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PHYSICAL CHARACTERIZATION OF VITREOUS STATE LABORATORY AY102/C106 AND AZ102 HIGH LEVEL WASTE MELTER FEED SIMULANTS (U)

MARCH 2005

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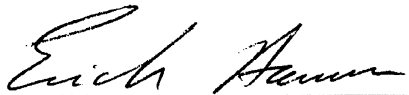
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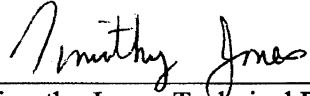
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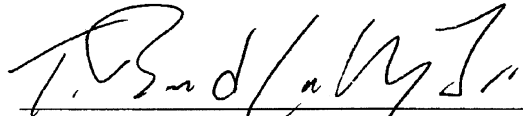
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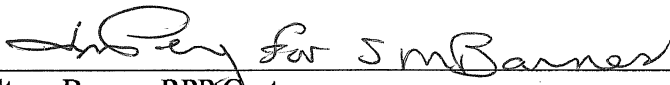
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LIST OF ACRONYMS AND ABBREVIATIONS

Avg.	Average
BP	Bingham Plastic
DI	Deionized
GFC	Glass Former Chemical
HLW	High Level Waste
ID	Identification
ITS	Immobilization Technology Section
MF	Melter Feeds
NIST	National Institute of Standards and Technology
QA	Quality Assurance
Ref.	Reference
RPP	River Protection Project
R&T	Research and Technology
R ²	Correlation Coefficient
SRNL	Savannah River National Laboratory
Stdev.	Standard Deviation
TS	Total Solids
UDS	Undissolved solids
Vol%	Volume Percent
VSL	Vitreous State Laboratory
Wt%	Weight Percent
WTP	Waste Treatment Plant
η_{BP}	Plastic Viscosity
τ_{BP}	Bingham Plastic Yield Stress

LIST OF DEFINITIONS

Variable	Definition
Density	Mass per unit volume.
Flow curve/rheogram	Plot of shear stress versus shear rate.
Interstitial solution	Solution contained between suspended, settled, or centrifuged solids.
Newtonian Fluid	A fluid whose apparent viscosity is independent of shear rate.
Non-Newtonian Fluid	A fluid whose apparent viscosity varies with shear rate.
Settled solids shear strength	The maximum settled solids shear stress as determined by the vane method for a slurry sample that is allowed to settle for a specified amount of time.
Solution	A liquid phase that can contain soluble solids.
Slurry	A mixture of insoluble solids and solution
Supernatant Liquid	A liquid phase overlying material deposited by settling, precipitation, or centrifugation.
Solids Settling Rate	Rate at which solids in a homogenized sample settle. The change in the interface height between the supernate and settled solids as a function of time.
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% 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.
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% oven dried solids	The percent mass of the centrifuged solids remaining after removing volatiles including free water by drying at $105 \pm 5^{\circ}\text{C}$ for at least 24 hours.
wt% settled solids	The percentage (mass basis) of settled solids present in the sample. Calculated on a percentage basis by dividing the mass of the settled solids by the mass of sample.
wt% soluble solids in supernatant	Calculated on a percentage basis by dividing the mass of the dried supernatant by the mass of the supernatant prior to drying.
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 $1050^{\circ}\text{C} \pm 50^{\circ}\text{C}$ for 1 hour.
wt% total solids	The percentage of the mass of dried solids divided by the mass of the slurry.
wt% undissolved solids	Calculated on a percentage basis by dividing the calculated mass of the undissolved solids by the mass of the bulk solids.
wt% water content	The percentage of the mass of water divided by the mass of the slurry.
Yield Stress	The minimum stress required to initiate flow. Determined by fitting measured flow curve using non-Newtonian rheological models.

ABSTRACT

The objective of this task is to characterize and report specified physical properties and pH of simulant high level waste (HLW) melter feeds (MF) processed through the scaled melters at Vitreous State Laboratories (VSL). The HLW MF simulants characterized are VSL AZ102 straight hydroxide melter feed, VSL AZ102 straight hydroxide rheology adjusted melter feed, VSL AY102/C106 straight hydroxide melter feed, VSL AY102/C106 straight hydroxide rheology adjusted melter feed, and Savannah River National Laboratory (SRNL) AY102/C106 precipitated hydroxide processed sludge blended with glass former chemicals at VSL to make melter feed. The physical properties and pH were characterized using the methods stated in the Waste Treatment Plant (WTP) characterization procedure (Ref. 7).

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1.0 TESTING SUMMARY

1.1 OBJECTIVES

The River Protection Project (RPP) Waste Treatment Plant (WTP) Research and Technology (R&T) requested the determination of physical properties and the pH of various HLW melter feed simulants that have been processed at VSL using different scaled glass melters. The HLW melter feed simulants to be characterized are VSL AZ102 straight hydroxide melter feed, VSL AZ102 straight hydroxide rheology adjusted melter feed, VSL AY102/C106 straight hydroxide melter feed, VSL AY102/C106 straight hydroxide rheology adjusted melter feed, and SRNL AY102/C106 precipitated hydroxide processed sludge blended with glass former chemicals at VSL to make melter feed. The original task described in the test specification [Ref. 5] has been completed [Ref. 6] and will not be described in this document. All the objectives in this task are provided in the test exception [ref. 1] and the objectives are listed in Table 1-1 for each of the melter feed simulants. The task plan [Ref. 2] is a generically written task plan that specifies the objectives are stated in either the test specification or test exception. All the objectives were met.

Table 1-1 RPP WTP R&T Test Objectives				
Test Objectives Stated In Test Exception [Ref. 1]			Objective Met (Y/N)	Discussion
AZ102 prepared via straight hydroxide (VSL/vendor prepared) characterize for:			Y	See Table 1-3, Table 2-7, Table 2-8 and Table 2-9 for summary of properties. See Appendix D for PSD histograms and Appendix E for individual flow curves. Section 2.1.1 determines the grams of oxides per liter melter feed of radioactive AZ102 melter feed and section 2.1.2 determines the required dilution required to obtain the equivalent grams of oxides per liter of the VSL AZ102 melter feed. The values in the parentheses in the wt% column in the table to the left are the calculated wt% undissolved solids (UDS).
wt% UDS ¹	# of meas. ²	Property to Measure		
5 (4.1)	3	Bulk density		
	3	Volume % settled solids		
	3	Flow Curves at 25°C, report Bingham Plastic Parameters		
10 (7.9)	3	Bulk density		
	3	Volume % settled solids		
	3	Flow Curves at 25°C, report Bingham Plastic Parameters		
15 (11.5)	3	Water Content and Oxide Content		
	1	pH		
	3	Bulk, settled solids, centrifuged solids, and supernatant densities		
	3	Total dried solids, centrifuged solids, oven dried, undissolved, and dissolved weight percent (wt%) solids		
	3	Volume % settled solids, centrifuged solids, and settling rate		
	3	Particle size distribution (PSD)		
	3	Flow Curves at 25°C, report Bingham Plastic Parameters		
	Mixing/Aging aging per TRPT-W374-00-00029, report Bingham Plastic Parameters.			
	1	Flow curve at 25°C after 1-hr of mixing.		
	1	Flow curve at 25°C after 1-day of mixing.		
	1	Flow curve at 25°C after 1-week of mixing.		
	1	Flow curve at 25°C after 1-week of aging (after having mixed for 1 week).		
20	3	Bulk density		
	3	Volume % settled solids		
	3	Flow Curves at 25°C, report Bingham Plastic Parameters		

¹ Starting wt% UDS is based on the sludge wt% UDS.

² # of meas. = the number of sample(s) measured for the property to measure at the given wt% UDS.

Table 1-1 RPP WTP R&T Test Objectives

Test Objectives Stated In Test Exception [Ref. 1]			Objective Met (Y/N)	Discussion
AZ102 prepared via straight hydroxide rheology adjusted (NOAH prepared) characterize for:			Y	See Table 1-4, Table 2-11, Table 2-12 and Table 2-13, for summary of properties. See Appendix E for individual flow curves. Section 2.1.1 determines the grams of oxides per liter melter feed of radioactive AZ102 melter feed and section 2.1.2 determines the required dilution required to obtain the equivalent grams of oxides per liter of the VSL AZ102 melter feed. The values in the parentheses in the wt% column in the table to the left are the calculated wt% undissolved solids
Wt% UDS ¹	# of meas.	Property to Measure		
5 (4.1)	3	Settling rate and volume % settled solids		
15 (11.5)	3	Water Content and Oxide Content		
	1	pH		
	3	Bulk density		
	3	Settling rate and volume % settled solids		
	3	Flow Curves at 25°C, report Bingham Plastic Parameters		
	Mixing/Aging aging per TRPT-W374-00-00029, report Bingham Plastic Parameters			
	1	Flow curve at 25°C after 1-hr of mixing.		
	1	Flow curve at 25°C after 1-day of mixing.		
	1	Flow curve at 25°C after 1-week of mixing.		
1	Flow curve at 25°C after 1-week of aging (after having mixed for 1 week).			
VSL AY102/C106 prepared via straight hydroxide characterize for:			Y	See Table 1-5, Table 2-15, Table 2-16, Table 2-17 and Table 2-18 for summary of properties. See Appendix D for PSD histograms and Appendix E for individual flow curves.
Wt% UDS	# of meas.	Property to Measure		
15	3	Bulk density		
	3	Volume % settled solids		
	3	Flow Curves at 25°C, report Bingham Plastic Parameters		
20	6	Water Content and Oxide Content		
	1	pH		
	6	Bulk density		
	6	Volume % settled solids, centrifuged solids, and settling rate		
	6	Particle size distribution		
	6	Flow Curves at 25°C, report Bingham Plastic Parameters		
	2	Shear Strength 0 hr gel time at 25°C		
	2	Shear Strength 9 hr gel times at 25°C		
	2	Shear Strength 24 hr gel time at 25°C		
VSL AY102/C106 prepared via straight hydroxide rheology adjusted characterize for:			Y	See Table 1-6, Table 2-19, Table 2-20 and Table 2-21 for summary of properties. See Appendix D for PSD histograms and Appendix E for individual flow curves.
Wt% UDS	# of meas.	Property to Measure		
20	3	Water Content and Oxide Content		
	1	pH		
	3	Bulk density		
	3	Volume % settled solids and settling rate		
	3	Particle size distribution		
	3	Flow Curves at 25°C, report Bingham Plastic Parameters		
	1	Shear Strength 0 hr gel time at 25°C		
	1	Shear Strength 9 hr gel times at 25°C		
	1	Shear Strength 24 hr gel time at 25°C		
SRNL AY102/C106 precipitated hydroxide characterize for:				
Wt% UDS	# of meas.	Property to Measure		
20	3	Water Content and Oxide Content		
	1	pH		
	3	Bulk density		
	3	Volume % settled solids and settling rate		
	3	Particle size distribution		
	3	Flow Curves at 25°C, report Bingham Plastic Parameters		
	1	Shear Strength 0 hr gel time at 25°C		
	1	Shear Strength 9 hr gel times at 25°C		
	1	Shear Strength 24 hr gel time at 25°C		

1.2 TEST EXCEPTIONS

The objectives performed in this report are described in section 1.1. This work is directly related to the test exception listed in Table 1-2. This test exception was written against the test specification [Ref. 5] as a means to perform the objectives stated in Table 1-1. There are at least two other test exceptions against this test specification, but they are not related to this task and will not be included in this report.

Table 1-2 Test Exception

List Test Exceptions	Describe Test Exceptions
24590-WTP-TEF-RT-04-00027, Rev. 0	Perform physical characterization and pH measurement of HLW MF simulants processed at VSL using different scale glass melters. The HLW sludge simulants are either made based on straight hydroxide or precipitated hydroxide preparation methods and then blended with GFCs to make melter feed. The HLW MF simulants and the properties to measure are listed in Table 1-1.

1.3 RESULTS AND PERFORMANCE AGAINST SUCCESS CRITERIA

The averaged physical properties and pH for VSL AZ102 straight hydroxide melter feed are shown in Table 1-3. The rheological properties of the individual flow curves are shown in Table 2-7. The settling data and the mean particle size distribution (PSD) parameters for the 11.5 wt% undissolved solids (UDS) are shown in Table 2-8 and Table 2-9 respectively. Section 2.1 provides the method used to process and dilute this simulant to the targeted grams of oxides per liter of melter feed given the radioactive AZ102 melter feed data. Additional details are located in section 2.2.

The averaged physical properties and pH for VSL AZ102 straight hydroxide rheology adjusted melter feed are shown in Table 1-4. The rheological properties of the individual flow curves are shown in Table 2-11, the settling data for the 4.1 wt% UDS melter feed in Table 2-12 and the settling data for the 11.5 wt% UDS melter feed in Table 2-13. Section 2.1 provides the method used to process and dilute this simulant to the targeted grams of oxides per liter of melter feed given the radioactive AZ102 melter feed data. Additional details are located in section 2.3.

The averaged physical properties and pH for VSL AY102/C106 straight hydroxide melter feed are shown in Table 1-5. The rheological properties of the individual flow curves are shown in Table 2-15. Two sets of settling data for the 20 wt% UDS melter feed are provided in Table 2-16 and Table 2-17. The PSD for the 20 wt% UDS is shown in Table 2-18. Additional details are located in section 2.4.

The averaged physical properties and pH for VSL AY102/C106 straight hydroxide rheology adjusted melter feed are shown in Table 1-6. The rheological properties of the individual flow curves are shown in Table 2-19. The settling data and PSD for the 20 wt% UDS melter feed are shown in Table 2-20 and Table 2-21 respectively. Additional details are located in section 2.5.

The averaged physical properties and pH for SRNL AY102/C106 precipitated hydroxide melter feed are shown in Table 1-7. The rheological properties of the individual flow curves are shown in Table 2-22. The settling data and PSD for the 20 wt% UDS melter feed are shown in Table 2-23 and Table 2-24 respectively. Additional details are located in section 2.6.

The individual flow curves are provided in Appendix E. The individual volume and number PSDs histograms are provided in Appendix D.

Table 1-3 Averaged VSL AZ102 Straight Hydroxide Melter Feed Properties

Property	Unit	Wt% Insoluble Solids Starting Sludge							
		4.1		7.9		11.5		20	
		Avg.	Stdev.	Avg.	Stdev.	Avg.	Stdev.	Avg.	Stdev.
pH	—	—	—	—	—	9.52	—	—	—
Wt % water content	%	—	—	—	—	71.5	0.2	—	—
Wt% oxide	%	—	—	—	—	26.1	0.2	—	—
Bulk density	g/mL	1.10	0.00	1.19	0.01	1.25	0.01	1.45	0.01
Settled solids density	g/mL	—	—	—	—	1.40	0.01	—	—
Centrifuged solids density	g/mL	—	—	—	—	1.61	0.01	—	—
Supernatant density	g/mL	—	—	—	—	1.07	0.00	—	—
Wt% total dried solids	%	—	—	—	—	28.5	0.2	—	—
Wt% centrifuged solids	%	—	—	—	—	42.0	0.5	—	—
Wt% oven dried solids	%	—	—	—	—	56.1	0.1	—	—
Wt% undissolved solids	%	—	—	—	—	21.8	0.2	—	—
Wt% dissolved solids in supernatant	%	—	—	—	—	8.59	0.05	—	—
Vol. % settled solids	%	24.8	0.6	42.4	1.1	56.7	0.4	90.7	1.2
Vol. % centrifuged solids	%	—	—	—	—	32.4	0.2	—	—
Settling Rate	—	—	—	—	—	Table 2-8		—	—
Particle Size Distribution	—	—	—	—	—	Table 2-9		—	—
Rheological Properties									
Bingham Plastic Yield Stress*	Pa	0.00	0.01	0.07	0.02	0.32	0.04	4.89	0.08
Plastic Viscosity*	mPa·s	1.49	0.02	2.29	0.03	3.90	0.10	18.3	4.9
Bingham Plastic Yield Stress – 1 hr mixing**	Pa	—	—	—	—	0.37	0.01	—	—
Plastic Viscosity – 1 hr mixing**	mPa·s	—	—	—	—	4.37	0.02	—	—
Bingham Plastic Yield Stress – 1 day mixing**	Pa	—	—	—	—	0.47	0.01	—	—
Plastic Viscosity – 1 day mixing**	mPa·s	—	—	—	—	4.62	0.03	—	—
Bingham Plastic Yield Stress – 1 week mixing**	Pa	—	—	—	—	0.54	0.00	—	—
Plastic Viscosity – 1 week mixing**	mPa·s	—	—	—	—	4.78	0.04	—	—
Bingham Plastic Yield Stress – 1 week mixing, 1 week age, decant supernatant**	Pa	—	—	—	—	6.74	0.06	—	—
Plastic Viscosity – 1 week mixing, decant, 1 week age, decant supernatant**	mPa·s	—	—	—	—	18.6	0.4	—	—

* Data averaged from both up and down curve results.

** Data averaged from down curve results only.

Table 1-4 Averaged VSL AZ102 Straight Hydroxide Rheology Adjusted Melter Feed Properties

Description	Unit	Wt% Insoluble Solids Starting Sludge			
		4.1		11.5	
		Avg.	Stdev.	Avg.	Stdev.
pH	—	—	—	9.49	—
Percent water content	%	—	—	69.0	0.2
Percent Oxide	%	—	—	26.6	0.1
Bulk density	g/mL	—	—	1.29	0.01
Vol. % settled solids	%	59.4	1.1	92.7	0.9
Settling Rate	—	Table 2-12		Table 2-13	
Rheological Properties*					
Bingham Plastic Yield Stress	Pa	—	—	6.02	0.02
Plastic Viscosity	mPa·s	—	—	13.4	0.1
Bingham Plastic Yield Stress – 1 hr mixing	Pa	—	—	6.48	0.07
Plastic Viscosity – 1 hr mixing	mPa·s	—	—	14.0	0.7
Bingham Plastic Yield Stress – 1 day mixing	Pa	—	—	7.47	0.11
Plastic Viscosity – 1 day mixing	mPa·s	—	—	15.2	0.4
Bingham Plastic Yield Stress – 1 week mixing	Pa	—	—	5.97	0.13
Plastic Viscosity – 1 week mixing	mPa·s	—	—	13.8	0.4
Bingham Plastic Yield Stress – 1 week mixing, 1 week age, decant supernatant	Pa	—	—	12.6	0.1
Plastic Viscosity – 1 week mixing, 1 week age, decant supernatant	mPa·s	—	—	23.3	0.1

* Data averaged from both up and down curve results.

Table 1-5 Averaged VSL AY102/C106 Straight Hydroxide Melter Feed Properties

Description	Unit	Wt% Insoluble Solids Starting Sludge					
		15		20 (1 st set)		20 (2 nd Set)	
		Avg.	Stdev.	Avg.	Stdev.	Avg.	Stdev.
pH	—	—	—	10.0	—	—	—
Percent water content	%	—	—	61.6	0.0	61.5	0.1
Percent Oxide	%	—	—	33.5	0.0	33.5	0.1
Bulk density	g/mL	1.28	0.01	1.38	0.02	1.37	0.01
Vol. % settled solids	%	79.8	0.4	90.1	0.4	90.0	0.3
Vol. % centrifuged solids	%	—	—	52.2	1.5	52.1	2.4
Settling Rate	—	—	—	Table 2-16		Table 2-17	
Particle Size Distribution	—	—	—	Table 2-18		Table 2-18	
Rheological Properties*							
Bingham Plastic Yield Stress	Pa	0.93	0.13	3.44	0.28	3.39	0.19
Plastic Viscosity	mPa·s	4.46	0.46	7.70	1.21	7.90	0.85
Settled Solids Shear Strength – 0 hrs of settling	Pa	—	—	5.04	—	5.36	—
Settled Solids Shear Strength – 9 hrs of settling	Pa	—	—	7.57	—	7.74	—
Settled Solids Shear Strength – 24 hrs of settling	Pa	—	—	30.9	—	28.1	—

* Data averaged from both up and down curve results.

Table 1-6 Averaged VSL AY102/C106 Straight Hydroxide Rheology Adjusted Melter Feed Properties

Description	Unit	Wt% Insoluble Solids Starting Sludge	
		20	
		Avg.	Stdev.
pH	—	9.57	—
Percent water content	%	56.1	0.1
Percent Oxide	%	37.6	0.0
Bulk density	g/mL	1.45	0.01
Vol. % settled solids	%	98.1	0.7
Settling Rate	—	Table 2-20	
Particle Size Distribution	—	Table 2-21	
Rheological Properties*			
Bingham Plastic Yield Stress	Pa	60.5	6.5
Plastic Viscosity	mPa·s	90.2	17.6
Settled Solids Shear Strength – 0 hrs of settling	Pa	42.6	—
Settled Solids Shear Strength – 9 hrs of settling	Pa	54.3	—
Settled Solids Shear Strength – 24 hrs of settling	Pa	68.2	—

Table 1-7 Averaged SRNL AY102/C106 Precipitated Hydroxide Melter Feed Properties

Description	Unit	Wt% Insoluble Solids Starting Sludge	
		20	
		Avg.	Stdev.
pH	—	9.93	—
Percent water content	%	58.2	0.0
Percent Oxide	%	36.5	0.0
Bulk density	g/mL	1.40	0.01
Vol. % settled solids	%	100	0
Settling Rate	—	Table 2-23	
Particle Size Distribution	—	Table 2-24	
Rheological Properties			
Bingham Plastic Yield Stress	Pa	11.0	0.7
Plastic Viscosity	mPa·s	27.9	2.3
Settled Solids Shear Strength – 0 hrs of settling	Pa	15.1	—
Settled Solids Shear Strength – 9 hrs of settling	Pa	18.0	—
Settled Solids Shear Strength – 24 hrs of settling	Pa	23.8	—

1.4 QUALITY REQUIREMENTS

This work was conducted in accordance with the RPP-WTP QA requirements specified for work conducted by SRNL as identified in DOE IWO M0SRLE60. SRNL has provided matrices to WTP demonstrating compliance of the SRNL QA program with the requirements specified by WTP. Specific information regarding the compliance of the SRNL QA program with RW-0333P, Revision 13, NQA-1 1989, Part 1, Basic and Supplementary Requirements and NQA-2a 1990, Subpart 2.7 is contained in these matrices.

The Task Technical and Quality Assurance Plan used to conduct this work are specified in reference 2.

1.5 R&T TEST CONDITIONS

The test exception (Ref. 1) establishes conditions to ensure that results are valid for project needs. Table 1-8 lists those conditions and indicates whether the conditions were followed. It describes the circumstances and consequences where deviations may have been necessary. (Note that deviations from specified conditions would normally require a Test Exception.)

Table 1-8 R&T Test Conditions

List R&T Test Conditions	Were Test Conditions Followed?
1. Perform solids, oxides and pH measurements using RPP characterization procedure [Ref. 7]	Yes.
2. All rheological measurements to follow RPP-WTP characterization procedure [Ref. 7]. Flow curves to be fitted to the Bingham Plastic model. Flow curves to be performed with an up curve shear rate range of 0 to 300 sec ⁻¹ and linear ramp time of two minutes, no hold, and a down curve shear rate range of 300 to 0 sec ⁻¹ and linear ramp time of two minutes. All flow curves to be measured at 25°C.	Yes.
3. Mass of mixing system (vessel, sample, impeller, and shaft) will be weighed. SRNL mixing system is not completely sealed, any weight loss other than from sampling to be maintained with DI water.	Yes.
4. Vane measurements to be performed at 25°C.	Yes.

1.6 SIMULANT USE

The HLW MF simulant characterized in this task were provided by VSL and authorized by WTP for characterization. Simulant usage by VSL was determined by WTP as appropriate.

1.7 DISCREPANCIES, FOLLOW-ON TESTS AND OBSERVATIONS

No discrepancies were encountered while performing this task with respect to the type of measurements and testing requested from WTP. No follow-on tests are necessary.

A visual observation of the as-received melter feed samples indicated they all contained large particles that could impact rheological measurements. This was also verified rheologically. The as-received samples (prior to dilution) were initially analyzed using the Z41 rotor, which has the smallest gap between the rotor and cup and is the recommended rotor to use per the RPP-WTP characterization procedure (Ref. 7). During the flow curve measurement, particle jamming was observed for all the samples due to these larger particles. This required the initial as-received samples to be analyzed using the Z38 rotor, which has a larger gap. For diluted samples, either the Z38 or Z41 rotor was utilized based on the viscosity of the sample. If the Z41 was utilized, most of the larger particles settled out of the gap and into the lower section of the cup, with no impact to the flow curve measurement. Additionally, the Z41 rotor was required if the diluted sample was too thin rheologically and its flow properties could not be measured using the Z38 rotor. The larger particles could be due to how the GFCs were processed prior to blending with the waste stream; how they were delivered to the mixing vessel; the type of mixing equipment used to entrain and disperse the GFCs; or due to agglomeration after mixing ceased. The 7 day mixing tests on the AZ102 MF simulants did not show any improvement in reducing these larger particles. No attempt was made by SRNL to quantify the size of these larger particles. Appendix E contains all the flow curves. The type of rotor (Z38 or Z41) utilized is clearly specified in each of these figures.

2.0 DISCUSSION

The scope of this work is to characterize the physical properties and pH of five different HLW MF simulants that have been processed at VSL (Ref. 3 and 4). The detailed objectives of this task are summarized in Table 1-1 for each simulant. Each HLW MF simulant provided by VSL came in two one liter bottles; both were homogenized and combined into one two liter bottle at SRNL. For the different samples provided by VSL, the sample identification (ID) used in this report, VSL sample ID, waste group, simulant preparation method, VSL melter simulant was tested, and if analyzed in this report are shown in Table 2-1. The VSL AY102/C106 rheology adjusted simulant, which came from the DM1200 (Ref. 3) melter test, was reddish in color and different from the simulant, which was brownish in color, used in the DM100 (Ref. 4) melter test (see Figure 2-1). The VSL AY102/C106 rheology adjusted sample from the DM1200 melter test was received and characterized prior to receiving the AY102/C106 rheology adjusted DM100 samples at SRNL; hence the samples from the DM100 test were not characterized. Per email confirmation with WTP R&T, the end of run SRNL precipitated hydroxide slurry was to be characterized.

