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Characterization of Settler Tank and KW Container Sludge Simulants

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May 2009



Pacific Northwest
NATIONAL LABORATORY

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Abstract

The Sludge Treatment Project (STP), managed by CH2M Hill Plateau Remediation Company (CHPRC) has specified base formulations for non-radioactive sludge simulants for use in the development and testing of equipment for sludge sampling, retrieval, transport, and processing. In general, the simulant formulations are based on the average or design-basis physical and chemical properties obtained by characterizing sludge samples. The simulants include surrogates for uranium metal, uranium oxides (agglomerates and fine particulate), and the predominant chemical phases (iron and aluminum hydroxides, sand). Specific surrogate components were selected to match the nominal particle-size distribution and particle-density data obtained from sludge sample analysis.

Under contract to CHPRC, Pacific Northwest National Laboratory (PNNL) has performed physical and rheological characterization of simulants, and the results are reported here. Two base simulant types (dry) were prepared by STP staff at the Maintenance and Storage Facility and received by PNNL on February 12, 2009: Settler Tank Simulant and KW Container Sludge Simulant. The objectives of this simulant characterization effort were to provide baseline characterization data on simulants being used by STP for process development and equipment testing and provide a high-level comparison of the simulant characteristics to the targets used to formulate the simulants.

Summary

The Sludge Treatment Project (STP), managed by CH2M Hill Plateau Remediation Company (CHPRC), has specified base formulations for non-radioactive sludge simulants for use in developing and testing equipment for sludge sampling, retrieval, transport, and processing. The simulant compositions are documented in a memorandum that is included in Appendix A.^(a) In general, the simulant formulations are based on the average or design-basis physical and chemical properties obtained through characterization of actual sludge samples. The simulants include surrogates for uranium metal, uranium oxides (agglomerates and fine particulate), and the predominant chemical phases (iron and aluminum hydroxides, sand). Specific surrogate components were selected to match the nominal particle-size distribution (PSD) and particle-density data obtained from sludge sample analysis.

Under contract to CHPRC, Pacific Northwest National Laboratory (PNNL) has performed physical and rheological characterization of simulants, and the results are reported here. Two base simulant types (dry) were prepared by STP staff at the Maintenance and Storage Facility (MASF) and received by PNNL on February 12, 2009: settler tank simulant, and KW container sludge simulant. To support rheological characterization, the KW container simulant was provided as several discrete samples: whole PSD and simulant sieved at 500 μm . Settler sludge simulant only contains particles less than 600 μm ; therefore, size fractionation of this simulant was not required.

The objectives of this simulant characterization effort were:

- 1) Provide baseline characterization data on simulants being used by STP for process development and equipment testing.
- 2) Provide a high-level comparison of the simulant characteristics to the targets used to formulate the simulants.
- 3) New data will be acquired from characterization of actual sludge samples during the second half of FY 2009 (sludge from KW containers 240, 250, 260, and 220) and in FY 2010 (sludge from settler tanks and KW container 210). If significant discrepancies in properties are found between the simulant and new sludge samples, and these differences are important to the STP equipment testing objectives, then base simulant formulations may be adjusted.

The simulant characterization approach used by PNNL was based on the physical and rheological characterization approach described within the Sampling Analysis Plan (SAP) for sludge in the KW engineered containers (Baker 2009). Thus, this characterization work also serves as an opportunity to refine and optimize the sample handling and rheological characterization techniques that will be used with actual sludge samples

S.1 Settler-Sludge-Simulant Characterization

Tables S.1 summarizes the PNNL characterization results obtained from the settler simulant sample (ST-A) provided by STP in February 2009 and compares the results to other simulants and data. The

(a) GT MacLean. 2008. *K Basin Sludge Simulants*, Letter Report, From GT MacLean (Fluor Government Group) to R Lokken, August 7, 2008, Fluor Government Group, Richland, WA (See Attachment A).

properties and parameters are compared to the values established in Schmidt and Zacher (2007) (Composition and Technical Basis for K Basin settler sludge simulant for Inspection Retrieval and Pump Testing) and incorporated into the MacLean memorandum (Attachment A).^(a) In Schmidt and Zacher (2007), the simulant was formulated based on data (i.e., composition and PSD) from KE and KW canister sludge samples (i.e., predominant source streams to the settler tanks). Settled density was also identified as a key parameter during the simulant development, and an iron hydroxide slurry was added to control this parameter. Targets for shear strength and yield stress were not specified in Schmidt and Zacher (2007); however, the simulant sample prepared in 2007 exhibited a relatively high shear strength relative to actual sludge samples (Plys and Schmidt 2004).

Characterization of settler simulant (ST A) (Table S.1) demonstrates that it adequately meets most targets established during simulant development. However, the measured shear strength does not bound the maximum values measured for all sludge samples (i.e., including KE Canister sludge samples with particles greater than 250 μm) and is lower than the settler simulant prepared in 2007 (using essentially the same formulation). With granular sludges, shear-strength measurements are highly sensitive to sample history, vessel geometry, and water content. Differences in handling (i.e., higher water content of ST-A) and characterization techniques likely contributed to the differences in the characterization results between settler simulant prepared in 2009 and 2007.

Larger batches of settler simulant and KW container simulant have been prepared by CHPRC and loaded into large-scale mock-up test systems and 55-gal drums at MASF. In these configurations, the CHPRC operators and test engineers have observed very high strengths in the simulant (qualitative observations). These observations are consistent with predictions of the behavior of materials with significant granularity under a lithostatic head (i.e., contribution of fractional forces to shear strength).

The PSD of Sample ST-A meets the target values established in Schmidt and Zacher (2007). To match the established PSD targets, mass-weighted PSDs of the individual simulant components were summed (values in Table S.1), and PSD measurements of the resulting simulant were not performed in 2007. The PSDs of the individual simulant components used for the ST-A simulant are known based on vendor data, sieving, and PSD measurement performed on individual components. Due to sample size limitations (very small), sub-sampling challenges, and instrument limitations (i.e., use of optical light-scattering technique for samples with complex composition, size, and density), to obtain a representative PSD (by volume), multiple sub-samples need to be analyzed and averaged.

(a) GT MacLean. 2008. *K Basin Sludge Simulants*, Letter Report, From GT MacLean (Fluor Government Group) to R Lokken, August 7, 2008, Fluor Government Group, Richland, WA (See Attachment A).

Table S.1. Settler Sludge Simulant Characterization Summary and Comparison

Property/Parameter	Unit	Settler Simulant ST-A (2-12-09)	Settler Simulant ^(a) 2007	Parameters given in Schmidt and Zacher, 2007, unless otherwise noted	
				Target	Range
U metal/surrogate, dry	Wt%	6.0	6.0	6.0	2.7 to 7.1
U Oxides/surrogate	Wt%	82	82	82	77 to 94
Ave Particle Density ^(b)	g/cm ³	5.9 to 6.0 ^(b)	6.0 to 6.2 ^(b)	6.0	4.6 to 9.6
Settled Density	g/cm ³	2.4 to 2.5	2.7	2.7	1.9 to 4.0
Volume % Water	%	69	67	6.7	63 to 75
Shear Strength					
Average	Pa	450 to 600	3650 ± 700	Not Specified	~280 ± 110 ^(c)
High Measurement	Pa	1200 ^(d)	6920 ± 230 ^(e)		8200 ± 4000 ^(c)
Bingham Fits					
Yield Stress	Pa	6 to 7	NM	Not Specified	5 to 40 ^(f)
Viscosity	Pa.sec	1.9 to 2.4	NM		Not provided
Particle Size Distribution					
D ₉₀ (90% <)	µm	334	Calculated from	350	50 to 350
D ₅₀ (50% <)	µm	14	individual	13	6 to 20
D ₂₀ (20% <)	µm	5	components	2	<1 to 5

NM = not measured.

(a) Settler simulant prepared and characterized in Schmidt and Zacher (2007).

(b) Calculated from settled density and volume-fraction water measurements.

(c) Plys and Schmidt (2006), Table C-2. 280 ± 110 Pa is based on sieved KE Canister sludge samples containing only particles less than at 250 µm. 8200 ± 4000 Pa measured on sample with whole PSD.

(d) Measured near bottom of sample container.

(e) Measured after sample was transported. Sample may have compacted during transport.

(f) Range in yield stress values for KE and KW canister sludge. Makenas et al. (1997, pp. I-80 to I-82); and Makenas et al. (1998, p F-52, F-53).

