTP engineering agitator specifications.

3.9.1 Settled Solids Shear Strength

The basis for the maximum shear strength limit is provided in WTP-RPT-075 and is described in detail in section 4.1.3. The basis assumes a 100 HP motor starting torque, 36” impeller diameter, 7.2” impeller width, and only one impeller completely immersed into settled slurry. The agitator parameters are based on the DWPF agitator design. The calculated maximum shear strength is 625Pa, based on these parameters and using the equation in Figure 4.2 of WTP-RPT-075. This maximum shear strength is also stated in 24590-LAW-M6C-LFP-00001, with the provision that the motor be sized to provide 1.5 times the maximum shear strength of 625 Pa. The LAW AZ-102 1.3M Na melter feed settled shear strength was measured at 2349Pa, which well exceeds the basis calculation and agitator design limit. Table 3-27 provides a list of issues that should be considered relative to the settled solids shear strength measurements.

Table 3-27. Issues with Shear Strength

#	Issue
1	WTP-RPT-075: Calculation was based on DWPF design, not on actual physical data.
2	WTP-RPT-075: Calculations used to determine the minimum yield stress necessary to prevent cavern mixing, have not been performed. How this compares to the shear strength and physical data needs to be considered. Problems with the development of a new tank bottom can develop if the settled solids are continually not being re-suspended by the agitator. Cavern formation can be determined using calculations outlined in references 26 through 29.
3	WTP-RPT-075: Calculations in this document must be performed using WTP design information, as it becomes available.
4	2490-LAW-M6C-LFP-00001: Shows that the top surface of the upper impeller for the LAW melter feed preparation vessel (MFPV) is approximately a factor of 0.653 of the nominal operating volume. If that condition exists, and electrical power was lost when the vessel was full, then both impellers would be totally covered by settled solids. Basis calculation and design do not consider that the top impeller has the potential to be covered by settled solids. Covering of the top impeller must be considered.
5	24590-WTP-3PS-MPCO-T0006: Pump operation is undetermined at the time of this review. One of the pumps uses an eductor priming line. Inactive use of this pump could be an issue, especially if the LAW material builds strength in inactive mixing zones, such as the pump suction line, pump casing, and eductor priming line. Physical testing should be performed to determine the frequency at which the inactive pump must be operated.
6	General Issue: Settled solids in the pipe line will become an issue, e.g. in the case where power is lost during transfer or if materials are not properly flushed from process lines. Flushing capabilities (e.g. operational requirements) are unknown, at this time. Minimum flush velocity should be measured and conditions of testing to cover potential plant operating upset conditions..

3.9.2 Transport Velocities and Homogeneous Mixing

The LAW AZ-102 melter feeds are settling heterogeneous slurries which was obvious based on the results of the settling and rheology tests. For these types of slurries, a transitional (deposition) velocity as a minimum must be determined for pipe flow. The pipe velocity for homogenous flow may be required based on actual process conditions, and may be calculated using existing correlations. Existing correlations used to determine the transitional and homogenous flow velocities are available, but due to the complex combination of solids in the melter feed, it may be difficult to apply these correlations with a high level of confidence. Typically, pipe test flow loops are used to properly quantify these velocity limits and to validate the utilization of a correlation. References 30 through 32 provide methods that can be used to determine the necessary velocities in the pipeline. The lower viscosity limit as specified in WTP-RPT-075 should be used in these calculations.

WTP-RPT-075 calculates a lower viscosity of 0.4 cP for homogenous mixing in an agitated tank, although no calculation was performed to determine agitator speed that would provide such homogeneity. Calculations or experimental data testing should be performed to determine the critical speed to achieve suspension (just off-bottom suspension), given the present design of the LAW MFPV and compared to the agitator speeds used in WTP-RPT-075. References 33 through 36 provide methods that can be used to determine the mixing speeds. If calculations are to be performed, they should be performed in WTP-RPT-075.

Use of any correlation may require obtaining the original technical document to determine the applicability of the correlation to the RPP-WTP process. The references listed are not all inclusive on work that has been performed concerning heterogeneous/homogenous mixing and transport issues.

3.9.3 Rheology Issues

The following rheological properties should be added to project-approved Guidelines [Ref. 1] or studied in more detail using simulants.

- The viscosity of the melter feed supernatant should be characterized. This would provide information about the LAW carrier fluid and could potentially be used to determine operating conditions. This can also be used for performing settling calculations.

- The settled solids shear strength as both a function of time and depth of settled solids, should also be determined. It was observed in this task that depth of settle solids shear strength and the rate at which the solid build strength are factors. Setting up of the melter feed settled solids were clearly evident within 15 to 30 minutes of lost agitation. The time at which the settled solids shear strength measurements are taken in settling slurry is also a factor. Settled solids shear strength needs to be studied to determine if mixing issues, such as agitator restart and solids resuspension, are possible given the current baseline design conditions.

3.9.4 Other Issues

The bubbles (foaming) due to the addition of GFCs (section 3.3.2) were from the addition of dry GFCs to the AZ-102 LAW pretreated waste stream. Foaming during processing could be an issue, if the foam gets up into areas of the MFPV where wetting would impact the operations of installed instrumentation or processes (e.g. GFC addition line) attached to the MFPV. The present RPP-WTP GFC delivery system at the vitrification facility will blend the dry GFCs with a predetermined amount of water prior to adding the GFCs to the MFPV via a chute. The wetted GFCs process has been designed to mitigate potential dusting issues related to delivering GFCs to the MFPV. No studies have been completed to date that investigated the potential impact of foaming relative to wetted (or dry) GFCs being blended into the MFPV. Because the final GFC delivery system and its interface with the MFPV are still being developed, the foaming issue will have to be addressed at a later date, preferably on a pilot scale process.

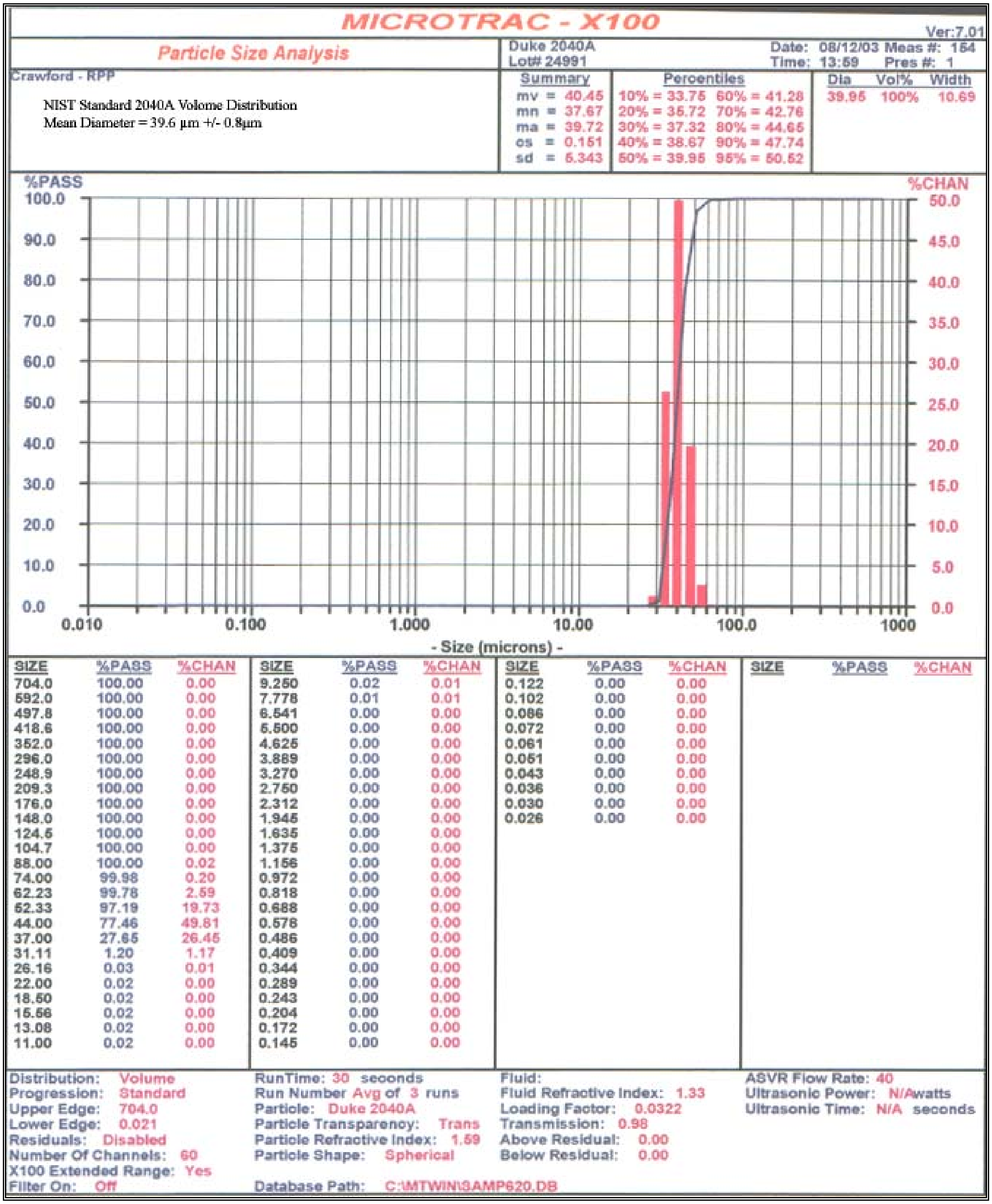
This page intentionally left blank.

4.0 REFERENCES

1. Smith, G.L. and Prindiville, K., *"Guidelines for Performing Chemical, Physical, and Rheological Properties Measurements"*, 24590-WTP-GPG-RTD-001, Rev. 0, May 20, 2002
2. *"Test Exception to AZ-102 (Envelope B) LAW Vitrification Product Testing 24590-LAW-TSP-RT-01-024, Rev. 0"*, 24590-WTP-TEF-RT-03-005, April 3, 2003
3. Crawford, C.L., "RPP Test Specification: AZ102 (Envelope B) LAW Vitrification, Product Testing; Document # 24590-LAW-TSP-RT-01-024, Rev. 0", WSRC-TR-2002-00153, Rev. 0, May 6, 2002
4. Schumacher, R. *"Characterization of HLW and LAW Glass Formers – Final Report"*, WSCR-TR-2002-00282, Rev. 1, July 15, 2003
5. Crawford, C.L., Ferrara, D.M., Schumacher, R.F. and Bibler, N.E., *"Interim Report for Crucible Scale Active Vitrification of Waste Envelop B (AZ-102) (U)"*, WSRC-TR-2001-00395, March 25, 2002
6. Email from Tommy Edwards to Erich Hansen, Subject Re: Follow Up, 8/28/03
7. Liddell, P.V. and Boger D.V., "*Yield Stress Measurements with the vane*", Journal of Non-Newtonian Fluid Mechanics, Vol. 63, pp 235-261, 1996
8. Nguyen, Q.D. and Boger D.V., "*Yield Stress Measurements for Concentrated Suspensions*", Journal of Rheology, Vol. 27, pp 321-349, 1983
9. Nguyen, Q.D. and Boger D.V., "*Direct Yield Stress Measurement with the Vane Method", Journal of Rheology*", Vol. 29, pp 335-347, 1985
10. Barnes, H.A. and Nguyen, Q.D., "*Review Rotating vane rheometry – a review*", Journal of Non-Newtonian Fluid Mechanics, Vol. 98, pp 1-14, 2001
11. Saak, A.W., Jennings, H.M., and Shah S.P., "*The influence of wall slip on yield stress and viscoelastic measurements of cement paste*", Cement and Concrete Research, Vol. 31, pp 205-212, 2001
12. Barnes, H. A. and Carnali, J. O., "*The vane-in-cup as a novel rheometer geometry for shear thinning and thixotropic materials*", Journal of Rheology, Vol. 34, pp 841-866, 1990
13. Yoshimura, A. S., and Prud'Homme, P.K., "*A Comparison of Techniques for Measuring Yield Stresses",* Journal of Rheology, Vol. 31, pp 699-710, 1987
14. Alderman, N. J., Meeten, G. H., and Sherwood, J. D., "*Vane rheometry of bentonite gel",* Journal of Non-Newtonian Fluid Mechanics, Vol. 91, pp 291-310, 1991
15. Mohseni, M., Kautola, H., and Allen D.G., "*The Viscoelastic Nature of Filamentous Fermentation Broths and Its Influence on the Directly Measured Yield Stress*", Journal of Fermentation and Bioengineering, Vol. 83, pp 281-286, 1997
16. Keating, J. and Hannant, D. J., "*The Effect of Rotation Rate on Gel Strength and Dynamic Yield Strength of Thixotropic Oil Well Cements Measured Using a Shear Vane*", Journal of Rheology, Vol. 33, pp 1011-1020, 1989
17. Wardhaugh, L. T. and Boger, D. V., "*The measurement and description of the yielding behavior of waxy crude oil*", Journal of Rheology, Vol. 35, pp 1121-1165, 1991
18. Yan, J. and Jame, A.E., 'The yield surface of viscoelastic and plastic fluids in a vane viscometer", Journal of Non-Newtonian Fluid Mechanics", Vol. 70, pp 237-253, 1997
19. Turian, R. M., Ma, T. W., Hsu, F.L.G., and Sung, D. J., "*Characterization, settling, and rheology of concentrated fine particle mineral slurries*", Power Technology, Vol. 93, pp. 219-233, 1997