Table 2-1 Simulant HLW MF Samples Provide By VSL

Sample ID used in this document	VSL Sample ID	Waste Group	Simulant Preparation Method	VSL Melter	Analyzed
VSLAZ102	1V2-F-111A 1V2-F-111B	AZ102	Straight hydroxide (VSL prepared).	DM1200	Yes
VSLAZ102RA	1U2-F-137A 1U2-F-137B	AZ102	Straight hydroxide rheology adjusted (VSL prepared).	DM1200	Yes
VSLAY102/C106	BLJ-F-81C-1 BLJ-F-81C-2	AY102/C106	Straight hydroxide (VSL prepared).	DM100	Yes
—	BLJ-F-135B-1 BLJ-F-135B-2	AY102/C106	Straight hydroxide rheology adjusted (VSL prepared).	DM100	No
VSLAY102/C106RA	1X2-F-48A 1X2-F-48B	AY102/C106	Straight hydroxide rheology adjusted (VSL prepared).	DM1200	Yes
—	BLK-F-17B-1 BLK-F-17B-2	AY102/C106	Precipitated hydroxide (SRNL prepared) and blended with GFCs at VSL – beginning of run sample.	DM100	No
SRNLAY102/C106	BLK-F-28B-1 BLK-F-28B-2	AY102/C106	Precipitated hydroxide (SRNL prepared) and blended with GFCs at VSL – end of run sample.	DM100	Yes

The characterization methods used to determine the water content, oxide content, density (bulk, settled solids, centrifuged solids, and supernatant), weight percent (total dried solids, centrifuged solids, oven dried solids, undissolved solids, and dissolved solids), and volume percent (settled solids, centrifuged solids, and settling rate) are described in detail in Appendix A and are the methods recommended in reference 7 for characterizing WTP waste streams. Since all the samples did not require the full suite of characterization, only specified characterization methods were performed for a given sample as listed in Table 1-1. The rheological methods used to analyze the samples are described in detail in Appendix B. The method used to determine the particle size distribution (PSD) is provided in Appendix D. Appendix D also contains both the volume PSD and number PSD for the samples that required analyses.

The processing of the VSL samples and resulting data will be discussed in the sample ID order as shown in Table 2-1.

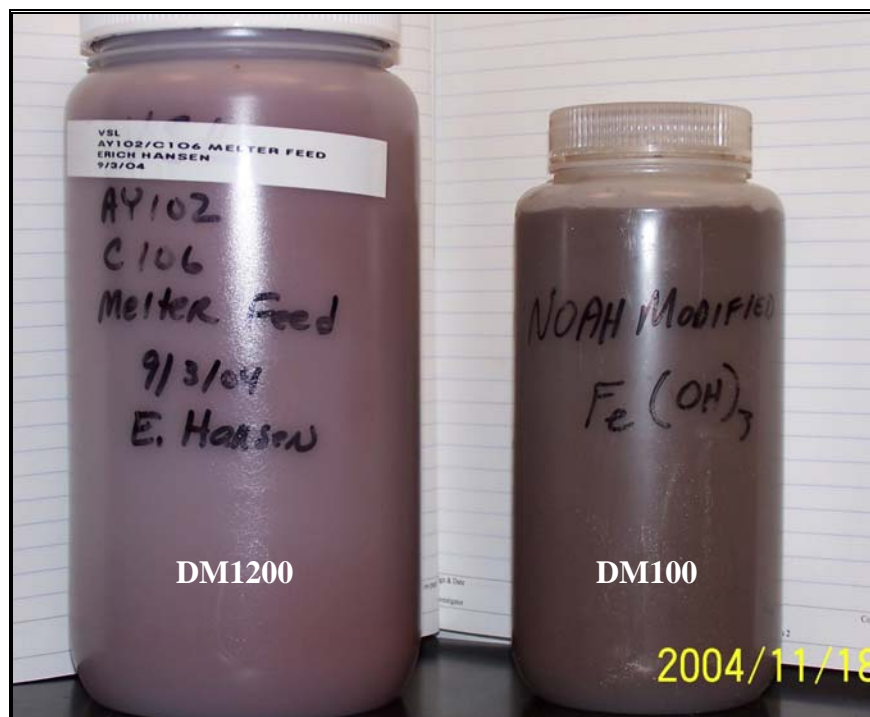


Figure 2-1 VSL AY102/C106 Rheology Adjusted Melter Feed

2.1 PROCESSING VSL AZ102 MELTER FEED SAMPLES

The composition of the VSL AZ102 straight hydroxide MF simulant can be found in reference 3. The basis for processing this simulant, as well as the AZ102 straight hydroxide rheology adjusted MF, was to process the simulants at the same grams of oxides per liter of MF of the actual radioactive AZ102 MF that was processed by Battelle, per WTP R&T direction. The method used for determining the grams of oxides per liter of MF of the radioactive AZ102 processed by Battelle is first described in section 2.1.1. The amount of dilution required in targeting the same grams of oxides per liter in the simulant AZ102 MF with that of the radioactive AZ102 MF is described in section 2.1.2. The extended mixing test is described in section 2.1.3.

2.1.1 Determining Grams Oxides Per Liter, Radioactive AZ102 Melter Feed

As directed by the WTP R&T (Eugene Morrey), the VSL AZ102 MF simulants were to be processed on the same grams oxides per liter MF basis as that of the radioactive AZ102 MF processed by Battelle (Ref. 9). The radioactive AZ102 sludge was blended with an AN107 Sr/TRU precipitate sludge simulant and glass former chemicals (Ref. 9). The elemental analyses of the radioactive AZ102 sludge (Ref. 10) and the Sr/TRU precipitated sludge (Ref. 9) were provided on a dry basis and were converted to an oxides bases to determine the dry calcined (grams oxide per grams dry mass at 105°C) values for each waste stream. Table 2-2 contains the radioactive AZ102 sludge results and Table 2-3 contains the Sr/TRU precipitated sludge results.

Table 2-2 Calculated Dry Calcined Value for Radioactive AZ102 Sludge

Elemental and Oxide of Processed AZ102 Radioactive Sludge							
Element	µg elemental /g-dry	Oxide	µg oxides /g-dry	Element	µg elemental /g-dry	Oxide	µg oxides /g-dry
Ag	470	Ag ₂ O	505	Mn	5380	MnO	6947
Al	107000	Al ₂ O ₃	202175	Na	83300	Na ₂ O	112286
B	225	B ₂ O ₃	724	Nd	5220	Nd ₂ O ₃	6089
Ba	958	BaO	1070	Ni	16200	NiO	20615
Be	23	BeO	62	P	2800	P ₂ O ₅	6416
Ca	11000	CaO	15391	Pb	2250	PbO	2424
Cd	33000	CdO	37697	Si	7100	SiO ₂	15189
Ce	1550	Ce ₂ O ₃	1815	Sr	473	SrO	559
Co	120	CoO	153	Ti	185	TiO ₂	309
Cr	1640	Cr ₂ O ₃	2397	U	42400	U ₃ O ₈	49999
Cu	585	CuO	732	Y	310	Y ₂ O ₃	394
Fe	221000	Fe ₂ O ₃	315973	Zn	970	ZnO	1207
La	7410	La ₂ O ₃	8690	Zr	32700	ZrO ₂	44171
Mg	1950	MgO	3233	Dry calcined:			0.8572

Table 2-3 Calculated Dry Calcined Value for Simulant AN107 Sr/TRU Precipitate

Simulant AN107 Sr/TRU Precipitate			
Element	µg elemental /g-dry	Oxide	µg oxides /g-dry
Ca	3400	CaO	4757
Cr	590	Cr ₂ O ₃	862
Fe	27000	Fe ₂ O ₃	38603
Mn	160000	MnO	206597
Na	39000	Na ₂ O	52571
P	710	P ₂ O ₅	1627
Pb	1200	PbO	1293
Sr	390000	SrO	461216
Dry calcined:			0.7675

The calculated dry calcined values were then used to determine the oxide contributions from the radioactive AZ102 sludge and Sr/TRU sludge used for making the different wt% TS starting sludge AZ102 melter feeds at Battelle. Appendix C contains the details used to determine the grams of oxides per liter of the different AZ102 melter feeds processed by Battelle. Note that the glass former chemicals (GFCs) used by Battelle are not the baseline GFCs (Ref. 8). Battelle was not required, at that time, to use baseline GFCs. It is assumed that the GFCs used by Battelle have an assay of 100% and yield the oxides as listed in Appendix C. The calculated grams of oxides per liter for a given wt% TS starting AZ102 sludge is provided in Table 2-4.

2.1.2 Dilution of VSL AZ102 Melter Feeds

The composition (wt% UDS, mass of sludge and mass of GFCs) of the VSL AZ102 MF are provided in reference 3. The densities of the VSL AZ102 melter feeds were determined using the WTP cone method and the results were used to determine the grams of oxides per liter. This information was used to determine the amount of de-ionized (DI) water necessary for diluting 100 grams of VSL AZ102 MF to the same grams of oxide per liter of the radioactive AZ102 MF processed by Battelle. Appendix C contains the detailed calculations and the amount of DI water necessary to dilute 100 grams of VSL AZ102 MF to targeted grams of oxides per liter of MF. Table 2-4 contains the grams of oxides per liter of MF as well as the calculated wt% UDS of the VSL AZ102 MF simulants after being diluted with DI water. Note that the wt% solids for the VSL AZ102 MF are provided as UDS.

Table 2-4 Gram Oxides per Liter MF and wt% Sludge for AZ102 Sludges

g-oxides/liter MF	AZ102 Sludge	
	Radioactive (wt% TS)	Calculated VSL (wt% UDS)
125	5	4.1
236	10	7.9
338	15	11.5
560	—	20.0 (given)

The VSL AZ102 MF simulants were first homogenized and a predetermined mass of MF was pulled and placed into an appropriate sized bottle and then diluted with the appropriate mass of DI water to the targeted gram oxides per liter for 4.1, 7.9 and 11.5 wt% UDS.

2.1.3 Setup of Extended Mixing Test

The 11.5 wt% UDS VSL AZ102 melter feeds were selected for extended mixing and aging tests. The mixing tests were performed to determine if agitation would impact the rheological properties over time. The resulting product was then allowed to settle for 7 additional days. Next the standing supernatant was decanted and the settled solid homogenized and analyzed for flow properties [Ref. 9]. The mass of simulant (plus DI water), mass of mixing vessel, mass of impeller and shaft were measured and recorded. The agitator, mixing vessel, mass of VSL AZ102 melter feed at 11.5 wt% UDS, and agitator speed to maintain adequate mixing (slurry motion evident throughout the mixing system) throughout the 7 day mixing tests are provided in Table 2-5. Approximately 70 to 80 mL of sample was pulled at 1 hour, 1 day and 1 week of mixing and analyzed rheologically. These pulled samples were not returned to the mixing vessel. Approximately one hour prior to pulling samples at 1 day and 1 week, the mixing system was weighed and any difference in mass was made up with DI water. After the mixing tests were completed, the sample was allowed to sit undisturbed for 7 days prior to decanting the supernatant and then performing flow curves on the settled solids.

Table 2-5 VSL AZ102 Melter Feed Extended Mixing Test Conditions

Description	11.5 wt% UDS VSL AZ102 Straight Hydroxide Slurries	
	Regular VSLAZ102	Rheology Adjusted VSLAZ102RA
Mixing Time	7 days	7 days
Impeller Type	Lightnin A-100 3 blade axial flow	Lightnin A-100 3 blade axial flow
Impeller Width W (inches)	2.7	2.7
Tank Type	1000 mL large mouth Nalgene bottle	1000 mL large mouth Nalgene bottle
Tank Inside Diameter (inches)	3.5	3.5
Tank Height (inches)	6.75	6.75
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
Initial sample size (grams)	913	726
Agitator Speed (Revolutions Per Minute) During Week Long Test		
Initial – 1 hr	500	650
1 hr to 1 day	500	650
1 day to 7 days	500	600

2.2 VSL AZ102 STRAIGHT HYDROXIDE MELTER FEED RESULTS

The VSL AZ102 straight hydroxide melter feed (VSLAZ102) averaged physical properties and pH results are provided in Table 1-3. The Bingham Plastic model results fitted to both the up and down curves for each flow curve are shown in Table 2-7. Appendix E contains each flow curve measurement. Table 2-8 contains the cone settling data. The mean PSD variables are provided in Table 2-9 and the volume and number PSD histograms are provided in Appendix D.

The calculated (from Appendix C) and the measured densities for the VSL AZ102 straight hydroxide melter feeds are shown in Table 2-6. The results are similar.

Table 2-6 Measured and Calculated VSLAZ102 Melter feeds

VSL AZ102 Straight Hydroxide Melter Feed		
Wt% (UDS)	Density (g/mL)	
	Measured	Calculated
4.1	1.10	1.10
7.9	1.19	1.19
11.5	1.25	1.28

Table 2-7 VSLAZ102 Melter Feed Bingham Plastic Data

VSL AZ102 Straight Hydroxide Melter Feed									
Wt% UDS or Condition (Figure related to data)	Run	Up Curve				Down Curve			
		τ_{BP} (Pa)	μ_{BP} (mPa·s)	R^2	Fitted $\dot{\gamma}$ range (sec ⁻¹)	τ_{BP} (Pa)	μ_{BP} (mPa·s)	R^2	Fitted $\dot{\gamma}$ range (sec ⁻¹)
4.1 (Figure E - 1)	1	0.01	1.48	0.9983	25 - 270	-0.01	1.51	0.9969	25 - 270
	2	0.01	1.47	0.9986	0 - 270	0.00	1.50	0.9973	0 - 270
	3	0.01	1.47	0.9986	0 - 270	0.00	1.50	0.9978	0 - 270
	Avg.	0.01	1.48	—	—	0.00	1.51	—	—
	Stdev.	0.00	0.00	—	—	0.00	0.01	—	—
7.9 (Figure E - 2)	1	0.08	2.30	0.9962	0 - 300	0.05	2.32	0.9984	0 - 300
	2	0.08	2.26	0.9977	0 - 300	0.05	2.31	0.9984	0 - 300
	3	0.08	2.25	0.9977	0 - 300	0.05	2.29	0.9983	0 - 300
	Avg.	0.08	2.27	—	—	0.05	2.31	—	—
	Stdev.	0.00	0.03	—	—	0.00	0.01	—	—
11.5 (Figure E - 3)	1	0.38	3.73	0.9951	25 - 300	0.28	3.98	0.9949	0 - 300
	2	0.35	3.88	0.9903	0 - 300	0.29	3.98	0.9949	0 - 300
	3	0.34	3.87	0.9902	0 - 300	0.28	3.97	0.9948	0 - 300
	Avg.	0.36	3.83	—	—	0.28	3.98	—	—
	Stdev.	0.02	0.09	—	—	0.00	0.01	—	—
20 (Figure E - 4)	1	4.78	18.8	0.9715	0 - 300	4.97	18.1	0.9681	0 - 300
	2	4.83	18.5	0.9755	0 - 300	4.97	18.0	0.9781	0 - 300
	3	4.83	18.5	0.9768	0 - 300	4.95	18.0	0.9710	0 - 300
	Avg.	4.82	18.6	—	—	4.96	18.0	—	—
	Stdev.	0.03	0.2	—	—	0.01	0.1	—	—
Extended Mixing Test – 11.5 wt% UDS									
1 hour of mixing (Figure E - 5)	1	0.47	4.11	0.9849	0 - 300	0.36	4.35	0.9943	0 - 300
	2	1.63	-1.23	0.0086	0 - 300	0.38	4.38	0.9968	0 - 300
	Avg.	1.05	1.44	—	—	0.37	4.37	—	—
	Stdev.	0.82	3.78	—	—	0.01	0.02	—	—
1 day of mixing (Figure E - 5)	1	0.60	4.32	0.9812	0 - 300	0.47	4.60	0.9930	0 - 300
	2	1.72	-1.02	0.0088	0 - 300	0.46	4.64	0.9934	0 - 300
	Avg.	1.16	1.65	—	—	0.47	4.62	—	—
	Stdev.	0.79	3.78	—	—	0.01	0.03	—	—
1 week of mixing (Figure E - 5)	1	0.78	4.04	0.7961	0 - 300	0.54	4.81	0.9917	0 - 300
	2	0.70	4.31	0.9462	0 - 300	0.54	4.75	0.9954	0 - 300
	Avg.	0.74	4.17	—	—	0.54	4.78	—	—
	Stdev.	0.05	0.19	—	—	0.00	0.04	—	—
1 week mixing, 1 week aging, decant supernatant (Figure E - 5)	1	6.78	18.3	0.9614	0 - 300	6.74	19.1	0.9482	0 - 300
	2	6.78	18.3	0.9652	0 - 300	6.66	18.8	0.9570	0 - 300
	Avg.	6.78	18.3	—	—	6.70	18.9	—	—
	Stdev.	0.00	0.01	—	—	0.05	0.23	—	—

Table 2-8 Settling Data 11.5 wt% UDS VSLAZ102 MF Settling Test

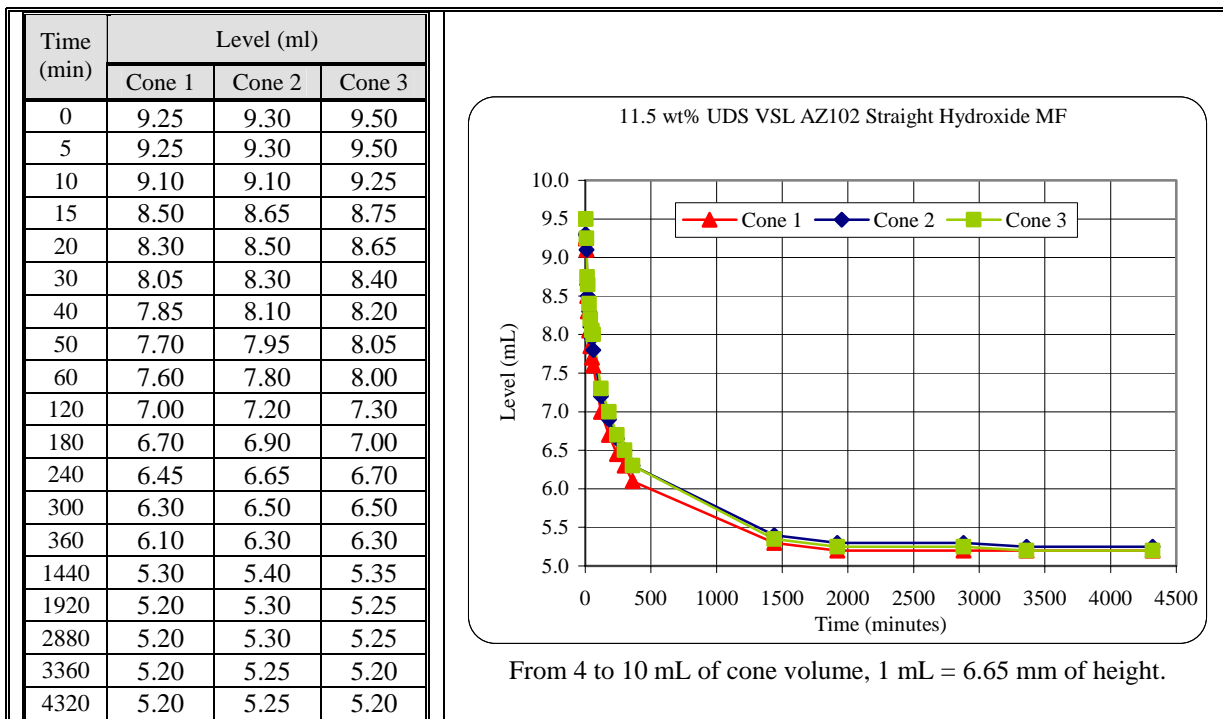


Table 2-9 Mean PSD Variables for 11.5 wt% UDS VSLAZ102 MF

11.5 wt% UDS VSL AZ102 Straight Hydroxide Melter Feed			
Variable	1 st Run	2 nd Run	3 rd Run
Mean Number (μm)	0.258	0.416	0.378
Mean Surface Area (μm)	1.475	1.689	1.878
Mean Volume (μm)	26.09	11.38	21.79
Reference Figure	Figure D - 1	Figure D - 2	Figure D - 3

Originally, the Z41 rotor was selected for all the measurements, but it was evident during the 20 wt% UDS VSL AZ102 MF flow curve measurement that the particles were causing significant jamming issues as shown in Figure 2-2; consequently, the data could not be properly analyzed. Because the Z41 rotor has the smallest gap between the rotor and cup, the as-received samples were analyzed using the Z38 rotor, which has a larger gap than the Z41 rotor; therefore, the larger particles did not impact the measurement with respect to jamming the rotor. For the diluted samples, utilization of the Z41 rotor was permitted, since these large particles were allowed to settle to the bottom of the cup. Additional, the Z41 rotor was required if the diluted sample was too thin rheologically, where its flow properties could not be measured using the Z38 rotor. This issue of jamming was consistent when using the Z41 rotor for the as-received VSL MF samples. Appendix E contains all the flow curves and the type of rotor (Z38 or Z41) utilized is clearly specified in each of these figures.

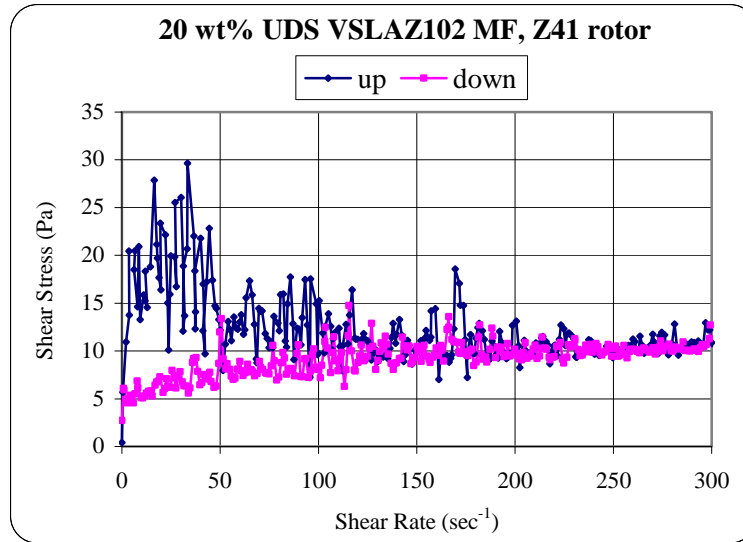


Figure 2-2 20 Wt% UDS VSLAZ102 MF Using Z41 Rotor

During the flow curve measurement of the 4.1 wt% UDS VSLAZ102 MF, Taylor vortices were noticed. The Taylor vortices equation in reference 7 was plotted against a representative 4.1 wt% UDS VSLAZ102 MF flow curve as shown in Figure 2-3. Measured data below the Taylor vortices curve is neglected, limiting the Bingham Plastic fit to an upper limit of 270 sec^{-1} .

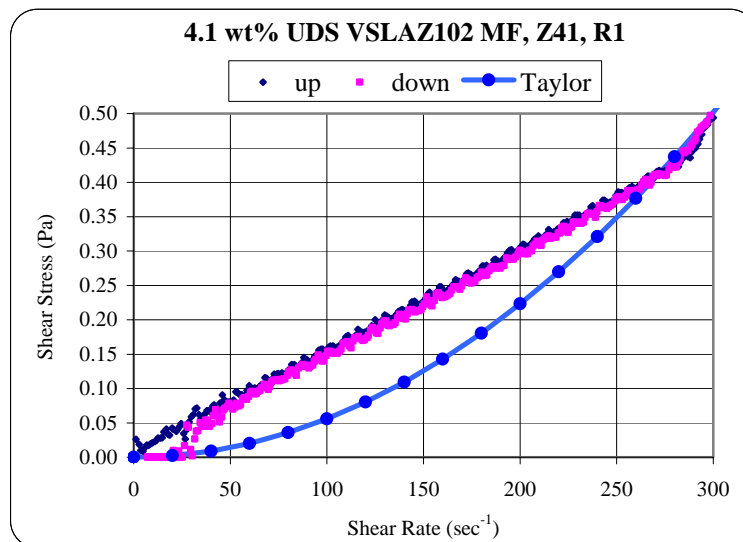


Figure 2-3 5 wt% UDS VSLAZ102 MF Using Z41 Rotor

2.3 VSL AZ102 STRAIGHT HYDROXIDE RHEOLOGY ADJUSTED MELTER FEED RESULTS

The VSL AZ102 straight hydroxide rheology adjusted melter feed (VSLAZ102RA) averaged physical properties and pH results are provided in Table 1-4. The Bingham Plastic model results fitted to both the up and down curves for each flow curve are shown in Table 2-11. Appendix E contains each flow curve measurement. Table 2-12 and Table 2-13 contain the cone settling data.

The calculated (from Appendix C) and the measured densities for the 11.5 wt% UDS VSLAZ102RA MF is shown in Table 2-6. The results are similar.