S.2 KW Container Sludge Simulant Characterization

Table S.2 summarizes the PNNL characterization results obtained from the KW container simulant samples, KW-A (whole) and KW-B (<500 µm), provided by STP in February 2009 and compares the results to targets (MacLean memorandum) and the Sludge Databook (Schmidt 2009) parameters. The KW container simulant was formulated to represent the full sludge PSD of sludge within engineered containers 210 and 220 (i.e., maximum size, up to 6350 µm [¼ in.]). Therefore, consistent with the SAP for KW containerized sludge (Baker 2009), and to better conform to instrumentation limits/recommendations on particle size, much of the rheological characterization of the KW container sludge simulant was performed on size-segregated simulant (KW-B). Particles greater than 500 µm (approximately 25 wt% of whole sample) were removed by passing the simulant through a sieve. Because of the sieving, characterization results from the KW-B sample (<500 µm) are not directly comparable to targets established for the “whole” simulant.

Comparison of the KW container simulant to Sludge Databook (Schmidt 2009) parameters (Table S.2) shows that the simulant was formulated based on design-basis values for the KW originating (container 210 and 220) sludge. Because the composition of the safety-basis KW-originating sludge approaches that of settler sludge (and exceeds it with respect to uranium metal); design-basis values were

used to provide a distinct mobilization/retrieval challenge (i.e., includes significant concentrations of other components: iron hydroxides, aggregate, sand, aluminum hydroxide vs. a simulant with very high concentration of uranium oxide surrogate, such as cerium oxide).

For most parameters of interest to sludge retrieval/mobilization equipment, the results from laboratory characterization of KW container simulants meet or exceed targets established based on existing characterization data. Relatively high shear-strength values were measured for the KW-B sample (sieved to remove all particle greater than 500 μm). The settled density and volume fraction solids are higher (conservative for mobilization and retrieval) than safety-basis KW originating container sludge. The PSD appears to be low for the coarsest particle-size material (100 to 500 μm).

Conclusions and Recommendations

The simulant characterization results provide base data to document and better understand simulant properties and behavior.

Challenges encountered during characterization of the simulants (PSD and rheological characterization) need to be considered and addressed before initiating rheological characterization of the new samples of containerized sludge being taken in 2009. Measurement set-up conditions and sample geometry must be defined and standardized.

STP equipment testing activities conducted at MASF (documented in STP project records) have shown that the settler sludge and KW container sludge simulants present appreciable challenges to mobilization and retrieval approaches. However, it is not possible for a single simulant to bound all parameters of interest. For each specific process equipment test, careful consideration of each application must be used to guide the selection of simulants and potential modification to the base simulant formulations.

Table S.2. KW Container Sludge Simulant Characterization Summary and Comparison

Property/Parameter	Unit	KW Container Samples ^(a)		Parameters from MacLean, ^(b) (unless otherwise noted)		
		< 500 μm KW-B	Whole KW-A	Target Whole	Databook KW Con 210 & 220 ^(c)	
					Design	Safety
Fraction of Whole Sample Mass	Wt%	75	100	100	100	100
U metal/surrogate, dry	Wt%	<1 (Est) ^(d)	3.6	3.6	3.6	7.7
U Oxides/surrogate, dry	Wt%	~40 (Est) ^(d)	35.1	35.1	~35 ^(e)	58 ^(e)
Ave Particle Density	g/cm^3	4.7 (calc) ^(f)	3.7 (calc) ^(f)	3.29	3.3	4.1
Settled Density	g/cm^3	2.4	2.4	Not Specified	1.6	1.8
Volume % Water	%	62	48	Not Specified	74	74
Shear Strength						
< 500 μm	Pa	800 to 2400	NM	Not Specified	~500 ^(g)	
High Measurement	Pa	5700	NM		8200 ^(g)	
Bingham Fits						
Yield Stress	Pa	21 to 27	NM	Not Specified	1 to 40 ^(h)	
Viscosity	Pa.sec	1.6 to 3.4	NM		Not provided	
Particle-Size Distribution						
D ₉₀ (90% <)	μm	345	NM	2200	+/- 20%	
D ₅₀ (50% <)	μm	25	NM	27	+/- 20%	
D ₁₀ (10% <)	μm	3	NM	2	+/- 20%	

NM = not measured

(a) See Attachment A. Whole PSD sample(KW-A) and < 500- μm sample (KW-B).

(b) Simulant composition provided in MacLean memorandum. See Attachment A.

(c) Schmidt (2009), Table 5.2.

(d) Estimated by simulant make-up in Attachment A.

(e) Assumes U oxides are a $\frac{1}{3} \text{UO}_2 + \frac{1}{3} \text{U}_4\text{O}_9 + \frac{1}{3} \text{UO}_3 \cdot 2\text{H}_2\text{O}$ mixture (U mole basis mixture).

(f) Calculated from settled density and volume fraction water measurements.

(g) Plys and Schmidt (2006), Table C-2. 500 Pa is based on sieved sludge samples containing only particles less than at 250 μm . 8200 Pa measured on sample with whole PSD.

(h) Range in yield stress values for KE floor and KE/ KW canister sludge. Makenas et al. (1996, pp I-14 I-15); Makenas et al. (1997, pp I-80 to I-82); and Makenas et al. (1998), pp F-52, F-53.

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Acronyms

CHPRC	CH2M Hill Plateau Remediation Company
KW-B	KW Basin
MASF	Maintenance and Storage Facility
NIST	National Institute of Standards and Technology
PNNL	Pacific Northwest National Laboratory
PSD	particle-size distribution
SAP	Sampling Analysis Plan
STDEV	standard deviation
STP	Sludge Treatment Project

Contents

Abstract.....	iii
Summary.....	v
Acronyms.....	xi
1.0 Introduction.....	1.1
2.0 Simulant Samples and Initial Preparations.....	2.1
3.0 Characterization Methods.....	3.1
3.1 Solids Content.....	3.2
3.2 Particle-Size Distribution.....	3.4
3.3 Rheological Measurements.....	3.7
4.0 Reporting.....	4.1
5.0 References.....	5.1
Appendix A: Simulant Material Provided to Pacific Northwest National Laboratory.....	A.1
Appendix B: Particle-Size Distribution Data.....	B.1
Appendix C: Rheology Plots.....	C.1

Figures

3.1. Graphical Depiction of Approach to Simulant Characterization.....	3.2
3.2. Cumulative Percentage of Particles as a Function of Particles Size for both Settler and KB <500 Micron Simulant.....	3.5
3.3. Percent in Range as a Function of Particle Size in Microns for a) Black Bars, Settler Simulant and b) Blue Bars KW-B <500 Micron Simulant.....	3.6
3.4. Typical Stress-Versus-Time Profile for a Shear Vane at Constant Shear Rate.....	3.8
3.5. Shear Stress Versus Shear Rate of Settler Simulant.....	3.11
3.6. Viscosity Versus Shear Rate Obtained for the Settler Simulant shown 3.5.....	3.12

Tables

1.1. Settler Tank and KW Container Sludge Simulant Compositions.....	1.2
2.1. Simulant Received for Characterization.....	2.2
3.1. Summary of Simulant Characterization Measurements and Calculations.....	3.3
3.2. Simulant Properties.....	3.4
3.3. Particle-Size Analysis.....	3.5
3.4. Sieve Analysis of KW-B (P500).....	3.6
3.5. Shear-Strength Measurements.....	3.10
3.6. Bingham Down Fits.....	3.12

1.0 Introduction

The Sludge Treatment Project (STP), managed by CH2M Hill Plateau Remediation Company (CHPRC), has specified base formulations for non-radioactive sludge simulants to use in developing and testing equipment for sludge sampling, retrieval, transport, and processing. Under contract to CHPRC, Pacific Northwest National Laboratory (PNNL) has performed physical and rheological characterization of simulants, and the results are reported here.

The STP base simulant compositions are designed to represent three primary K Basin sludge streams and are documented in a memorandum^(a) (MacLean 2008) that is included in Appendix A.

The simulant formulations are:

1. KE Basin containerized sludge simulant (KE container simulant), to represent sludge originating in the KE Basin pits and floor that now resides in KW containers 240, 250, and 260
2. KW Basin container sludge simulant (KW container simulant), to represent sludge originating from the KWest pit and floors that currently (or in the future) resides in KW containers 210 and 220
3. Settler sludge simulant, to represent KW settler tank sludge that will be recovered into KW container 230.

The first two formulations are similar; however, the KW sludge in containers 210 and 220 is expected to exhibit a higher uranium content (metal and oxide) than the KE Basin originating sludge contained within containers 240, 250, and 260. For equipment testing, the STP has elected to use the more aggressive KW container simulant to conservatively represent both the KW and KE Basin container sludge. Therefore, for simulant characterization reported here, only the latter two of the base simulant types (KW container and settler tank simulant) were characterized.

In addition to the summary formulation provided in the MacLean memorandum (Appendix A), a more detailed description of the technical basis for the settler tank simulant is provided in Schmidt and Zacher (2007). This simulant was formulated based on examination of design- and safety-basis mixtures of KE and KW Canister sludge samples (i.e., the predominant source stream to the settler tanks). The KW container simulant formulation is based on the average- or design-basis physical and chemical properties of the source streams that make up the sludge in containers 210 and 220 (source streams make-up described in the Sludge Technical Databook [Schmidt 2009]).