20. Iyer, R.S. and Stanmore B., "*The effect of water absorption and the role of fines on the yield stress of dense fly ash slurries*", Cement and Concrete Research, Volume 29, pp 765-767, 1999
21. Poloski, A., Smith, H., Smith., G., and Calloway B., WTP-RPT-075, Rev. 0, "*Interim Report: Development of LAW and HLW Vitrification Physical Property Bounding Conditions and Simulant Verification Criteria*", March 2003
22. 24590-WTP-3PS-MPCO-T0006, "*RPP-WTP Engineering Specification for Vessel-Mounted Vertical Transfer Pumps",* Rev. 0, August 15, 2002
23. Calc. No. 24590-LAW-MPC-LFP-00001, "*Melter 1 Feed Prep Vessel Pumps LFP-PMP-00001A, 1B",* Rev. C, June 29, 2003
24. Calc. No. 24590-LAW-M6C-LFP-00001, "*LFP-VSL-00001, 00003, 00005 Sizing*", Rev. 0A, December 13, 2002
25. 24590-WTP-3PS-MACS-T0003, "*RPP-WTP Engineering Specification For Mechanical Agitators*", Rev. 1,
26. Elson, T. P., Cheesman, D. J, and A. W. Nienow, "*X-Ray Studies of Cavern Sizes and Mixing Performance With Fluids Possessing a Yield Stress*", Chemical Engineering Science, Vol. 41, pp. 2555-2562, 1986
27. Etchells, A. W, Ford, W.N, and Short, D. G. R., "*Mixing of Bingham Plastics On An Industrial Scale*", Fluid Mixing III, pp. 271-285, Institution of Chemical Engineers, 1988
28. Elson, T.P., "*The Growth of Caverns Formed Around Rotating Impellers During the Mixing of Yield Stress Fluids*", Vol. 96, pp. 303-319, Chemical Engineering Communications, 1990
29. Hirata, Y. and Aoshima Y., "*Formation and Growth of Cavern in Yield Stress Fluids Agitated Under Baffled and Non-Baffled Conditions*", Vol. 74, pp. 438-444, Institution of Chemical Engineers, May 1996
30. Wasp, E. J., "*Solid-Liquid Flow Slurry Pipeline Transportation*", Vol. 1, No. 4, Trans Tech Publications, 1977
31. Govier, G. W. and Aziz, K., "*The Flow of Complex Mixtures in Pipe*", Van Nostrand Reinhold Company, 1972
32. Shook, C. A. and Roco, M. C., "*Slurry Flow Principle and Practice*", Butterworth Heinemann, 1991
33. Zwietering, T. N., "*Suspending of solid particles in liquid by agitators*", Vol. 8, pp 244-253, Chemical Engineering Science, 1958
34. Uhl, V. W. and Gray, J. B., "*Mixing Theory and Practice – Volume II*", Academic Press, 1967
35. Oldshue, J. Y., "*Fluid Mixing Technology*", McGraw-Hill, 1983
36. Baldi, G., Conti, R., and Alaria, E., "*Complete Suspension of Particles in Mechanically Agitated Vessels*", Vol. 33, pp 21-25, Chemical Engineering Science, 1978

APPENDIX A.
PARTICLE SIZE DISTRIBUTION DATA



MICROTRAC - X100

Ver:7.01

Particle Size Analysis

Duke 2040A
Lot# 24991

Date: 08/12/03 Meas #: 154
Time: 13:59 Pres #: 1

Crawford - RPP

NIST Standard 2040A Volome Distribution
Mean Diameter = 39.6 µm +/- 0.8µm

Summary	Percentiles		Dia	Vol%	Width
mv = 40.45	10% = 33.75	60% = 41.28	39.95	100%	10.69
mn = 37.67	20% = 35.72	70% = 42.76			
ma = 39.72	30% = 37.32	80% = 44.65			
cs = 0.151	40% = 38.67	90% = 47.74			
sd = 5.343	50% = 39.95	95% = 50.52			

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	0.02	0.01	0.122	0.00	0.00			
592.0	100.00	0.00	7.778	0.01	0.01	0.102	0.00	0.00			
497.8	100.00	0.00	6.541	0.00	0.00	0.086	0.00	0.00			
418.6	100.00	0.00	5.500	0.00	0.00	0.072	0.00	0.00			
352.0	100.00	0.00	4.625	0.00	0.00	0.061	0.00	0.00			
296.0	100.00	0.00	3.889	0.00	0.00	0.051	0.00	0.00			
248.9	100.00	0.00	3.270	0.00	0.00	0.043	0.00	0.00			
209.3	100.00	0.00	2.750	0.00	0.00	0.036	0.00	0.00			
176.0	100.00	0.00	2.312	0.00	0.00	0.030	0.00	0.00			
148.0	100.00	0.00	1.945	0.00	0.00	0.026	0.00	0.00			
124.5	100.00	0.00	1.635	0.00	0.00						
104.7	100.00	0.00	1.375	0.00	0.00						
88.00	100.00	0.02	1.156	0.00	0.00						
74.00	99.98	0.20	0.972	0.00	0.00						
62.23	99.78	2.59	0.818	0.00	0.00						
52.33	97.19	19.73	0.688	0.00	0.00						
44.00	77.46	49.81	0.578	0.00	0.00						
37.00	27.65	26.45	0.486	0.00	0.00						
31.11	1.20	1.17	0.409	0.00	0.00						
26.16	0.03	0.01	0.344	0.00	0.00						
22.00	0.02	0.00	0.289	0.00	0.00						
18.50	0.02	0.00	0.243	0.00	0.00						
15.56	0.02	0.00	0.204	0.00	0.00						
13.08	0.02	0.00	0.172	0.00	0.00						
11.00	0.02	0.00	0.145	0.00	0.00						

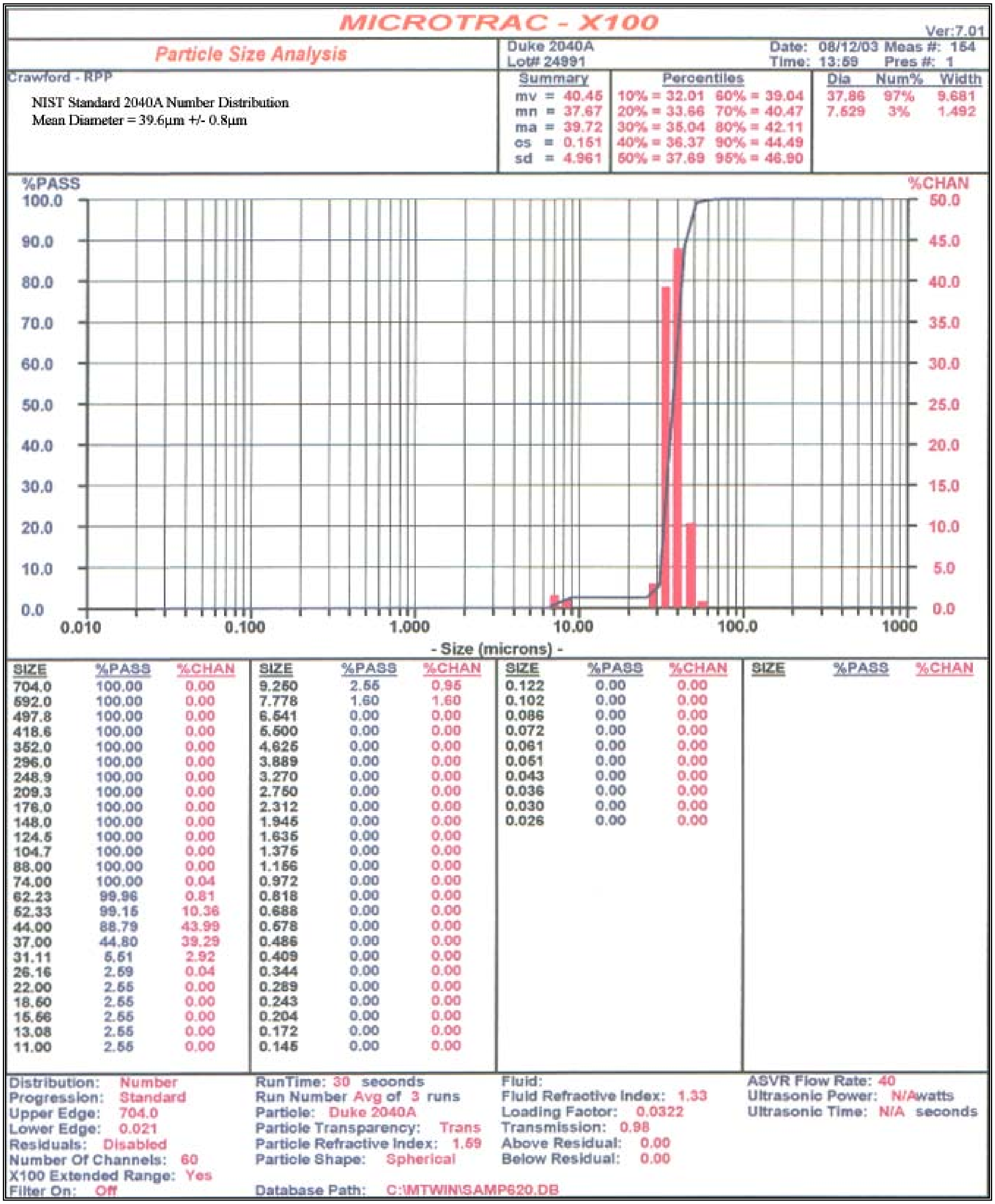
Distribution: Volume
Progression: Standard
Upper Edge: 704.0
Lower Edge: 0.021
Residuals: Disabled
Number Of Channels: 60
X100 Extended Range: Yes
Filter On: Off

RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Duke 2040A
Particle Transparency: Trans
Particle Refractive Index: 1.59
Particle Shape: Spherical
Database Path: C:\MTWIN\SAMP620.DB

Fluid:
Fluid Refractive Index: 1.33
Loading Factor: 0.0322
Transmission: 0.98
Above Residual: 0.00
Below Residual: 0.00

ASVR Flow Rate: 40
Ultrasonic Power: N/Awatts
Ultrasonic Time: N/A seconds

Figure A- 1. Initial Volume PSD – 2040A Standard



MICROTRAC - X100

Ver:7.01

Particle Size Analysis

Duke 2040A
Lot# 24991

Date: 08/12/03 Meas #: 154
Time: 13:59 Pres #: 1

Crawford - RPP

NIST Standard 2040A Number Distribution
Mean Diameter = 39.6µm +/- 0.8µm

Summary	Percentiles		Dia	Num%	Width
mv = 40.45	10% = 32.01	60% = 39.04	37.86	97%	9.681
mn = 37.67	20% = 33.66	70% = 40.47	7.629	3%	1.492
ma = 39.72	30% = 35.04	80% = 42.11			
cs = 0.151	40% = 36.37	90% = 44.49			
sd = 4.961	50% = 37.69	95% = 46.90			

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	2.55	0.95	0.122	0.00	0.00			
592.0	100.00	0.00	7.778	1.60	1.60	0.102	0.00	0.00			
497.8	100.00	0.00	6.541	0.00	0.00	0.086	0.00	0.00			
418.6	100.00	0.00	5.500	0.00	0.00	0.072	0.00	0.00			
352.0	100.00	0.00	4.625	0.00	0.00	0.061	0.00	0.00			
296.0	100.00	0.00	3.889	0.00	0.00	0.051	0.00	0.00			
248.9	100.00	0.00	3.270	0.00	0.00	0.043	0.00	0.00			
209.3	100.00	0.00	2.750	0.00	0.00	0.036	0.00	0.00			
176.0	100.00	0.00	2.312	0.00	0.00	0.030	0.00	0.00			
148.0	100.00	0.00	1.945	0.00	0.00	0.026	0.00	0.00			
124.5	100.00	0.00	1.635	0.00	0.00						
104.7	100.00	0.00	1.375	0.00	0.00						
88.00	100.00	0.00	1.156	0.00	0.00						
74.00	100.00	0.04	0.972	0.00	0.00						
62.23	99.96	0.81	0.818	0.00	0.00						
52.33	99.15	10.36	0.688	0.00	0.00						
44.00	88.79	43.99	0.578	0.00	0.00						
37.00	44.80	39.29	0.486	0.00	0.00						
31.11	5.51	2.92	0.409	0.00	0.00						
26.16	2.59	0.04	0.344	0.00	0.00						
22.00	2.55	0.00	0.289	0.00	0.00						
18.50	2.55	0.00	0.243	0.00	0.00						
15.56	2.55	0.00	0.204	0.00	0.00						
13.08	2.55	0.00	0.172	0.00	0.00						
11.00	2.55	0.00	0.145	0.00	0.00						

Distribution: Number
Progression: Standard
Upper Edge: 704.0
Lower Edge: 0.021
Residuals: Disabled
Number Of Channels: 60
X100 Extended Range: Yes
Filter On: Off

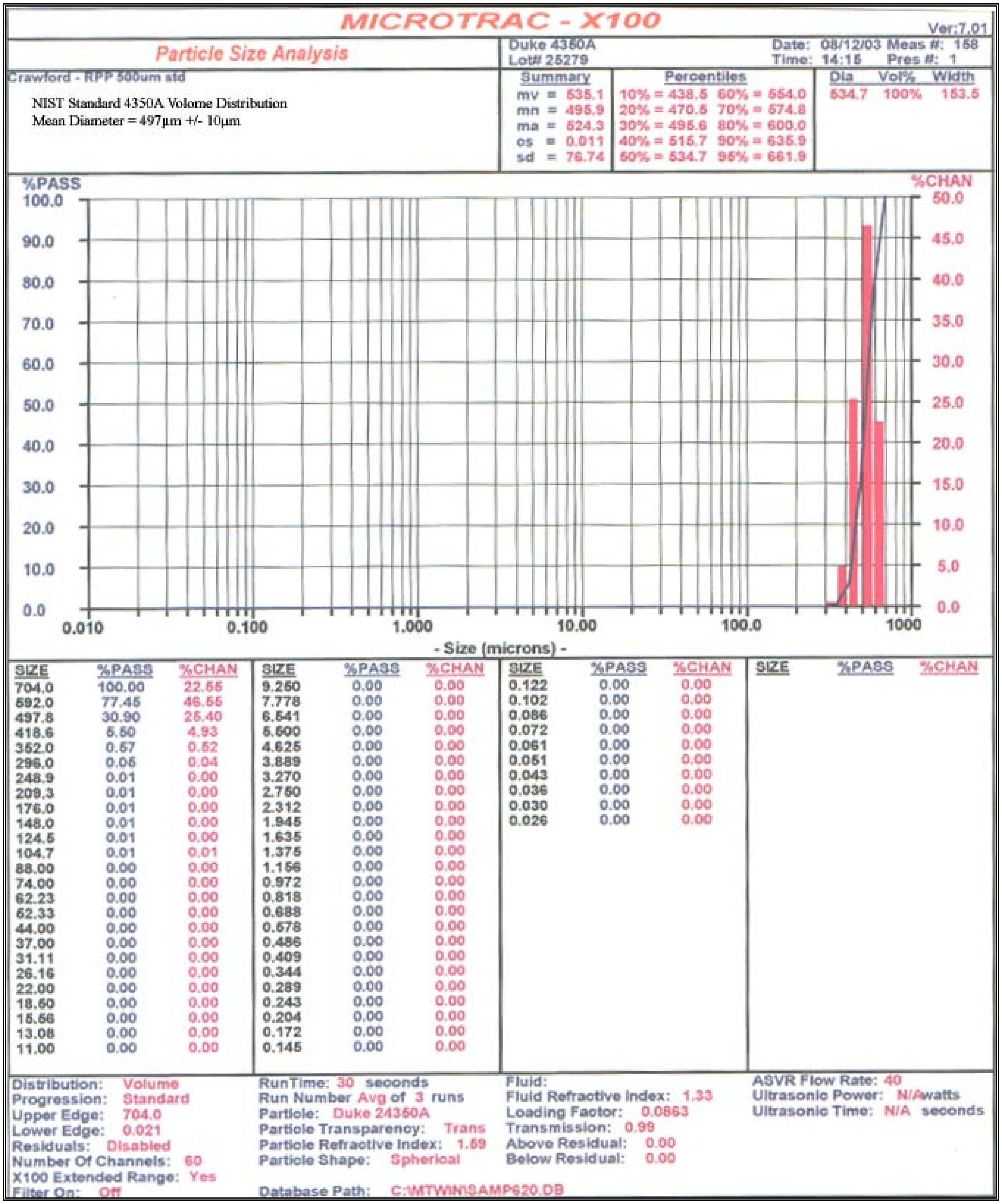
RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Duke 2040A
Particle Transparency: Trans
Particle Refractive Index: 1.59
Particle Shape: Spherical