Table 2-10 Measured and Calculated of 11.5 wt% UDS VSLAZ102RA Melter Feed

VSL AZ102 Straight Hydroxide Rheology Adjusted Melter Feed		
Wt% (UDS)	Density (g/mL)	
	Measured	Calculated
11.5	1.29	1.28

Table 2-11 VSLAZ102RA Melter Feed Bingham Plastic Data

VSL AZ102 Straight Hydroxide Rheology Adjusted Melter Feed									
11.5 Wt% UDS Condition (Figure related to data)	Run	Up Curve				Down Curve			
		τ_{BP} (Pa)	μ_{BP} (mPa·s)	R^2	Fitted $\dot{\gamma}$ range (sec ⁻¹)	τ_{BP} (Pa)	μ_{BP} (mPa·s)	R^2	Fitted $\dot{\gamma}$ range (sec ⁻¹)
As Received (Figure E - 6)	1	6.02	13.4	0.9882	20 - 300	6.04	13.3	0.9888	20 - 300
	2	6.00	13.5	0.9884		6.05	13.3	0.9865	
	3	6.01	13.4	0.9897		6.01	13.4	0.9888	
	Avg.	6.01	13.4	—	—	6.03	13.3	—	—
	Stdev.	0.01	0.0	—	—	0.02	0.0	—	—
Extended Mixing Test – 11.5 wt% UDS									
1 hour of mixing (Figure E - 7)	1	6.46	13.2	0.9965	20 - 300	6.52	13.6	0.9863	20 - 300
	2	6.39	14.6	0.9949		6.54	14.5	0.9887	
	Avg.	6.43	13.9	—	—	6.53	14.0	—	—
	Stdev.	0.05	1.00	—	—	0.02	0.6	—	—
1 day of mixing (Figure E - 7)	1	7.46	15.0	0.9876	20 - 300	7.59	14.7	0.9837	20 - 300
	2	7.33	15.6	0.9930		7.50	15.5	0.9860	
	Avg.	7.40	15.3	—	—	7.55	15.1	—	—
	Stdev.	0.09	0.45	—	—	0.06	0.6	—	—
1 week of mixing (Figure E - 7)	1	5.85	14.2	0.9925	20 - 300	6.08	13.7	0.9868	20 - 300
	2	5.87	13.9	0.9879		6.09	13.3	0.9852	
	Avg.	5.86	14.1	—	—	6.08	13.5	—	—
	Stdev.	0.01	0.15	—	—	0.01	0.3	—	—
1 week mixing, 1 week aging, decant supernatant (Figure E - 7)	1	12.6	23.2	0.9860	20 - 300	12.7	23.4	0.9831	20 - 300
	2	12.5	23.4	0.9827		12.5	23.3	0.9827	
	Avg.	12.55	23.3	—	—	12.61	23.4	—	—
	Stdev.	0.12	0.19	—	—	0.11	0.0	—	—

Table 2-12 Settling Data 4.1 wt% UDS VSLAZ102RA MF Settling Test

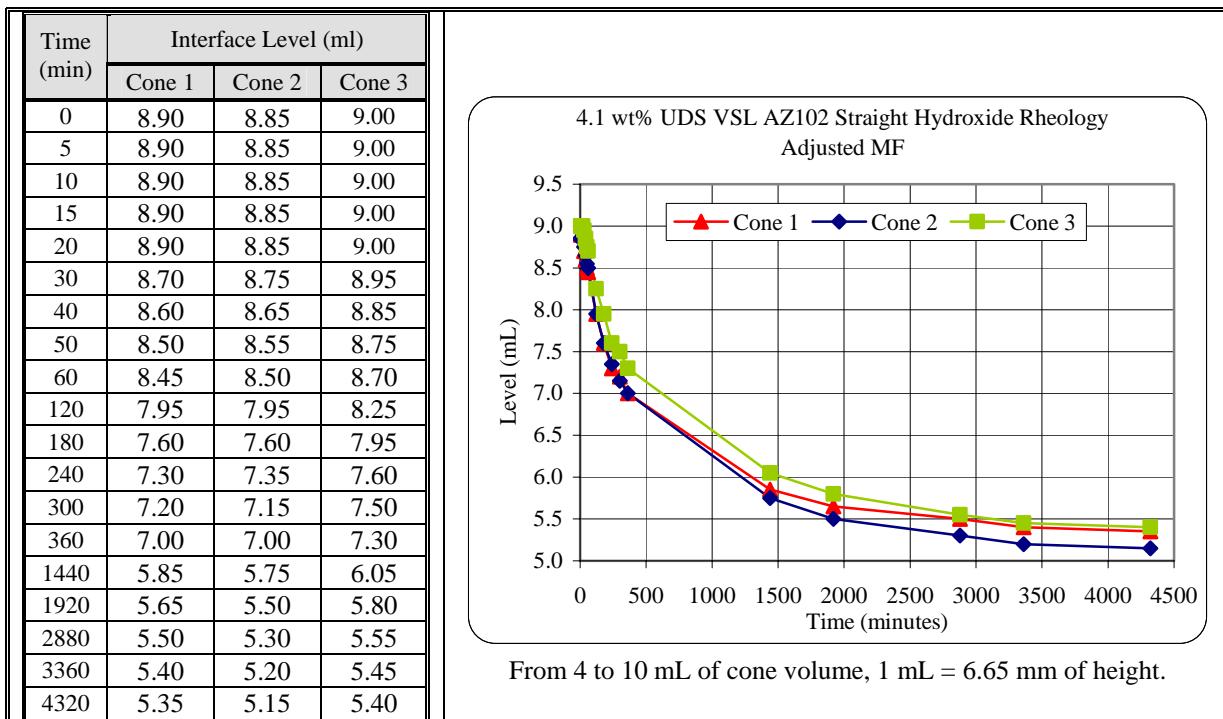
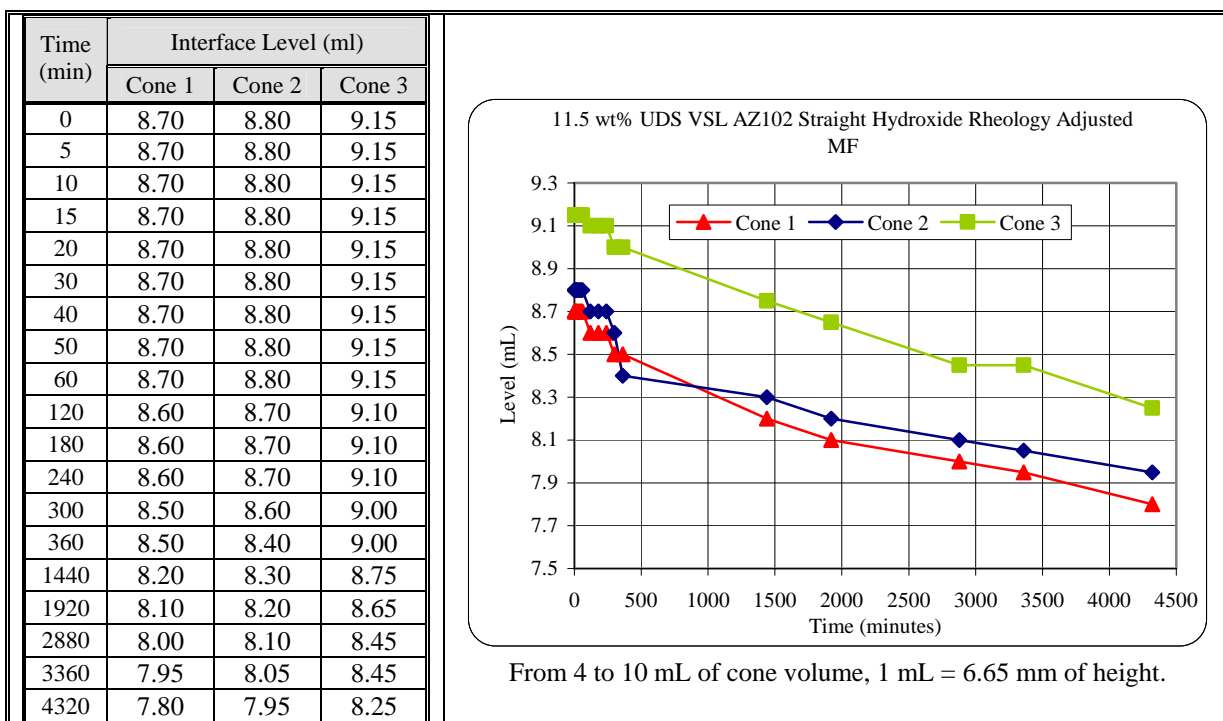


Table 2-13 Settling Data 11.5 wt% UDS VSLAZ102RA MF Settling Test



2.4 VSL AY102/C106 STRAIGHT HYDROXIDE MELTER FEED RESULTS

The VSL AY102/C106 straight hydroxide melter feed (VSLAY102/C106) was also diluted to 15 wt% UDS starting sludge. Reference 3 provides the mass of sludge (2227.3 Kg) and mass of GFCs (828.12 Kg) with a starting UDS sludge concentration of 20 wt%. The batching of a 15 wt% UDS sludge basis feed consisted of 200 grams of this melter feed (yielding a total mass of 145.8 grams of which 29.2 grams are UDS in this 20 wt% UDS sludge) blended with 48.6 grams of DI water.

The VSL AY102/C106 straight hydroxide melter feeds averaged physical properties and pH results are provided in Table 1-5. The Bingham Plastic model results fitted to both the up and down curves for each flow curve are shown in Table 2-15. Appendix E contains each flow curve measurement. Two settling data sets for the 20 wt % UDS VSLAY102/C106 are shown in Table 2-16 and Table 2-17. The 20 wt % UDS VSLAY102/C106 mean PSD variables for two different sets are provided in Table 2-18 and the volume and number PSD histograms are provided Appendix D.

The measured and calculated (using the density of the measured MF at 20 wt% UDS and volume additivity) densities of the 15 wt% UDS VSLAY102/C106 MF are shown in Table 2-14. The results are similar.

Table 2-14 Measured and Calculated of 15 wt% UDS VSLAY102/C106 Melter Feed

VSL AY102/C106 Straight Hydroxide Melter Feed		
Wt% (UDS)	Density (g/mL)	
	Measured	Calculated
15	1.28	1.28

Table 2-15 VSLAY102/C106 MF Bingham Plastic Data

VSL AY102/C106 Straight Hydroxide Melter Feed									
Wt% UDS (Figure related to data)	Run	Up Curve				Down Curve			
		τ_{BP} (Pa)	μ_{BP} (mPa·s)	R^2	Fitted $\dot{\gamma}$ range (sec ⁻¹)	τ_{BP} (Pa)	μ_{BP} (mPa·s)	R^2	Fitted $\dot{\gamma}$ range (sec ⁻¹)
15 (Figure E - 8)	1	1.00	4.22	0.9782	0 - 300	0.819	4.80	0.999	0 - 300
	2	1.09	3.97	0.8656		0.817	4.96	0.993	
	3	1.04	3.97	0.6889		0.786	4.86	0.994	
	avg.	1.05	4.05	—	—	0.81	4.87	—	—
	std	0.04	0.15	—	—	0.02	0.08	—	—
20 – 1 st Set (Figure E - 9)	1	3.58	7.09	0.9485	20 - 250	3.21	8.84	0.9470	0 – 250
	2	3.57	7.14	0.9767		3.23	8.47	0.9724	
	3	3.55	7.17	0.9707		3.22	8.72	0.9546	
	Avg.	3.57	7.13	—	—	3.22	8.67	—	—
	Stdev.	0.02	0.04	—	—	0.01	0.19	—	—
20 – 2 nd Set (Figure E - 10)	1	3.67	6.65	0.9715	20 - 250	3.18	8.90	0.9655	0 - 250
	2	3.75	6.09	0.9511		3.19	8.70	0.9701	
	3	3.67	7.19	0.9698		3.19	8.69	0.9682	
	Avg.	3.70	6.64	—	—	3.19	8.76	—	—
	Stdev.	0.05	0.55	—	—	0.01	0.12	—	—

Table 2-16 Settling Data 20 wt% UDS VSLAY102/C106 MF, 1st Set

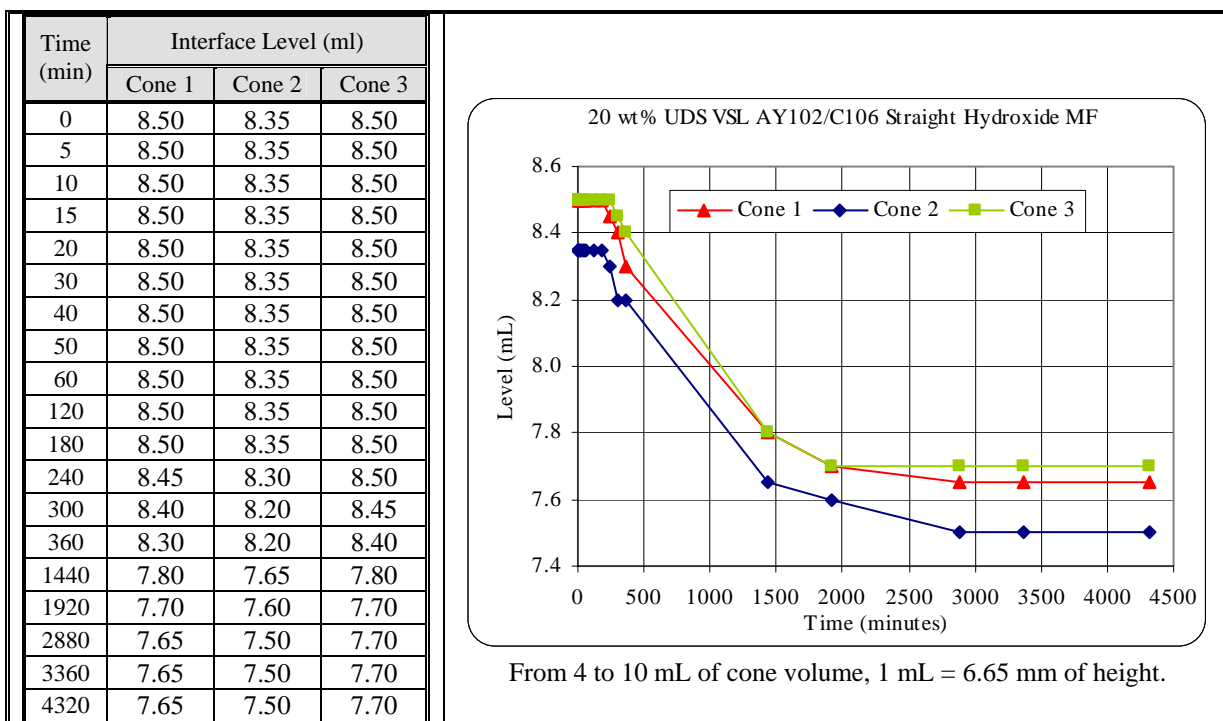


Table 2-17 Settling Data 20 wt% UDS AY102/C106 MF, 2nd Set

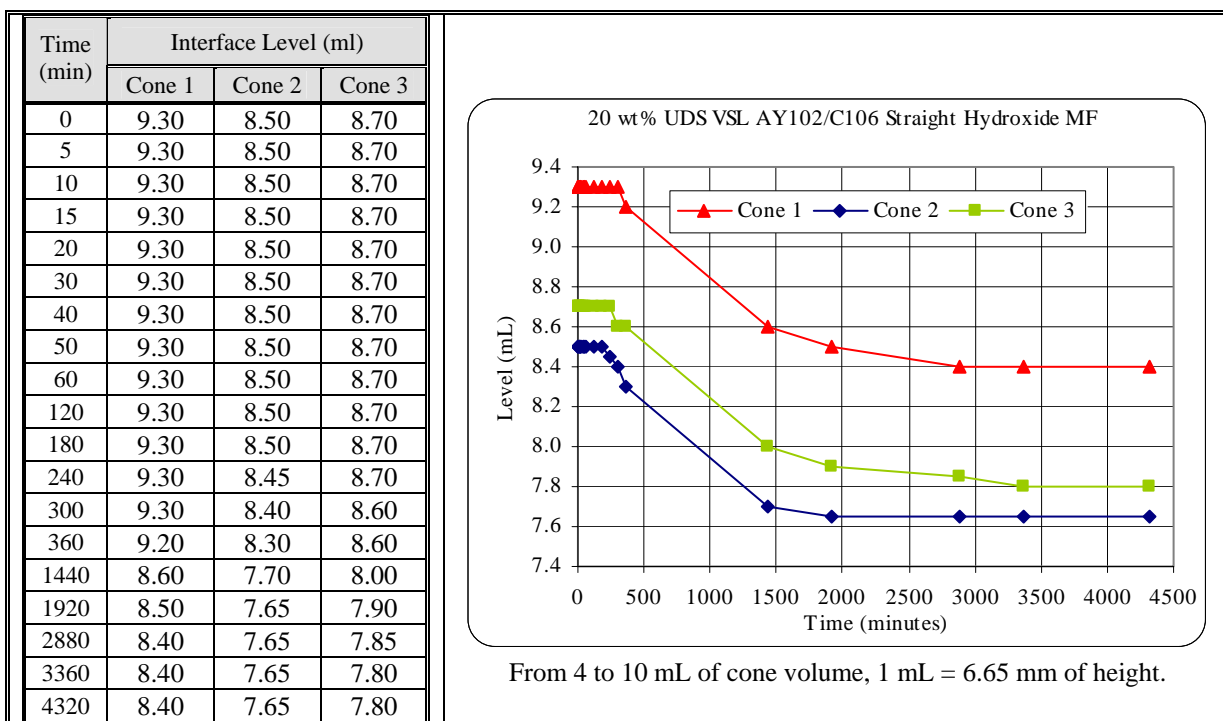


Table 2-18 Mean PSD Variables for 20 wt% UDS VSLAY102/C106 MF

20 wt% UDS VSL AY102/C106 Straight Hydroxide Melter Feed				
Set	Variable	1 st Run	2 nd Run	3 rd Run
1 st	Mean Number (μm)	0.472	0.493	0.410
	Mean Surface Area (μm)	2.010	2.089	2.138
	Mean Volume (μm)	13.33	14.84	27.13
	Reference Figure	Figure D - 4	Figure D - 5	Figure D - 6
2 nd	Mean Number (μm)	0.466	0.481	0.477
	Mean Surface Area (μm)	1.989	1.915	1.973
	Mean Volume (μm)	14.25	11.36	12.84
	Reference Figure	Figure D - 7	Figure D - 8	Figure D - 9

During the flow curve measurement of the 20 wt% UDS VSLAY102/C106 MF, Taylor vortices were observed. The Taylor vortices equation in reference 7 was plotted against a representative 20 wt% UDS VSLAY102/C106 MF flow curve as shown in Figure 2-3. Though the Taylor vortices equation was developed for a Newtonian fluid, it seems to be a good tool to determine when Taylor vortices occur in non-Newtonian fluids. Measured data below the Taylor vortices curve is neglected, limiting the Bingham Plastic fit to an upper limit of 250 sec^{-1} .

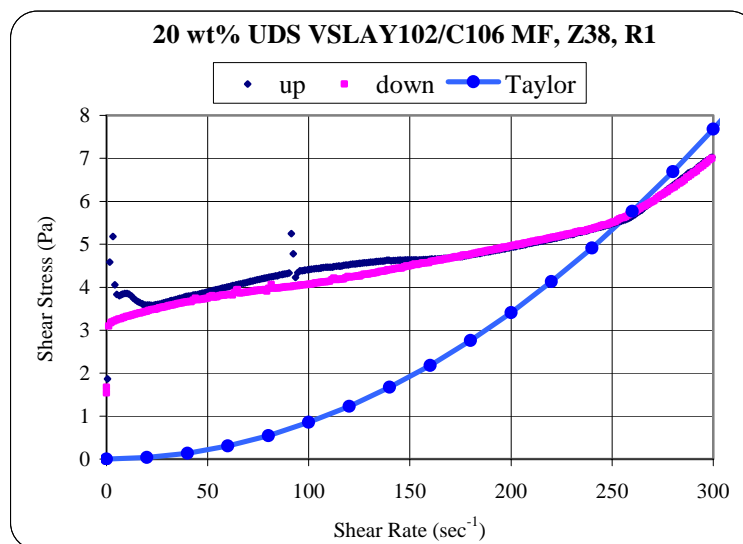


Figure 2-4 20 wt% UDS VSLAY102/C106 MF Flow Curve Impacted by Taylor Vortices

2.5 VSL AY102/C106 STRAIGHT HYDROXIDE RHEOLOGY ADJUSTED MELTER FEED RESULTS

The 20 wt% UDS VSL AY102/C106 straight hydroxide rheology adjusted melter feeds (VSLAY102/C105RA) averaged physical properties and pH results are provided in Table 1-6. The Bingham Plastic model results fitted to both the up and down curves for each flow curve are shown in Table 2-19. Of all the MFs characterized, this was the only MF that had thixotropic behavior. Appendix E contains each flow curve measurement. The settling data for the 20 wt % UDS VSLAY102/C106RA are shown in Table 2-20. The 20 wt % UDS VSLAY102/C106RA mean PSD

variables are provided in Table 2-21 and the volume and number PSD histograms are provided in Appendix D.

Table 2-19 VSLAY102/C106RA MF Bingham Plastic Data

VSL AY102/C106 Straight Hydroxide Rheology Adjusted Melter Feed									
Wt% UDS (Figure related to data)	Run	Up Curve				Down Curve			
		τ_{BP} (Pa)	μ_{BP} (mPa·s)	R^2	Fitted $\dot{\gamma}$ range (sec ⁻¹)	τ_{BP} (Pa)	μ_{BP} (mPa·s)	R^2	Fitted $\dot{\gamma}$ range (sec ⁻¹)
20 (Figure E - 11)	1	67.0	71.5	0.6090	0 – 300	54.3	106.7	0.9259	0 - 300
	2	66.5	76.7	0.6452		55.6	105.5	0.9646	
	3	65.6	74.3	0.7663		54.0	106.1	0.9267	
	Avg.	66.4	74.2	—	—	54.6	106.1	—	—
	Stdev.	0.7	2.6	—	—	0.8	0.6	—	—

Table 2-20 Settling Data 20 wt% UDS VSLAY102/C106RA MF

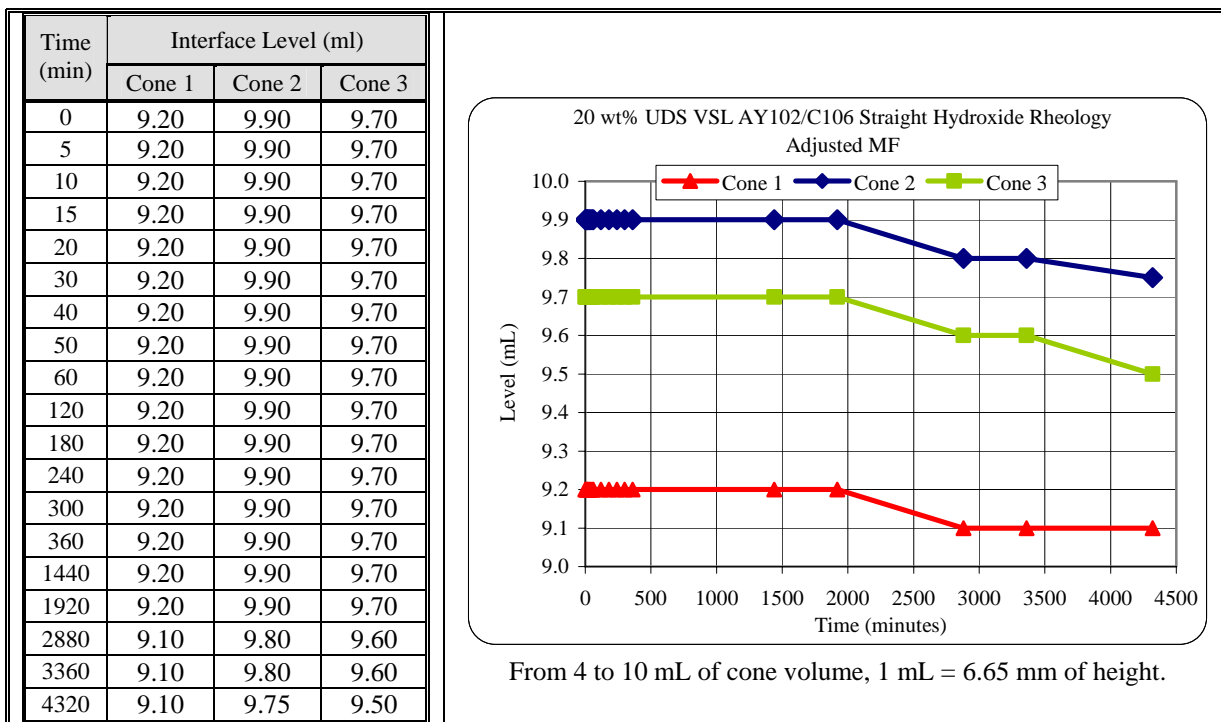


Table 2-21 Mean PSD Variables for 20 wt% UDS VSLAY102/C106RA MF

20 wt% UDS VSL AY102/C106 Straight Hydroxide Rheology Adjusted Melter Feed			
Variable	1 st Run	2 nd Run	3 rd Run
Mean Number (μm)	0.252	0.262	0.260
Mean Surface Area (μm)	1.128	1.117	1.104
Mean Volume (μm)	22.54	10.96	11.06
Reference Figure	Figure D - 10	Figure D - 11	Figure D - 12

2.6 SRNL AY102/C106 PRECIPITATED HYDROXIDE MELTER FEED RESULTS

The 20 wt% UDS SRNL AY102/C106 precipitated hydroxide melter feeds (SRNLAY102/C106) averaged physical properties and pH results are provided in Table 1-7. The Bingham Plastic model results fitted to both the up and down curves for each flow curve are shown in Table 2-22. Appendix E contains each flow curve measurement. The settling data for the 20 wt % UDS SRNLAY02/C106 are shown in Table 2-23 which indicates that no measurable settling occurred. The 20 wt % UDS SRNL102/C106 mean PSD variables are provided in Table 2-24 and the volume and number PSD histograms are provided Appendix D.

Table 2-22 SRNL AY102/C106M MF Bingham Plastic Data

SRNL AY102/C106 Melter Feed									
Wt% UDS (Figure related to data)	Run	Up Curve				Down Curve			
		τ_{BP} (Pa)	μ_{BP} (mPa·s)	R^2	$\dot{\gamma}$ range (sec ⁻¹)	τ_{BP} (Pa)	μ_{BP} (mPa·s)	R^2	$\dot{\gamma}$ range (sec ⁻¹)
20 (Figure E - 12)	1	11.8	26.5	0.9459	0 – 300	10.5	30.7	0.9607	0 - 300
	2	11.6	25.1	0.9425		10.4	29.6	0.9347	
	3	11.5	26.0	0.9474		10.5	29.6	0.9328	
	Avg.	11.6	25.9	—	—	10.4	29.9	—	—
	Stdev.	0.2	0.7	—	—	0.1	0.6	—	—

Table 2-23 Settling Data 20 wt% UDS SRNLAY102/C106 MF

20 wt% USD SRNL AY102/C106 MF			
Time (min)	Interface Level (ml)		
	Cone 1	Cone 2	Cone 3
0	7.30	7.70	7.75
5	7.30	7.70	7.75
10	7.30	7.70	7.75
15	7.30	7.70	7.75
20	7.30	7.70	7.75
30	7.30	7.70	7.75
40	7.30	7.70	7.75
50	7.30	7.70	7.75
60	7.30	7.70	7.75
120	7.30	7.70	7.75
180	7.30	7.70	7.75
240	7.30	7.70	7.75
300	7.30	7.70	7.75
360	7.30	7.70	7.75
1440	7.30	7.70	7.75
1920	7.30	7.70	7.75
2880	7.30	7.70	7.75
3360	7.30	7.70	7.75
4320	7.30	7.70	7.75

Table 2-24 Mean PSD Variables for 20 wt% UDS SRNLAY102/C106 MF

20 wt% UDS SRNL AY102/C106 Melter Feed			
Variable	1 st Run	2 nd Run	3 rd Run
Mean Number (μm)	0.530	0.541	0.535
Mean Surface Area (μm)	1.782	1.535	1.518
Mean Volume (μm)	28.07	12.83	12.94
Reference Figure	Figure D - 13	Figure D - 14	Figure D - 15

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APPENDIX A

**MEASUREMENT TECHNIQUES OTHER THAN RHEOLOGICAL
MEASUREMENTS**

The physical properties of the VSL HLW simulant melter feeds were measured in accordance with the project Guidelines [Ref. 7]. 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 (18 to 23°C) unless otherwise specified. Methods for obtaining the rheological properties are discussed separately in Appendix B.