The simulants include surrogates for uranium metal, uranium oxides (agglomerates and fine particulate), and the predominant chemical phases (iron and aluminum hydroxides, sand). Specific surrogate components were selected to match the nominal particle-size distribution (PSD) and particle density data obtained from sludge sample analysis. Table 1.1 provides a summary of the simulant compositions. Additional details on the simulant make-up are provided in Appendix A.

(a) GT MacLean. 2008. *K Basin Sludge Simulants*. Letter Report from GT MacLean (Fluor Government Group) to R Lokken, August 7, 2008, Fluor Government Group, Richland, Washington.

Table 1.1. Settler Tank and KW Container Sludge Simulant Compositions

Sludge Component Represented	Simulant Component Used	Simulant (Dry Basis)	
		Settler Tank, Wt%	KW Container, Wt%
Uranium metal	Tungsten particles	6	3.6
Uranium oxide agglomerates	Steel grit	14	4.2
Uranium Oxide fine particles	Cerium oxide	68	30.9
Aluminum hydroxides and blow sand	Flyash	11	0
Non U larger particles	Aggregate (rocks)	0	16.9
Blow sand	Sand	0	14.7
Iron Phases	Iron hydroxide (added as a slurry)	1	0
	Iron oxide hydroxide	0	21.9
Aluminum Phases	Gibbsite, Al(OH) ₃	0	7.8
Total		100%	100%

Settler tank sludge is expected to exhibit a much higher uranium total concentration than the KE/KW container sludge, and as shown in Table 1.1, the compositions of these simulant are very different. Also, while the KW container simulant contains particles up to 6350 μm , the settler sludge simulant only contains particles less than 600 μm . Therefore, these two simulants represent distinct challenges for the development and testing of sludge handling equipment.

To support design and testing goals of the STP, the physical properties and rheology of the KW containerized sludge and settler sludge simulants have been characterized. This report outlines the approach that was used to characterize these simulants and documents the results of the characterization efforts. The simulant characterization approach used by PNNL was based on the physical and rheological characterization approach described within the Sampling Analysis Plan (SAP) for sludge in the KW engineered containers (Baker 2009). Thus, this characterization work also serves as an opportunity to refine and optimize the sample handling and rheological characterization techniques that will be used with actual sludge samples.

New data will be acquired from characterization of actual sludge samples during the second half of FY 2009 (sludge from KW containers 240, 250, 260 and 220) and in FY 2010 (sludge from settler tanks and KW container 210). If significant discrepancies in properties are found between the simulant and new sludge samples, and these differences are important to the STP equipment testing objectives, then base simulant formulations may be adjusted.

2.0 Simulant Samples and Initial Preparations

Simulants were prepared by STP staff at the Maintenance and Storage Facility (MASF). Each simulant component was extracted from the vendor-supplied container with a small shovel or spatula, weighed, and then added to a sample bottle. All weight measurements were performed to the nearest 0.1 gram, using a calibrated balance.

For the settler sludge simulant, ~600 g of the dry simulant components were provided in a single container. Iron hydroxide slurry, 13 wt% Fe(OH)₃ in water (Noah Technologies Corporation), was provided in a separate container with instructions on the quantity to add to complete the simulant.

STP staff prepared two equal 600-g batches of KW container sludge sample. One batch was then subjected to dry sieving (through a #35 [500 micron] sieve using mechanical agitation for approximately 15 minutes) to create the “fine” and “coarse” KW container simulant fractions. These two sub-fractions were placed into separate bottles.

The simulants (Table 2.1) were received by PNNL on February 12, 2009, under a chain of custody (included in Attachment A). At PNNL, the simulant components were mixed, an excess of water was added, and they were allowed to settle for 24 hours. After this settling period was complete, excess water was decanted from the top of the settled solids. This process of mixing, settling (for 24-hours), and decanting excess water was repeated two more times. To avoid loss of fine particles in the mixed simulant slurry as a result of repeated decanting operations, any solid particles removed with the decant liquid were recovered and returned to the simulant test mixture. Because of the large range in particle sizes and densities of the individual sludge components, dispersions of these materials may be subject to significant size and density segregation. Dilute suspensions of the slurry are likely to yield stratification of simulant components based on the overall particle/aggregate settling velocities. However, well-mixed thickened sludge simulant has sufficient shear strength to uniformly suspend dense particles, and concentration-hindered particle settling also limits the degree of component segregation. For this reason, sub-sampling the settled and thickened slurry provides the most representative sampling.

This process of dry weighing and combining components before adding water is consistent with the manner in which large batches of simulant are prepared by STP during testing at MASF; however, due to scale differences, the process may have some inherent differences, particularly in the area of hydration of the solids.

Table 2.1. Simulant Received for Characterization

Sample Identification	Quantity (Dry Basis)	Description
ST-A	600 g	KW Settler Simulant (note PNNL combined dry Fe(OH) ₃ slurry with dry components)
KW-A	600 g	KW Container Simulant, whole PSD
KW-B (M500)	449.2 g	Size fractionated KW Container Simulant, < 500 μm (Note: unless otherwise noted, KW-B (M500) is referred to as KW-B in this report)
KW-B (P500)	148.8 g	Size fractionated KW Container Simulant, -6350, +500 μm

3.0 Characterization Methods

A graphical depiction of how the simulant samples were handled and the approach for simulant characterization used at PNNL are given in Figure 3.1. The KW container simulant was formulated to represent the full sludge PSD of sludge (i.e., maximum size, up to 6350 μm [$\frac{1}{4}$ in.]). Therefore, consistent with the SAP for KW containerized sludge (Baker 2009), and to better conform to instrumentation limits/recommendations on particle size, as shown in Figure 3.1, much of the rheological characterization of the KW container sludge simulant was performed on size-segregated simulant (KW-B). Particles greater than 500 μm (approximately 25 wt% of whole sample), were removed by passing the simulant through a sieve.

The physical and rheological properties of the simulants were determined according to PNNL technical procedure RPL-Colloid-02 Rev. 1, "Measurement of Physical and Rheological Properties of Solutions, Slurries, and Sludges." The simulants prepared as described in the preceding paragraphs were to be tested to determine the physical properties outlined in Table 3.1. The physical and rheological properties measured include settled density, solids content (both volume and weight fraction), settling rate, PSD, viscosity as a function of shear rate, and shear strength. The average particle density of the simulants was calculated from the simulant composition and particle density of the individual simulant components. It should be noted that due to difficulties in measuring the shear stress vs. shear rate, the proposed dilutions outlined in Table 3.1 were not attempted.

For the determination of settled density, settling rate, and volume fraction of both the water and solids, duplicate aliquots of the simulants were transferred into 100-mL graduated cylinders, and the sediment volume was monitored as a function of time. The sediment volume is the volume from the bottom of the suspension column to the interface between the clear supernatant and the cloudy suspension.