Database Path: C:\MTWIN\SAMP620.DB

Fluid:
Fluid Refractive Index: 1.33
Loading Factor: 0.0322
Transmission: 0.98
Above Residual: 0.00
Below Residual: 0.00

ASVR Flow Rate: 40
Ultrasonic Power: N/Awatts
Ultrasonic Time: N/A seconds

Figure A- 2. Initial Number PSD – 2040A Standard



MICROTRAC - X100

Ver:7.01

Particle Size Analysis

Duke 4350A
Lot# 25279

Date: 08/12/03 Meas #: 158
Time: 14:15 Pres #: 1

Crawford - RPP 500um std

NIST Standard 4350A Volome Distribution
Mean Diameter = 497µm +/- 10µm

Summary	Percentiles	Percentiles	Dia	Vol%	Width
mv = 535.1	10% = 438.5	60% = 554.0	534.7	100%	153.5
mn = 495.9	20% = 470.5	70% = 574.8			
ma = 524.3	30% = 495.6	80% = 600.0			
cs = 0.011	40% = 515.7	90% = 635.9			
sd = 76.74	50% = 534.7	95% = 661.9			

SIZE	%PASS	%CHAN
704.0	100.00	22.55
592.0	77.45	46.55
497.8	30.90	25.40
418.6	5.50	4.93
352.0	0.57	0.52
296.0	0.05	0.04
248.9	0.01	0.00
209.3	0.01	0.00
176.0	0.01	0.00
148.0	0.01	0.00
124.5	0.01	0.00
104.7	0.01	0.01
88.00	0.00	0.00
74.00	0.00	0.00
62.23	0.00	0.00
52.33	0.00	0.00
44.00	0.00	0.00
37.00	0.00	0.00
31.11	0.00	0.00
26.16	0.00	0.00
22.00	0.00	0.00
18.50	0.00	0.00
15.56	0.00	0.00
13.08	0.00	0.00
11.00	0.00	0.00
9.250	0.00	0.00
7.778	0.00	0.00
6.541	0.00	0.00
5.500	0.00	0.00
4.625	0.00	0.00
3.889	0.00	0.00
3.270	0.00	0.00
2.750	0.00	0.00
2.312	0.00	0.00
1.945	0.00	0.00
1.635	0.00	0.00
1.375	0.00	0.00
1.156	0.00	0.00
0.972	0.00	0.00
0.818	0.00	0.00
0.688	0.00	0.00
0.578	0.00	0.00
0.486	0.00	0.00
0.409	0.00	0.00
0.344	0.00	0.00
0.289	0.00	0.00
0.243	0.00	0.00
0.204	0.00	0.00
0.172	0.00	0.00
0.145	0.00	0.00
0.122	0.00	0.00
0.102	0.00	0.00
0.086	0.00	0.00
0.072	0.00	0.00
0.061	0.00	0.00
0.051	0.00	0.00
0.043	0.00	0.00
0.036	0.00	0.00
0.030	0.00	0.00
0.026	0.00	0.00

Distribution: Volume
Progression: Standard
Upper Edge: 704.0
Lower Edge: 0.021
Residuals: Disabled
Number Of Channels: 60
X100 Extended Range: Yes
Filter On: Off

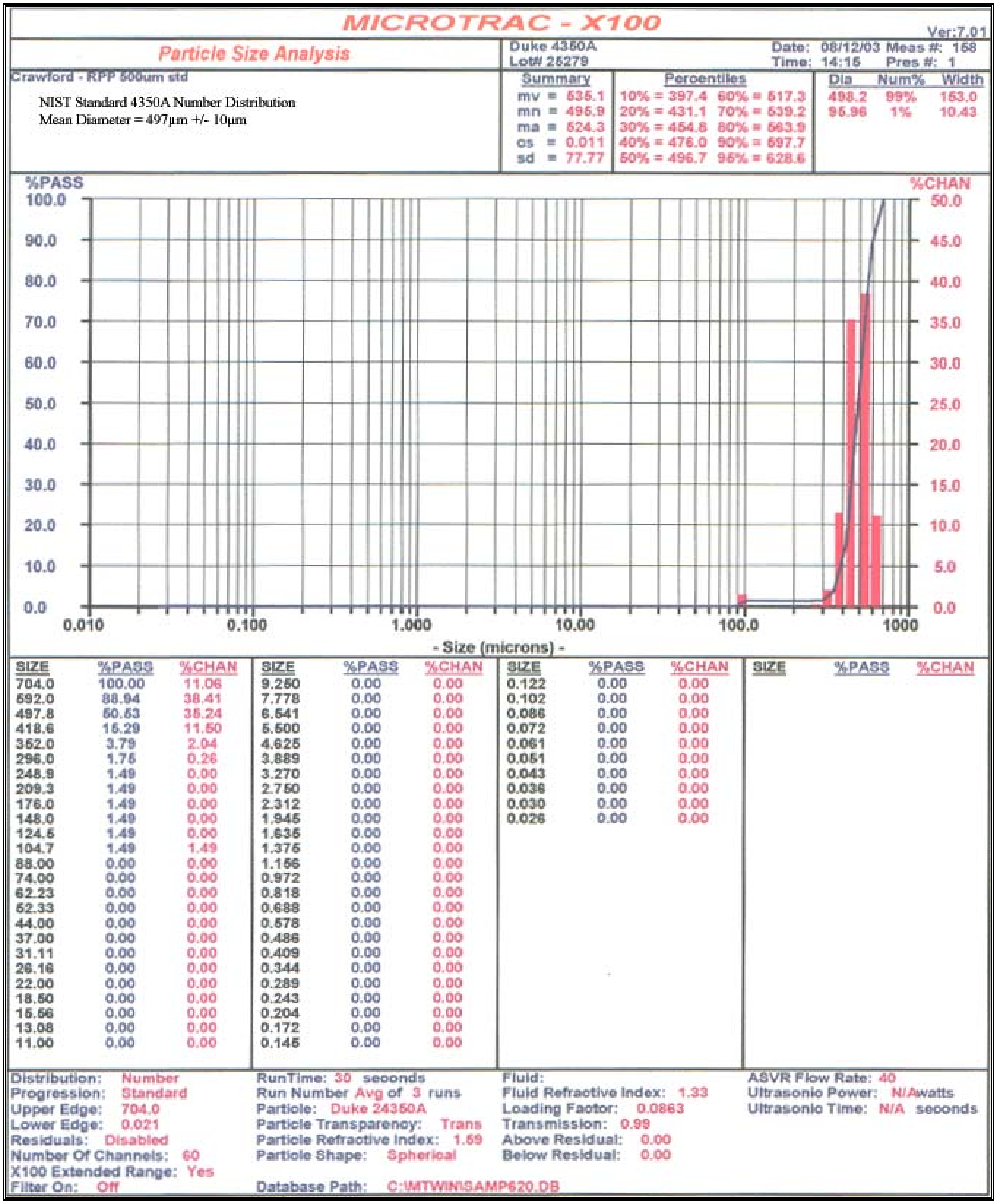
RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Duke 24350A
Particle Transparency: Trans
Particle Refractive Index: 1.59
Particle Shape: Spherical

Database Path: C:\MTWIN\SAMP620.DB

Fluid:
Fluid Refractive Index: 1.33
Loading Factor: 0.0863
Transmission: 0.99
Above Residual: 0.00
Below Residual: 0.00

ASVR Flow Rate: 40
Ultrasonic Power: N/Awatts
Ultrasonic Time: N/A seconds

Figure A- 3. Initial Volume PSD – 4350A Standard



MICROTRAC - X100

Ver:7.01

Particle Size Analysis

Duke 4350A
Lot# 25279

Date: 08/12/03 Meas #: 158
Time: 14:15 Pres #: 1

Crawford - RPP 500um std

NIST Standard 4350A Number Distribution
Mean Diameter = 497µm +/- 10µm

Summary
mv = 535.1
mn = 495.9
ma = 524.3
cs = 0.011
sd = 77.77

Percentiles
10% = 397.4
20% = 431.1
30% = 454.8
40% = 476.0
50% = 496.7
60% = 517.3
70% = 539.2
80% = 563.9
90% = 597.7
95% = 628.6

Dia	Num%	Width
498.2	99%	153.0
95.96	1%	10.43

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	11.06	9.250	0.00	0.00	0.122	0.00	0.00			
592.0	88.94	38.41	7.778	0.00	0.00	0.102	0.00	0.00			
497.8	50.53	35.24	6.541	0.00	0.00	0.086	0.00	0.00			
418.6	15.29	11.50	5.500	0.00	0.00	0.072	0.00	0.00			
352.0	3.79	2.04	4.625	0.00	0.00	0.061	0.00	0.00			
296.0	1.75	0.26	3.889	0.00	0.00	0.051	0.00	0.00			
248.9	1.49	0.00	3.270	0.00	0.00	0.043	0.00	0.00			
209.3	1.49	0.00	2.750	0.00	0.00	0.036	0.00	0.00			
176.0	1.49	0.00	2.312	0.00	0.00	0.030	0.00	0.00			
148.0	1.49	0.00	1.945	0.00	0.00	0.026	0.00	0.00			
124.5	1.49	0.00	1.635	0.00	0.00						
104.7	1.49	1.49	1.375	0.00	0.00						
88.00	0.00	0.00	1.156	0.00	0.00						
74.00	0.00	0.00	0.972	0.00	0.00						
62.23	0.00	0.00	0.818	0.00	0.00						
52.33	0.00	0.00	0.688	0.00	0.00						
44.00	0.00	0.00	0.578	0.00	0.00						
37.00	0.00	0.00	0.486	0.00	0.00						
31.11	0.00	0.00	0.409	0.00	0.00						
26.16	0.00	0.00	0.344	0.00	0.00						
22.00	0.00	0.00	0.289	0.00	0.00						
18.50	0.00	0.00	0.243	0.00	0.00						
15.56	0.00	0.00	0.204	0.00	0.00						
13.08	0.00	0.00	0.172	0.00	0.00						
11.00	0.00	0.00	0.145	0.00	0.00						

Distribution: Number
Progression: Standard
Upper Edge: 704.0
Lower Edge: 0.021
Residuals: Disabled
Number Of Channels: 60
X100 Extended Range: Yes
Filter On: Off

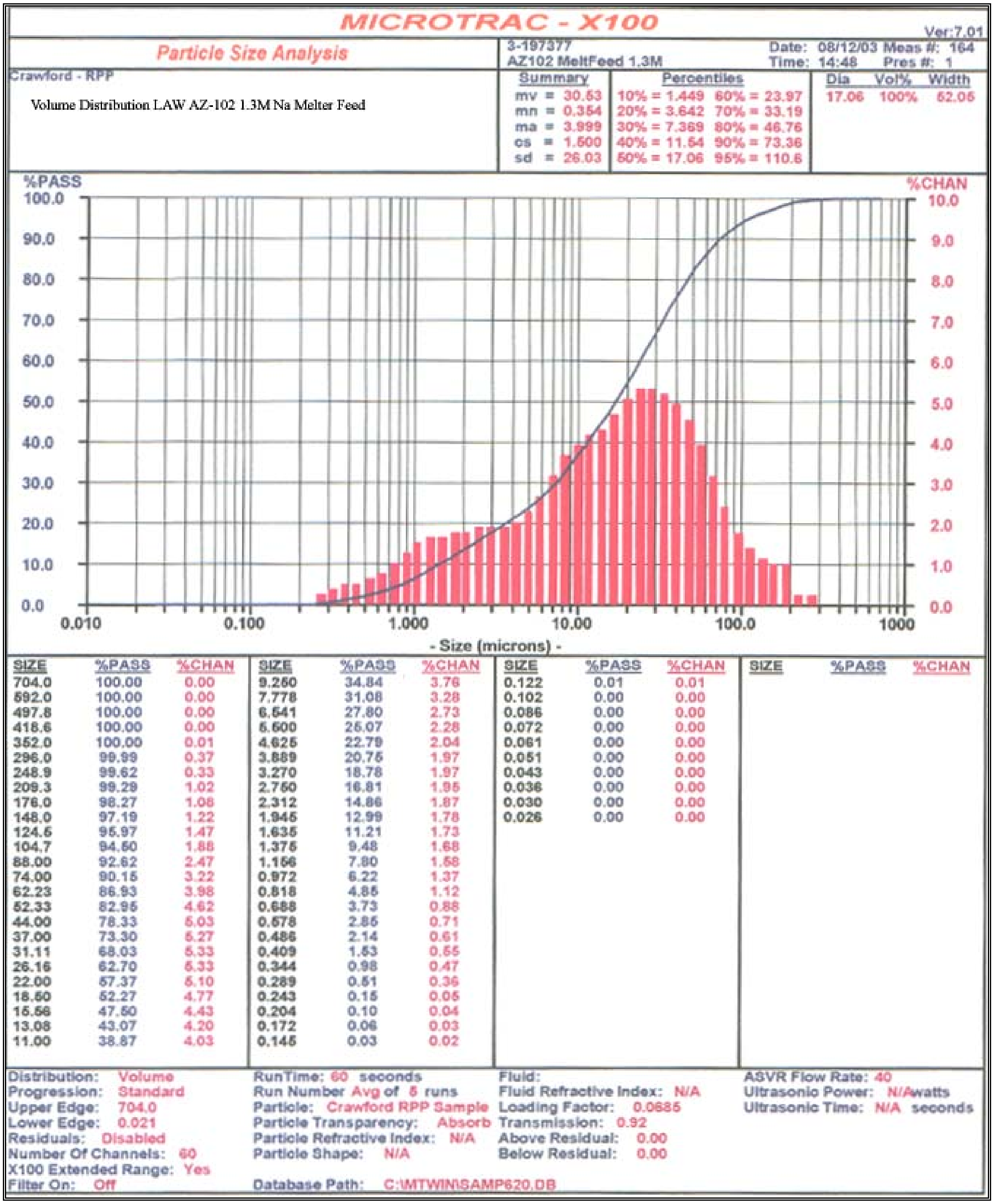
RunTime: 30 seconds
Run Number Avg of 3 runs
Particle: Duke 24350A
Particle Transparency: Trans
Particle Refractive Index: 1.59
Particle Shape: Spherical