This section describes how a melter feed sample was processed and the variables calculated, in accordance with the project Guidelines procedure [Ref. 7]. Typically three sub-samples were run for each waste stream at room temperature, unless otherwise noted. The slurry samples required for the physical properties measurement were obtained by physically shaking the contents in the bottle prior to sampling. After homogenization, a sample was extracted from the bottle using a 5-mL slurry pipette, which had its suction tip enlarged.

Between 7 to 10 mL of a sample was placed into a 10 mL volumetric graduated centrifuge cone and the cone shaken to re-homogenize the sample. The mass of the graduated centrifuge cone and cap were measured and recorded. The mass (M_B) and volume (if possible) of the sample were then recorded. The sample was then allowed to settle for at least 3 days. A detailed description of the settling test is provided at the end of appendix. After the settling tests were 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 estimating the total volume, which may be under or over estimated, based on how the meniscus was read. In this report, the bottom of the meniscus was read. Additionally, the materials hanging onto the sides of the tube would slightly decrease the level of settled slurry and the total volume.

The contents in the settled centrifuge tube were 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 (Equation A.1), vol% settled solids (Equation A.2) and vol% centrifuged solids (Equation A.3) were then calculated.

$$\rho_B = \frac{M_B}{V_B} \quad [A.1]$$

$$P_{VSS} = \frac{V_{SS}}{V_{SB}} \cdot 100\% \quad [A.2]$$

$$P_{VCS} = \frac{V_{CS}}{V_B} \cdot 100\% \quad [A.3]$$

The transparent 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 (Equation A.4), the density of centrifuged solids (Equation A.5), the wt% centrifuged solids (Equation A.6), the mass of settled solids (Equation A.7), density of settled solids (Equation A.8) and wt% of settled solids (Equation A.9) 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.
- 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.

$$\rho_S = \frac{M_S}{V_S} \quad [A.4]$$

$$\rho_{CS} = \frac{M_{CS}}{V_{CS}} \quad [A.5]$$

$$P_{VCS} = \frac{M_{CS}}{M_B} \cdot 100\% \quad [A.6]$$

$$M_{ss} = M_B - \rho_S \cdot (V_{SB} - V_{SS}) \quad [A.7]$$

$$\rho_{SS} = \frac{M_{SS}}{V_{SS}} \quad [A.8]$$

$$P_{SS} = \frac{M_{SS}}{M_B} \cdot 100\% \quad [A.9]$$

The graduated cylinder containing the supernatant and the centrifuged cone containing the centrifuged solids were then placed overnight into a forced convection 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 (Equation A.10), wt% total dried solids (Equation A.11), wt% oven dried solids (Equation A.12) and wt% UDS (Equation A.13) were then calculated. Variables that could potentially impact these results are:

- Volatiles (organics) lost during the oven drying process. Organics could be lost in both the supernatant and centrifuged solids and could 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.

$$P_{SSS} = \frac{M_{DCL}}{M_{VL}} \cdot 100\% \quad [A.10]$$

$$P_{MTS} = \left[\frac{P_{SSS}}{100\%} \cdot \frac{M_S}{M_B} + \frac{M_{DCS}}{M_B} \right] \cdot 100\% \quad [A.11]$$

$$P_{ODS} = \frac{M_{DCS}}{M_{CS}} \cdot 100\% \quad [A.12]$$

$$P_{MUS} = \left[1 - \frac{1 - \frac{M_{DCS}}{M_{CS}}}{1 - \frac{M_{DCL}}{M_{VL}}} \right] \cdot \frac{M_{CS}}{M_B} \cdot 100\% \quad [A.13]$$

Homogenized samples were also transferred to a 50-mL pre-fired (1100°C) high-purity alumina crucible. The mass of the sample transferred was recorded (M_{WCS}) and the crucible placed overnight into a forced convection oven at 90°C. The oven temperature was then increased to 105°C the next day and the sample was maintained in the oven until the dried weight stabilized (about 2 days). The oven-dried mass (M_{OSC}) was recorded and the wt% dried sample (Equation A.14) calculated. The sample was then placed into a 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 to room temperature. The weight of the oven fired sample (M_{FSC}) was recorded and the wt% total oxides (Equation A.15) calculated. The same issues as described earlier 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%.

$$P_{MDS} = \frac{M_{OSC}}{M_{WCS}} \cdot 100\% \quad [A.14]$$

$$P_{MOX} = \frac{M_{FSC}}{M_{WCS}} \cdot 100\% \quad [A.15]$$

The wt% water content can be calculated using the two different methods used to determine the wt% total solids, one from the cone (equation A.16) and the other from the crucible (equation A.17).

$$P_{WC} (Cone) = 100\% - P_{MTS} \quad [A.16]$$

$$P_{WC}(Crucible) = 100\% - P_{MDS} \quad [A.17]$$

The standard deviations of the measured and calculated values are reported. Calculations to estimate uncertainty in the mass and volume measurements were not included.

Settling Tests

Settling tests were performed as described in reference 7. First a sub-sample of the homogenized sample is transferred to the graduated centrifuging cone using a 5-mL slurry pipette (with the tip of the pipette cut for a larger opening). Approximately 7 to 10 mL of slurry is transferred to the cone. The cap is placed on the cone and the cone is shaken and/or rolled to re-homogenize the contents in the cone. The time at which re-homogenization occurs is recorded and then volume measurements were recorded of the solids interface layer, which is the layer between the clear supernatant and settling solids at the time intervals specified in reference 7. When the settling test is completed after 3 days of settling, the sample is processed for solids analysis as described above.

The 10 mL centrifuge cones have a linear region, between the 4 to 10 mL volume markings, where each mL of volume in this region is equivalent to 6.65 mm of vertical height. The diameter of the cone in this region is 13.8 mm. Wall and hinder settling effects most likely impact the solids interface layer, due to the small diameter of the centrifuge cone and the samples having high solids content.

pH Measurements

The pHs were measured using an IQ Scientific Instruments pH meter fitted with an IQ Scientific Instruments stainless steel electrode reference pH probe at ambient conditions. The functionality of the pH meter and probe were confirmed by measuring three different pH buffers of pH 4, 7, and 10 before 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.

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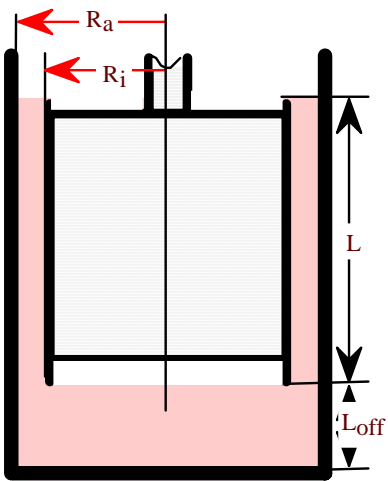
APPENDIX B

RHEOLOGICAL EQUIPMENT, METHOD, AND ANALYSIS

Flow Curve Measurements

Flow curve measurements were obtained using the Z41 or Z38 cylindrical rotor, Z43 cup and Haake RS600 rheometer. The rotor specifications are shown in Table B - 1. The bob and its cup are initially installed onto the rheometer. The zero reference point (point at which the rotor makes full contact with the bottom of the cup) is then determined by the rheometer. The rotor and cup are removed and the appropriate volume of sample is added to the cup. The cup and rotor are then reinstalled onto the rheometer. The rheometer drives the rotor into the predetermined bottom off-set position and excess sample is trimmed from the bob. A cooling/heating bath is used to control the temperature of the rotor/sample/cup at 25°C. The rheometer is programmed to control the rate at which the rotor spins 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 bob. The shear rate of the rotating rotor is calculated as the product of the measured speed and geometry (this M-factor assumes the fluid is Newtonian) of the rotor. The A-factor, M-factor, shear rate range and the linear ramp up time, hold time at maximum shear rate, and linear ramp down time are provided in Table B - 1. The linear ramp rates (or acceleration) is $\pm 150 \text{ sec}^{-1}$ per minute. The shear rate range and linear ramp rates differ than those specified in reference 7 and are those specified in the test exception (Ref. 1).

Table B - 1 Z41 Rotor Specification and Program Ramp Rates

Design of Rotor	Z38 and Z41 Rotor Specifications		
	Rotor	Z38	Z41
	Rotor radius (mm)	$R_i = 19.010$	$R_i = 20.710$
	Cup Radius (mm)	$R_a = 21.700$	$R_a = 21.700$
	Height of rotor (mm)	$L = 55$	$L = 55$
	Sample Volume (cm^3)	$V = 33$	$V = 14$
	Bottom off-set (mm)	$L_{\text{off}} = 8$	$L_{\text{off}} = 3$
	A factor ($\text{Pa}/(\text{N}\cdot\text{m})$)	8010	6750
	M factor ($\text{s}^{-1}/\text{rad s}^{-1}$)	8.60	22.40
	Shear rate measuring range (s^{-1})	0 – 300	0 – 300
	Ramp up time (min)	2	2
	Hold time (min)	0	0
	Ramp down time (min)	2	2

Prior to any flow curve measurement, the rotor and cup are inspected for visual damage that could potentially impact the rheological measurement. A National Institute of Standards and Technology (NIST) traceable Newtonian oil standard is then used to verify the operability of the rheometer at a measurement temperature of 25°C. The resulting flow curves are analyzed as a Newtonian fluid and the calculated viscosity is compared to that of the NIST traceable Newtonian oil standard. The rheometer is considered operable if the calculated viscosity is within $\pm 10\%$ of the NIST traceable Newtonian oil standard viscosity. The NIST traceable Newtonian oil standard was run each day that a flow curve or vane measurements was required. The measured viscosity was always within the $\pm 10\%$ of the standard.

Upon completion of a flow curve measurement, the bottom of the rotor is inspected to determine if the sample completely filled the void space at the bottom of the rotor. This void space is an air buffer, where the shear stress contribution from the air is negligible compared to the shear stress contribution from the cylindrical section of the rotor, which is in full contact with the fluid during the flow curve measurement. If the void space is completely filled with the sample, after the measurement is completed, then a fresh sample is loaded and the rotor inserted into the sample at a slower insertion rate. This process is repeated until there is evidence that the sample is not impacting the measurement due to the bottom void space being completely filled. If at the lowest insertion speed, the bottom is still full of sample after the measurement is complete, a large gap rotor (e.g. going from the Z41 to the Z38) is used and the same sequence as described in the above sentences is repeated for this larger gapped rotor. If this void space is completely filled, the resulting flow curve is more viscous.

If, during the measurement, the flow curve has multiple spiking (jamming of the particles between the cup and bob) when measuring with the Z41 rotor, the sample was then analyzed using the Z38 rotor to minimize the impact of particles during the flow curve measurement.

A minimum of three flow curves of each VSL melter feed were measured at 25°C. The VSL melter feed samples were homogenized, using care to avoid the introduction of air into the sample, and then immediately transferred to the cup. The rotor was inserted into the cup, excess sample trimmed, and the measurement performed. During these measurements, settling was not an issue, but jamming did occur in the Z41 rotor/cup, requiring the utilizing of the Z38 rotor/cup. Jamming during rheological measurements is discussed in the appropriate sections of this document.

The resulting flow curves are fitted to the Bingham Plastic rheological model (equation B.1). Unless otherwise noted in the tables that report the fitted data, the data is fitted to the complete shear rate range.

Bingham Plastic:

$$\tau = \tau_{BP} + \eta_{BP} \cdot \dot{\gamma} \quad (\text{B.1})$$

Where: τ_{BP} = Bingham Plastic yield stress (Pa)

η_{BP} = Bingham Plastic Viscosity (or consistency) (cP)

Settled Solids Shear Strength Measurement

The RS600 rheometer was used to perform the vane measurements. The RS600 was functionally verified using the Z41 rotor/cup and a NIST traceable Newtonian oil standard within the frequency as specified in reference 7.

Vanes have been used (Ref. 11 through 26) to measure the yield stress of non-Newtonian fluids as shown in Figure B - 1. 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, which is cylindrical. It has been shown that this is a good assumption (Ref. 12, 13, 16, and 17) for determining the yield stress of the fluid as the vane slowly rotates through the sample. The derived equation B.1 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 when meeting the criteria as shown in Figure B - 1. The length of immersed shaft will need to be considered if it length starts to impact the measured stress. The exclusion of the shear stress contribution of the immersed shaft length over-estimates the shear stress.

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

Where Γ = measured torque (N·m)
 D = diameter of vane (m)
 H = height of vane (m)
 A = geometric constant (Pa/(N·m))
 τ_{vane} = shear stress (Pa)

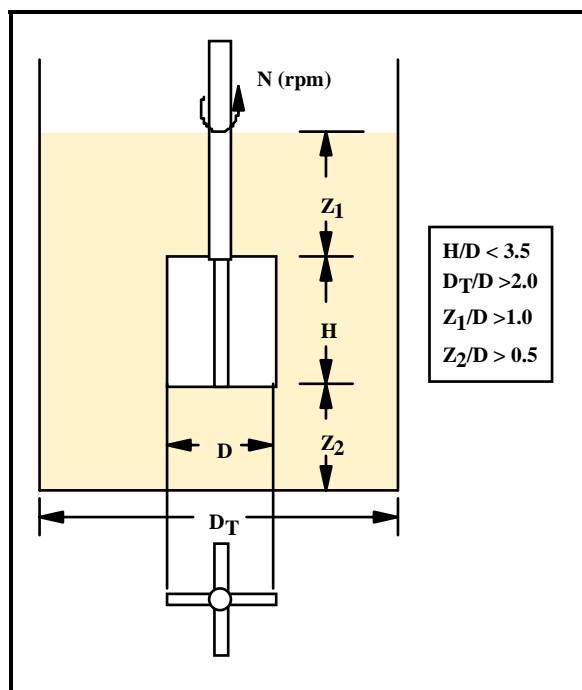


Figure B - 1 Vane Geometric Requirements

A typical stress versus time (or displacement) curve is shown in Figure B - 1. 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. 11) 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 (also known as the dynamic yield stress).

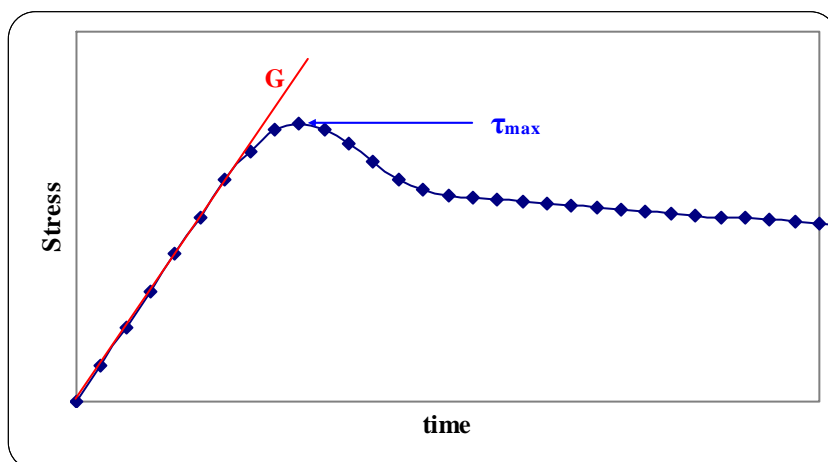


Figure B - 2 Typical Vane Torque versus Time/Displacement Curve

The vane dimensions used in this task were $D = 22 \text{ mm}$ and $H = 16 \text{ mm}$. The A factor for this vane was calculated (equation B.2) and used in the RS600 to calculate the shear stress from the measured torque, given the measured torque is in N-m.

$$A = \frac{A}{N \cdot m} = \frac{2}{\pi \cdot (0.022m)^3 \cdot \left(\left(\frac{16}{22} \right) + \frac{1}{3} \right)} \cdot \frac{N \cdot m}{N \cdot m} \cdot \frac{Pa}{\frac{N}{m^2}} = 56,370 \frac{Pa}{N \cdot m} \quad [B.2]$$

The M factor for this vane was set at $1.0 \text{ sec}^{-1}/(\text{rad} \cdot \text{s}^{-1})$. Going through the same exercise as that shown in equation B.2, for a rotational speed of 0.3 RPM, the controlled shear rate was 0.03 sec^{-1} . The rotational speed was also visually verified at approximately 0.3 revolutions per minute (RPM). This is the recommended rotational speed for the vane measurement.

The WTP terminology for yield stress obtained using the vane method is the settled solids shear strength.

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APPENDIX C
OXIDE CALCULATIONS FOR AZ102 MELTER FEEDS

Method for determining g-oxide/liter for both radioactive and VSL simulant AZ102 melter feeds.

From radioactive AZ102 melter feed (Ref. 9 and 10):

- a: Determine dry calcined value (grams oxide per grams dry solids at 105°C) for AZ102 sludge (use PNNL ICP-ES analyses Ref. 9).
- b: Determine dry calcined value for Sr/TRU precipitate (use PNNL ICP-ES analysis Ref. 10).
- c: Determine grams of GFC oxides from GFCs used (use PNNL GFC and assume 100% assay Ref. 9) for each batch (5 and 15 wt%).
- d: Given the batch makeup from PNNL (Ref. 9), calculate oxide/L for the 5 and 15 wt% total solids (TS) melter feeds.
- e: Verify batch dry calcined values for the 5 and 15 wt% 's are the same (this is to oxide method is consistent).
- f: For 10 wt% TS sludge, calculate the following
 - i: Use 10 wt% TS MF.
 - ii: Calculate density of sludge using 15 wt% sludge data using volume additivity (DI water and sludge)
 - iii: Maintain same mass ratio of total solids of Sr/TRU to total solids sludge
 - iv: Verify calcined values of sludge/Sr/TRU waste is same for all waste
 - v: Calculate GFCs used to blend 10 wt% TS MF, using GFC masses of the 5 wt% TS MF. Adjust GFCs addition to maintain same calcined value ratios as in the 5 and 15 wt% TS cases.
 - vi: Verify GFC calcined values.
 - vii: Verify overall calcined values for melter feeds are all the same.
 - viii: Calculate density of 10 wt% TS MF.
 - ix: Use density to determine volume of feed material and then g-oxide/liter.

An example calculation for the 15 wt% TS radioactive AZ102 MF is shown in Figure C - 1. The grams oxide per liter of MF for the 5, 10 and 15 wt% TS starting sludge are provided in Table C - 1.

All pages are from PNNL-13359			
Green = Input		Red = Calculated	
Calcine Factor		g oxide/g total dry solids	
AZ-102 simulant	0.8572	Calculated	
AN-107 Sr/TRU	0.7675	Calculated	
Blend for PNNLAZ102-15wt			
AZ-102	77.59	grams	Page 2.7
wt. % total solids	15.00%	wt.% T.S.	Page 2.7
total solids	11.64	grams	
Calcine solids	9.98	grams	
Density	1.14	g/mL	
Sr/TRU	5.367	grams	Page 2.7
wt. % total solids	25.00%	wt.% T.S.	Page 2.7
total solids	1.34	grams	
Calcine solids	1.03	grams	
Total mass of sludge	82.96	grams	Sludge Blended Data Results
Total solids	12.98	grams	
wt. % total solids	15.65%	wt.% T.S.	
Total calcine solids	11.01	grams	
Calculated calcine factor	0.848	g-oxide/g-total	
Glass former Chemicals	g	Oxide	g
LiOH-H ₂ O	4.33	Li ₂ O	1.54
H ₂ O	1.86		
Na ₂ B ₄ O ₇ ·10H ₂ O	3.01	B ₂ O ₃	1.10
10H ₂ O	1.42	Na ₂ O	0.49
Na ₂ SiO ₃ +5H ₂ O	8.96	Na ₂ O	2.62
10H ₂ O	3.80	SiO ₂	2.54
SiO ₂	11.1	SiO ₂	11.1
Sugar C ₁₂ H ₂₂ O ₁₁	0.374		
Total	20.69	Total	19.39
Total + water	27.77		
GFCs - oxides	19.39	grams	Melter Feed Blended Data Results
Sludge - oxides	11.01	grams	
Actual Masses Require			
GFCs	27.77	grams	
Sludge - Total	82.96	grams	
Sludge - total solids	12.98	grams	
Total Mass	110.73	grams	
Density	1.23	g/mL	Page 3.2
Wt. % Total Solids	30.41%	Wt. % total solids	
Calcine Factor Wet Basis	27.45%	g-oxide/g-feed	
Calcine Factor Dry Basis	0.9027	g-oxide/g-total solids	
Mass of oxide	30.4	grams	
Volume of Melter Feed	90.0	mL	

Figure C - 1 Sample Calculation (g-oxide/liter) for 15 wt% TS Starting Sludge Radioactive AZ102 MF

For the VSL AZ102 MF simulants:

From reference 3, determine the g-oxides/L of the 20 wt% UDS AZ102 simulant (use the AZ102 MF without rheology modifier as an example).

Obtain the following from reference 3:

- a: Mass of sludge.
- b: Mass of GFCs.
- c: Mass of glass produced (oxides).

Determine the following:

- d: Calculate Total Mass.
- e: Calculate wet calcined ratio (kg-oxide/kg total mass).
- f: Measure density of 20 wt% UDS AZ102 MF using WTP cone method.
- g: Calculate volume of 20 wt% UDS AZ102 MF.
- h: Calculate g-oxide/liter of 20 wt% UDS AZ102 MF.

Calculate volume of DI water required to reduce g-oxides/L to targeted values calculated for actual melter feed. Use radioactive AZ102 MF wt% TS starting sludge as a reference.

Perform the following:

- i: Using 100 grams as a 20 wt% UDS MF as a basis.
- j: Calculate mass of oxides.
- k: Determine mass of DI water addition (Goal seek cell with step q below)..
- l: Calculate total mass of targeted MF.
- m: Calculate density (using volume additivity) of targeted MF.

$$\frac{1}{\rho_{newMF}} = \left(\frac{\frac{m_{MF}}{\rho_{MF}} + \frac{m_{DIwater}}{\rho_{water}}}{m_{MF} + m_{DIwater}} \right)$$

- n: Calculate volume of targeted MF.
- o: Calculate g-oxide/L of targeted MF.
- p: Calculate mass of UDS in the targeted AZ102 sludge used.

$$= m_{MF} (step i) \times 0.20 \frac{g-oxide}{g-sludge} \times \frac{M_{sludge} (step a)}{M_{sludge} (step a) + M_{GFCs} (step b)}$$

- q: Calculate wt% UDS of targeted AZ102 sludge. The dilution is only applicable to the sludge.

$$= \frac{m_{UDS} (step p)}{m_{MF} (step i) \times \frac{M_{sludge} (step a)}{M_{sludge} (step a) + M_{GFCs} (step b)} + m_{DI} (step k)} \times 100\%$$

The densities of the as-received VSL AZ102 Melter feeds were measured at 1.462 and 1.461 g/mL. An average of 1.462 was used for either the straight hydroxide or straight hydroxide rheology modified melter feeds for determine the dilution required for the targeted g-oxides/L radioactive AZ102 MF and the results are shown in Table C - 1.

Table C - 1 Dilution of VSL AZ102 MF to Target g-oxides/L

VSL Data From Table 3.3 (VSL-04T4800-1, Rev.0)				
AZ102 HLW Simulant	1594.03	Kg		
GFCs	1013.96	Kg		
Calculate Total Mass	2607.99	Kg		
Glass Yield	1000	Kg		
Calculate wet calcined	0.3834	kg oxide/kg feed		
Calculate wt% UDS	20.00%	wt% UDS		
Measured Density	1.461	g/mL		
Calculated Volume of Melter Feed	1785	L		
Calculate g-oxides/L melter feed	560	g-oxide/L MF		
Assume 100 grams of starting MF				
Radioactive AZ102 sludge wt% TS	15	10	5	wt% TS
Mass of MF	100	100	100	Grams
Mass of oxides	38.3	38.3	38.3	Grams
Mass of DI addition	45.0	94.1	238.3	Grams
Total Mass	145.0	194.1	338.3	Grams
Calculate Density Using Additivity	1.278	1.194	1.103	g/mL
Calculate Volume	113.5	162.5	306.8	mL
Calculate g - oxide/L	338	236	125	g-oxide/L MF
Calculate mass of UDS in AZ102 sludge	12.22	12.22	12.22	Grams
Calculate wt% UDS of AZ102 sludge	11.5%	7.9%	4.1%	wt% UDS

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APPENDIX D

PARTICLE SIZE DISTRIBUTION METHOD AND DATA

A Microtrac S3000 particle size analyzer was used to measure the particle size distribution (PSD) of the sludge sample. The Microtrac S3000 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 S3000 measuring range is 0.026 to 1408 μm . Sonication (ultrasonic energy that breaks up agglomerations) was used (25 watts for 60 seconds). The Microtrac S3000 stores the recorded data into volume bins, where a bin is defined by an upper and lower diameter and the average bin size is reported. This volume data is then normalized to 100% after the measurement is complete. For the Microtrac S3000, there are 64 bins covering the measuring range. The number distribution is determined from equation (2.1). The Microtrac XS3000 software then calculates the mean number, mean surface area, mean volume, number distribution and volume distribution data for the standards, pretreated sludge and melter feed. The mean volume, mean surface area and mean number are determined using equation (2.2).

$$\left[\begin{array}{l} n_i = \frac{6 \cdot V_i}{\pi \cdot d_i^3} \\ N_i = \frac{n_i}{\sum n_i} \times 100\% \end{array} \right] \quad (2.1)$$

$$\left[\begin{array}{l} mv(\text{mean volume}) = \frac{\sum V_i d_i}{\sum V_i} \\ ma(\text{mean surface area}) = \frac{\sum V_i}{\sum (V_i / d_i)} \\ mn(\text{mean number}) = \frac{\sum (V_i / d_i^2)}{\sum (V_i / d_i^3)} \end{array} \right] \quad (2.2)$$

where: V_i = volume % in the i^{th} bin

d_i = average bin diameter

n_i = number of particles in the i^{th} bin, given volume %

N_i = number % in the i^{th} bin (data normalized)

The S3000 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.