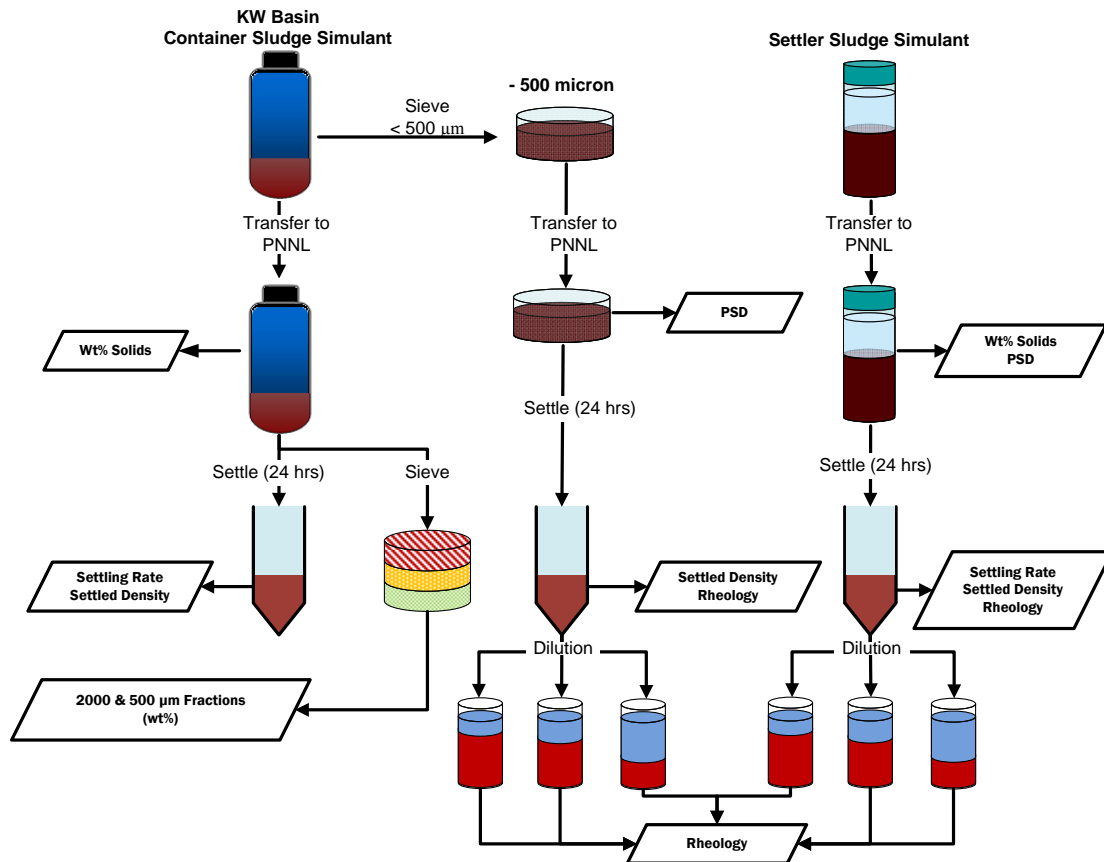


Figure 3.1. Graphical Depiction of Approach to Simulant Characterization

Under the force of gravity, the solids in the suspension sink to the bottom of the cylinder, forming a sludge layer and a clear supernatant layer. The final sediment-bed volume is measured after no significant change in the height of this sludge layer is observed over 4 hours. The volume percent settled solids is then determined by dividing the final sediment bed volume by the total volume of the slurry. The settling rate was determined on the dewatered simulant used for all tests outlined in Table 3.1.

3.1 Solids Content

The solids content was analyzed with a gravimetric method after oven drying at 105°C. The procedure for using this method is PNNL Technical Procedure RPL-COLLOID-02 (Daniel 2007).

The solids content of a sample is the mass of the dried sample divided by the original mass of the settled sample.

Table 3.1. Summary of Simulant Characterization Measurements and Calculations

Property/Parameter	Measurement/Calculation Approach
Settled Density	Prep simulant with excess water; allow sludge to settle for 24 hr; measure volume and mass.
Wt % Total Solids (in settled sludge)	Dry aliquot (known mass and volume) of as-settled sludge at 105°C, measure mass, and record dry bulk volume.
Volume Fraction Water (in settled sludge)	Calculated from settled density and wt% solids. Assumption: all mass loss during drying at 105°C is from loss of water.
Volume Fraction Solids (in settled sludge)	Calculated from volume fraction water.
Settling Rate	Graduated cylinder and stop watch.
Average Particle Density	Calculated from vendor/handbook data on simulant components and simulant make up.
Particle-Size Distribution	Calculated from vendor data on components and simulant make up < 500 µm ^(a) —Particle size analyzer.
Viscosity vs Shear Rate, Shear Stress vs. Shear Rate (Rheograms)	Sieve > 500-µm sample using the following: Sieves 4000 µm, 2000 µm, and 500 µm—dry mass of resulting three fractions. < 500 µm fraction ^(a) : Concentrations: as settled, 75%, 50%, and 25% volume ratio of as-settled + water. Rheology of each concentration was measured at 72°F.
Shear Strength	< 500 µm fraction ^(a) : Settled sludge 24, 48- to 91-hour gel time, vane rheometer.

(a) Note: for settler sludge simulant, all particles are <600 µm. Therefore, for these analyses, settler simulant will not be size fractionated.

A summary of the characterization data obtained is given in Table 3.2.

Table 3.2. Simulant Properties

Simulant	ST-A (settler)	KW-B < 500 μm	KW-A (complete KW)	Unit
Property				
Average Particle Density ^(a)	6.00	3.56	3.56	g/cm^3
Average Particle Density ^(b)	5.91	nd	3.74	g/cm^3
Settled sludge density, ^(c) ST-A ^(f)	2.52	insufficient sample	2.41 ± 0.01	g/cm^3
Settled sludge density ^(g)	2.42 ± 0.03	2.38 ± 0.05	nd	g/cm^3
Settling rate, First 24 hours	0.17	0.04	0.09	mL/hr
Wt% water ^(e)	27.40 ± 0.43	25.51 ± 0.16	20.05 ± 0.82	Percent
Volume fraction water ^(b,c)	69.07	61.59	48.15	Percent
Shear strength ^(b)			nd	
Mixed, ~21 h gel time, middle ^(d)	491 ± 36	1719 ± 580		Pascal
Mixed, ~21 h gel time, bottom	627	1597		Pascal
Mixed, 48 h gel time, middle	612	986 ± 266		Pascal
Mixed, 91 h gel time, middle	604	1519 ± 34		Pascal
Mixed, 91 h gel time, bottom	1214	1495		Pascal

(a) Calculated based on vendor-provided data.

(b) Based on measurements performed on simulant prepared in laboratory.

(c) Performed with Haake – RS600, 1.6 cm diameter by 1.6 cm height shear vane.

(d) Mean \pm STDEV of 2 (3 for St-A, 21 hr) measurements.

(e) Mean \pm STDEV of 2 measurements.

(f) Measurement (1) made on unused simulant.

(g) Duplicates measured on sample used for Rheology, KW-B measured on Rheology sample only.

Note: these samples have been mixed and measured several times.

3.2 Particle-Size Distribution

The PSD of the “fines” (KW-B <500 μm) fraction of the KW container simulant and the “whole” settler simulant (maximum particle size \sim 600 μm) were measured using laser diffraction technology (Table 3.3 and Figure 3.2 and Figure 3.3). A Malvern Mastersizer 2000 equipped with a Hydro S dispersion unit was used to analyze samples, and software for the particle size analyzer calculates the PSD (i.e., fractional volume contribution versus particle diameter) from the light-scattering patterns using Mie scattering theory.

Particles with diameters between 0.02 and 1400 microns (μm) can be analyzed by the instrument to determine the PSD of the simulants. However, because of the high density of some of the simulant components, large size particles (greater than 600 μm) were not introduced to the instrument.

The distribution of particles greater than 500 μm for the KW simulant was determined by sieving methods, and results are also provided in Table 3.4.

Before conducting any simulant slurry particle-size measurements, the PSD of a National Institute of Standards and Technology (NIST)-traceable particle-size standard was measured. The standard consisted of polydispersed (in size) silica particles with diameters falling primarily between 10 and 100 microns. The result of this measurement was compared to the standard’s certificate of analysis provided by NIST, and the acceptable performance of the size analyzer was confirmed. The Malvern Mastersizer 2000 requires the particle refractive index (RI) to calculate the particles size. In the case of complex simulants, such as those used in this study, a trial and error approach is used to determine the particle refractive

index. It was determined that little change in the PSD was observed when using an RI of 2.2 (cerium oxide) – 2.94 (ferric oxide hydroxide) with a particle-absorption index of 0 or 1.

Table 3.3. Particle-Size Analysis

Size, μm	Cumulative Percent Less Than		
	Settler Simulant ST-A	KW-B (wet) < 500 μm	KW-B (dry) < 500 μm
800	99.10	99.99	100
600	97.07	98.42	99.94
500	94.95	96.21	99.3
250	88.23	84.86	95.8
100	87.08	74.24	92.5
40	84.01	59.30	80.2
20	66.23	45.56	62.6
10	38.00	29.83	41.6
5	19.22	17.01	24.1
2.5	9.60	3.03	12.2
1	3.01	3.03	3.9
0.5	0.69	0.73	0.985

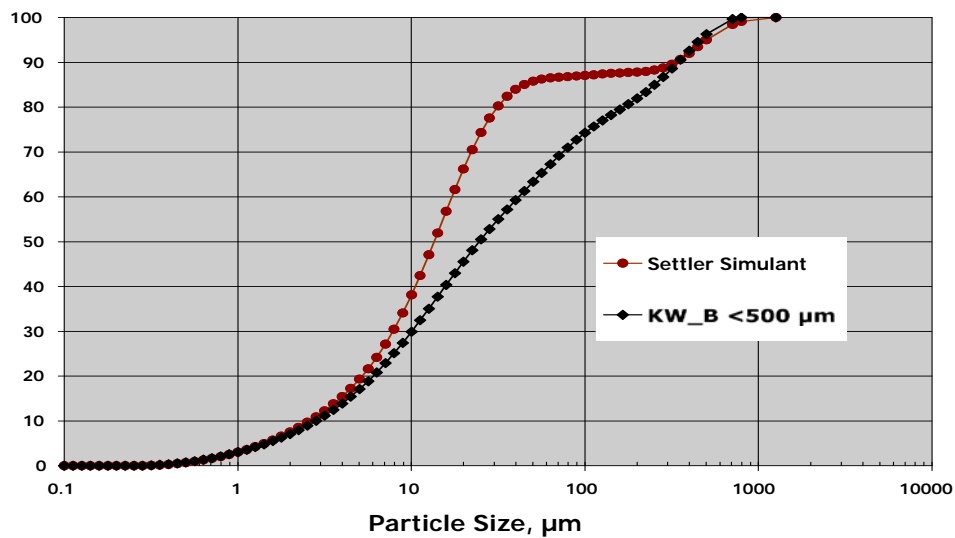


Figure 3.2. Cumulative Percentage of Particles as a Function of Particles Size for both Settler and KB <500 Micron Simulant