Database Path: C:\MTWIN\SAMP620.DB

Fluid:
Fluid Refractive Index: 1.33
Loading Factor: 0.0863
Transmission: 0.99
Above Residual: 0.00
Below Residual: 0.00

ASVR Flow Rate: 40
Ultrasonic Power: N/Awatts
Ultrasonic Time: N/A seconds

Figure A- 4. Initial Number PSD – 4350A Standard



MICROTRAC - X100

Ver:7.01

Particle Size Analysis

3-197377
AZ102 MeltFeed 1.3M

Date: 08/12/03 Meas #: 164
Time: 14:48 Pres #: 1

Crawford - RPP

Volume Distribution LAW AZ-102 1.3M Na Melter Feed

Summary	Percentiles	
mv = 30.53	10% = 1.449	60% = 23.97
mn = 0.354	20% = 3.642	70% = 33.19
ma = 3.999	30% = 7.369	80% = 46.76
cs = 1.500	40% = 11.54	90% = 73.36
sd = 26.03	50% = 17.06	95% = 110.6

Dia	Vol%	Width
17.06	100%	52.05

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	34.84	3.76	0.122	0.01	0.01			
592.0	100.00	0.00	7.778	31.08	3.28	0.102	0.00	0.00			
497.8	100.00	0.00	6.541	27.80	2.73	0.086	0.00	0.00			
418.6	100.00	0.00	5.500	25.07	2.28	0.072	0.00	0.00			
352.0	100.00	0.01	4.625	22.79	2.04	0.061	0.00	0.00			
296.0	99.99	0.37	3.889	20.75	1.97	0.051	0.00	0.00			
248.9	99.62	0.33	3.270	18.78	1.97	0.043	0.00	0.00			
209.3	99.29	1.02	2.750	16.81	1.95	0.036	0.00	0.00			
176.0	98.27	1.08	2.312	14.86	1.87	0.030	0.00	0.00			
148.0	97.19	1.22	1.945	12.99	1.78	0.026	0.00	0.00			
124.5	95.97	1.47	1.635	11.21	1.73						
104.7	94.50	1.88	1.375	9.48	1.68						
88.00	92.62	2.47	1.156	7.80	1.58						
74.00	90.15	3.22	0.972	6.22	1.37						
62.23	86.93	3.98	0.818	4.85	1.12						
52.33	82.95	4.62	0.688	3.73	0.88						
44.00	78.33	5.03	0.578	2.85	0.71						
37.00	73.30	5.27	0.486	2.14	0.61						
31.11	68.03	5.33	0.409	1.53	0.55						
26.16	62.70	5.33	0.344	0.98	0.47						
22.00	57.37	5.10	0.289	0.51	0.36						
18.50	52.27	4.77	0.243	0.15	0.05						
15.56	47.50	4.43	0.204	0.10	0.04						
13.08	43.07	4.20	0.172	0.06	0.03						
11.00	38.87	4.03	0.145	0.03	0.02						

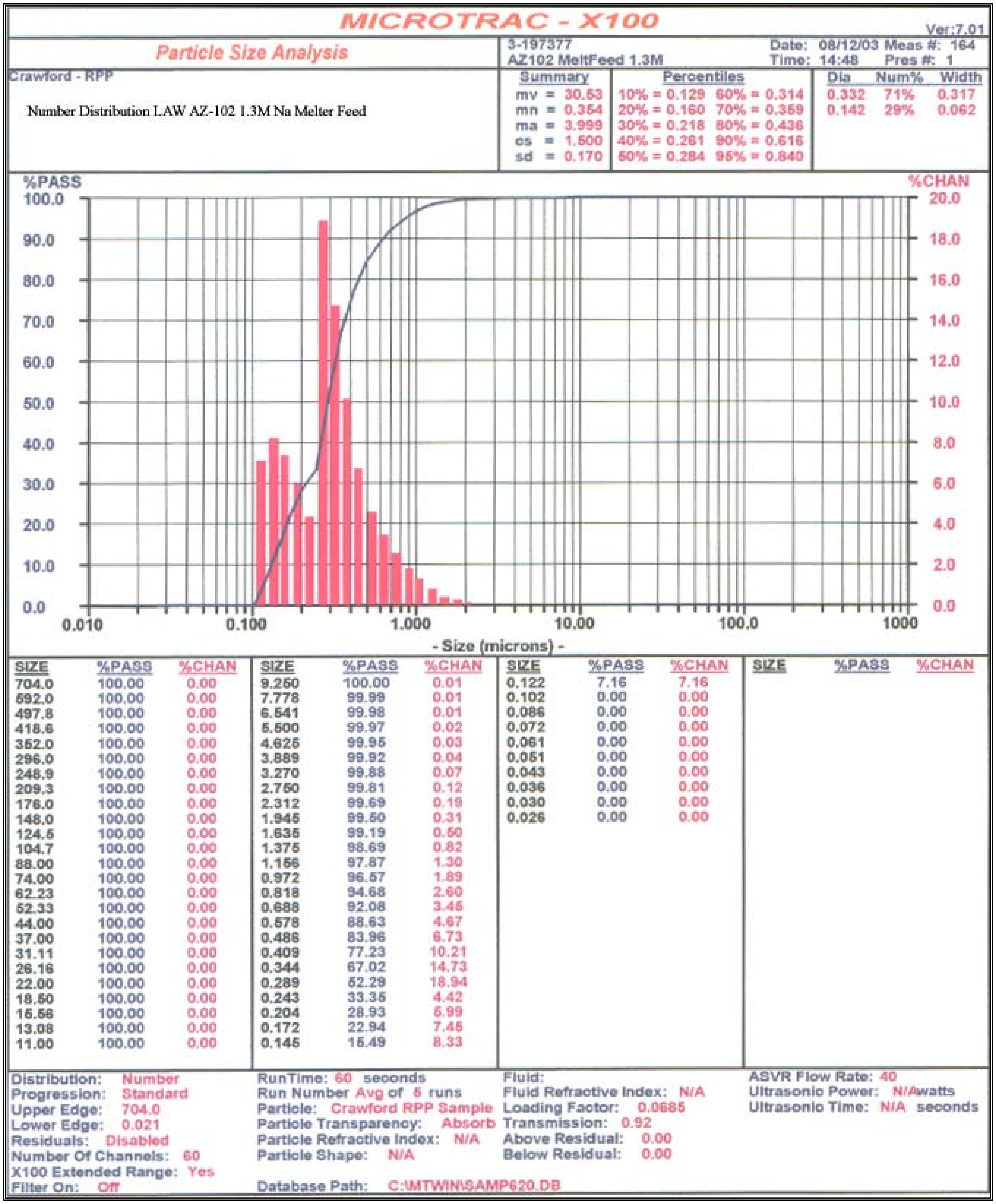
Distribution: Volume
Progression: Standard
Upper Edge: 704.0
Lower Edge: 0.021
Residuals: Disabled
Number Of Channels: 60
X100 Extended Range: Yes
Filter On: Off

RunTime: 60 seconds
Run Number Avg of 5 runs
Particle: Crawford RPP Sample
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A
Database Path: C:\MTWIN\SAMP620.DB

Fluid:
Fluid Refractive Index: N/A
Loading Factor: 0.0685
Transmission: 0.92
Above Residual: 0.00
Below Residual: 0.00

ASVR Flow Rate: 40
Ultrasonic Power: N/Awatts
Ultrasonic Time: N/A seconds

Figure A- 5. Volume PSD – LAW AZ-102 1.3M Na Melter Feed – 1 day mixing



MICROTRAC - X100

Ver:7.01

Particle Size Analysis

3-197377
AZ102 MeltFeed 1.3M

Date: 08/12/03 Meas #: 164
Time: 14:48 Pres #: 1

Crawford - RPP

Number Distribution LAW AZ-102 1.3M Na Melter Feed

Summary
mv = 30.53
mn = 0.354
ma = 3.999
cs = 1.500
sd = 0.170

Percentiles	
10% = 0.129	60% = 0.314
20% = 0.160	70% = 0.359
30% = 0.218	80% = 0.436
40% = 0.261	90% = 0.616
50% = 0.284	95% = 0.840

Dia	Num%	Width
0.332	71%	0.317
0.142	29%	0.062

SIZE	%PASS	%CHAN
704.0	100.00	0.00
592.0	100.00	0.00
497.8	100.00	0.00
418.6	100.00	0.00
352.0	100.00	0.00
296.0	100.00	0.00
248.9	100.00	0.00
209.3	100.00	0.00
176.0	100.00	0.00
148.0	100.00	0.00
124.5	100.00	0.00
104.7	100.00	0.00
88.00	100.00	0.00
74.00	100.00	0.00
62.23	100.00	0.00
52.33	100.00	0.00
44.00	100.00	0.00
37.00	100.00	0.00
31.11	100.00	0.00
26.16	100.00	0.00
22.00	100.00	0.00
18.50	100.00	0.00
15.56	100.00	0.00
13.08	100.00	0.00
11.00	100.00	0.00
9.250	100.00	0.01
7.778	99.99	0.01
6.541	99.98	0.01
5.500	99.97	0.02
4.625	99.95	0.03
3.889	99.92	0.04
3.270	99.88	0.07
2.750	99.81	0.12
2.312	99.69	0.19
1.945	99.50	0.31
1.635	99.19	0.50
1.375	98.69	0.82
1.156	97.87	1.30
0.972	96.57	1.89
0.818	94.68	2.60
0.688	92.08	3.45
0.578	88.63	4.67
0.486	83.96	6.73
0.409	77.23	10.21
0.344	67.02	14.73
0.289	52.29	18.94
0.243	33.35	4.42
0.204	28.93	5.99
0.172	22.94	7.45
0.145	15.49	8.33
0.122	7.16	7.16
0.102	0.00	0.00
0.086	0.00	0.00
0.072	0.00	0.00
0.061	0.00	0.00
0.051	0.00	0.00
0.043	0.00	0.00
0.036	0.00	0.00
0.030	0.00	0.00
0.026	0.00	0.00

Distribution: Number
Progression: Standard
Upper Edge: 704.0
Lower Edge: 0.021
Residuals: Disabled
Number Of Channels: 60
X100 Extended Range: Yes
Filter On: Off

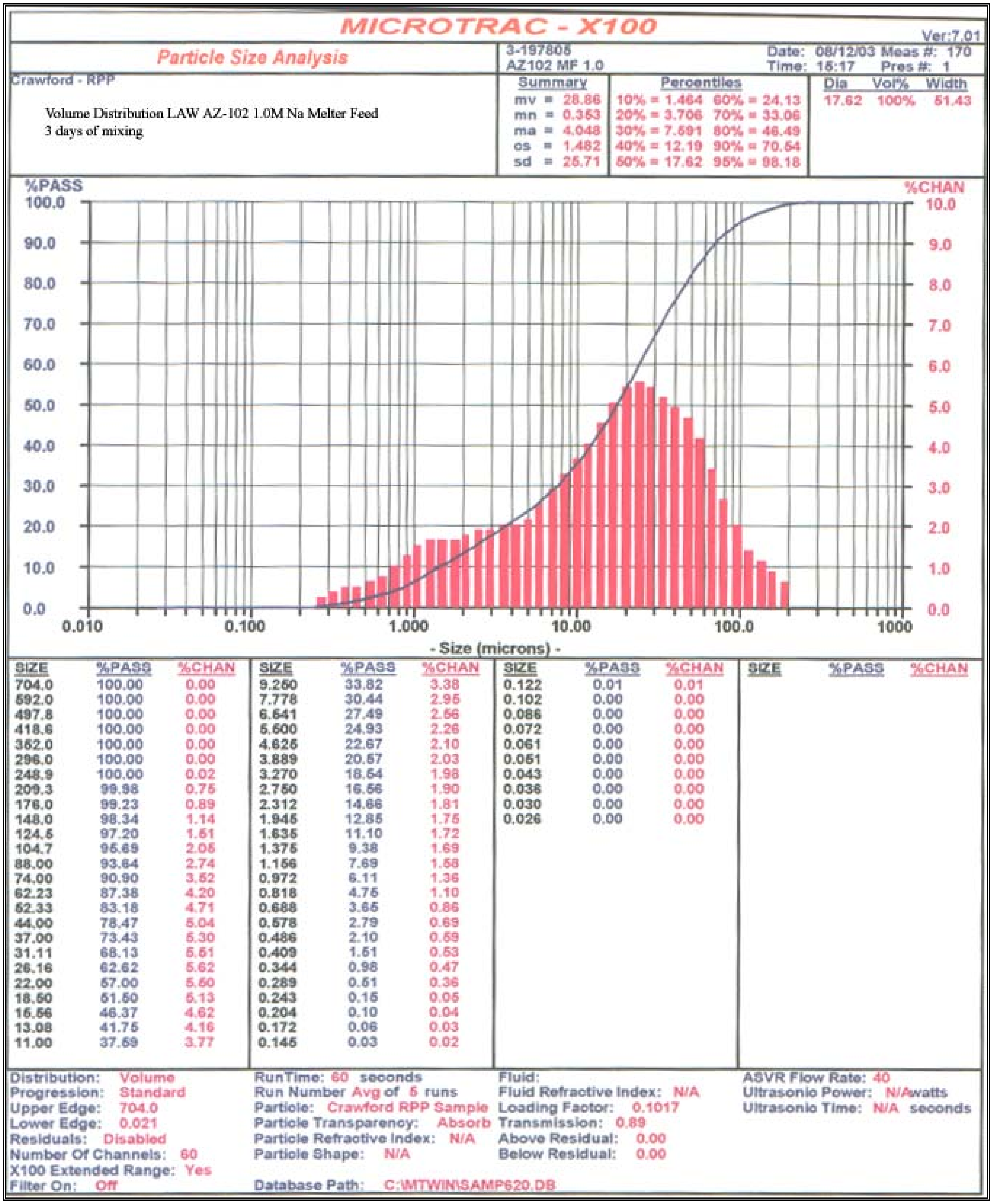
RunTime: 60 seconds
Run Number Avg of 5 runs
Particle: Crawford RPP Sample
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Database Path: C:\MTWIN\SAMP620.DB

Fluid:
Fluid Refractive Index: N/A
Loading Factor: 0.0685
Transmission: 0.92
Above Residual: 0.00
Below Residual: 0.00