Prior to performing the PSD measurement, the samples were diluted using DI water. Once the operability of the Microtrac S3000 was confirmed, the melter feed measurements were performed. The results, both number and volume PSD basis are provided in this Appendix.

The following is a brief description of how to interpret the information from both the volume and number histograms provided in this Appendix. Description refers to Figure D - 1 volume distribution.

The “% PASS” (blue colored) is the percentage of particles smaller or equal to the corresponding particle size. The “% CHAN” (reddish color) is the percentage of particles between the particle size and the closest smaller particle size in the table. In the example above, 48.06% of the particles are below 6.541 microns and 1.75% of the particles are between 5.500 and 6.541 microns. The table labeled Dia, Vol, and Width are the distribution for one or more peaks/modes within the data. In this case, there is only one mode (e.g. one can have bi-modal, tri-modal, etc distribution). “mv” is the mean diameter of the volume distribution. “mn” is the mean diameter of the number distribution. “ma” is the mean diameter of the area distribution. “cs” is the calculated specific surface area (m^2/cc) of the sample based on smooth, solid spherical particles. This number can in no way be related to a measured surface area, for example BET by adsorption. “sd” is the standard deviation for the distribution (not the variation about the mean). Specifically, “sd” is the difference between the diameters of the 84% and 16% particles that have passed divided by 2. “sd” does not provide an indication of the statistical error about the mean of multiple measurements. The table on the bottom provides the type of graph provided, operating parameters of the particle size analyzer and the number of sample runs.

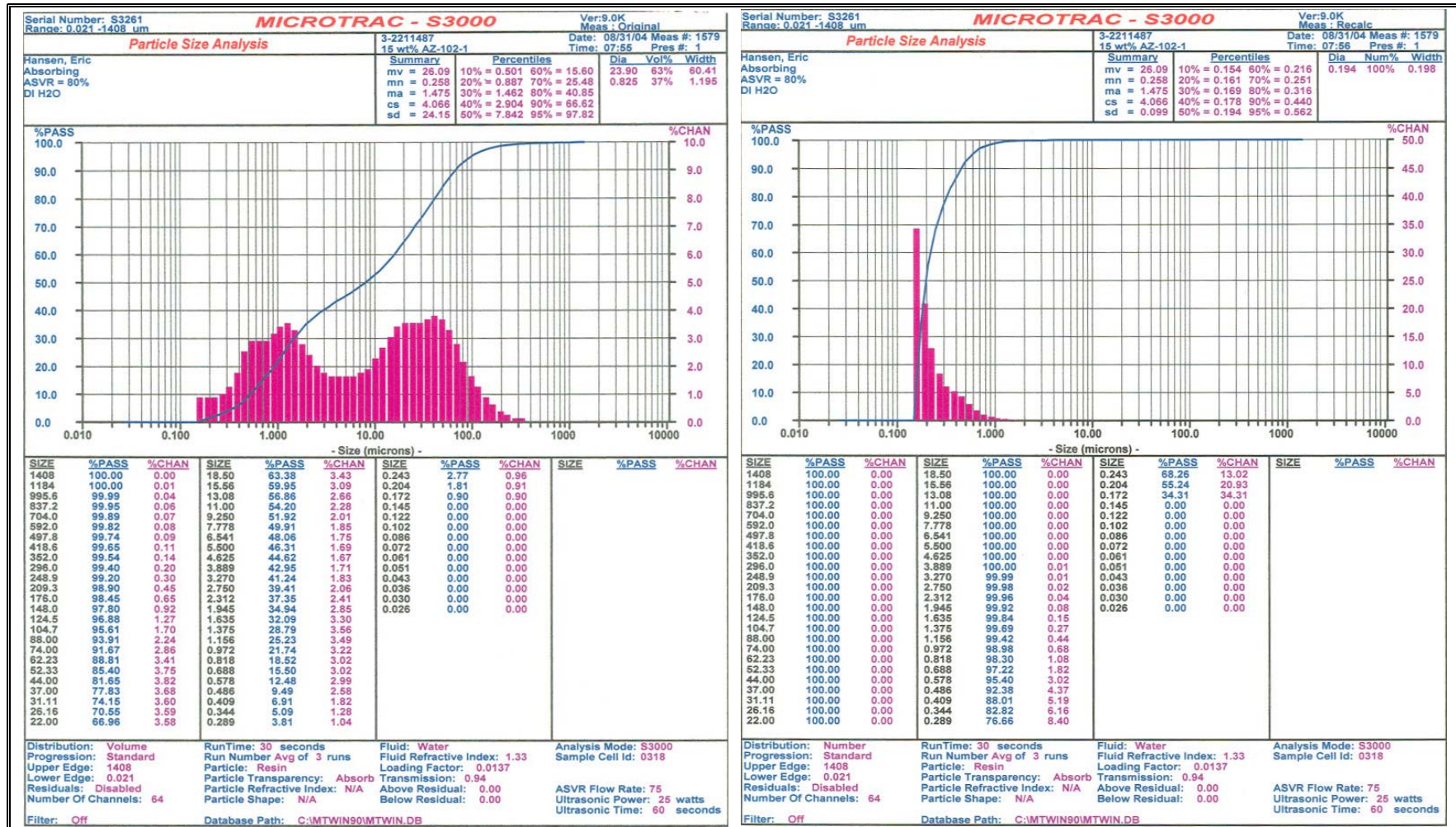


Figure D - 1 Volume and Number PSDs for 11.5 wt% UDS VSLAZ102 MF – 1st Run

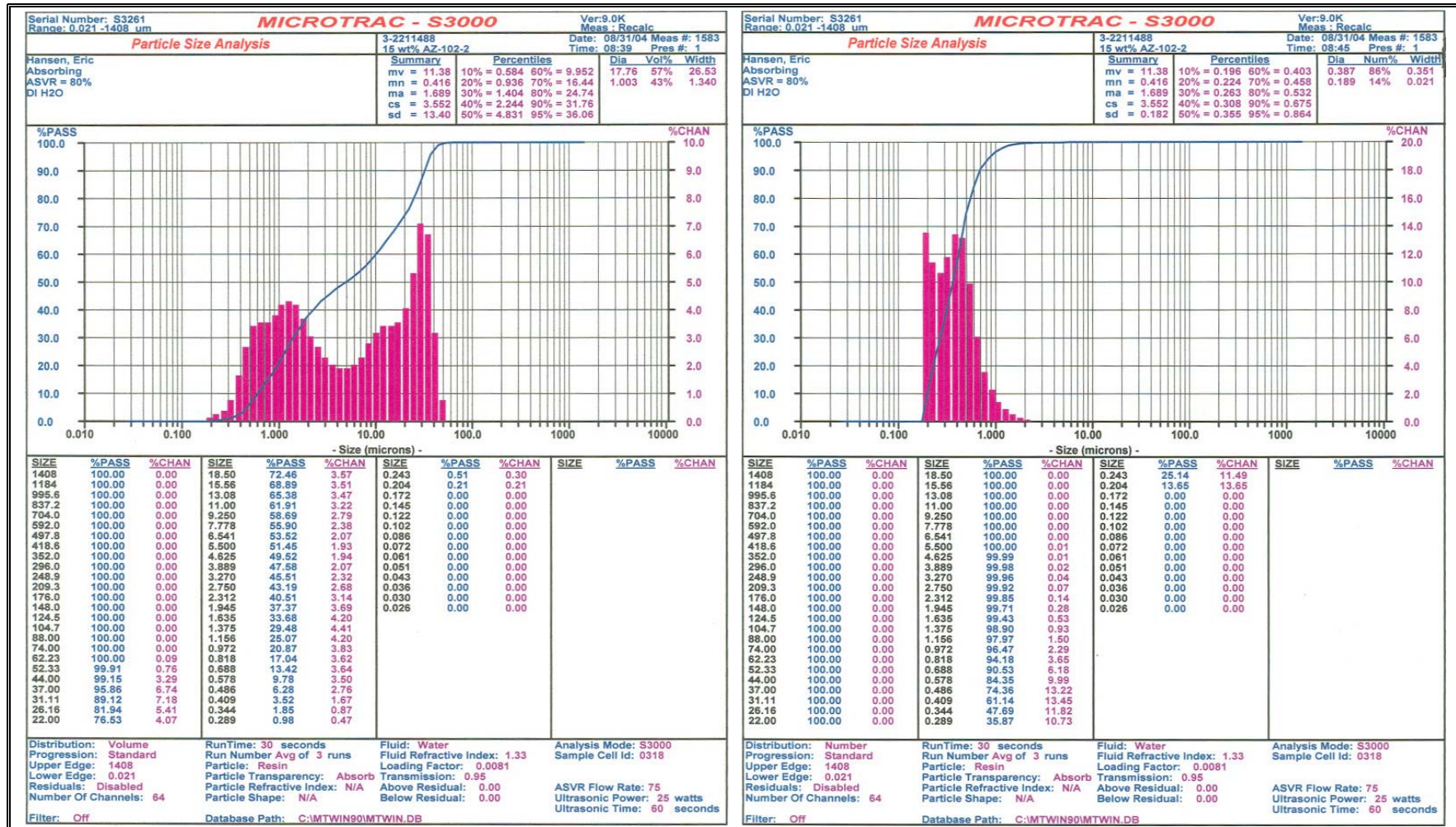


Figure D - 2 Volume and Number PSDs for 11.5 wt% UDS VSLAZI02 MF – 2nd Run

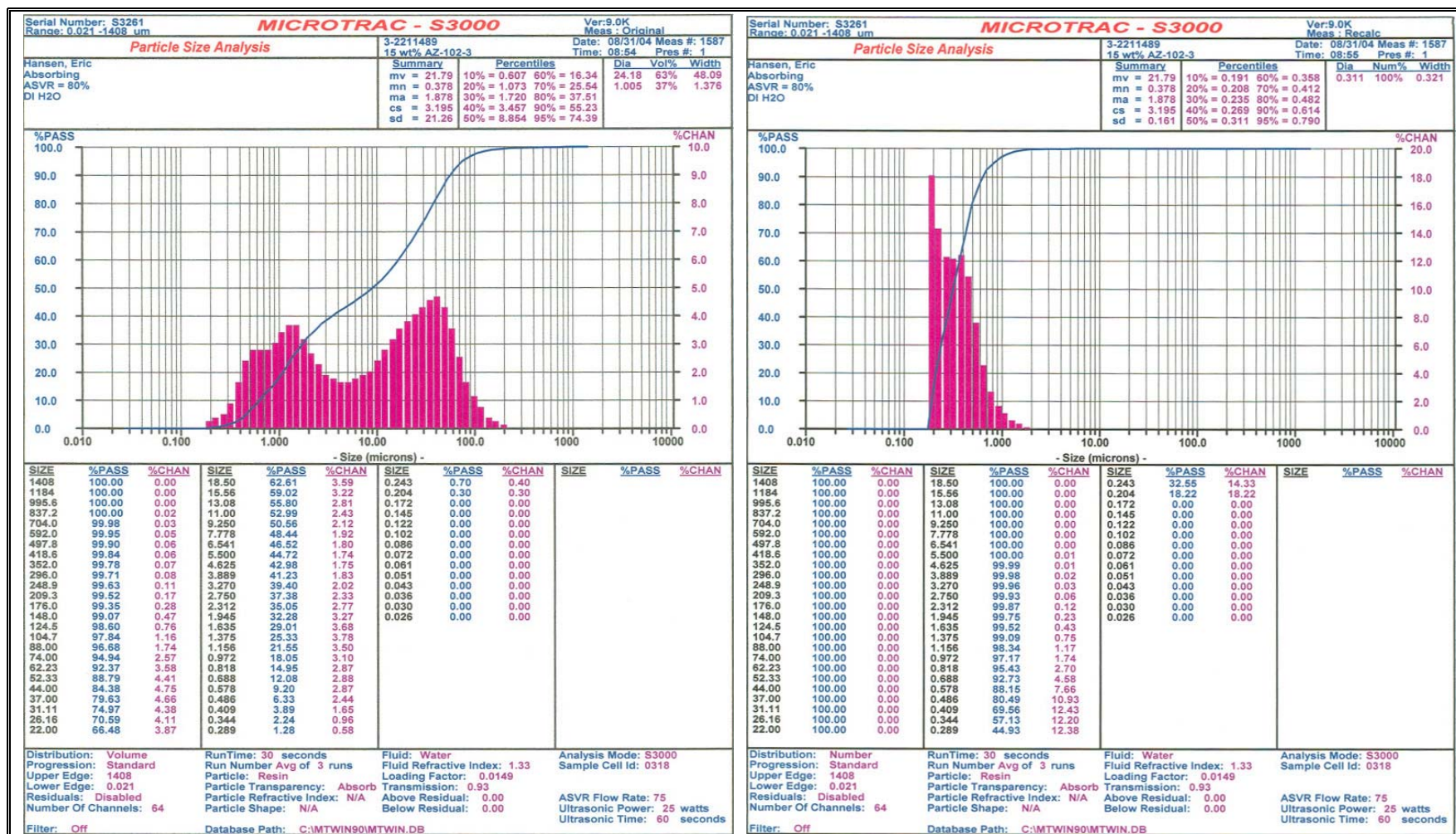


Figure D - 3 Volume and Number PSDs for 11.5 wt% UDS VSLAZ102 MF – 3rd Run

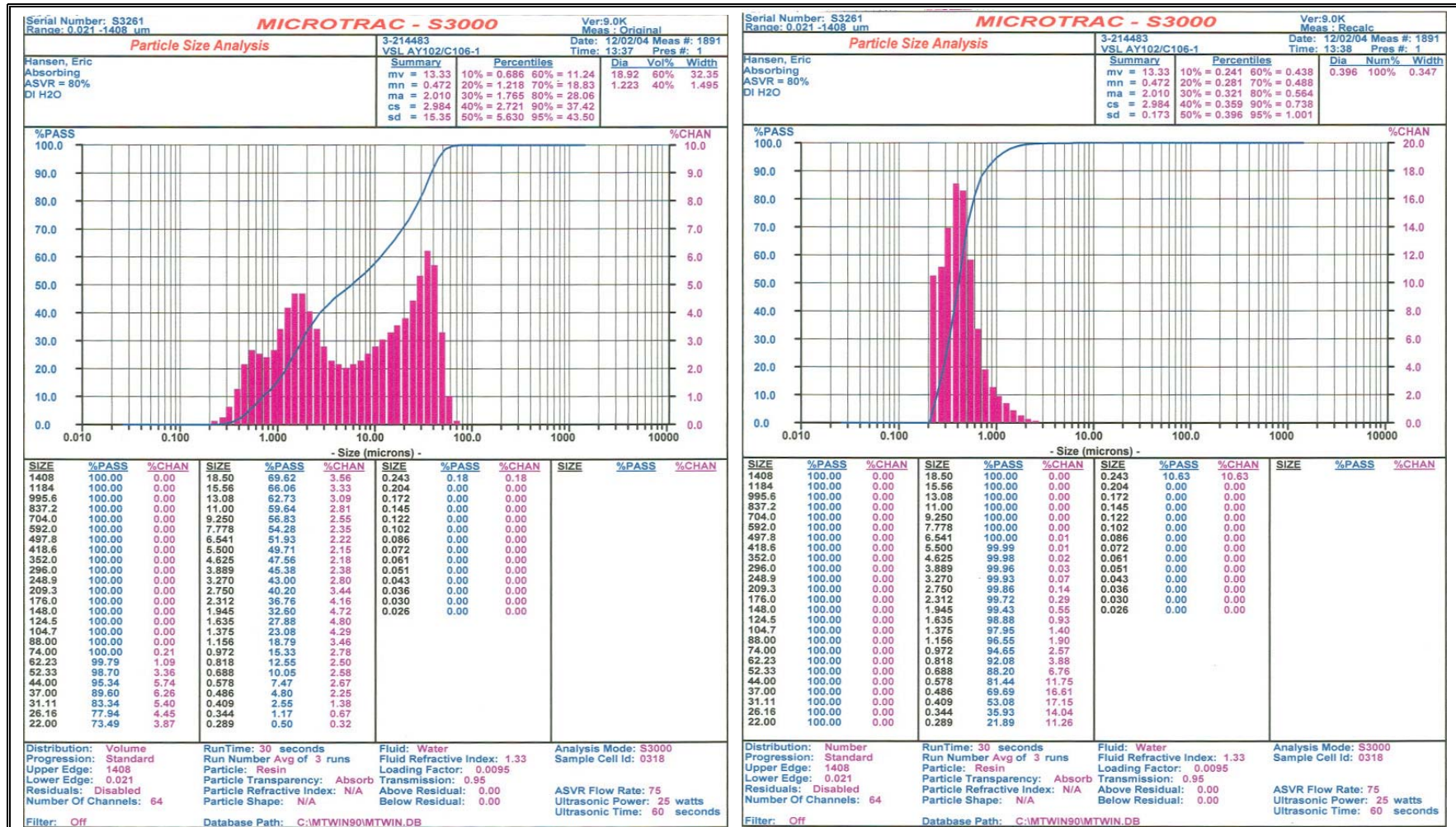


Figure D - 4 Volume and Number PSDs for 20 wt% UDS VSL AY102/106 MF – 1st Set 1st Run

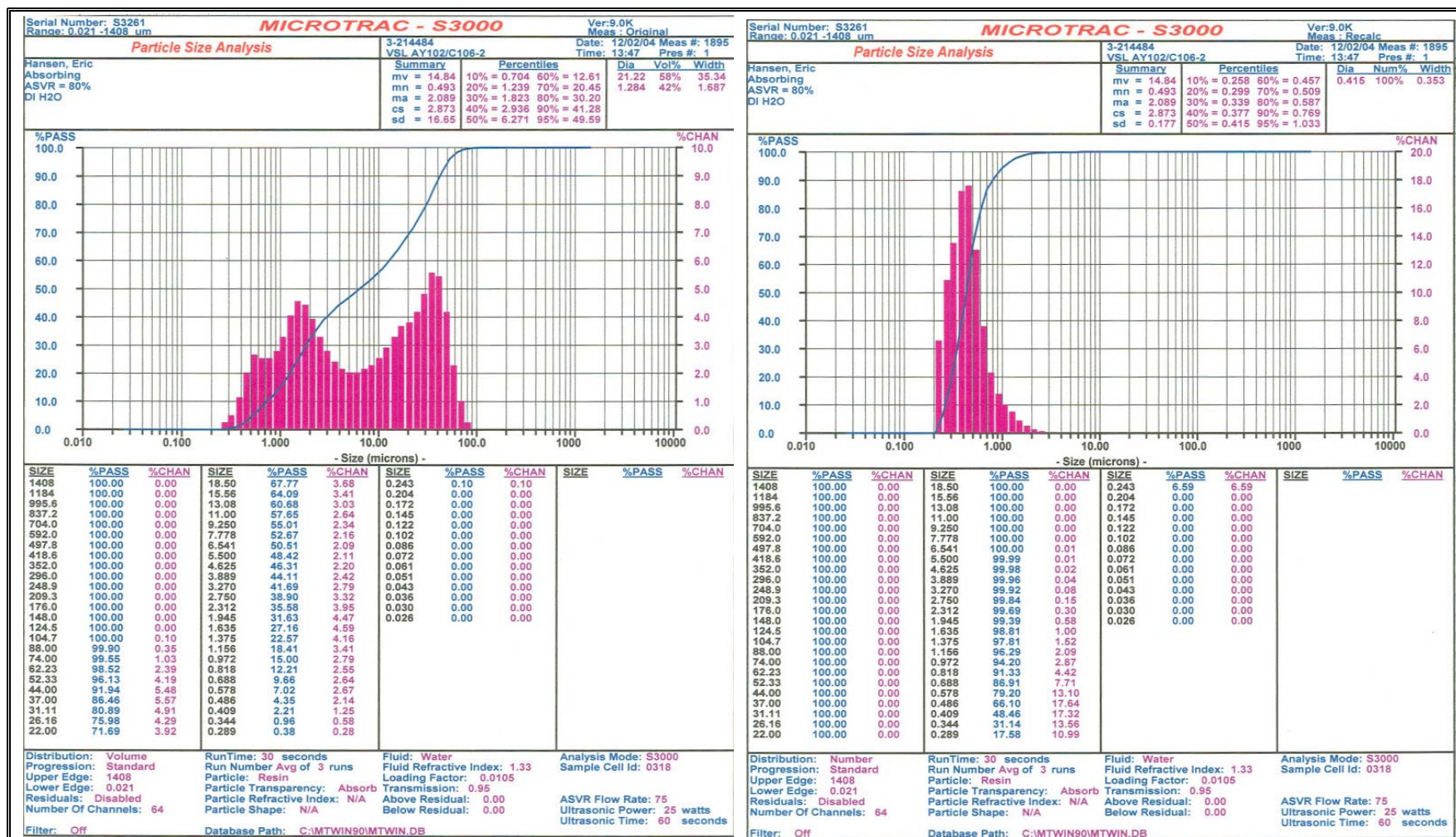


Figure D - 5 Volume and Number PSDs for 20 wt% UDS VSLAY102/106 MF – 1st Set 2nd Run

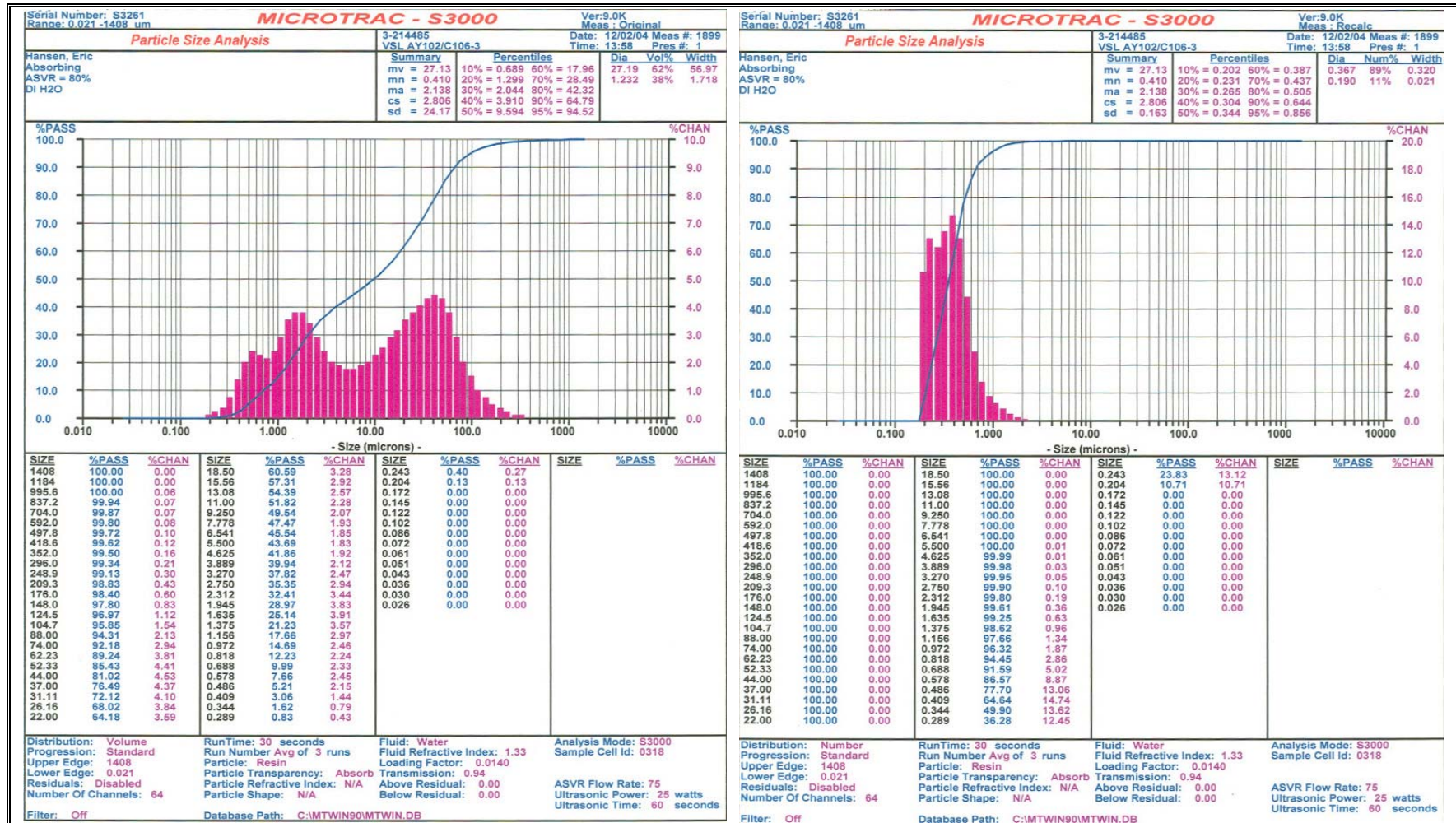


Figure D - 6 Volume and Number PSDs for 20 wt% UDS VSLAY102/106 MF – 1st Set 3rd Run