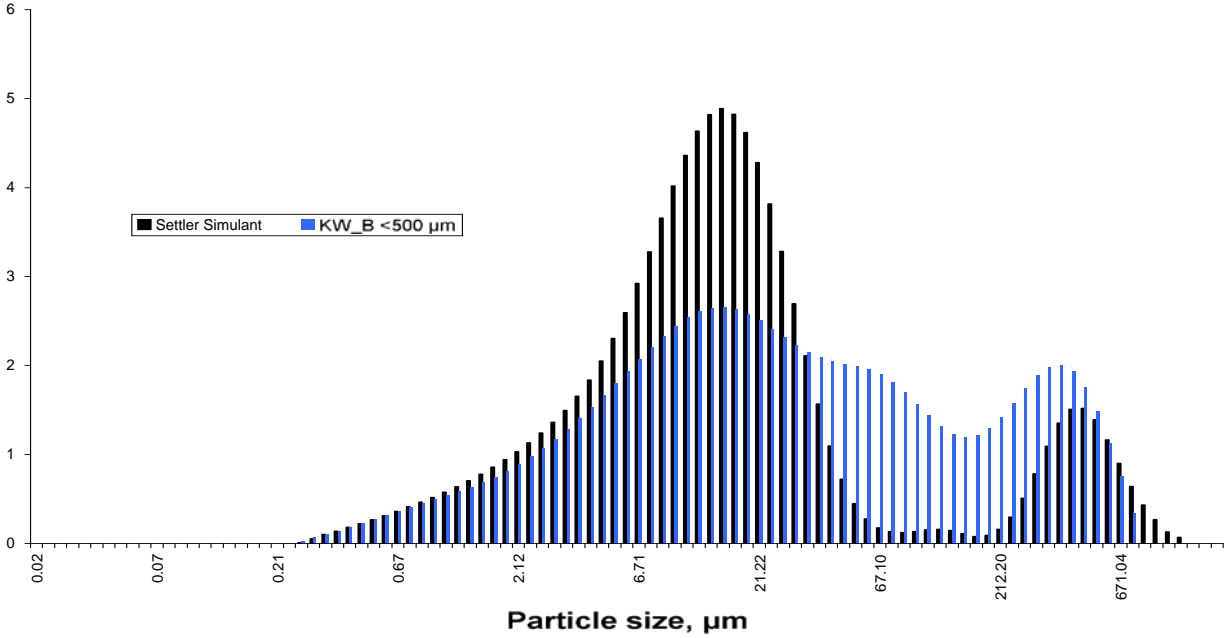


Figure 3.3. Percent in Range as a Function of Particle Size in Microns for a) Black Bars, Settler Simulant and b) Blue Bars KW-B <500 Micron Simulant

Table 3.4. Sieve Analysis of KW-B (P500)

Sample Weight (g)	149.87
Sieve	Cumulative
μm	% less than
4000	78.31
2000	57.90
1000	25.42
500	0.31

When measuring the particle size of simulant materials, small aliquots of the concentrated simulant dispersion (< 1 mL) were diluted in deionized water in a variable-speed recirculator before taking the particle-size measurements (Hydro S dispersion unit). The dilution factor is determined by monitoring the amount of light passing through the diluted material—this is referred to as obscuration. Obscuration is a function of the amount of particles present and is used to determine the amount of material used for an analysis. Sufficient sample dilution to yield obscuration values of 5 to 20% are generally considered acceptable for size measurements, which corresponds to sample sizes in the range of 0.1 g (for fine/less-dense particles) to 2 g for larger/more-dense particles. Due to the presence of the cerium oxide and iron hydroxide in our simulants, very little sample was required to reach the maximum obscuration value of the instrument. Measurements were performed at a pump speed of 2000 rpm. Particle-size analyzers measure volume distributions and thus are highly dependent on the size of particles present, given that particle volume is proportional to the cubed root of its diameter. Particles that have the greatest impact on the volume distribution are also those that are the most difficult to sub-sample in complex, mixed simulants like those used in this study. This is well illustrated in the PSDs obtained for the settler simulant (ST-A) in Appendix B. For comparison purposes and to illustrate the relationship between

particle size and volume, an example of the PSD transformed to number % has been included for each simulant.

Simulant samples were shaken before taking aliquots for PSD analysis. Measurements were made on samples with no additional chemical treatment apart from the sample dilution required to obtain acceptable obscuration values. To determine the stability of the particles with respect to mechanical forces, measurements of particle size before, during, and after the application of sonication were taken. The use of sonication helps verify that the simulant particles are well dispersed, and no particle agglomeration or breaking or particle settling occurs during particle-size measurements.

Measuring the particle size of the settler simulant proved challenging. Specifically, it was difficult to obtain a representative sample of the settler simulant that contained both the larger particles and the finer particles without creating a bias in the results. The heterogeneity of the simulant and the variability in the component density and size range made it very difficult to obtain a representative PSD of the settler simulant. Multiple sub-samples were measured, and the cumulative average PSD along with the individual averages of each aliquot are given in Appendix B. The results given in Table 3.3 and Figure 3.2 and Figure 3.3 are based on the average of all sub-samples (settler simulants, four aliquots, Appendix B). The PSDs obtained for both the settler simulant and the KW-B <500 micron compare well with the target distributions given in the memorandum (MacLean 2008) that is included in Appendix A. The PSDs given in Appendix B illustrate the difficulties encountered obtaining a representative sample for the settler simulant; this is due to the optical properties of the complex simulant and the sampling difficulties. The abundance of fine particles likely causes saturation of the optical detector before the larger particles are at a concentration that can be measured. This is illustrated in sub-samples where particles greater than 100 microns were not detected. To verify that representative PSDs of such simulants are captured, multiple sub-sampling is required.

Additional consideration is needed on dilution approaches that should be pursued for PSD measurements for complex simulants and actual sludge samples. The dispersion unit used in the hot cell at PNNL that will be used for actual K Basin sludge calls for a 10-fold reduction in sample size. This will increase the difficulties in obtaining a representative sample and capturing the larger, higher density particles.

3.3 Rheological Measurements

Shear strength is a semi-quantitative measure of the force required to move the sample and is dependent on sample history. Shear strength can be measured directly by slowly rotating a vane immersed in the sample material and recording the resulting torque as a function of time. The measured torque is converted to a shear stress by equations 3.1 and 3.2.

$$\tau = T / K \quad (3.1)$$

where

$$K = \frac{\pi D^3}{2} \left(\frac{H}{D} + \frac{1}{3} \right) \quad (3.2)$$

where τ = calculated shear stress in Pascal
 T = measured torque in Newton-meters
 K = shear vane constant in cubic meters
 D = shear vane diameter in meters
 H = shear vane height in meters.

A typical stress/time profile is shown in Figure 3.4. The profile shows an initial linear region (τ_y) followed by a nonlinear region, a stress maximum (τ_s), and a stress decay region. The stress maximum is the transition between the visco-elastic and fully viscous flow. Shear strength is defined as the transition between these two flows and is measured at the stress maximum.

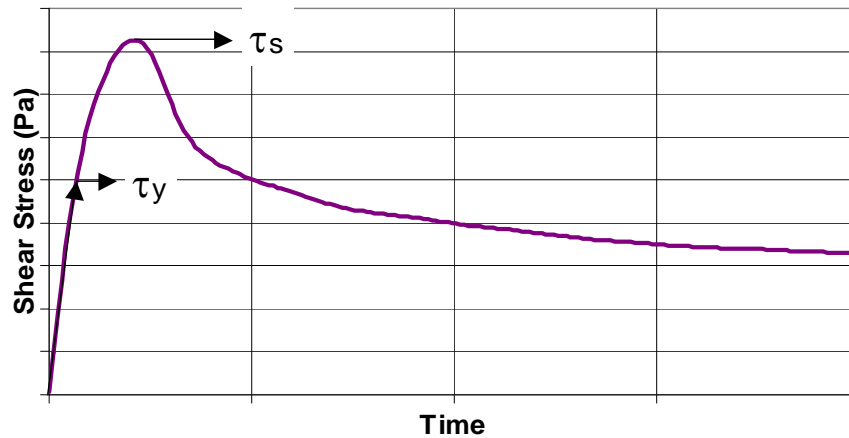


Figure 3.4. Typical Stress-Versus-Time Profile for a Shear Vane at Constant Shear Rate

In general, the test material should be saturated, fine grained, and homogeneous to provide reliable/consistent results from the shear vane test system. There are two primary force contributions to the torque measurement with the shear vane technique: 1) colloidal forces and 2) frictional forces. The colloidal forces will be dominant for slurries with smaller particles (generally under 1 to 10 μm). Frictional forces become important for slurries with large particles (generally greater than 50 microns). Friction will vary with the depth of the slurry and the test geometry of the sample container. The K Basin simulants and sludge contain both fine and larger grain materials, and frictional forces are expected to be a significant contributor to the shear-strength measurements.