ASVR Flow Rate: 40
Ultrasonic Power: N/Awatts
Ultrasonic Time: N/A seconds

Figure A- 6. Number PSD – LAW AZ-102 1.3M Na Melter Feed – 1 Day Mixing



MICROTRAC - X100

Ver:7.01

Particle Size Analysis

3-197808
AZ102 MF 1.0

Date: 08/12/03 Meas #: 170
Time: 15:17 Pres #: 1

Crawford - RPP

Volume Distribution LAW AZ-102 1.0M Na Melter Feed
3 days of mixing

Summary
mv = 28.86
mn = 0.353
ma = 4.048
cs = 1.482
sd = 25.71

Percentiles
10% = 1.464
20% = 3.706
30% = 7.691
40% = 12.19
50% = 17.62
60% = 24.13
70% = 33.06
80% = 46.49
90% = 70.54
95% = 98.18

Dia	Vol%	Width
17.62	100%	51.43

SIZE	%PASS	%CHAN
704.0	100.00	0.00
592.0	100.00	0.00
497.8	100.00	0.00
418.6	100.00	0.00
352.0	100.00	0.00
296.0	100.00	0.00
248.9	100.00	0.02
209.3	99.98	0.75
176.0	99.23	0.89
148.0	98.34	1.14
124.5	97.20	1.61
104.7	95.59	2.05
88.00	93.54	2.74
74.00	90.90	3.52
62.23	87.38	4.20
52.33	83.18	4.71
44.00	78.47	5.04
37.00	73.43	5.30
31.11	68.13	5.51
26.16	62.62	5.62
22.00	57.00	5.50
18.50	51.50	5.13
15.56	46.37	4.62
13.08	41.75	4.16
11.00	37.59	3.77
9.250	33.82	3.38
7.778	30.44	2.95
6.541	27.49	2.56
5.500	24.93	2.26
4.625	22.67	2.10
3.889	20.57	2.03
3.270	18.54	1.98
2.750	16.56	1.90
2.312	14.66	1.81
1.945	12.85	1.75
1.635	11.10	1.72
1.375	9.38	1.69
1.156	7.69	1.58
0.972	6.11	1.36
0.818	4.75	1.10
0.688	3.65	0.86
0.578	2.79	0.69
0.486	2.10	0.59
0.409	1.51	0.53
0.344	0.98	0.47
0.289	0.51	0.36
0.243	0.15	0.05
0.204	0.10	0.04
0.172	0.06	0.03
0.145	0.03	0.02
0.122	0.01	0.01
0.102	0.00	0.00
0.086	0.00	0.00
0.072	0.00	0.00
0.061	0.00	0.00
0.051	0.00	0.00
0.043	0.00	0.00
0.036	0.00	0.00
0.030	0.00	0.00
0.026	0.00	0.00

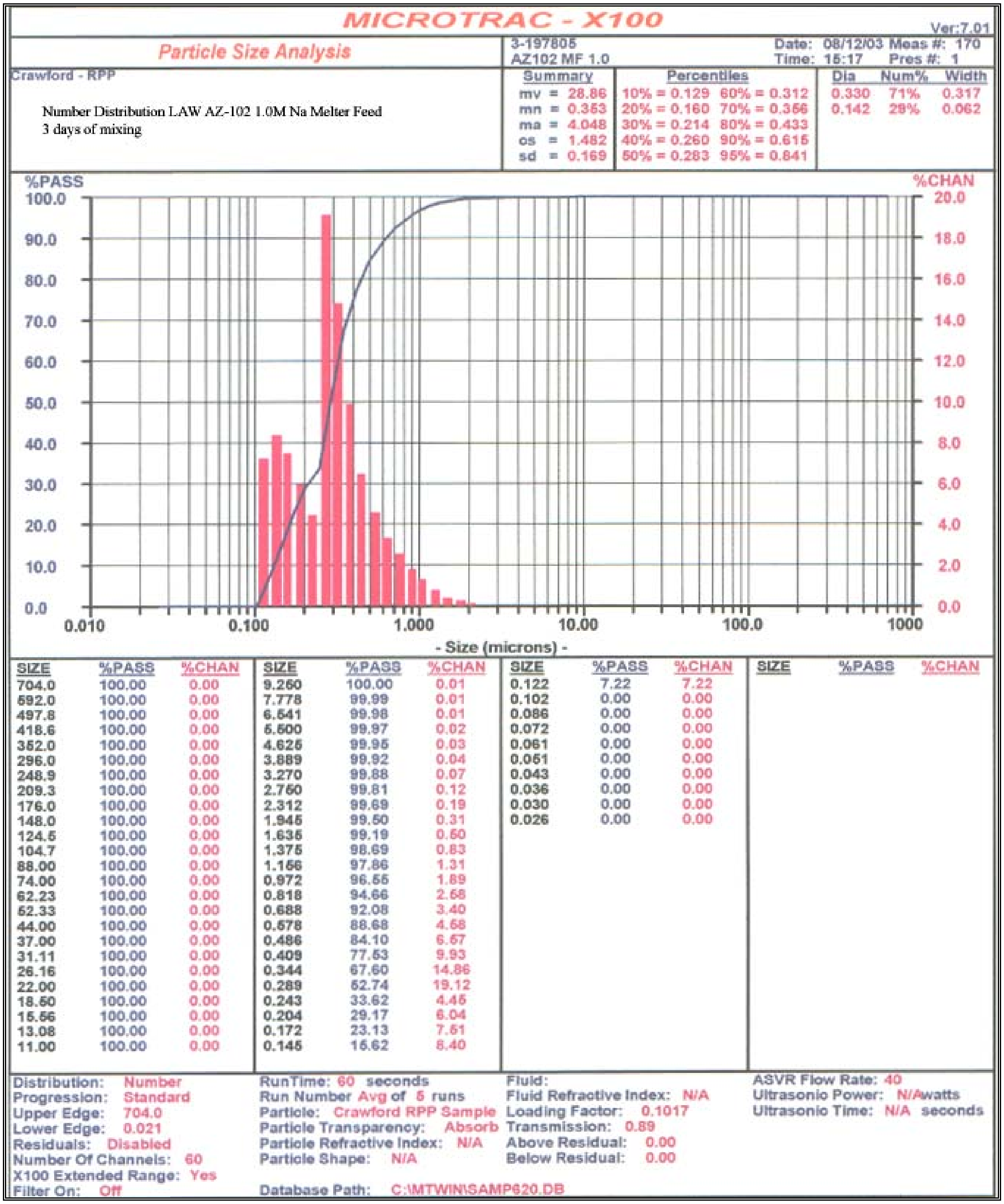
Distribution: Volume
Progression: Standard
Upper Edge: 704.0
Lower Edge: 0.021
Residuals: Disabled
Number Of Channels: 60
X100 Extended Range: Yes
Filter On: Off

RunTime: 60 seconds
Run Number Avg of 5 runs
Particle: Crawford RPP Sample
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A
Database Path: C:\MTWIN\SAMP620.DB

Fluid:
Fluid Refractive Index: N/A
Loading Factor: 0.1017
Transmission: 0.89
Above Residual: 0.00
Below Residual: 0.00

ASVR Flow Rate: 40
Ultrasonic Power: N/Awatts
Ultrasonic Time: N/A seconds

Figure A- 7. Volume PSD – LAW AZ-102 1.0M Na Melter Feed – 3 Days Mixing



MICROTRAC - X100

Ver:7.01

Particle Size Analysis

3-197805
AZ102 MF 1.0

Date: 08/12/03 Meas #: 170
Time: 15:17 Pres #: 1

Crawford - RPP

Number Distribution LAW AZ-102 1.0M Na Melter Feed
3 days of mixing

Summary
mv = 28.86
mn = 0.353
ma = 4.048
cs = 1.482
sd = 0.169

Percentiles	
10% = 0.129	60% = 0.312
20% = 0.160	70% = 0.356
30% = 0.214	80% = 0.433
40% = 0.260	90% = 0.615
50% = 0.283	95% = 0.841

Dia	Num%	Width
0.330	71%	0.317
0.142	29%	0.062

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.260	100.00	0.01	0.122	7.22	7.22			
592.0	100.00	0.00	7.778	99.99	0.01	0.102	0.00	0.00			
497.8	100.00	0.00	6.541	99.98	0.01	0.086	0.00	0.00			
418.6	100.00	0.00	5.500	99.97	0.02	0.072	0.00	0.00			
352.0	100.00	0.00	4.625	99.95	0.03	0.061	0.00	0.00			
296.0	100.00	0.00	3.889	99.92	0.04	0.051	0.00	0.00			
248.9	100.00	0.00	3.270	99.88	0.07	0.043	0.00	0.00			
209.3	100.00	0.00	2.750	99.81	0.12	0.036	0.00	0.00			
176.0	100.00	0.00	2.312	99.69	0.19	0.030	0.00	0.00			
148.0	100.00	0.00	1.945	99.50	0.31	0.026	0.00	0.00			
124.5	100.00	0.00	1.635	99.19	0.50						
104.7	100.00	0.00	1.375	98.69	0.83						
88.00	100.00	0.00	1.156	97.86	1.31						
74.00	100.00	0.00	0.972	96.55	1.89						
62.23	100.00	0.00	0.818	94.66	2.58						
52.33	100.00	0.00	0.688	92.08	3.40						
44.00	100.00	0.00	0.578	88.68	4.58						
37.00	100.00	0.00	0.486	84.10	6.57						
31.11	100.00	0.00	0.409	77.53	9.93						
26.16	100.00	0.00	0.344	67.60	14.86						
22.00	100.00	0.00	0.289	52.74	19.12						
18.50	100.00	0.00	0.243	33.62	4.45						
15.56	100.00	0.00	0.204	29.17	6.04						
13.08	100.00	0.00	0.172	23.13	7.61						
11.00	100.00	0.00	0.145	16.62	8.40						

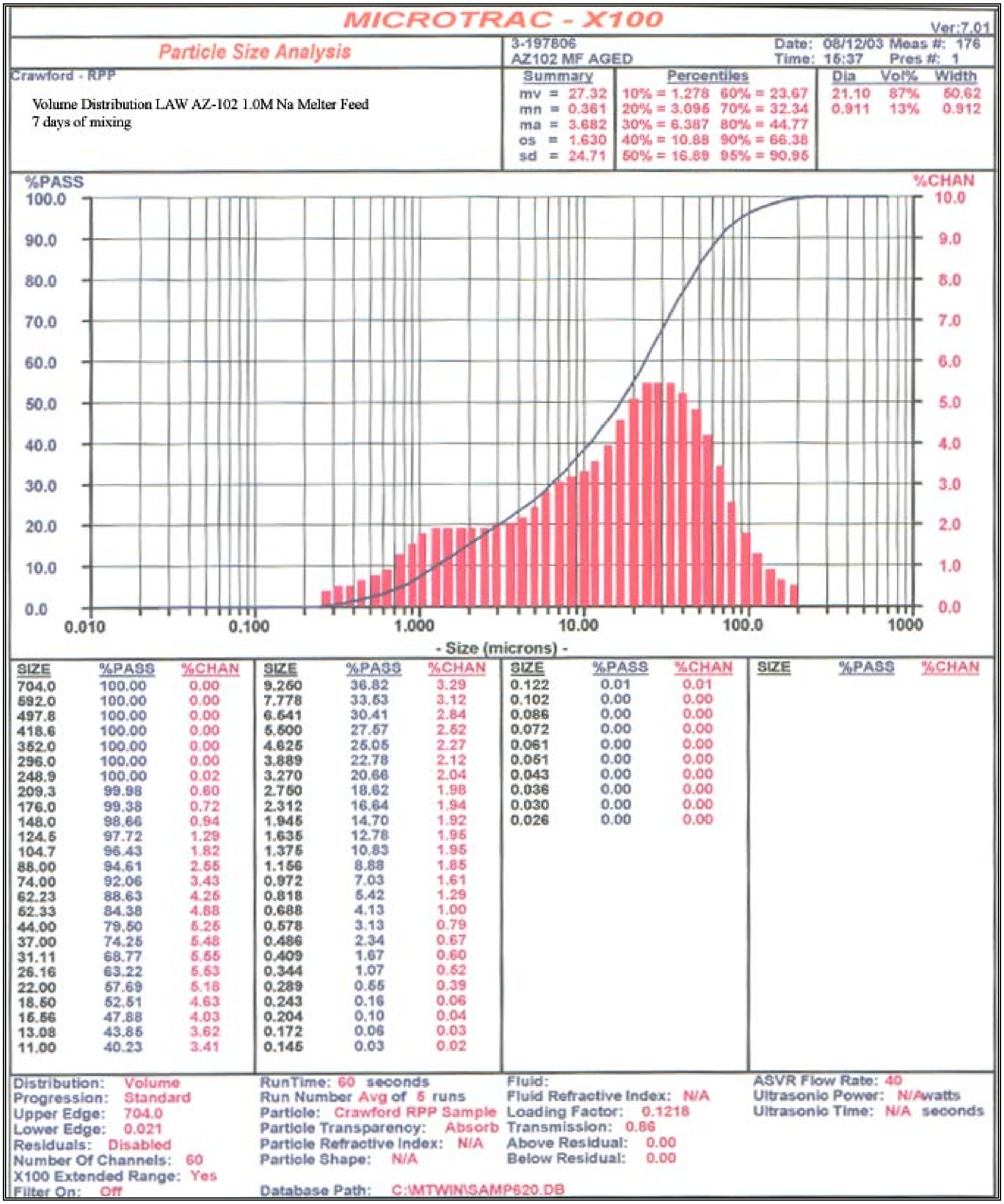
Distribution: Number
Progression: Standard
Upper Edge: 704.0
Lower Edge: 0.021
Residuals: Disabled
Number Of Channels: 60
X100 Extended Range: Yes
Filter On: Off

RunTime: 60 seconds
Run Number Avg of 5 runs
Particle: Crawford RPP Sample
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A
Database Path: C:\MTWIN\SAMP620.DB

Fluid:
Fluid Refractive Index: N/A
Loading Factor: 0.1017
Transmission: 0.89
Above Residual: 0.00
Below Residual: 0.00

ASVR Flow Rate: 40
Ultrasonic Power: N/Awatts
Ultrasonic Time: N/A seconds

Figure A- 8. Number PSD – LAW AZ-102 1.0M Na Melter Feed – 3 Days Mixing



MICROTRAC - X100

Ver:7.01

Particle Size Analysis

3-197806
AZ102 MF AGED

Date: 08/12/03 Meas #: 176
Time: 15:37 Pres #: 1

Crawford - RPP

Volume Distribution LAW AZ-102 1.0M Na Melter Feed
7 days of mixing

Summary
mv = 27.32
mn = 0.361
ma = 3.682
os = 1.630
sd = 24.71

Percentiles
10% = 1.278
20% = 3.096
30% = 6.387
40% = 10.88
50% = 16.89
60% = 23.67
70% = 32.34
80% = 44.77
90% = 66.38
95% = 90.95