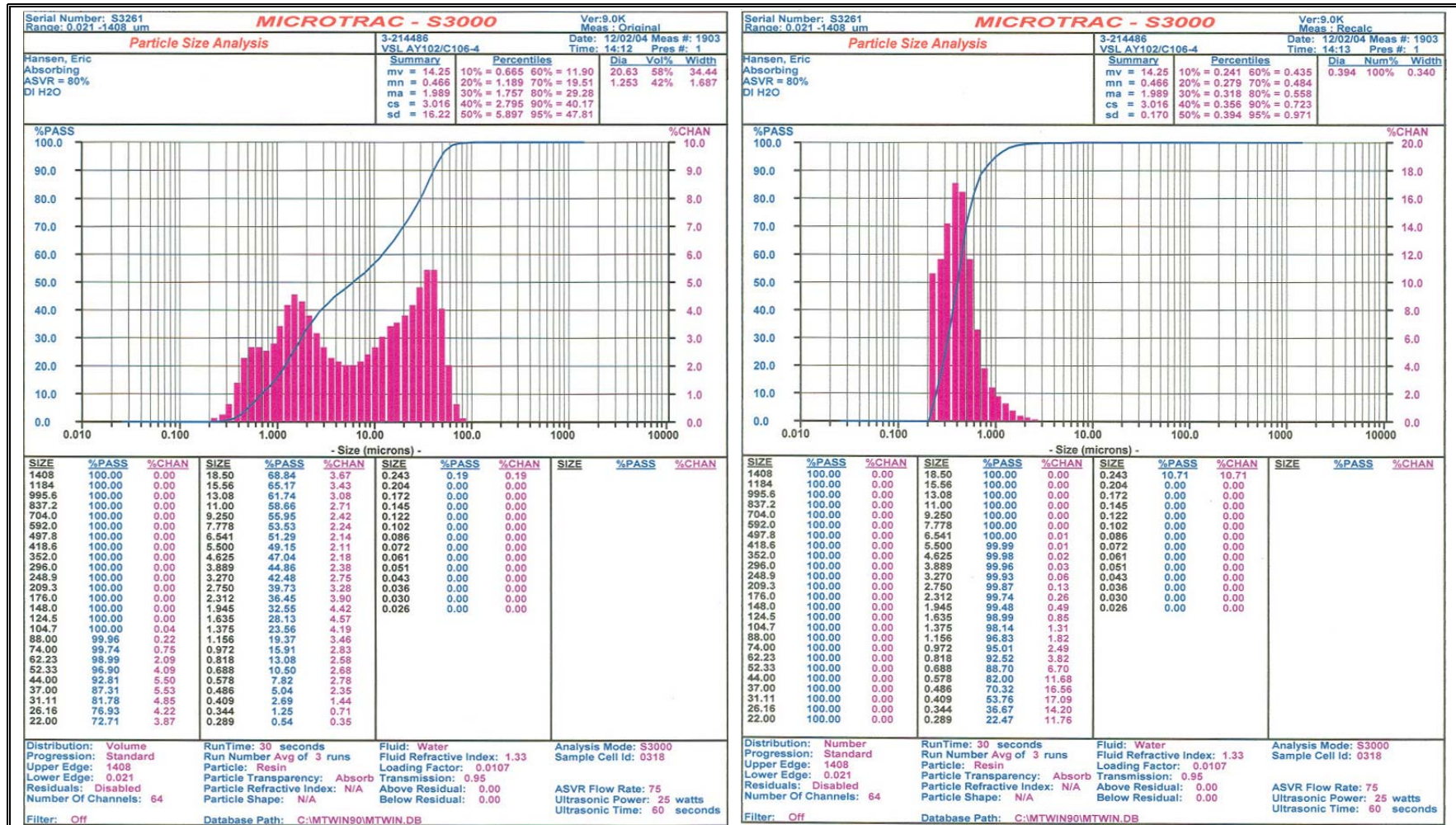


Figure D - 7 Volume and Number PSDs for 20 wt% UDS VSLAY102/106 MF – 2nd Set 1st Run

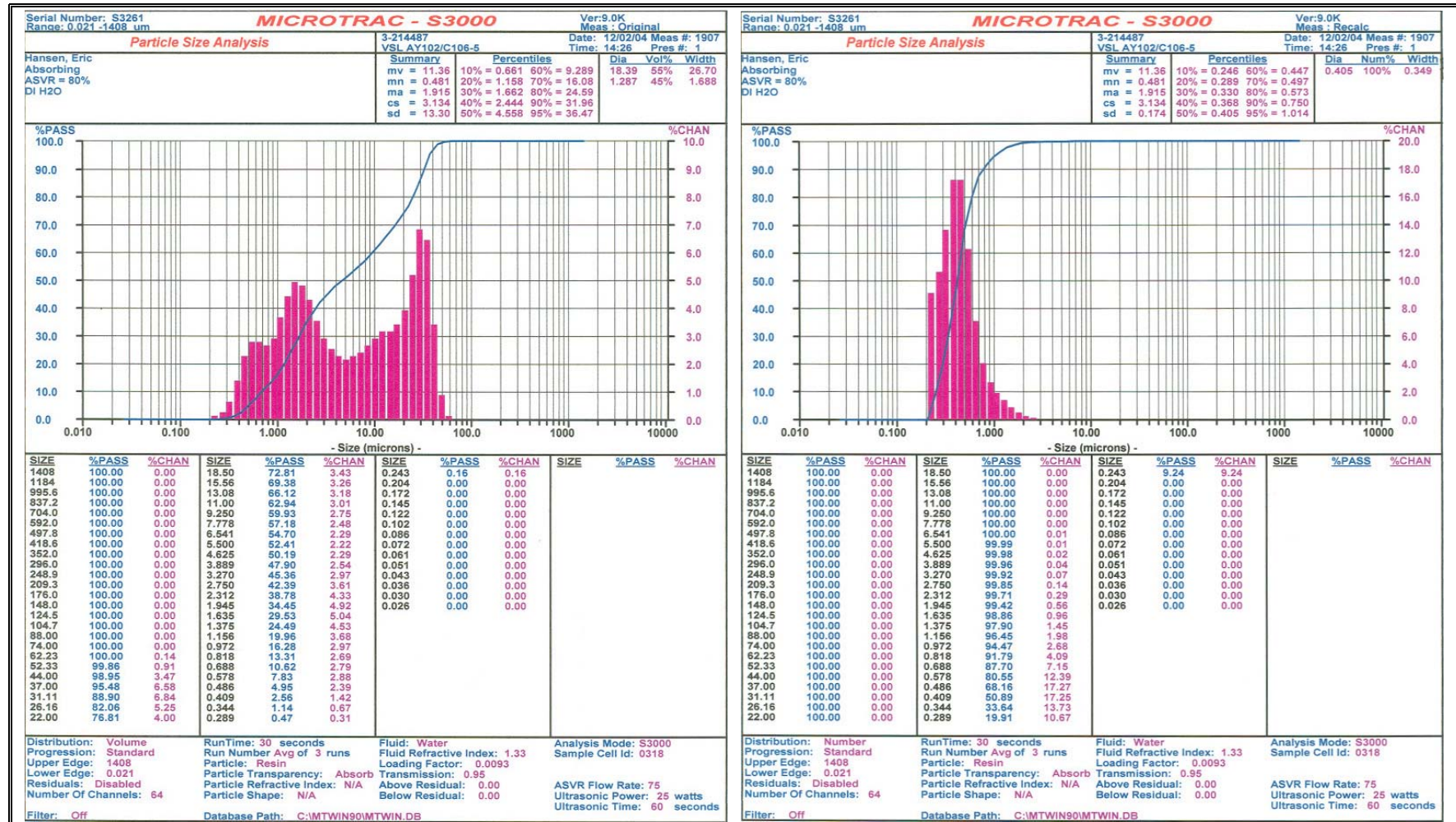


Figure D - 8 Volume and Number PSDs for 20 wt% UDS VSLAY102/106 MF – 2nd Set 2nd Run

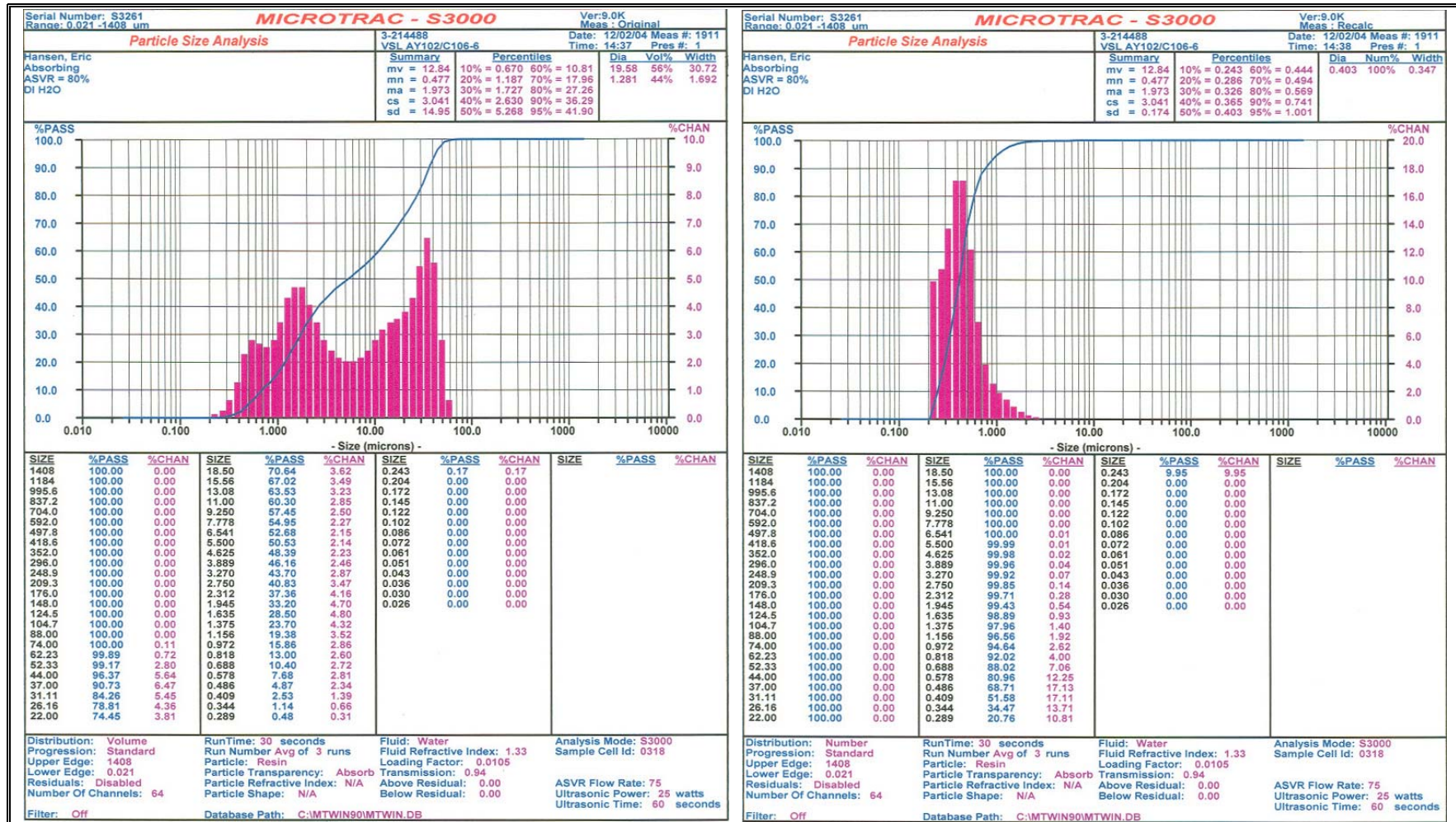


Figure D - 9 Volume and Number PSDs for 20 wt% UDS VSLAY102/106 MF – 2nd Set 3rd Run

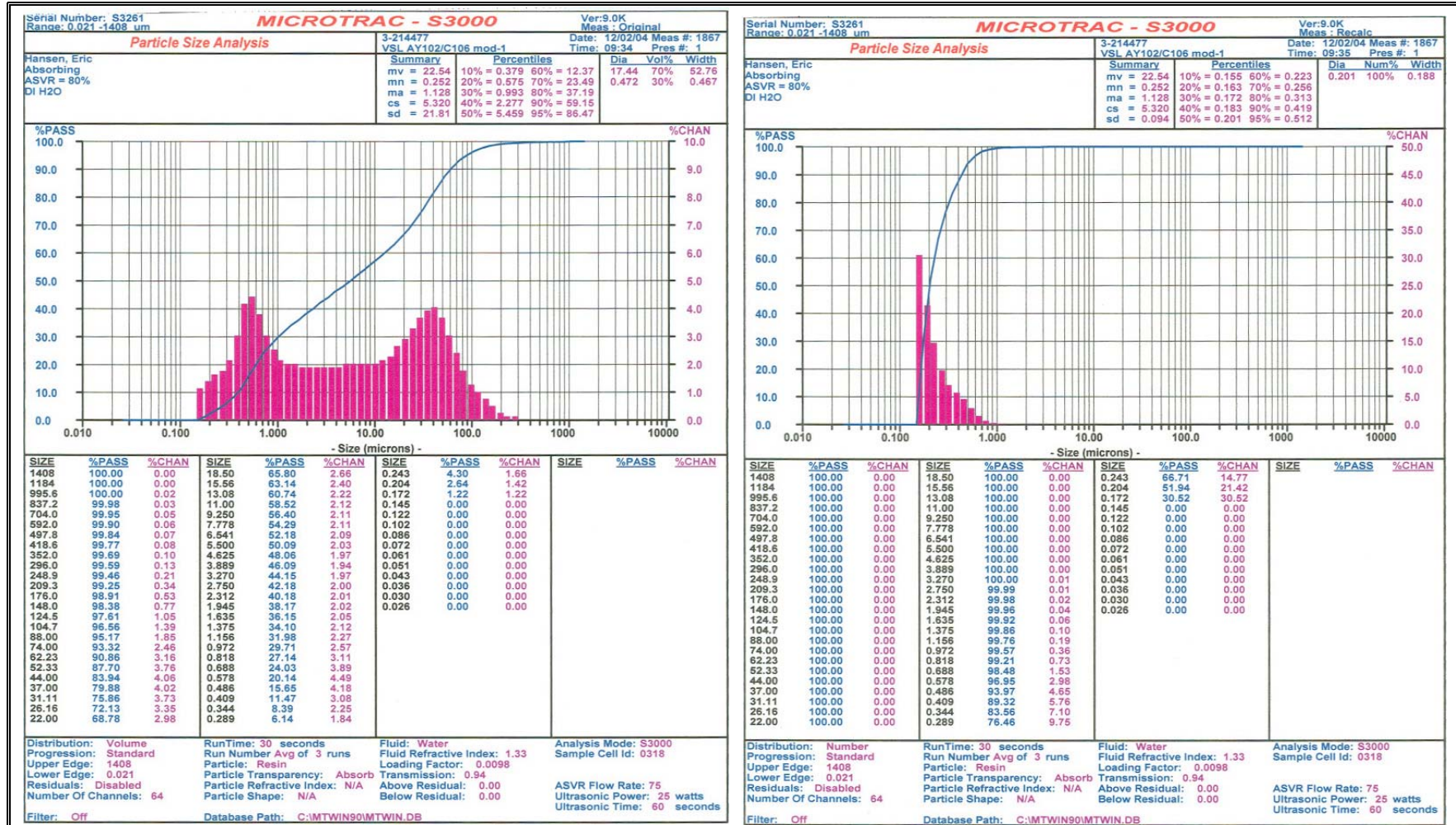


Figure D - 10 Volume and Number PSDs for 20 wt% UDS VSLAY102/106RA MF – 1st Run

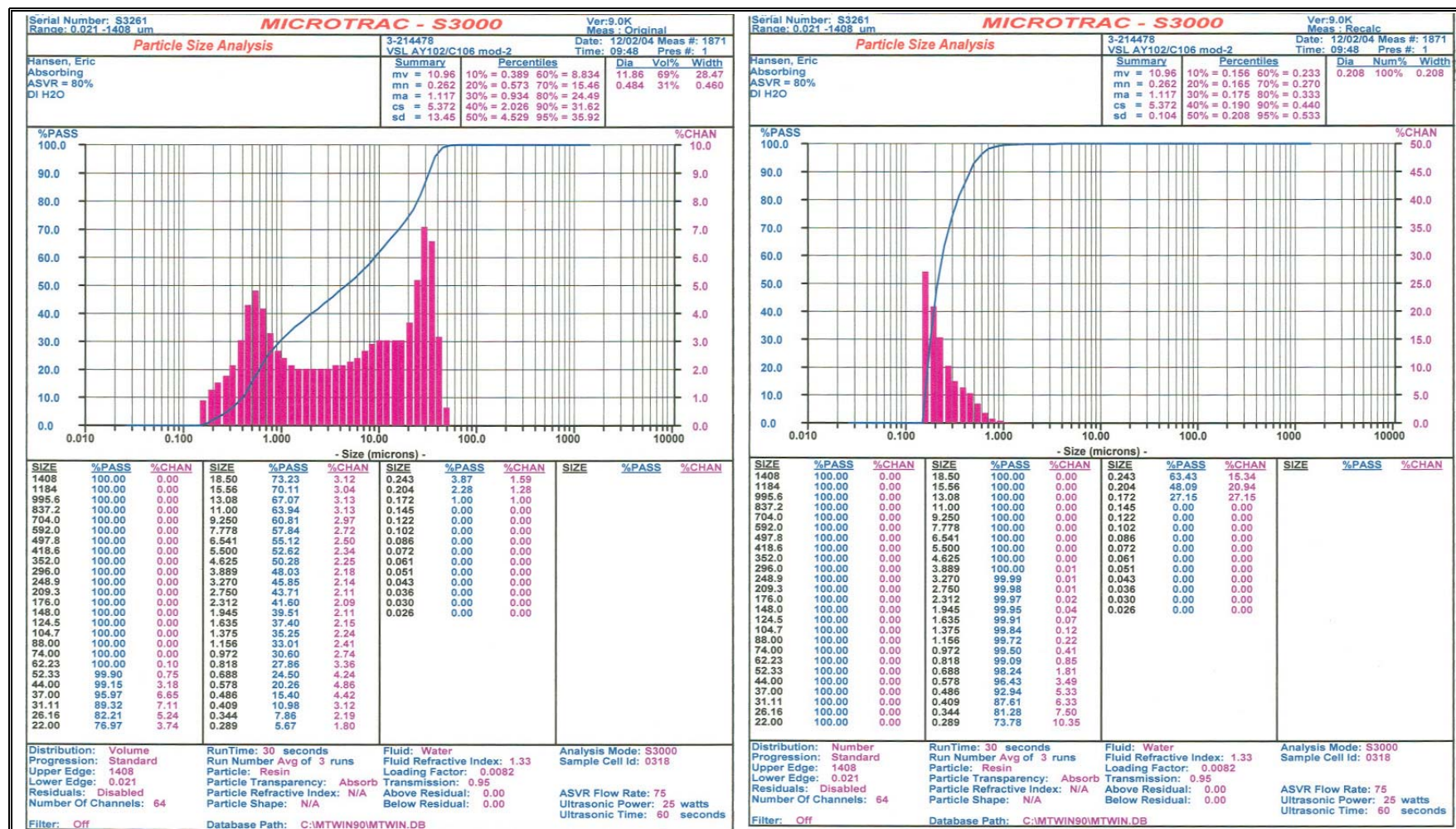


Figure D - 11 Volume and Number PSDs for 20 wt% UDS VSLAY102/106RA MF – 2nd Run

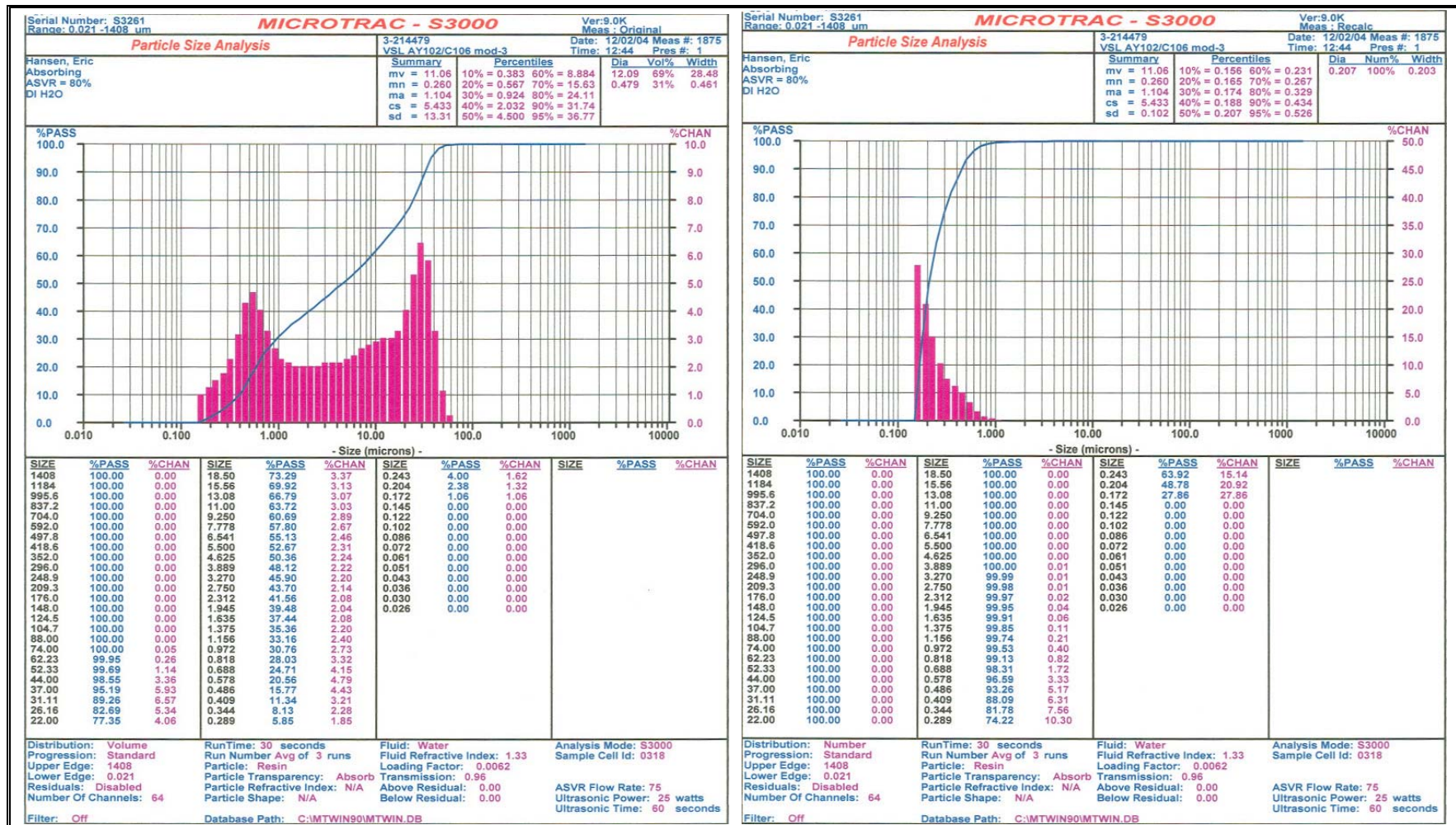


Figure D - 12 Volume and Number PSDs for 20 wt% UDS VSLAY102/106RA MF – 3rd Run

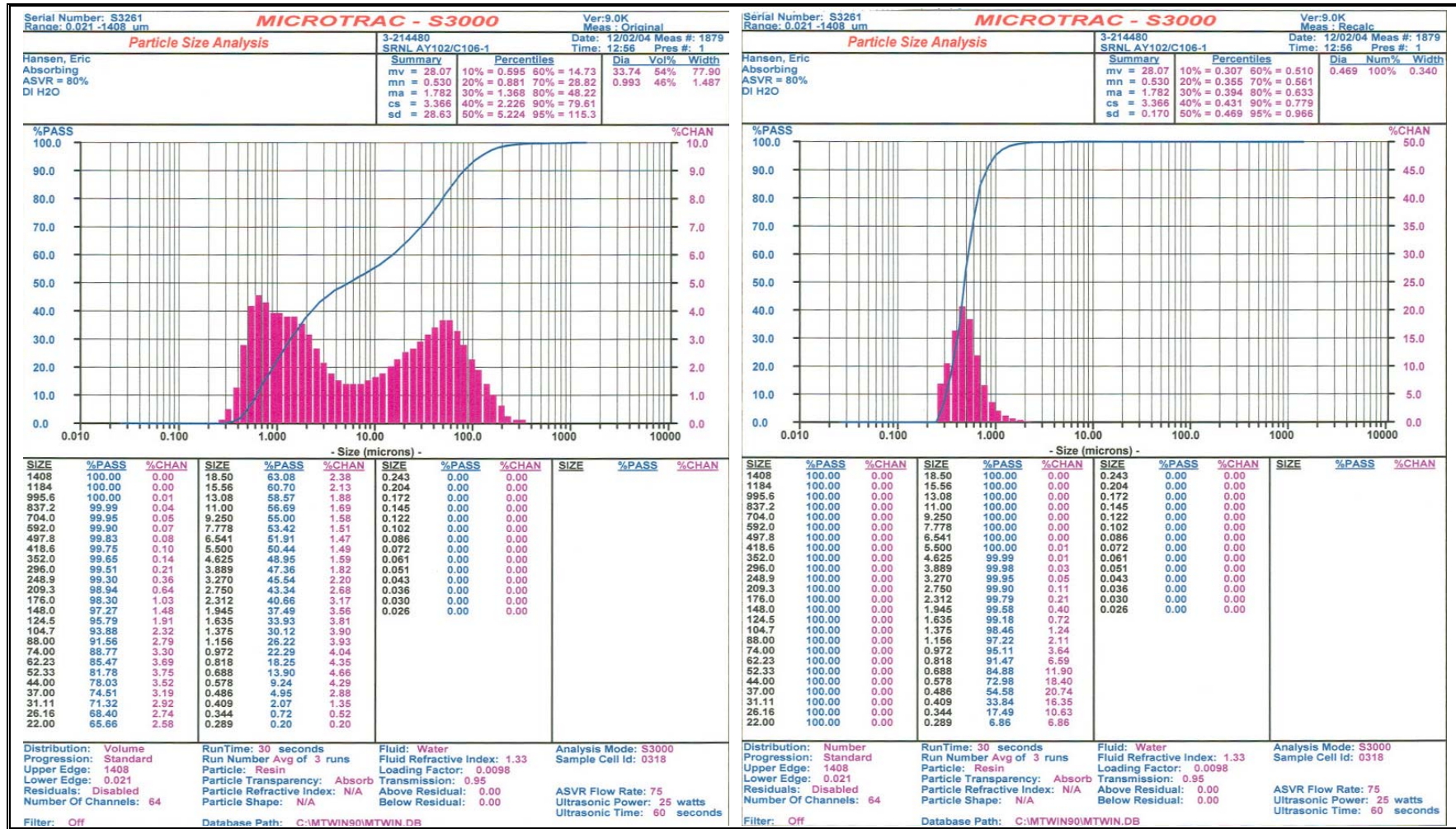


Figure D - 13 Volume and Number PSDs Distribution for 20 wt% UDS SRNLAY102/106 MF – 1st Run