Viscosity is determined by analyzing the flow curve. The flow curve was obtained on a rheometer where shear stress can be measured as a function of shear rate. The shear rate was ramped from 0 up to 1000 s^{-1} (or maximum obtainable shear rate for the tool) over a 5-minute period. The shear rate was held constant at 1000 s^{-1} (or maximum obtainable rate) for 1 minute and then ramped back down to 0 s^{-1} over another 5-minute period. Standard rheological models are used to fit the flow curves and determine the yield stress and viscosity of the material. The calibration was checked with certified viscosity standards to verify that the rheometer is operating within acceptable tolerance ranges.

A rheogram for a material with a yield stress has two portions to it. The first portion appears as a nearly vertical line beginning at the origin and running up the ordinate. This portion of the rheogram is

recording the behavior of the material as it acts like a solid or gel. When sufficient force is transmitted to the material to break the gel or make it yield, the rheogram angles sharply to the right, and from then on, the behavior of the material as a fluid is recorded. The point in the curve at which the sample transfers from a solid or gel to a fluid is the yield point. The stress at this point measured in Pascals on the ordinate is the value of the yield stress. The viscosity is the slope of the curve after the material has yielded.

The shear-strength measurements of both the ST-A (settler simulant) and the KW-B simulants (less than 500 μm) are summarized in Table 3.5. The corresponding stress versus time profiles can be found in Appendix C. The shear strength was measured for gel times of 21, 48, and 91 hours for both simulants. The Haake RS 600 Rheometer was used to measure shear strengths. Measurements employed a shear vane tool (16 mm diameter by 16 mm height four-blade vane). Samples were measured in 250-mL wide-neck Nalgene bottles at two different measurement depths, the middle of the container and the bottom (1 times the vane diameter from container bottom). No temperature control was employed during shear-strength measurement.

From Table 3.5, we can see that measurements taken at the bottom of the settler simulant container as opposed to the middle exhibited greater shear strengths. These values were not included in the average value given in Table 3.2. The settler simulant appeared to have a gradient throughout the container, most probably because of particle settling and continued dewatering of the simulant. This behavior was not observed in the KW-B simulant. Both simulants were very difficult to mix and required considerable force to resuspend them between measurements.

It should be noted that the shear strength of the KW-B simulant more than doubled after fines (settled out of the water removed during dewatering, <0.5 g) were reintroduced into the container and mixed on the top. This observation is most likely due to vibration/packing effects of the simulant during partial mixing and has been reported here because of the magnitude of the change observed. The simulant was split into two equal parts to facilitate the performance of parallel experiments; both parts underwent the same dewatering/mixing process with the final dewatering carried out on the split samples. Care was taken to handle both samples in the same manner with mass balance of the water removed per bulk mass of simulant present in the containers taken into account and kept constant for both samples. This observation/measurement provides an indication of the sensitivity of shear strength to very small changes in the simulant handling, make-up, and water content. While not included in the average value calculated and reported in Table 3.2, the high value for KW-B is reported in Table 3.5 because the magnitude of the measurement is comparable to that observed in Schmidt and Zacher (2007) after a settler tank simulant was transported in a vehicle.

Table 3.5. Shear-Strength Measurements

KW-B (M-500) Shear-Strength Measurements				
Conditions	Gel Time (hours)	Shear Strength (Pa)	ave	std
*Mid point	21	2350		
++Added fines and mixed top, Mid point	48	5715		
*Bottom	21	1597		
*Mid point	24	1209	1719	± 580
Mid point	48	797		
*Bottom	48	1174	986	± 266
*Mid point	91	1543		
*Bottom	91	1495	1519	± 34
++ not included in average value				
ST-A Shear-Strength Measurements				
Conditions	Gel Time (hours)	Shear Strength (Pa)	ave	std
*Mid point	21	516		
*Mid point	24	465		
*Bottom	24	627	536	± 83
Mid point	48	612		
*Mid point	91	604		
*Bottom	91	1214	909	± 431
* Average and STDEV include both midpoint and bottom measurements based on gel time .				

The flow-curve analyses for the settler sample and KW-B M500 sample were attempted several times using the concentric cylinder and vane sensor on the TA Rheometer and the Haake RS 600 Rheometer. Particle interaction within the sludge during analysis caused flocculation/agglomeration of the particles. It is suspected that the clustering of particles created a particle bridge in the 1-mm gap between cup and rotor, causing the sensors on both rheometers to stick. Sticking caused the instruments to exceed their maximum allowable torque, which triggers the instrument to terminate the flow-curve analysis to prevent damage to the instrument.

Flow-curve analyses were also attempted with the vane Rheometer setup. However, due to the nature of the rapidly settling simulant, consistent/reportable results were not obtained. Different geometries need to be investigated using this setup to determine if this method is suitable for these types of simulants.

One major consideration in performing a valid rheological measurement of a sample is to identify the necessary gap size between the sensor and the cup to verify that the particle size (in this case agglomeration of particles) does not affect the measurement of the flow curve. In our case, the ~1-mm gap was not sufficiently large, and as such, it was not possible to measure the flow properties of the sludge using the concentric cylinder and vane sensor. Therefore, the flow-curve analysis was performed on the TA Rheometer using a parallel plate geometry at room temperature. Sample dilution was not attempted using this setup because this technique is limited to concentrated/viscous samples. Initially, a plate distance of 1 mm was used, which once again resulted in particle bridging/jamming. The minimum plate gap required for this method is three times the largest particle present in the sample being analyzed. Optimally, a gap size of 10 times the largest particle is desired where practically possible. Next, a 3-mm plate gap was used to measure the flow curves and obtain viscosity plots (Figure 3.5 and Figure 3.6 and

Appendix C), Slurry particles were seen to agglomerate at the edges of the two plates while taking measurements. A summary of down fits using the Bingham model is given in Table 3.6. Both the settler and the KW-B simulants were measured in triplicate, and all plots associated with these measurements can be found in Appendix C. The up-ramp data indicate significant yield strength. From Figure 3.5, it can be seen that the up-ramp is highly non-linear, possibly indicating structural disruption or possibly bridging effects, even in the 3-mm-gap data.

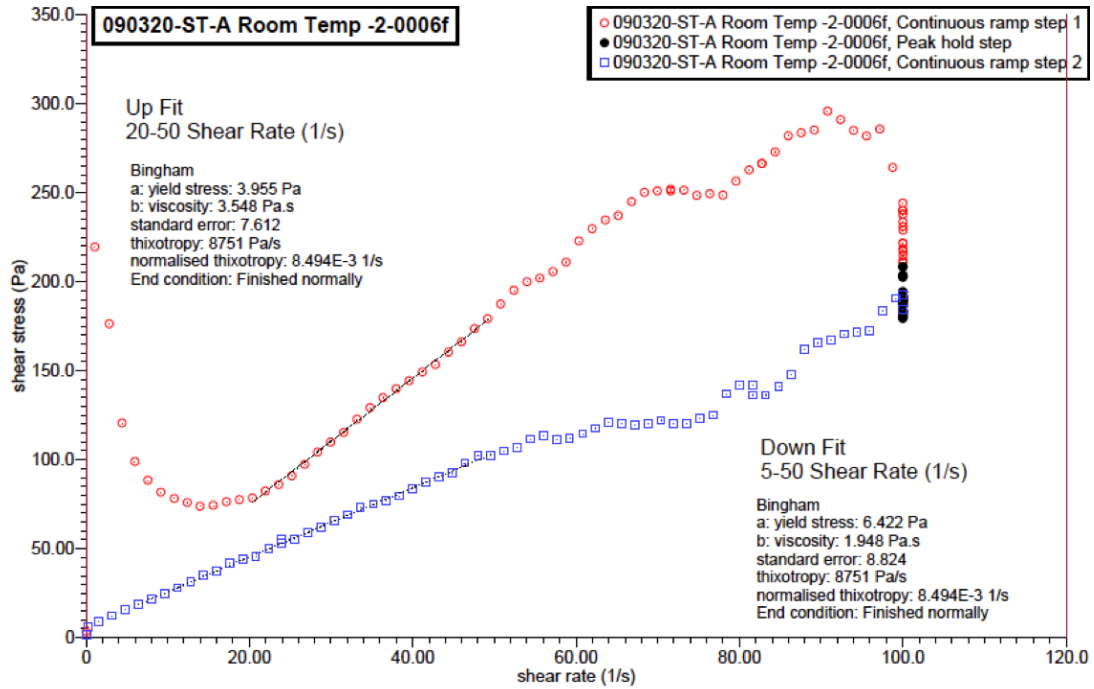


Figure 3.5. Shear Stress Versus Shear Rate of Settler Simulant

The observed hysteresis is indicative of significant structural changes to the sample upon shearing, which is typical for dense slurries. The down-ramp data obtained are most likely indicative of the well mixed, fully disrupted slurry rheology, and show a relatively linear, Bingham-type stress response. Optimization of measuring shear strength needs to be investigated for these difficult materials.