Dia	Vol%	Width
21.10	87%	50.62
0.911	13%	0.912

SIZE	%PASS	%CHAN
704.0	100.00	0.00
592.0	100.00	0.00
497.8	100.00	0.00
418.6	100.00	0.00
352.0	100.00	0.00
296.0	100.00	0.00
248.9	100.00	0.02
209.3	99.98	0.60
176.0	99.38	0.72
148.0	98.66	0.94
124.5	97.72	1.29
104.7	96.43	1.82
88.00	94.61	2.55
74.00	92.06	3.43
62.23	88.63	4.25
52.33	84.38	4.88
44.00	79.50	5.25
37.00	74.25	5.48
31.11	68.77	5.55
26.16	63.22	5.53
22.00	57.69	5.18
18.50	52.51	4.63
15.56	47.88	4.03
13.08	43.85	3.62
11.00	40.23	3.41

SIZE	%PASS	%CHAN
9.250	36.82	3.29
7.778	33.53	3.12
6.541	30.41	2.84
5.500	27.57	2.52
4.625	25.05	2.27
3.889	22.78	2.12
3.270	20.66	2.04
2.750	18.62	1.98
2.312	16.64	1.94
1.945	14.70	1.92
1.635	12.78	1.95
1.375	10.83	1.95
1.156	8.88	1.85
0.972	7.03	1.61
0.818	5.42	1.29
0.688	4.13	1.00
0.578	3.13	0.79
0.486	2.34	0.67
0.409	1.67	0.60
0.344	1.07	0.52
0.289	0.55	0.39
0.243	0.16	0.06
0.204	0.10	0.04
0.172	0.06	0.03
0.145	0.03	0.02

SIZE	%PASS	%CHAN
0.122	0.01	0.01
0.102	0.00	0.00
0.086	0.00	0.00
0.072	0.00	0.00
0.061	0.00	0.00
0.051	0.00	0.00
0.043	0.00	0.00
0.036	0.00	0.00
0.030	0.00	0.00
0.026	0.00	0.00

SIZE	%PASS	%CHAN

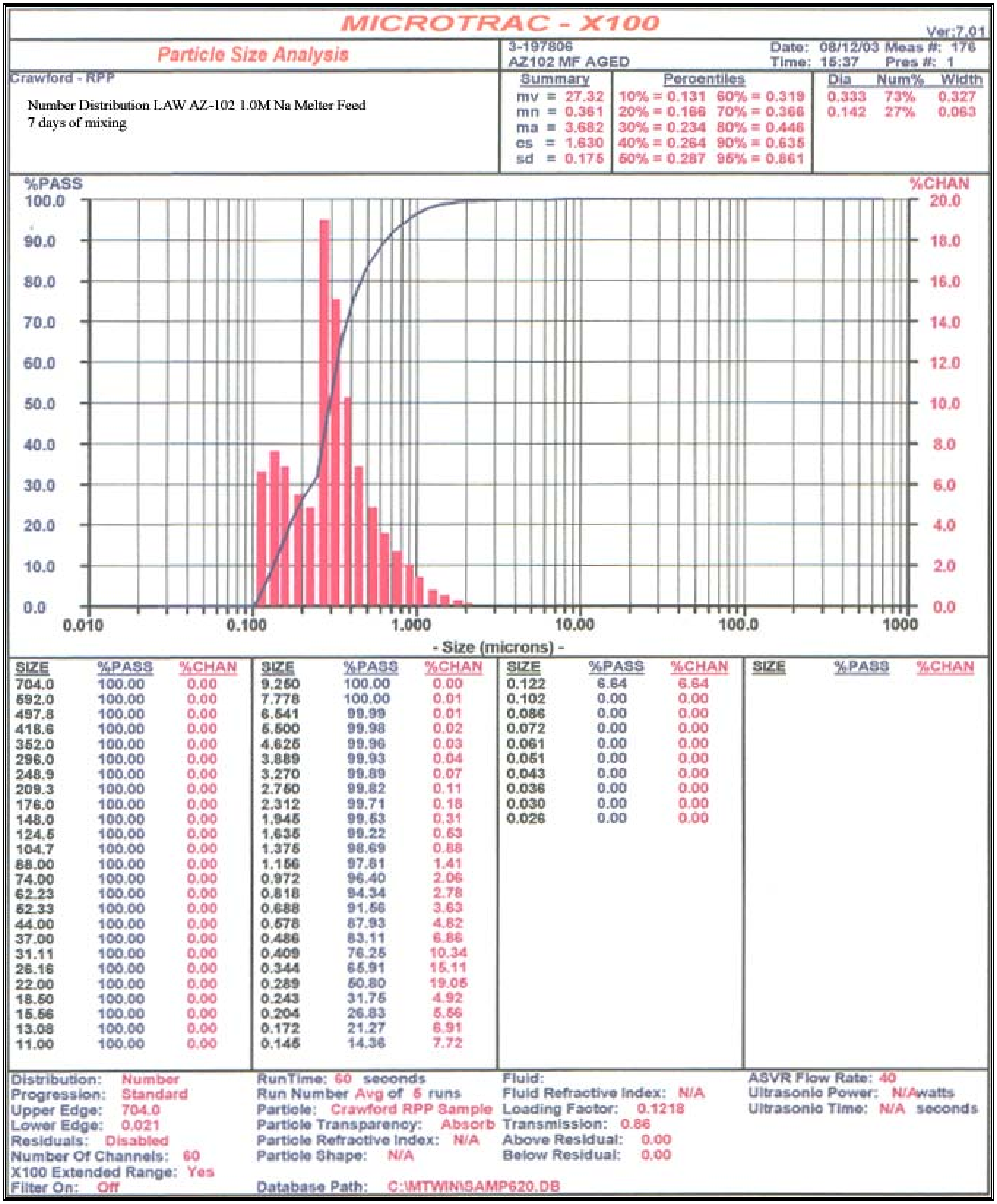
Distribution: Volume
Progression: Standard
Upper Edge: 704.0
Lower Edge: 0.021
Residuals: Disabled
Number Of Channels: 60
X100 Extended Range: Yes
Filter On: Off

RunTime: 60 seconds
Run Number Avg of 5 runs
Particle: Crawford RPP Sample
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A
Database Path: C:\MTWIN\SAMP620.DB

Fluid:
Fluid Refractive Index: N/A
Loading Factor: 0.1218
Transmission: 0.86
Above Residual: 0.00
Below Residual: 0.00

ASVR Flow Rate: 40
Ultrasonic Power: N/Awatts
Ultrasonic Time: N/A seconds

Figure A- 9. Volume PSD – LAW AZ-102 1.0M Na Melter Feed – 7 Days Mix



MICROTRAC - X100

Ver:7.01

Particle Size Analysis

3-197806
AZ102 MF AGED

Date: 08/12/03 Meas #: 176
Time: 15:37 Pres #: 1

Crawford - RPP

Number Distribution LAW AZ-102 1.0M Na Melter Feed
7 days of mixing

Summary
mv = 27.32
mn = 0.361
ma = 3.682
cs = 1.630
sd = 0.175

Percentiles
10% = 0.131
20% = 0.166
30% = 0.234
40% = 0.264
50% = 0.287
60% = 0.319
70% = 0.366
80% = 0.446
90% = 0.635
95% = 0.861

Dia	Num%	Width
0.333	73%	0.327
0.142	27%	0.063

SIZE	%PASS	%CHAN
704.0	100.00	0.00
592.0	100.00	0.00
497.8	100.00	0.00
418.6	100.00	0.00
352.0	100.00	0.00
296.0	100.00	0.00
248.9	100.00	0.00
209.3	100.00	0.00
176.0	100.00	0.00
148.0	100.00	0.00
124.5	100.00	0.00
104.7	100.00	0.00
88.00	100.00	0.00
74.00	100.00	0.00
62.23	100.00	0.00
52.33	100.00	0.00
44.00	100.00	0.00
37.00	100.00	0.00
31.11	100.00	0.00
26.16	100.00	0.00
22.00	100.00	0.00
18.50	100.00	0.00
15.56	100.00	0.00
13.08	100.00	0.00
11.00	100.00	0.00
9.250	100.00	0.00
7.778	100.00	0.01
6.541	99.99	0.01
5.500	99.98	0.02
4.625	99.96	0.03
3.889	99.93	0.04
3.270	99.89	0.07
2.750	99.82	0.11
2.312	99.71	0.18
1.945	99.53	0.31
1.635	99.22	0.53
1.375	98.69	0.88
1.156	97.81	1.41
0.972	96.40	2.06
0.818	94.34	2.78
0.688	91.56	3.63
0.578	87.93	4.82
0.486	83.11	6.86
0.409	76.25	10.34
0.344	65.91	15.11
0.289	50.80	19.05
0.243	31.75	4.92
0.204	26.83	5.56
0.172	21.27	6.91
0.145	14.36	7.72
0.122	6.64	6.64
0.102	0.00	0.00
0.086	0.00	0.00
0.072	0.00	0.00
0.061	0.00	0.00
0.051	0.00	0.00
0.043	0.00	0.00
0.036	0.00	0.00
0.030	0.00	0.00
0.026	0.00	0.00

Distribution: Number
Progression: Standard
Upper Edge: 704.0
Lower Edge: 0.021
Residuals: Disabled
Number Of Channels: 60
X100 Extended Range: Yes
Filter On: Off

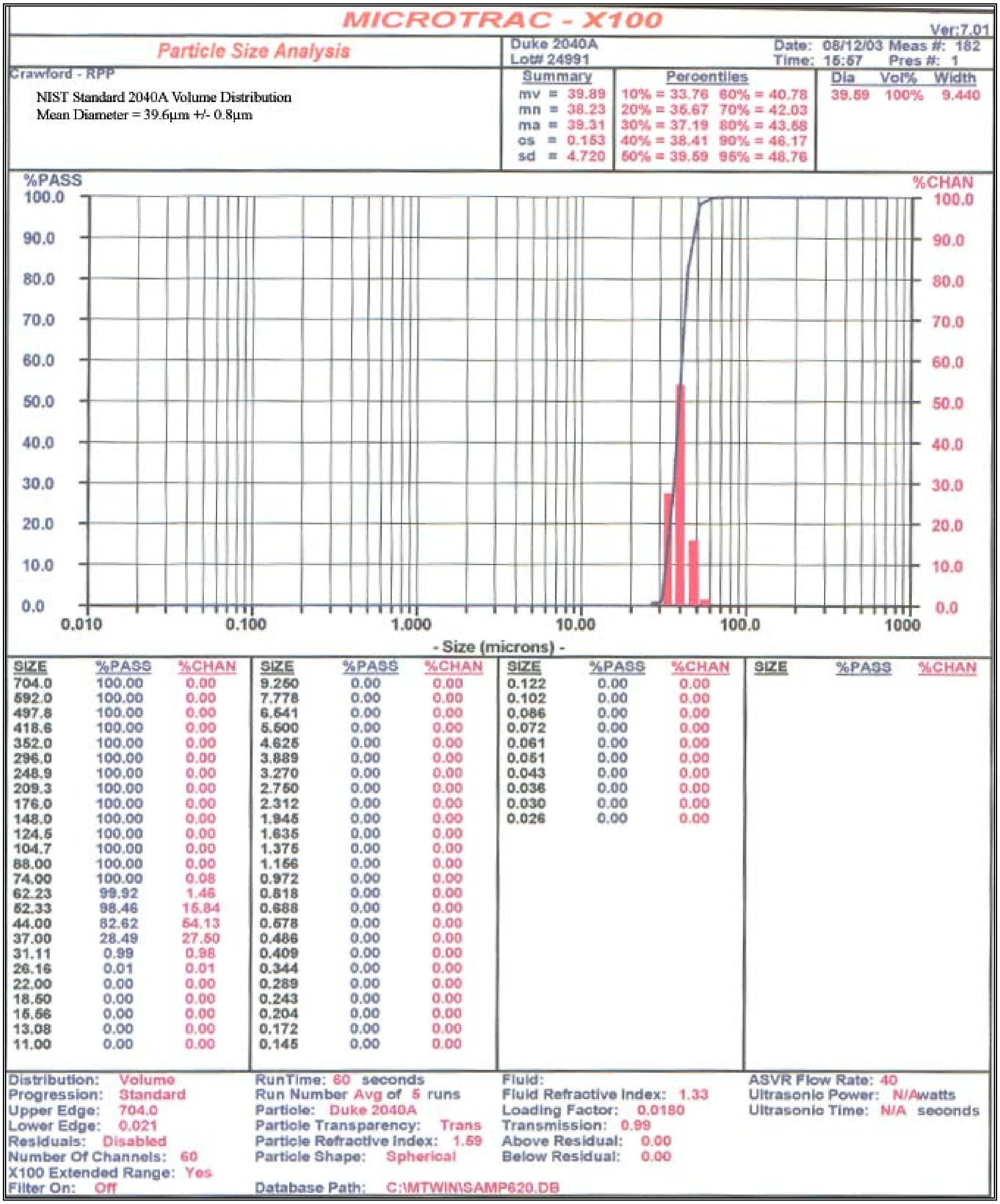
RunTime: 60 seconds
Run Number Avg of 5 runs
Particle: Crawford RPP Sample
Particle Transparency: Absorb
Particle Refractive Index: N/A
Particle Shape: N/A

Database Path: C:\MTWIN\SAMP620.DB

Fluid:
Fluid Refractive Index: N/A
Loading Factor: 0.1218
Transmission: 0.86
Above Residual: 0.00
Below Residual: 0.00

ASVR Flow Rate: 40
Ultrasonic Power: N/Awatts
Ultrasonic Time: N/A seconds

Figure A- 10. Number PSD – LAW AZ-102 1.0M Na Melter Feed – 7 Days Mixing



MICROTRAC - X100

Ver:7.01

Particle Size Analysis

Duke 2040A
Lot# 24991

Date: 08/12/03 Meas #: 182
Time: 15:57 Pres #: 1

Crawford - RPP

NIST Standard 2040A Volume Distribution
Mean Diameter = 39.6µm +/- 0.8µm

Summary		Percentiles	
mv =	39.89	10% = 33.76	60% = 40.78
mn =	38.23	20% = 35.67	70% = 42.03
ma =	39.31	30% = 37.19	80% = 43.68
cs =	0.153	40% = 38.41	90% = 46.17
sd =	4.720	50% = 39.59	95% = 48.76