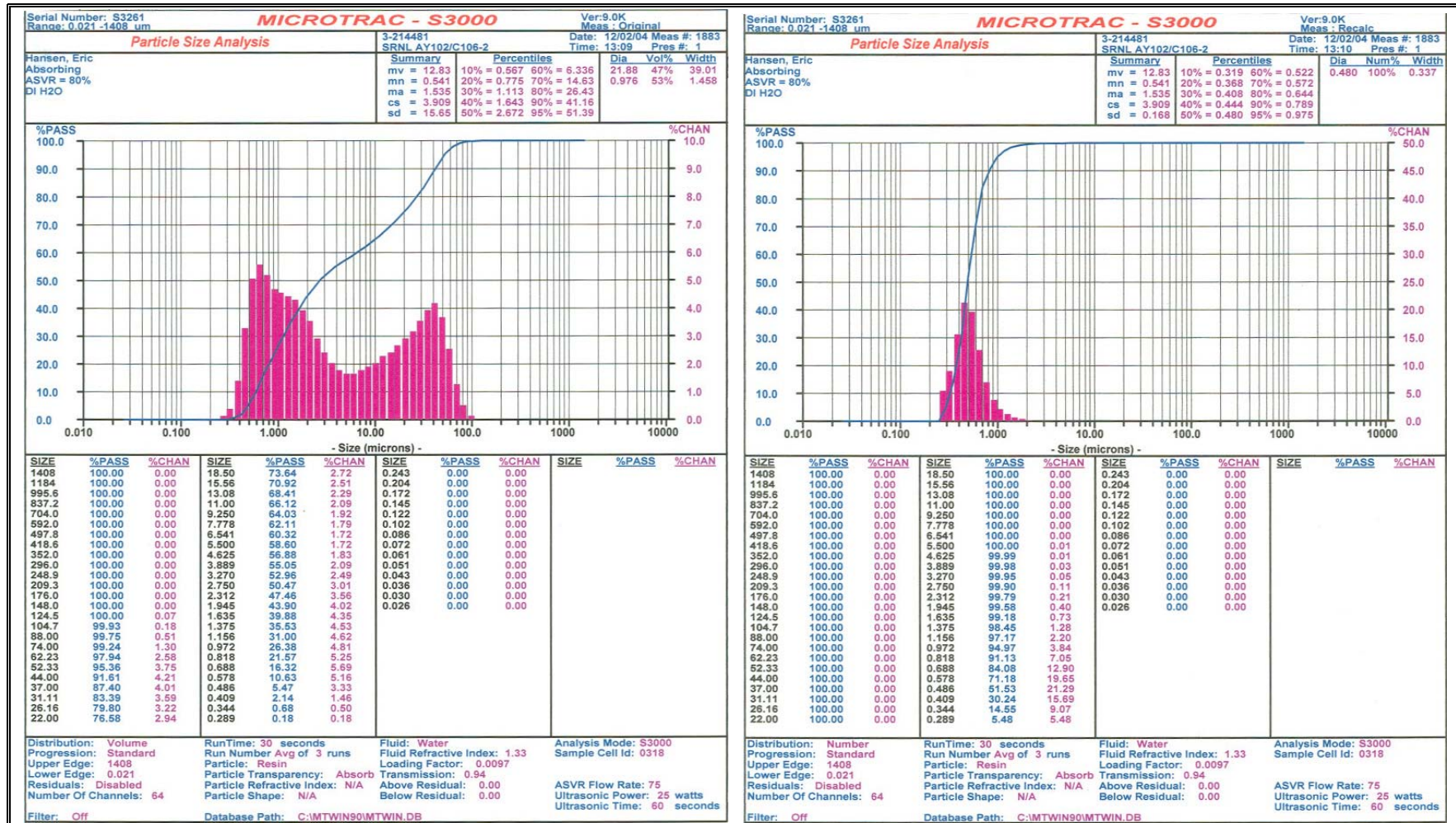


Figure D - 14 Volume and Number PSDs for 20 wt% UDS SRNLAY102/106 MF – 2nd Run

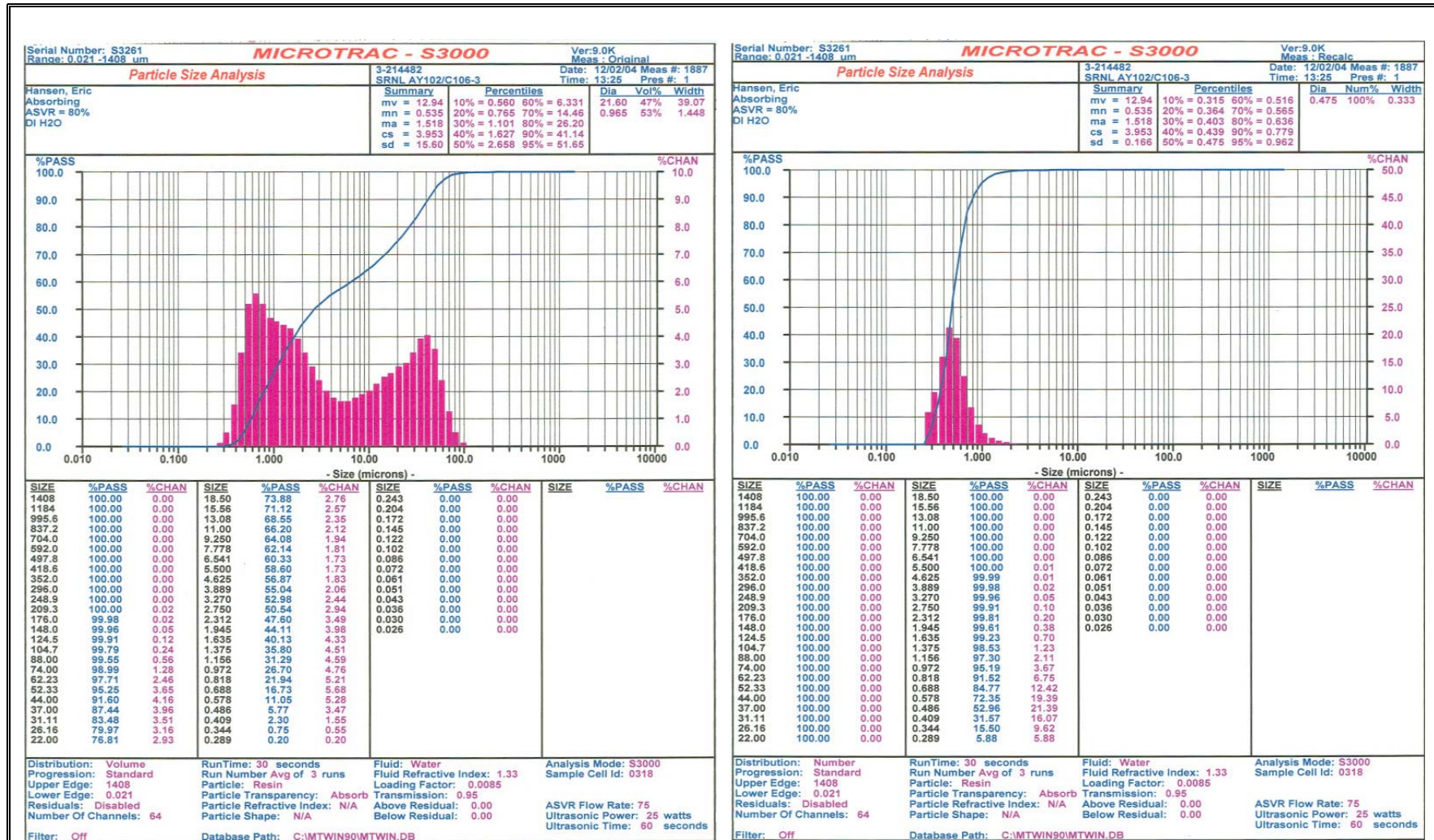


Figure D - 15 Volume and Number PSDs for 20 wt% UDS SRNLAY102/106 MF – 3rd Run

APPENDIX E
FLOW CURVES

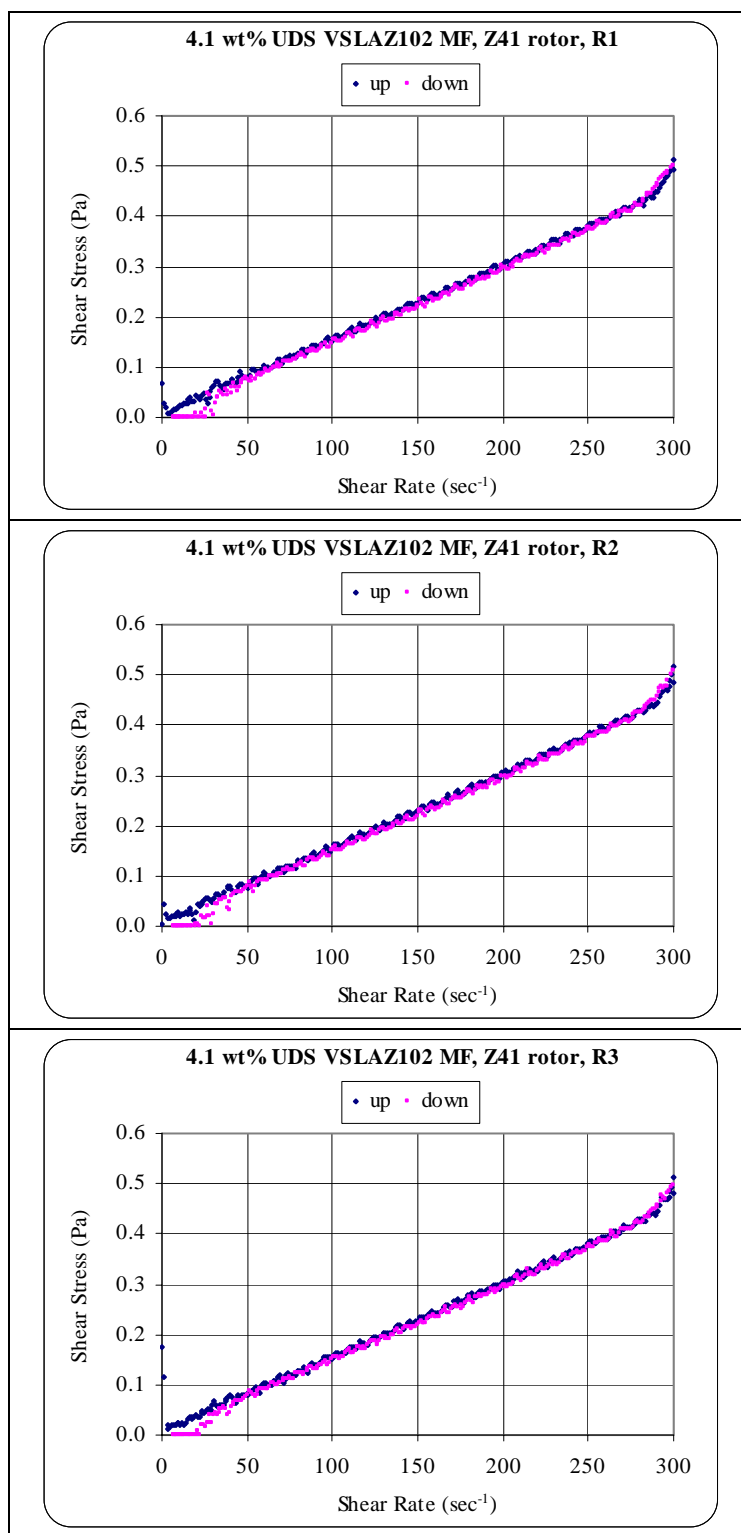


Figure E - 1 4.1 wt% UDS VSL AZ102 Straight Hydroxide MF Flow Curves

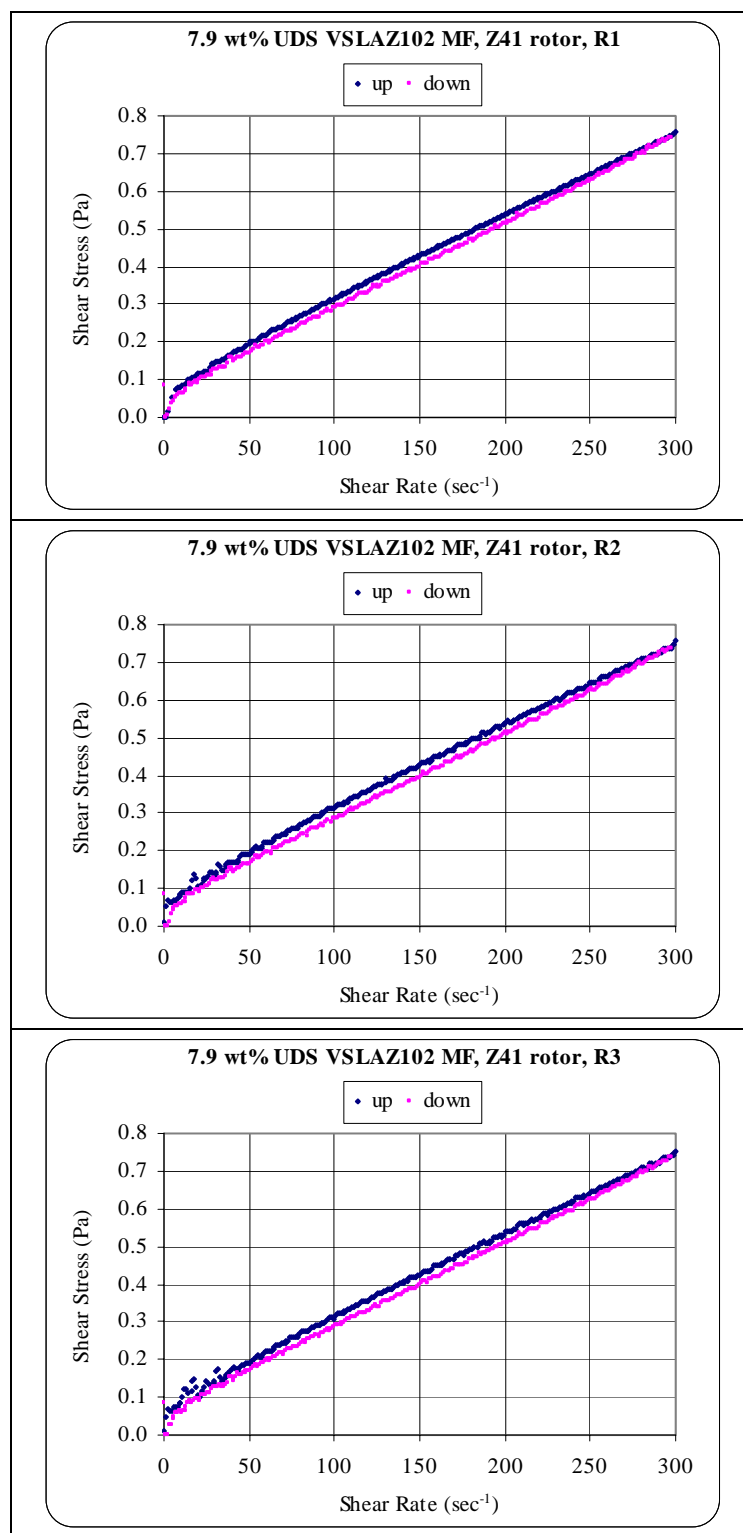


Figure E - 2 7.9 wt% UDS VSL AZ102 Straight Hydroxide MF Flow Curves

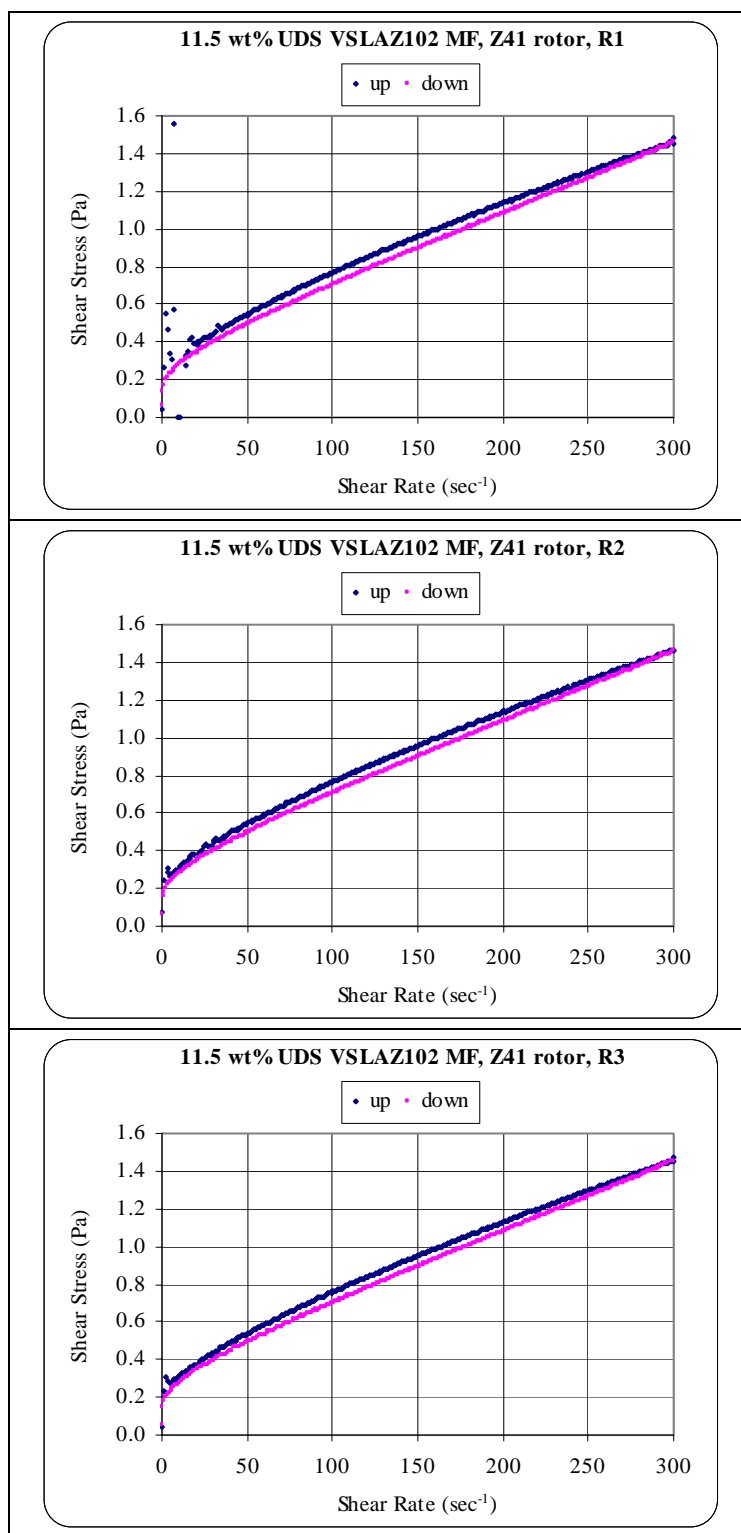


Figure E - 3 11.5 wt% UDS VSL AZ102 Straight Hydroxide MF Flow Curves

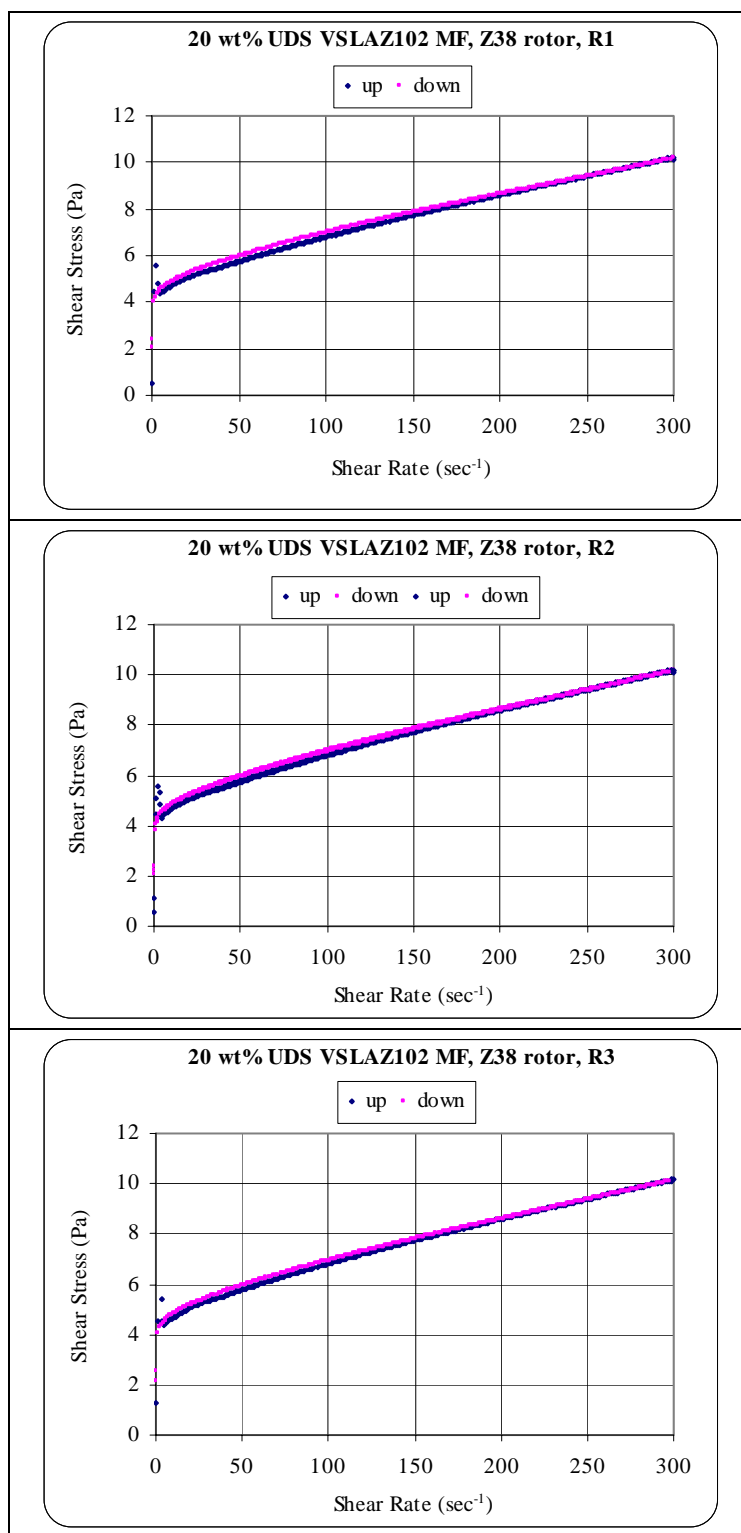


Figure E - 4 20 wt% UDS VSL AZ102 Straight Hydroxide MF Flow Curves

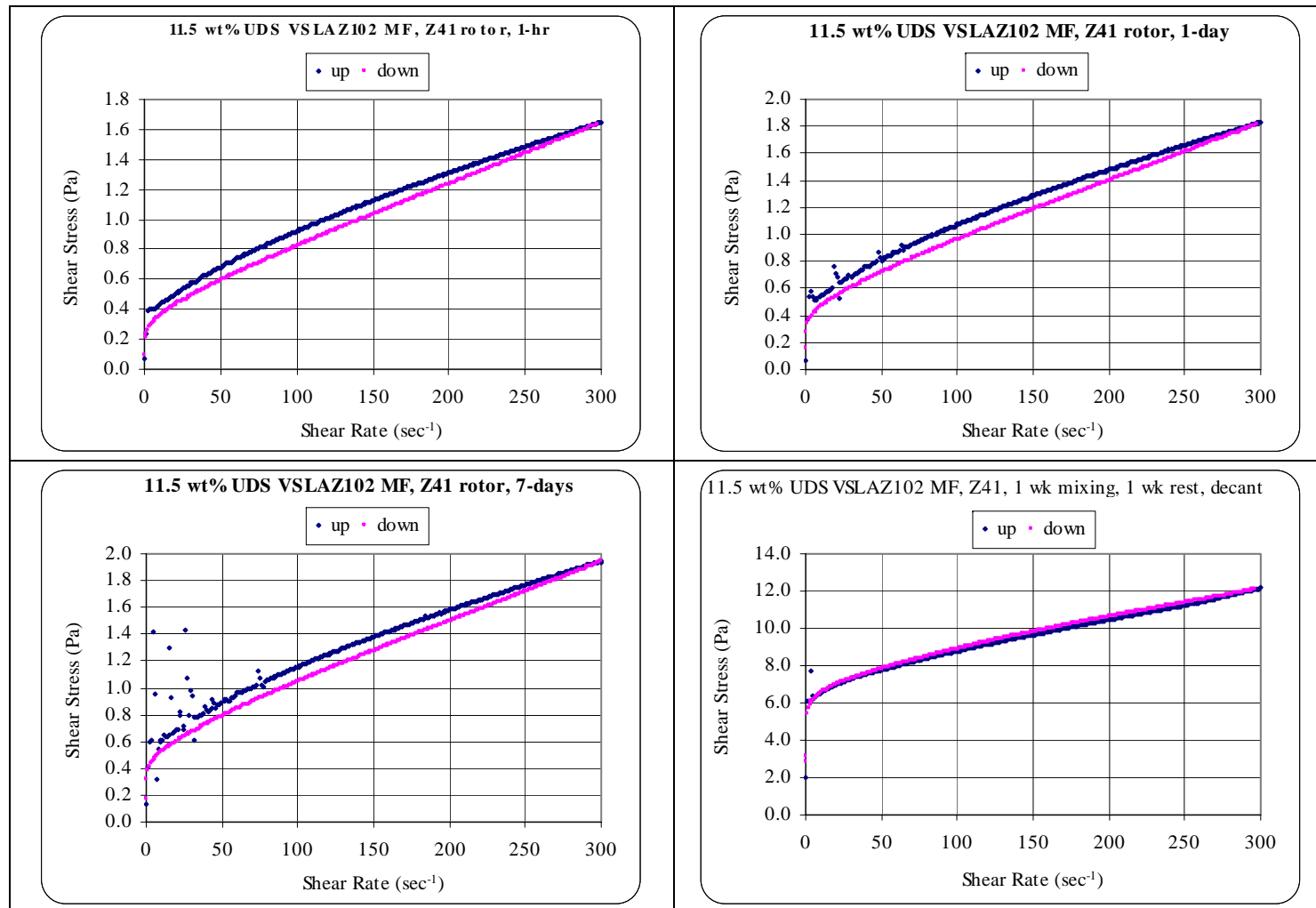


Figure E - 5 11.5 wt% UDS VSL AZ102 Straight Hydroxide MF, Mixing and Aging Flow Curves