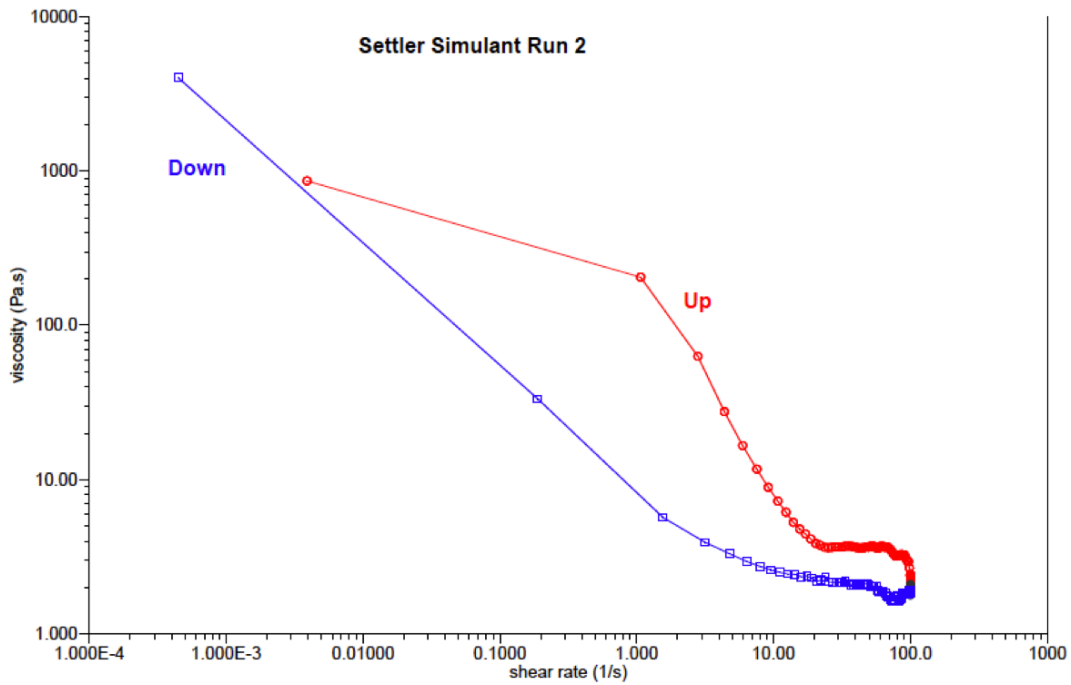


Figure 3.6. Viscosity Versus Shear Rate Obtained for the Settler Simulant shown Figure 3.5

Table 3.6. Bingham Down Fits

Run Number	Down Fit	
	Yield stress Pa	Viscosity Pa.s
ST-A		
1	6.007	2.35
2	6.422	1.948
3	6.692	2.396
KW-B (M-500)		
1	21.86	3.402
2	27.39	2.248
3	21.51	1.63

4.0 Reporting

Experimental data have been recorded in an official PNNL Laboratory Record Book where laboratory notes were taken.

An independent review of the electronic files used for data analysis has being executed.

5.0 References

RB Baker, JA Pottmeyer, JL Westcott, AJ Schmidt and TL Welsh. 2009. *Quality Assurance Project Plan/Sampling and Analysis Plan for Sludge in the KW Engineered Containers*. KBC-33786, Rev. 1, CH2MHill Plateau Remediation Company, Richland, Washington.

Daniel RC. 2007. PNNL Technical Procedure: Measurement of Physical and Rheological Properties of Solutions, Slurries and Sludges. RPL-Colloid-02 Rev. 1, Pacific Northwest National Laboratory, Richland, Washington.

Schmidt AJ. 2009. *Spent Nuclear Fuel Project Technical Databook*. Vol. 2, "Sludge," Rev. 14, HNF-SD-SNF-TI-015, Fluor Hanford, Inc. Richland, Washington.

Schmidt AJ and AH Zacker. 2007. *Composition and Technical Basis for K Basin Settler Sludge Simulant for Inspection, Retrieval and Pump Testing*. PNNL-16619, Rev 1., Pacific Northwest National Laboratory, Richland, Washington.

Appendix A

Simulant Material Provided to Pacific Northwest National Laboratory

Appendix A: Simulant Material Provided to Pacific Northwest National Laboratory

These simulant formulations and copies of chain-of-custody documents are included in this Appendix. The simulant compositions are documented in a memorandum.^(a)

- 1) Sludge Treatment Project Base Simulant Recipes
- 2) Settler Tank Simulant, ST-A
 - Chain of Custody
 - Simulant Preparation Data Sheet
 - Simulant Component Source Data
 - Characterization Request
- 3) KW Container Simulant, KW-A, Complete
 - Chain of Custody
 - Simulant Preparation Data Sheet
 - Simulant Component Source Data
 - Characterization Request
- 4) KW Container Simulant, KW-B, P500 and M500 (size fractionated)
 - Chain of Custody
 - Simulant Preparation Data Sheet
 - Simulant Component Source Data
 - Characterization Request

(a) GT MacLean. 2008. *K Basin Sludge Simulants*. Letter Report from GT MacLean (Fluor Government Group) to R Lokken, August 7, 2008, Fluor Government Group, Richland, Washington.

Fluor Government Group

Richland Office
1200 Jadwin Avenue, PO Box 1050
Richland, WA 99352-1050

509.372.0405
509.373.6471



Memorandum

To: Ryan Lokken
Location: 4710/309/400

Date: 08/07/2008
Reference:

From: Graham MacLean
Location: 1200 Jadwin/247

Client: FH

Phone: 372-0405
FAX: 373-6471

Subject: K-Basin Sludge Simulants

c: Andy Schmidt P8-60

Primary or base recipes (formulations) have been selected for mixtures that represent the combined sludges in three groupings:

1. KE Basin origin sludges that currently reside in KW Containers 240, 250, and 260
2. KW Basin sludges that currently and will reside in KW Containers 210 and 220
3. KW Settler-tube sludge that will reside in KW container 230

The KE Basin and KW settler-tube sludge recipes have been selected from those listed in Table 2 of A21C-STP-TI-0001, Rev. 0, "K Basin Sludge Simulant Recipe Book." The identifications are shown in parentheses in the tables below. The KW Basin sludge recipe, also shown below, was developed after the initial Recipe Book was issued. Data tables and particle size plots are include to show an example composition that meets the specifications.

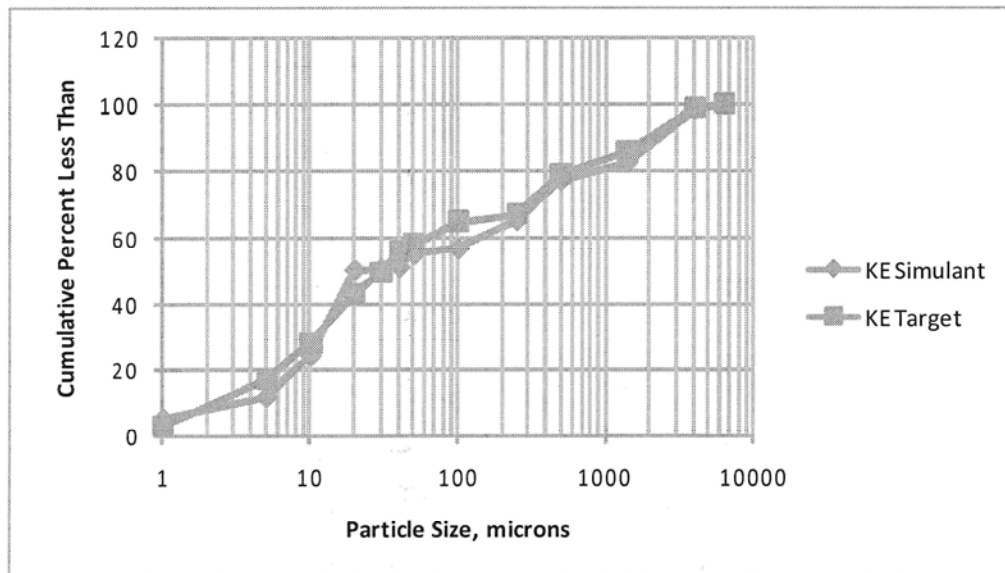
KE Basin Container Sludge Simulant (#11 – modified rheology simulant based on 93 vol.% KE floor sludge and 7 vol.% KE canister sludge)