Dia	Vol%	Width
39.59	100%	9.440

SIZE	%PASS	%CHAN
704.0	100.00	0.00
592.0	100.00	0.00
497.8	100.00	0.00
418.6	100.00	0.00
352.0	100.00	0.00
296.0	100.00	0.00
248.9	100.00	0.00
209.3	100.00	0.00
176.0	100.00	0.00
148.0	100.00	0.00
124.5	100.00	0.00
104.7	100.00	0.00
88.00	100.00	0.00
74.00	100.00	0.08
62.23	99.92	1.46
52.33	98.46	15.84
44.00	82.62	54.13
37.00	28.49	27.50
31.11	0.99	0.98
26.16	0.01	0.01
22.00	0.00	0.00
18.50	0.00	0.00
15.56	0.00	0.00
13.08	0.00	0.00
11.00	0.00	0.00
9.250	0.00	0.00
7.778	0.00	0.00
6.541	0.00	0.00
5.500	0.00	0.00
4.625	0.00	0.00
3.889	0.00	0.00
3.270	0.00	0.00
2.750	0.00	0.00
2.312	0.00	0.00
1.945	0.00	0.00
1.635	0.00	0.00
1.375	0.00	0.00
1.156	0.00	0.00
0.972	0.00	0.00
0.818	0.00	0.00
0.688	0.00	0.00
0.578	0.00	0.00
0.486	0.00	0.00
0.409	0.00	0.00
0.344	0.00	0.00
0.289	0.00	0.00
0.243	0.00	0.00
0.204	0.00	0.00
0.172	0.00	0.00
0.145	0.00	0.00
0.122	0.00	0.00
0.102	0.00	0.00
0.086	0.00	0.00
0.072	0.00	0.00
0.061	0.00	0.00
0.051	0.00	0.00
0.043	0.00	0.00
0.036	0.00	0.00
0.030	0.00	0.00
0.026	0.00	0.00

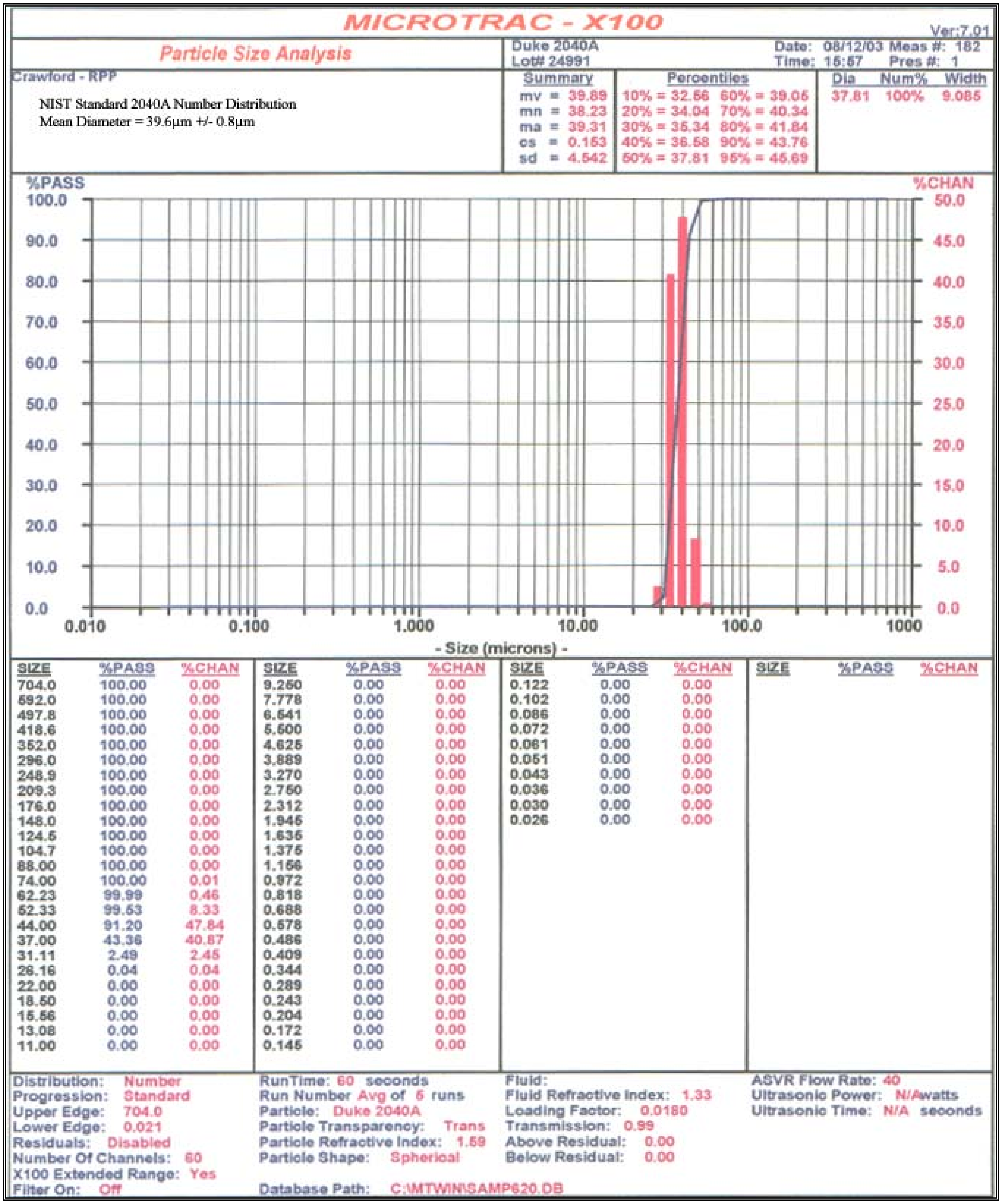
Distribution: Volume
Progression: Standard
Upper Edge: 704.0
Lower Edge: 0.021
Residuals: Disabled
Number Of Channels: 60
X100 Extended Range: Yes
Filter On: Off

RunTime: 60 seconds
Run Number Avg of 5 runs
Particle: Duke 2040A
Particle Transparency: Trans
Particle Refractive Index: 1.59
Particle Shape: Spherical
Database Path: C:\MTWIN\SAMP620.DB

Fluid:
Fluid Refractive Index: 1.33
Loading Factor: 0.0180
Transmission: 0.99
Above Residual: 0.00
Below Residual: 0.00

ASVR Flow Rate: 40
Ultrasonic Power: N/Awatts
Ultrasonic Time: N/A seconds

Figure A- 11. Final Volume PSD – 2040A Standard



MICROTRAC - X100

Ver:7.01

Particle Size Analysis

Duke 2040A
Lot# 24991

Date: 08/12/03 Meas #: 182
Time: 15:57 Pres #: 1

Crawford - RPP

NIST Standard 2040A Number Distribution
Mean Diameter = 39.6μm +/- 0.8μm

Summary
mv = 39.89
mn = 38.23
ma = 39.31
cs = 0.153
sd = 4.542

Percentiles
10% = 32.56
20% = 34.04
30% = 35.34
40% = 36.58
50% = 37.81
60% = 39.05
70% = 40.34
80% = 41.84
90% = 43.76
95% = 45.69

Dia	Num%	Width
37.81	100%	9.085

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
704.0	100.00	0.00	9.250	0.00	0.00	0.122	0.00	0.00			
592.0	100.00	0.00	7.778	0.00	0.00	0.102	0.00	0.00			
497.8	100.00	0.00	6.541	0.00	0.00	0.086	0.00	0.00			
418.6	100.00	0.00	5.500	0.00	0.00	0.072	0.00	0.00			
352.0	100.00	0.00	4.625	0.00	0.00	0.061	0.00	0.00			
296.0	100.00	0.00	3.889	0.00	0.00	0.051	0.00	0.00			
248.9	100.00	0.00	3.270	0.00	0.00	0.043	0.00	0.00			
209.3	100.00	0.00	2.750	0.00	0.00	0.036	0.00	0.00			
176.0	100.00	0.00	2.312	0.00	0.00	0.030	0.00	0.00			
148.0	100.00	0.00	1.945	0.00	0.00	0.026	0.00	0.00			
124.5	100.00	0.00	1.635	0.00	0.00						
104.7	100.00	0.00	1.375	0.00	0.00						
88.00	100.00	0.00	1.156	0.00	0.00						
74.00	100.00	0.01	0.972	0.00	0.00						
62.23	99.99	0.46	0.818	0.00	0.00						
52.33	99.53	8.33	0.688	0.00	0.00						
44.00	91.20	47.84	0.578	0.00	0.00						
37.00	43.36	40.87	0.486	0.00	0.00						
31.11	2.49	2.45	0.409	0.00	0.00						
26.16	0.04	0.04	0.344	0.00	0.00						
22.00	0.00	0.00	0.289	0.00	0.00						
18.50	0.00	0.00	0.243	0.00	0.00						
15.56	0.00	0.00	0.204	0.00	0.00						
13.08	0.00	0.00	0.172	0.00	0.00						
11.00	0.00	0.00	0.145	0.00	0.00						

Distribution: Number
Progression: Standard
Upper Edge: 704.0
Lower Edge: 0.021
Residuals: Disabled
Number Of Channels: 60
X100 Extended Range: Yes
Filter On: Off

RunTime: 60 seconds
Run Number Avg of 6 runs
Particle: Duke 2040A
Particle Transparency: Trans
Particle Refractive Index: 1.59
Particle Shape: Spherical
Database Path: C:\MTWIN\SAMP620.DB

Fluid:
Fluid Refractive Index: 1.33
Loading Factor: 0.0180
Transmission: 0.99
Above Residual: 0.00
Below Residual: 0.00

ASVR Flow Rate: 40
Ultrasonic Power: N/Awatts
Ultrasonic Time: N/A seconds

Figure A- 12. Final Number PSD – 2040A Standard

APPENDIX B.
COMPOSITION OF GLASS FORMER CHEMICALS

The composition of each GFC used was taken from reference 4 and is summarized below

Mineral	**nominal weight percent of compound in mineral**																			
	Al_2O_3	B_2O_3	CaO	CO_2	Cr_2O_3	Fe_2O_3	H_2O	Li_2O	MgO	MnO	Na_2O	Nb_2O_5	NiO	P	SiO_2	TiO_2	U+Th	V_2O_5	ZnO	ZrO_2
Kyanite - Al_2O_2-SiO_2	**57**	N/A	N/A	N/A	N/A	**0.71**	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	**40.5**	**1.05**	N/A	N/A	N/A	N/A
Boric Acid - H_3BO_3	N/A	**56.5**	N/A	N/A	N/A	N/A	**43.5**	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Na_2CO_3 Anhydrous	N/A	N/A	N/A	**41.4**	N/A	N/A	N/A	N/A	N/A	N/A	**58.4**	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Wollastonite – $CaSiO_3$	**0.2**	N/A	**47.5**	N/A	N/A	**0.4**	N/A	N/A	**0.1**	**0.1**	N/A	N/A	N/A	N/A	**51**	N/A	N/A	N/A	N/A	N/A
Fe_2O_3	**1.5**	N/A	N/A	N/A	N/A	**97**	N/A	N/A	**0.1**	N/A	N/A	N/A	N/A	**0.12**	**1.35**	N/A	N/A	N/A	N/A	N/A
Li_2CO_3	N/A	N/A	N/A	**59.2**	N/A	N/A	N/A	**40.2**	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Olivine – $MgSiO_3$	**0.19**	N/A	N/A	N/A	**0.13**	**7.68**	N/A	N/A	**48**	N/A	N/A	N/A	**0.37**	N/A	**42.5**	N/A	N/A	N/A	N/A	N/A
SiO_2	**0.14**	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	**99.7**	N/A	N/A	N/A	N/A	N/A
Rutile TiO_2/Fe_2O_3	**0.5**	N/A	N/A	N/A	**0.16**	**0.7**	N/A	N/A	N/A	N/A	N/A	**0.4**	N/A	N/A	**2.2**	**93.2**	N/A	**0.45**	N/A	**1.9**
ZnO	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	**99.8**	N/A
$ZrSiO_4$	**0.25**	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	**32.3**	**0.11**	**0.05**	N/A	N/A	**65.5**

This page intentionally left blank.

APPENDIX C.
TABLE 12, SHEETS #1, #2, #3 AND # 5

Table 12 – Sheet #1 – Waste Composition Of Each Sample

Sample Identification		LAW AZ-102 1.0M Na Pretreated Feed	LAW AZ-102 1.3M Na Pretreated Feed	LAW AZ-102 1.0M Na Melter Feed	LAW AZ-102 1.3M Na Melter Feed
Sample History		Diluted from original AZ-102 4.38M Na Pretreated Feed	Diluted from original AZ-102 4.38M Na Pretreated Feed	Glass Former Chemicals Added to 1.0M Na Pretreated Feed	Glass Former Chemicals Added to 1.3M Na Pretreated Feed
Sodium Molarity		1.02	1.33	1.29	1.54
Oxide Loading of HLW sludge or Pretreated Sludge		N/A	N/A	N/A	N/A
pH of the Waste		12.01	12.15	9.04	8.72
< MRQ = Below Minimum Reportable Quantity or not measured					
Analyte:	**Units**				
Cations					
Ag	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Al	mg/L	214	289	12931	15465
As	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
B	mg/L	< MRQ	< MRQ	23596	28194
Ba	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Be	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Bi	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Ca	mg/L	17.9	24.3	36434	43538
Cd	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Ce	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Co	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Cr	mg/L	273	370	236	290
Cs	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Cu	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Dy	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Eu	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Fe	mg/L	< MRQ	< MRQ	27877	33317
Hg	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
K	mg/L	1220	1653	816	1010
La	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Li	mg/L	< MRQ	< MRQ	5773	6896
Mg	mg/L	0.8	1.1	13672	16336
Mn	mg/L	< MRQ	< MRQ	107	129
Mo	mg/L	21	29	14	18
Na	mg/L	23450	30576	29572	35308
Nb	mg/L	< MRQ	< MRQ	16	19

Table 12 – Sheet #1 – Waste Composition Of Each Sample					
Sample Identification		LAW AZ-102 1.0M Na Pretreated Feed	LAW AZ-102 1.3M Na Pretreated Feed	LAW AZ-102 1.0M Na Melter Feed	LAW AZ-102 1.3M Na Melter Feed
Analyte:	**Units**				
Cations					
Nd	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Ni	mg/L	< MRQ	< MRQ	136	162
P	mg/L	62	83	83	101
Pb	mg/L	6.9	9.3	5	6
Pd	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Pr	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Pt	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Rb	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Rh	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Ru	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
S	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Sb	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Se	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Si	mg/L	15.0	20.4	171015	204310
Sn	mg/L	4.6	6.2	3	4
Sr	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Ta	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Te	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Th	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Ti	mg/L	< MRQ	< MRQ	6789	8116
Tl	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
U	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
V	mg/L	< MRQ	< MRQ	28	34
W	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Y	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Zn	mg/L	< MRQ	< MRQ	29123	34809
Zr	mg/L	< MRQ	< MRQ	17750	17750
Carbon Analyses					
TIC	mg/L	2516	3409	1683	2084
TOC	mg/L	5333	7225	3566	4417
Anions					
F	mg/L	367	497	245	304
Cl	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Br	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
NO_2	mg/L	13239	17937	8854	10965