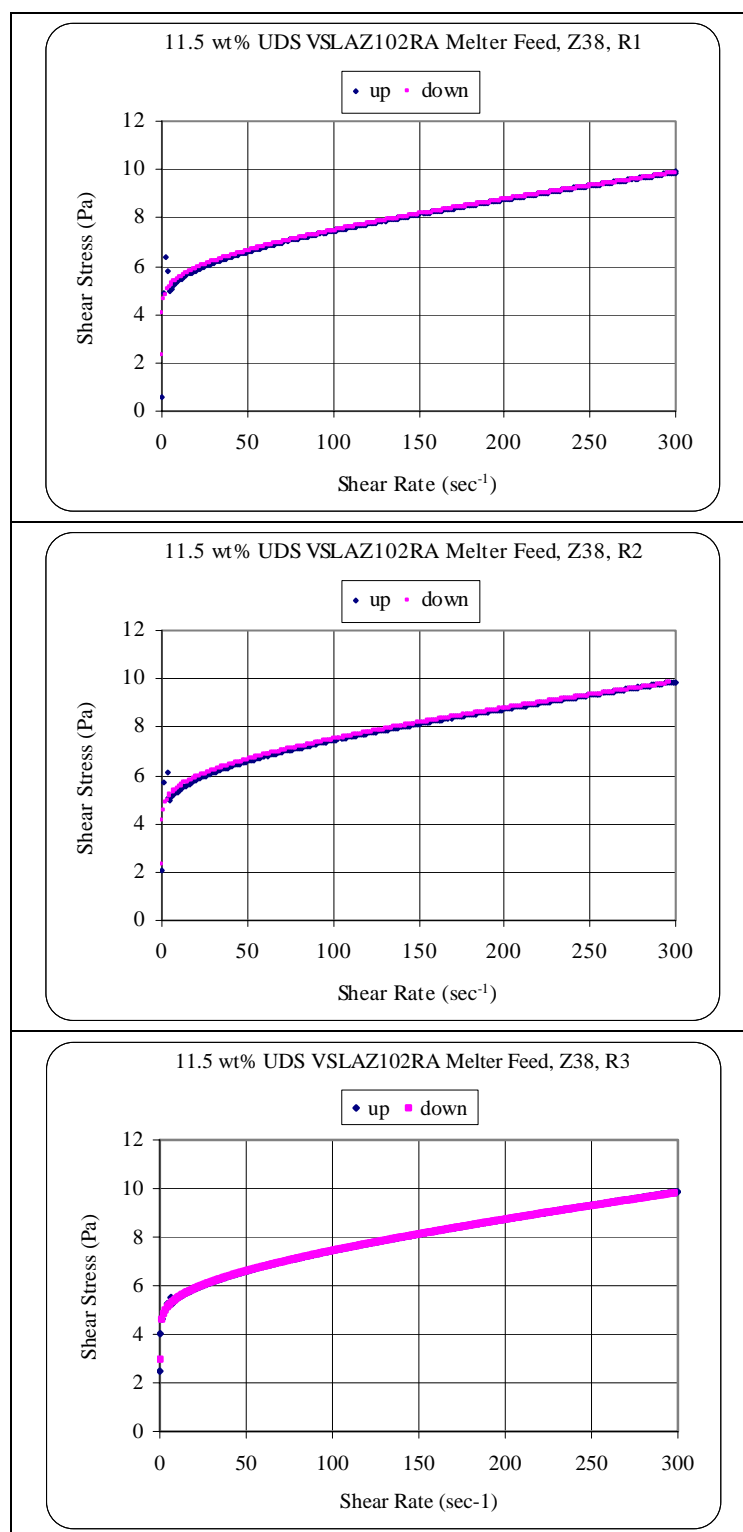


Figure E - 6 11.5 wt% UDS VSL AZ102 Straight Hydroxide Rheology Adjusted MF Flow Curves

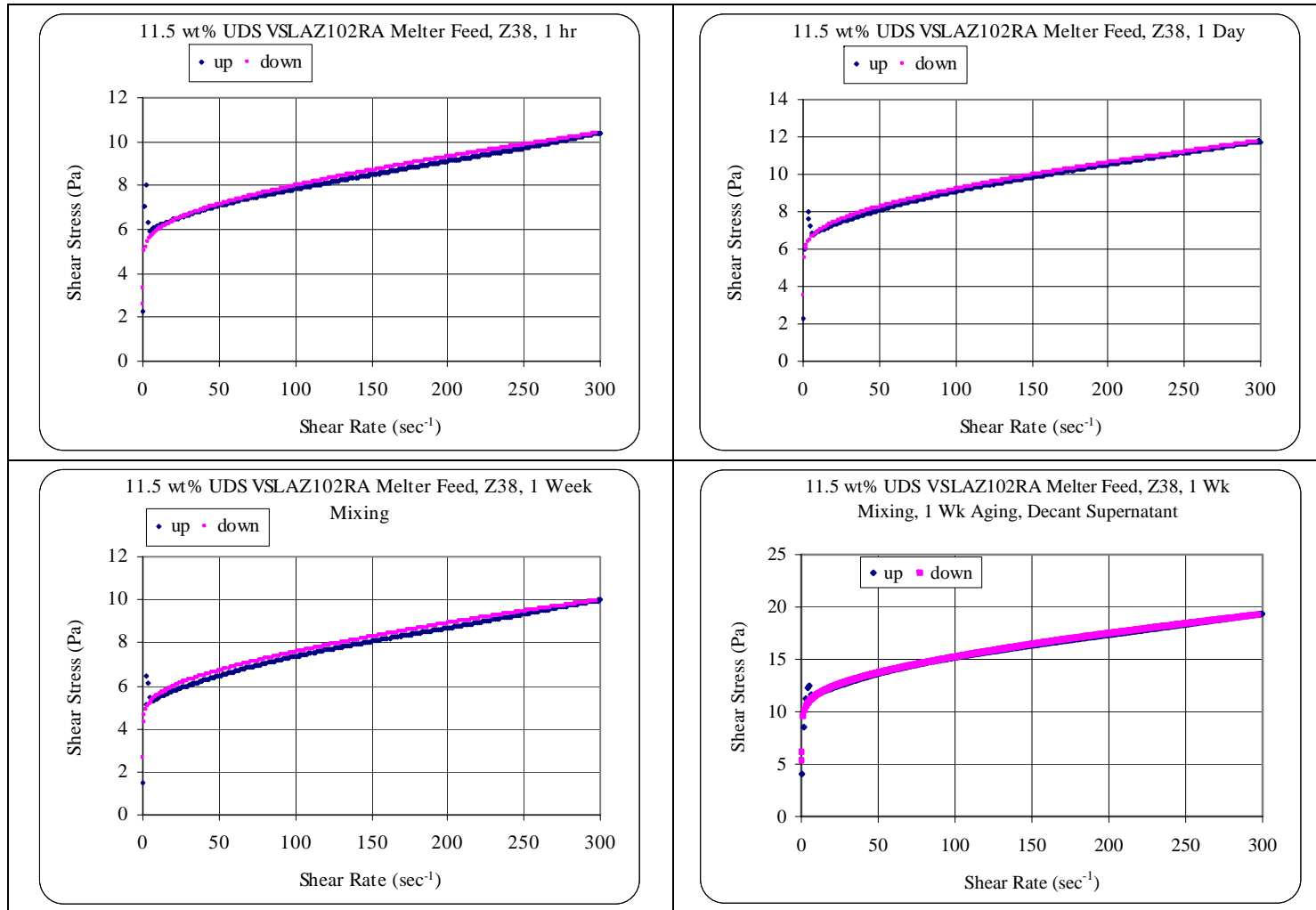


Figure E - 7 11.5 wt% UDS VSL AZ102 Straight Hydroxide Rheology Modified MF Mixing and Aging Flow Curves

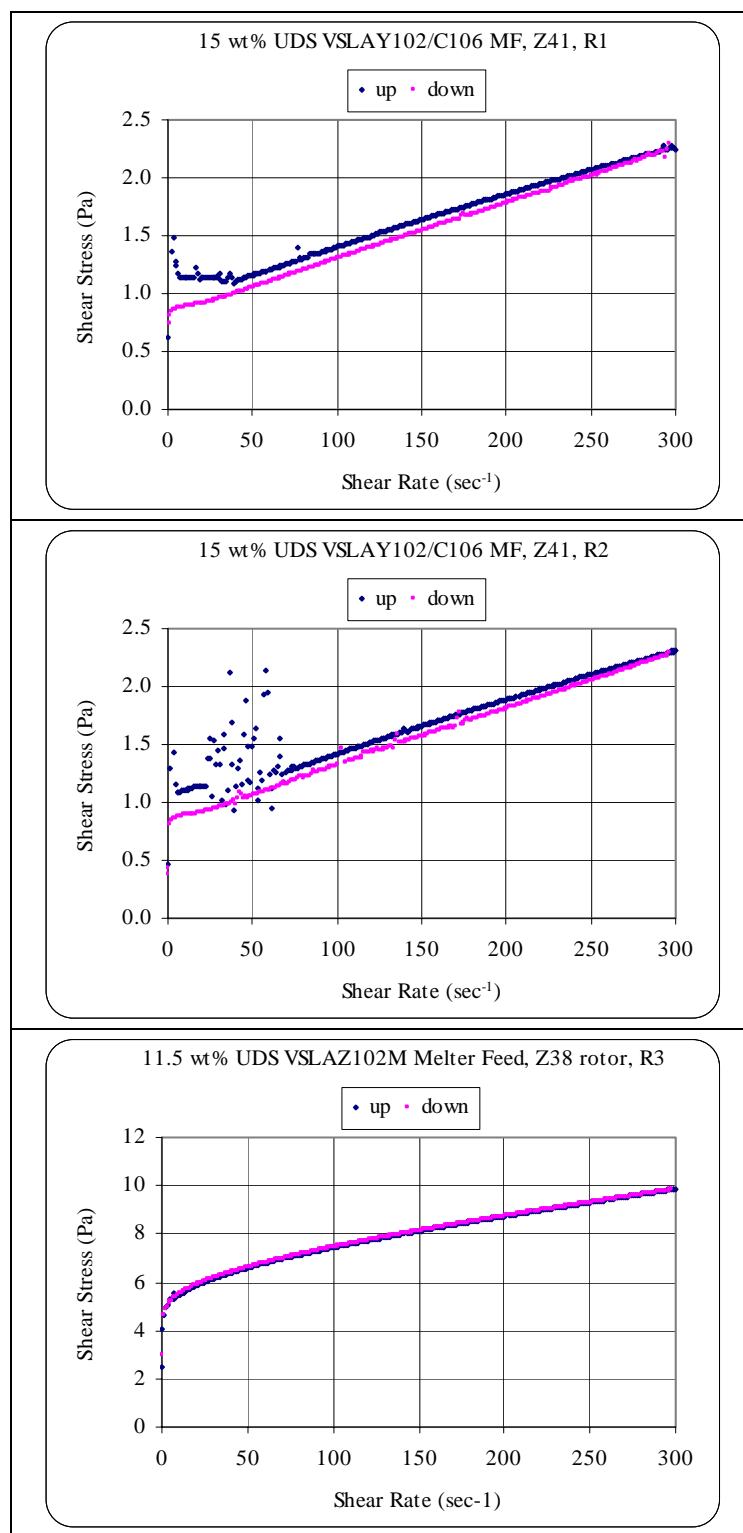


Figure E - 8 15 wt% UDS VSL AY102/C106 Straight Hydroxide MF Flow Curves

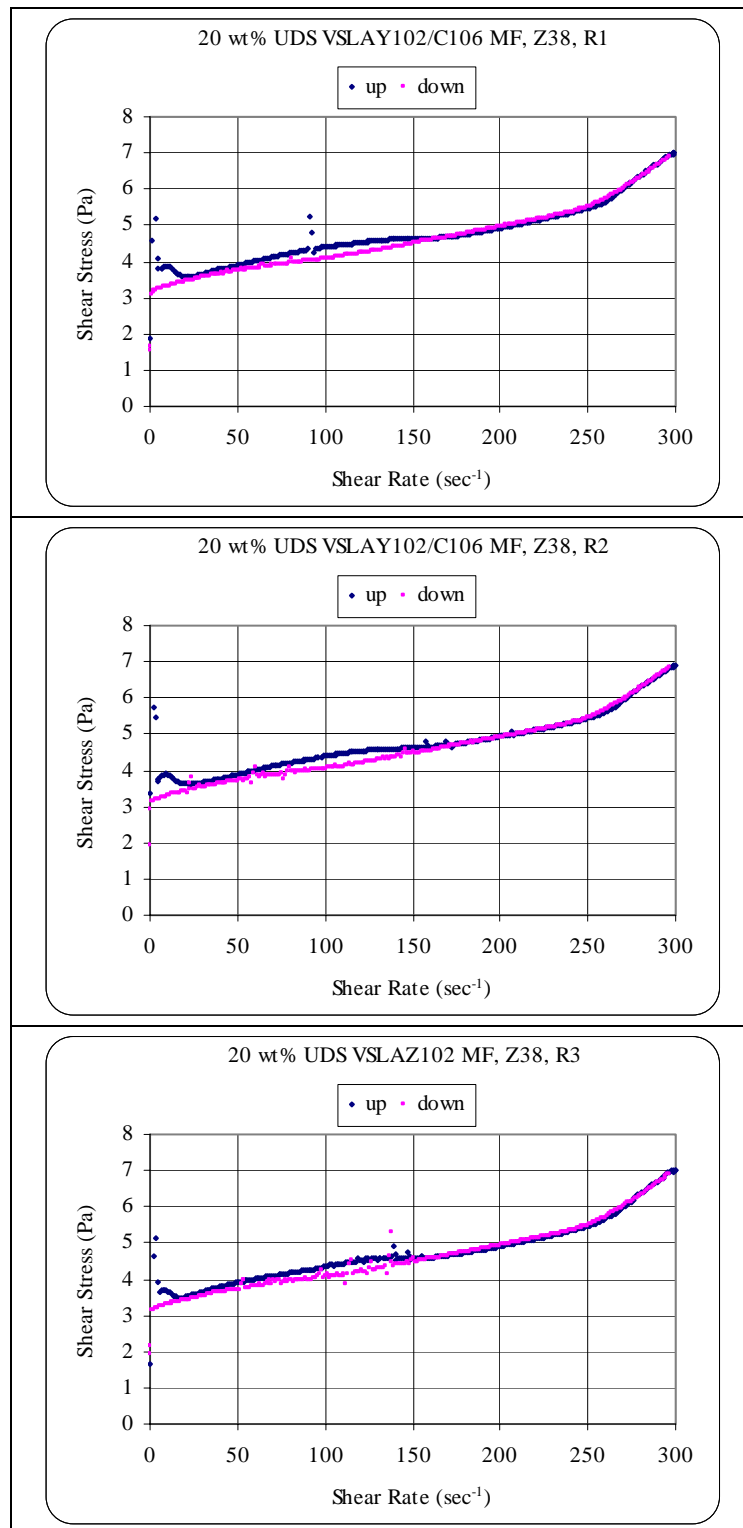


Figure E - 9 20 wt% UDS VSL AY102/C106 Straight Hydroxide MF Flow Curves 1st Set

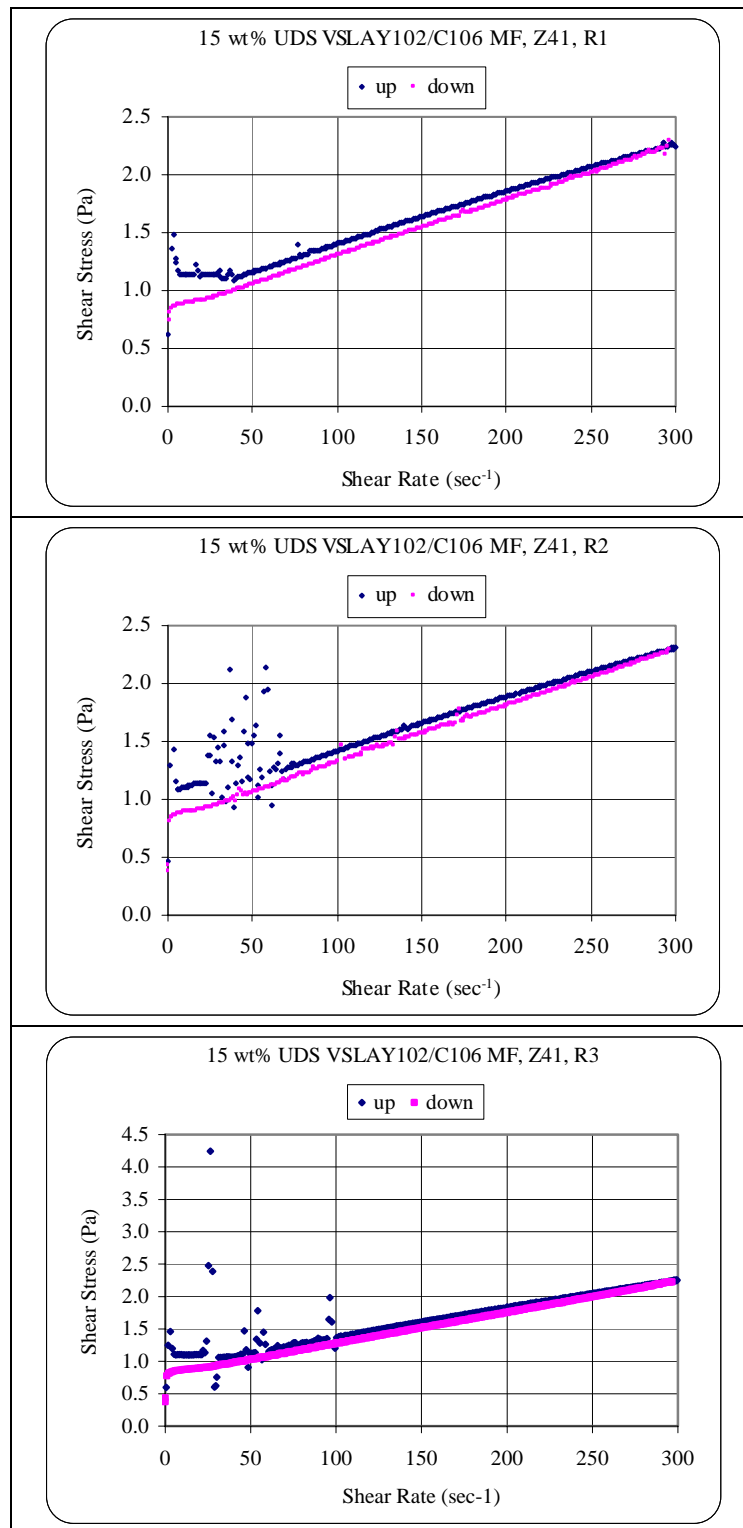


Figure E - 10 20 wt% UDS VSL AY102/C106 Straight Hydroxide MF Flow Curves 2nd Set

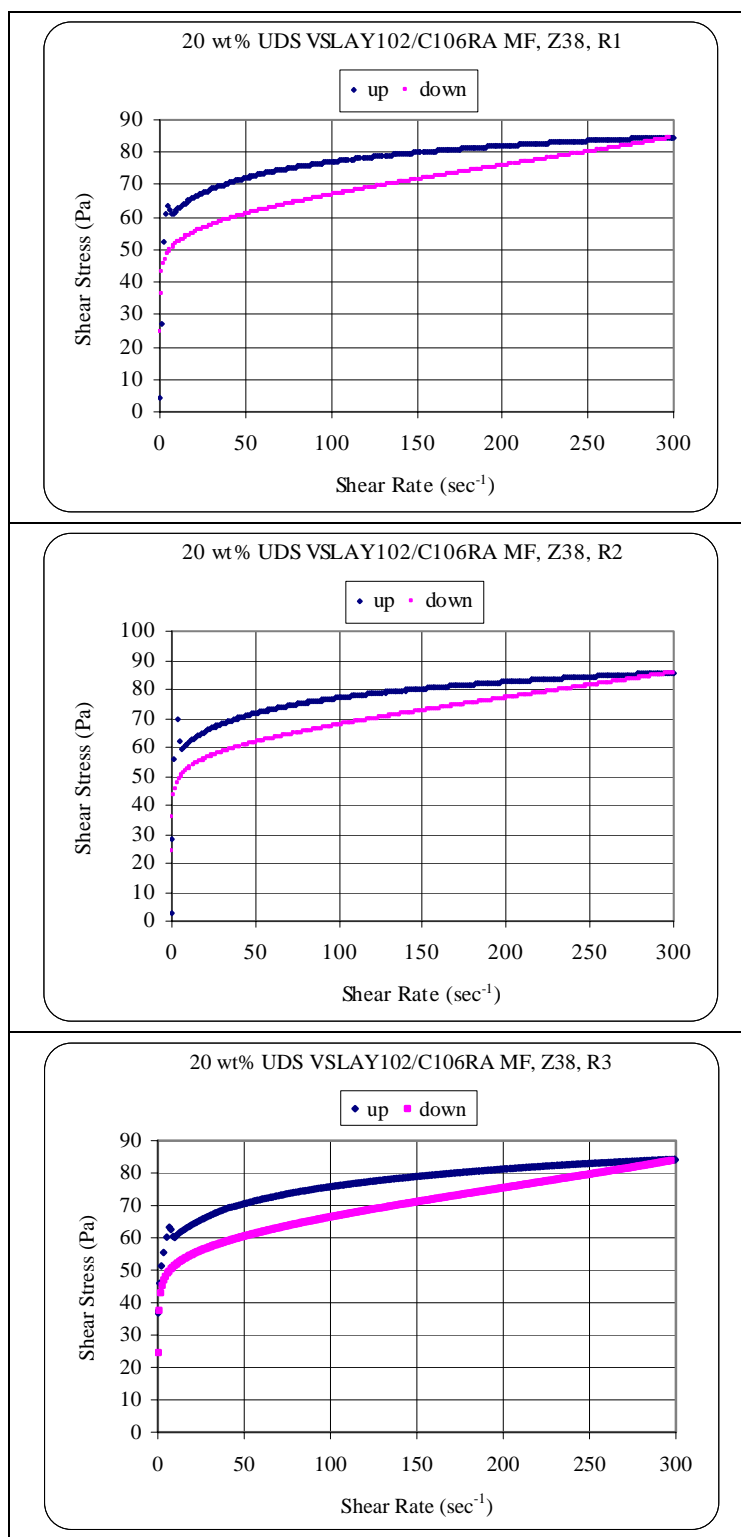


Figure E - 11 20 wt% UDS VSL AY102/C106 Straight Hydroxide Rheology Adjusted MF Flow Curves

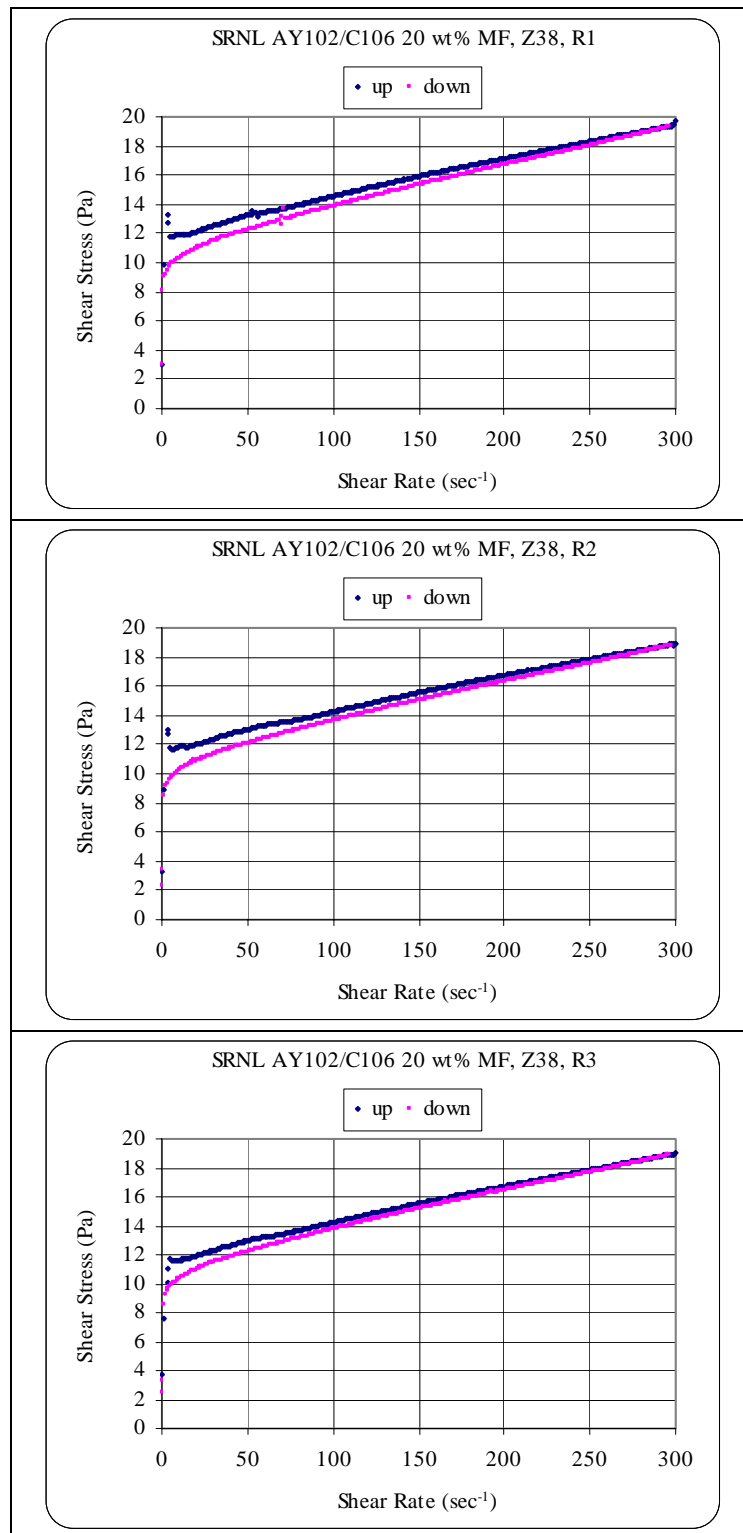


Figure E - 12 20 wt% UDS SRNL AY102/C106 MF Flow Curves