Table 12 – Sheet #1 – Waste Composition Of Each Sample

Sample Identification		LAW AZ-102 1.0M Na Pretreated Feed	LAW AZ-102 1.3M Na Pretreated Feed	LAW AZ-102 1.0M Na Melter Feed	LAW AZ-102 1.3M Na Melter Feed
Analyte:	**Units**				
Anions					
NO_3	mg/L	7312	9907	4890	6056
PO_4	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
SO_4	mg/L	7043	9542	4710	5833
CN	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
NH_3	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Anions					
Free OH	Molarity	0.29	0.40	0.20	0.24
Total OH	Molarity	< MRQ	< MRQ	< MRQ	< MRQ
Radioisotopes					
H-3	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
C-14	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Cr-51	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Fe-59	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Ni-59	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Co-60	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Ni-63	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Se-79	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Y-88	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Sr-90	µCi/L	332	450	222	275
Sr-90/Y-90	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Nb-94/95	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Tc-99	µCi/L	0	0.0	1.1	1
Ru-103	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Ru-106	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Sn-113	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Sb-125	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Sn-126	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Sb\Sn-126	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
I-127	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
I-129	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
C-133	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Cs-134	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Cs-135	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Cs-137	µCi/L	329	446	220	273
Ce-144	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ

Table 12 – Sheet #1 – Waste Composition Of Each Sample

Sample Identification		LAW AZ-102 1.0M Na Pretreated Feed	LAW AZ-102 1.3M Na Pretreated Feed	LAW AZ-102 1.0M Na Melter Feed	LAW AZ-102 1.3M Na Melter Feed
Analyte:	**Units**				
Radioisotopes					
Sm-151	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Eu-152	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Eu-154	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Eu-155	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Pa-231	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
U-233	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
U-234	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
U-235	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
U-236	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
U-238	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Np-237	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Pu-236	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Pu-238	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Pu-239	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Pu-240	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Pu-239/240	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Pu-241	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Pu-242	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Pu-241/Am-241	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Am-241	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Am-241, Am-243	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Am-242	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Am-243	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Cm-242	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Cm-243	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Cm-244	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Cm-243/244	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Sum of alpha (TRU) = S (Pu-238, Pu-239, Pu-240, Am-241)	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Total alpha	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Total beta	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Total gamma	µCi/L	< MRQ	< MRQ	< MRQ	< MRQ
Radioisotopes					
235	µg/L	9.3	12.7	35.8	44.3
237	µg/L	53.5	72.5	685.0	848.3

Table 12 – Sheet #1 – Waste Composition Of Each Sample					
Sample Identification		LAW AZ-102 1.0M Na Pretreated Feed	LAW AZ-102 1.3M Na Pretreated Feed	LAW AZ-102 1.0M Na Melter Feed	LAW AZ-102 1.3M Na Melter Feed
Analyte:	**Units**				
Radioisotopes					
238	µg/L	1024.3	1387.8	6.3	7.7
239	µg/L	20.1	27.3	13.5	16.7
Organic analytes:					
Oxalate	mg/L	1238	1678	828	1026
Citrate	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Formate	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Gluconate	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Glycolate	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
EDTA (ethylenediaminetetraacetic acid)	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
HEDTA (N-(2-hydroxyethyl)ethylenediaminetriacetic acid)	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
D2EHPA (bis-(2-ethylhexyl)phosphate	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
NTA (nitrilotriacetic acid)	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
IDA (iminodiacetic acid)	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Succinic Acid	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
ED3A (ethylenediaminetriacetic acid)	mg/L	< MRQ	< MRQ	< MRQ	< MRQ
Analytes Obtained on an Opportunistic Basis:					
CO_3	Molarity	0.13	0.18	0.09	0.11
CO_2	mg/L	< MRQ	< MRQ	60992	72887
U+Th	mg/L	< MRQ	< MRQ	16.3	19.5

Table 12 – Sheet#2 – LAW Melter Feed Preparation Description

Pretreated Waste or HLW Pretreated Sludge Identification			LAW AZ-102 1.0M Na Melter Feed		LAW AZ-102 1.3M Na Melter Feed	
Pretreatment History (include washing, leaching, chemical precipitation, mechanical agitation of any kind (time and intensity)			See WSRC-TR-2001-00395		See WSRC-TR-2001-00395	
Sodium Concentration of LAW Pretreated Waste		Na Molarity:	1.02		1.33	
Oxide Loading of HLW Pretreated Sludge		Total Grams Oxide Per Liter:	N/A		N/A	
Source Chemical	Manufacturer	Oxide	Target Mass (g)	Actual Mass Added (g)	Target Mass (g)	Actual Mass Added (g)
Kyanite	Kyanite Mining Corp	Al_2O_3	22.10	22.102	12.95	12.953
Boric Acid Technical	U.S. Borax	B_2O_3	37.08	37.083	21.72	21.720
10M Borax	U.S. Borax	Na_2O/B_2O_3	N.A	N/A	N.A	N/A
Soda Ash	Solvay Minerals	Na_2CO_3	8.85	8.85	5.19	5.190
Wollastonite	NYCO	CaO	29.60	29.602	17.34	17.340
Fe_2O_3 5001	Prince Mfg. Co.	Fe_2O_3	10.09	10.091	5.91	5.912
Li_2CO_3	Chemettal-Foote	Li_2O	22.23	22.233	13.02	13.020
Olivine	Unimin Corp	MgO	12.89	12.89	7.55	7.550
SCS-75	U.S. Silica	SiO_2	68.13	68.181	39.92	39.920
Rutile (Air floated)	Chemalloy Co.	TiO_2	3.085	3.092	1.81	1.812
Kadox	Zinc Corp Amer.	ZnO	10.02	10.022	5.87	5.872
Zircon	Amer. Miner, Inc.	ZrO_2	10.01	10.01	5.87	5.870
Sucrose	Amalgamated Sugar	Sugar	N/A	N/A	N/A	N/A

Table 12 – Sheet#2 – LAW Melter Feed Preparation Description Continued

Mixing Operation Data Needed to Compare Mixing of the Melter Feed			
Melter Feed ID:	AZ-1021.0 M LAW 3 days of Mixing	AZ-102 1.0 M LAW MF 7 day	AZ-102 1.3 M LAW Melter Feed + 24 Hrs
Processing Scale (lab/bench, pilot or full)	Lab/Bench	Lab Scale	Lab/Bench
Activity/Property			
Order of Chemical Additions	GFCs are blended	GFCs are blended	GFC are blended
Mixing Time	3 days	4 days after the initial 3 days of mixing	24 hours
Impeller Speed	325 RPM	200 RPM	600 RPM
Impeller Diameter	50.69 mm	50.69 mm	38.3 mm
Tank Diameter	83.4 mm	83.4 mm	63.25 mm
Number of Baffles	None	None	None
Size of Baffles	None	None	None
Depth of Impeller	Just of bottom	Just of bottom	Just of bottom
Comments	None	None	None

Table 12 – Sheet#3 – Physical Property Data

Physical Property	LAW AZ-102 1.0M Na Pretreated Feed	LAW AZ-102 1.3M Na Pretreated Feed	AZ-102 1.0 M LAW 3 days of Mixing	AZ-102 1.0 M LAW MF 7 day	AZ-102 1.3 M LAW Melter Feed + 24 Hrs
Sodium concentration of LAW waste or pretreated waste (Molar)	1.02	1.33	1.02	1.02	1.32
Oxide loading of HLW sludge or pretreated sludge (total grams oxide/Liter)	N/A	N/A	N/A	N/A	N/A
pH – (aging 1 day, 1 week, 1 mo)	12.01	12.15	9.04 (3 days)	9.22 (7 days)	8.72 (1 day)
Solid phases present	yes	Yes	Yes	Yes	yes
Particle size distribution - Mean Vol. Distribution (μm)	N/A	N/A	28.86	27.32	30.53
Particle size distribution - Mean No. Distribution (μm)	N/A	N/A	0.353	0.361	0.354
Density – Bulk slurry (g/mL)	N/A	N/A	1.55	1.53	1.67
Density – settled solids (g/mL)	N/A	N/A	1.76	1.75	1.82
Density – centrifuged solids (g/mL)	N/A	N/A	1.86	1.89	1.90
Density - supernatant liquid (g/mL)	1.05	1.07	1.09	1.08	1.10
Vol. % settled solids after 72 hours	N/A	N/A	69.37	67.39	78.65
Vol. % centrifuged solids	N/A	N/A	61.37	59.42	72.40
Wt % total dried solids	6.34	8.14	52.15	53.41	59.61
Wt % centrifuged solids	N/A	N/A	73.34	73.21	82.67
Wt % oven dried solids	N/A	N/A	67.68	69.46	70.06
Wt % undissolved solids	N/A	N/A	47.19	48.52	55.26
Wt % dissolved solids	N/A	N/A	4.96	4.89	4.35

Table 12 – Sheet #5 Rheological Property Model Parameter Data

Model/model Parameter	LAW AZ-102 1.0M Na Pretreated Feed		LAW AZ-102 1.0M Na Pretreated Feed	
Shear Strength (by Vane Method):				
$\tau_{o,vane}$ - Shear Strength (Pa)	N/A	N/A	N/A	N/A
Ostwald (or Power Law):				
Shear rate range data fitted (sec^{-1})	0 - 2000	0 - 2000	0 - 2000	0 - 2000
m – consistency coefficient ($Pa\text{-}sec^n$)	0.0012	0.0012	0.0010	0.0011
n – power law exponent	1 (forced)	1 (forced)	1 (forced)	1 (forced)
R^2 – correlation coefficient	0.9964	0.9972	0.9932	0.9925
Bingham Plastic:				
Shear rate range data fitted (sec^{-1})	N/A	N/A	N/A	N/A
$\tau_O{}^B$ - Bingham yield stress (Pa)	N/A	N/A	N/A	N/A
η_P – Bingham plastic viscosity (cP)	N/A	N/A	N/A	N/A
R^2 – correlation coefficient	N/A	N/A	N/A	N/A
Herschel-Bulkley:				
Shear rate range data fitted (sec^{-1})	N/A	N/A	N/A	N/A
$\tau_O{}^H$ - Herschel-Bulkley yield stress (Pa)	N/A	N/A	N/A	N/A
k - Herschel-Bulkely consistency coefficient (cP)	N/A	N/A	N/A	N/A
b - Hershel-Bulkely power law exponent	N/A	N/A	N/A	N/A
R^2 – correlation coefficient	N/A	N/A	N/A	N/A

Table 12 – Sheet #5 Rheological Property Model Parameter Data

Model/model Parameter	LAW AZ-102 1.0M Na Melter Feed – 3 days		LAW AZ-102 1.0M Na Melter Feed – 7 days		LAW AZ-102 1.3M Na Melter Feed – 1 day	
Shear Strength (by Vane Method):						
$\tau_{o,vane}$ - Shear Strength (Pa)	218 (72 hrs)	N/A	112 (48 hrs)*	494 (48 hrs)*	2349 (50 hrs)	N/A
Ostwald (or Power Law):						
Shear rate range data fitted (sec^{-1})	N/A	N/A	N/A	N/A	N/A	N/A
m – consistency coefficient ($Pa\text{-}sec^{n}$)	N/A	N/A	N/A	N/A	N/A	N/A
n – power law exponent	N/A	N/A	N/A	N/A	N/A	N/A
R^2 – correlation coefficient	N/A	N/A	N/A	N/A	N/A	N/A
Bingham Plastic:						
Shear rate range data fitted (sec^{-1})	N/A	N/A	N/A	N/A	N/A	N/A
$\tau_O{}^B$ - Bingham yield stress (Pa)	N/A	N/A	N/A	N/A	N/A	N/A
η_P – Bingham plastic viscosity (cP)	N/A	N/A	N/A	N/A	N/A	N/A
R^2 – correlation coefficient	N/A	N/A	N/A	N/A	N/A	N/A
Herschel-Bulkley:						
Shear rate range data fitted (sec^{-1})	N/A	N/A	N/A	N/A	N/A	N/A
$\tau_O{}^H$ - Herschel-Bulkley yield stress (Pa)	N/A	N/A	N/A	N/A	N/A	N/A
k - Herschel-Bulkely consistency coefficient (cP)	N/A	N/A	N/A	N/A	N/A	N/A
b - Hershel-Bulkely power law exponent	N/A	N/A	N/A	N/A	N/A	N/A
R^2 – correlation coefficient	N/A	N/A	N/A	N/A	N/A	N/A

N/A - Flow curves were not fitted to any of the above rheological models due to settling issues related to this specific melter feed.

*** Measurement taken in one cup. The 2nd measurement was taken at a deeper depth than the 1st measurement.**

Table 12 – Sheet #5 Rheological Property Model Parameter Data

Model/model Parameter	LAW AZ-102 1.0M Na Melter Feed Supernatant – 3 days		LAW AZ-102 1.0M Na Melter Feed Supernatant – 7 days		LAW AZ-102 1.3M Na Melter Feed Supernatant – 1 day	
Shear Strength (by Vane Method):						
$\tau_{o,vane}$ - Shear Strength (Pa)	N/A	N/A	N/A	N/A	N/A	N/A
Ostwald (or Power Law):						
Shear rate range data fitted (sec^{-1})	0 - 2000	0 - 2000	0 - 2000	0 - 2000	0 - 2000	0 - 2000
m – consistency coefficient ($Pa\text{-}sec^n$)	0.0018	0.0018	0.0018	0.0017	0.002	0.0019
n – power law exponent	1 (forced)	1 (forced)	1 (forced)	1 (forced)	1 (forced)	1 (forced)
R^2 – correlation coefficient	0.9956	0.9973	0.9981	0.9972	0.9988	0.9987
Bingham Plastic:						
Shear rate range data fitted (sec^{-1})	N/A	N/A	N/A	N/A	N/A	N/A
τ_O^B - Bingham yield stress (Pa)	N/A	N/A	N/A	N/A	N/A	N/A
η_P – Bingham plastic viscosity (cP)	N/A	N/A	N/A	N/A	N/A	N/A
R^2 – correlation coefficient	N/A	N/A	N/A	N/A	N/A	N/A
Herschel-Bulkley:						
Shear rate range data fitted (sec^{-1})	N/A	N/A	N/A	N/A	N/A	N/A
τ_O^H - Herschel-Bulkley yield stress (Pa)	N/A	N/A	N/A	N/A	N/A	N/A
k - Herschel-Bulkely consistency coefficient (cP)	N/A	N/A	N/A	N/A	N/A	N/A
b - Hershel-Bulkely power law exponent	N/A	N/A	N/A	N/A	N/A	N/A
R^2 – correlation coefficient	N/A	N/A	N/A	N/A	N/A	N/A