Material	Amount, wt. %	Particle Size Specification
FeOOH or Fe(OH) ₃	32.1	d ₁₀ = 6 μm, d ₅₀ = 13 μm, d ₉₀ = 19 μm
Sand	24.1	d ₁₀ = .17 mm, d ₅₀ = .30 mm, d ₉₀ = .56 mm
Aggregate	14.3	d ₁₀ = 2.0 mm, d ₅₀ = 2.8 mm, d ₉₀ = 4.0 mm
Al(OH) ₃	12.1	d ₁₀ = 2 μm, d ₅₀ = 13 μm, d ₉₀ = 47 μm
CeO ₂ or equivalent	12.1	d ₁₀ = .5 μm, d ₅₀ = 4 μm, d ₉₀ = 19 μm
Steel Grit or equivalent	4.1	d ₁₀ = 1.9 mm, d ₅₀ = 2.3 μm, d ₉₀ = 2.7 μm
Dense metal or alloy	1.2	d ₁₀ = .30 mm, d ₅₀ = .60 mm, d ₉₀ = 1.41 mm
Total	100.0	d ₁₀ = 4 μm, d ₅₀ = 20 μm, d ₉₀ = 2.6 mm

SO-NW-022 (03/06)

Simulant Component	Tungsten or Alloy	Cerium Oxide (CeO2)	Steel Grit -- G10	Al(OH)3 - (OC-1000)	Ferric Oxide hydroxide (FeOOH)	Aggregate (Gravel)	Lane Mt. Sand -- LM30	Calculated KE Simulant	KE Target	
Wt. %, dry	1.2	12.1	4.1	12.1	32.1	14.3	24.1	100.0		
Particle Density, g/cm ³	16.9	7.13	7.86	2.42	2.85	2.6	2.6	3.00	2.74	
Particle Size, microns	Cumulative Percent Finer Than									
6350	100	100	100	100	100	100	100	100	100	
4000	95.24	100	100	100	100	90	100	99	99	
1410	90	100	0	100	100	6	100	82	86	
500	36.67	100	0	100	100	0	84	77	79	
250	0	100	0	100	100	0	38	65	67	
100	0	100	0	98	100	0	2	57	65	
50	0	99	0	93	99	0	0	55	58	
40	0	97	0	80	92	0	0	51	56	
30	0	94	0	73	94	0	0	51	50	
20	0	91	0	66	97	0	0	50	43	
10	0	73	0	42	32	0	0	24	28	
5	0	57	0	24	6	0	0	12	17	
1	0	37	0	4	0	0	0	5	3	

d90	1410	19	2706	47	19	4000	562	2560	2207
d50	600	4	2329	13	13	2800	295	20	31
d10	300	0	1875	2	6	2000	166	4	3



KW Basin Container Sludge Simulant (based on mixture of all KW floor sludge and pit sludge sources)

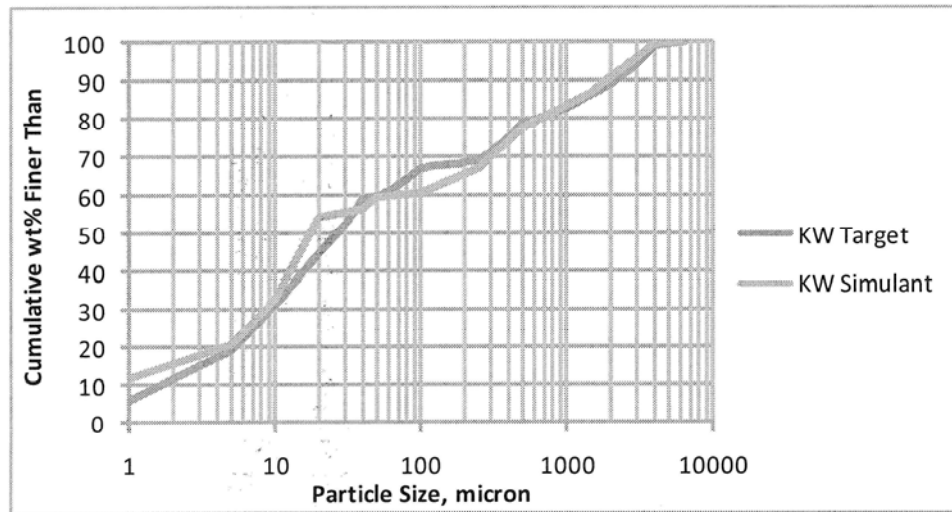
Material	Amount, wt. %	Particle Size Specification
CeO ₂ or equivalent	30.9	d ₁₀ = < 1 μm, d ₅₀ = 4 μm, d ₉₀ = 19 μm
FeOOH or Fe(OH) ₃	21.9	d ₁₀ = 6 μm, d ₅₀ = 13 μm, d ₉₀ = 19 μm
Sand	14.7	d ₁₀ = .17 mm, d ₅₀ = .30 mm, d ₉₀ = .56 mm
Aggregate	16.9	d ₁₀ = 1.3 mm, d ₅₀ = 2.2 mm, d ₉₀ = 3.7 mm
Al(OH) ₃	7.8	d ₁₀ = 2 μm, d ₅₀ = 13 μm, d ₉₀ = 47 μm
Steel Grit or equivalent	4.2	d ₁₀ = .18 mm, d ₅₀ = .39 mm, d ₉₀ = .50 mm
Dense metal or alloy	3.6	d ₁₀ = .41 mm, d ₅₀ = 1.8 mm, d ₉₀ = 4.4 mm
Total	100.0	d ₁₀ = 1 μm, d ₅₀ = 17 μm, d ₉₀ = 2.2 mm

Settler Sludge Simulant (#1 - retrieval simulant)

Material	Amount, wt. %	Particle Size Specification
CeO ₂ or equivalent	68	d ₁₀ = .3 μm, d ₅₀ = 4 μm, d ₉₀ = 19 μm
Steel Grit or equivalent	14	d ₁₀ = .10 mm, d ₅₀ = .34 mm, d ₉₀ = .53 mm
Fly Ash	11	d ₁₀ = 1.4 μm, d ₅₀ = 12 μm, d ₉₀ = 96 μm
Dense metal or alloy	6	d ₁₀ = .28 mm, d ₅₀ = .40 mm, d ₉₀ = .53 mm
Fe(OH) ₃	1	d ₁₀ = .2 μm, d ₅₀ = .6 μm, d ₉₀ = 1.0 μm
Total	100.0	d ₁₀ = .10 mm, d ₅₀ = .34 mm, d ₉₀ = 1.0 mm

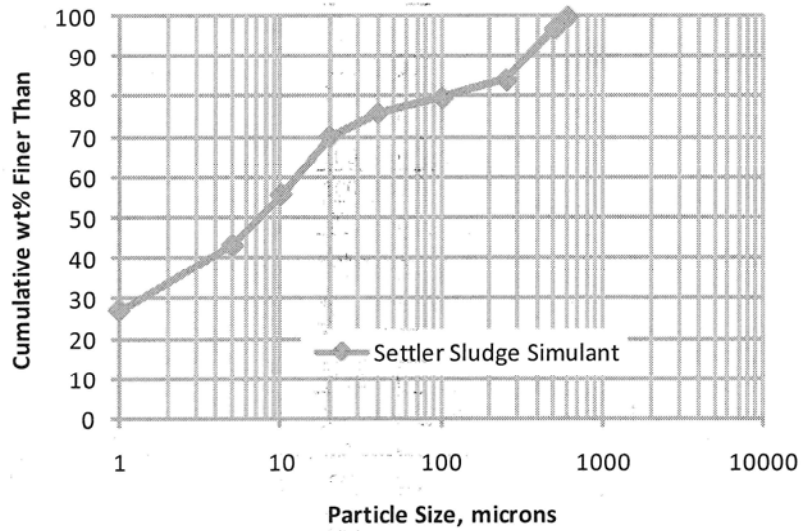
Simulant Component	Tungsten	Cerium Oxide (CeO2)	Steel Grit -- G120	Al(OH)3 - (OC-1000)	Ferric Oxide hydroxide (FeOOH)	Aggregate (Gravel)	Lane Mt. Sand -- LM30	Calculated Simulant	Overall Target
Wt. %, dry	3.6	30.9	4.2	7.8	21.9	16.9	14.7	100.0	
Particle Density, g/cm ³	16.9	7.13	7.86	2.42	2.85	2.6	2.6	3.55	3.29
Particle Size Distribution, Cumulative Percent Less Than									
6350	100	100	100	100	100	100	100	100	100
4000	88	100	100	100	100	98	100	99	99
1410	44	100	100	100	100	31	100	86	86
500	15	100	90	100	100	0	84	77	79
250	0	100	16	100	100	0	38	67	69
100	0	100	0	98	100	0	2	61	67
50	0	99	0	93	99	0	0	60	60
40	0	97	0	80	92	0	0	56	59
20	0	91	0	66	97	0	0	55	45
10	0	73	0	42	32	0	0	33	31
5	0	57	0	24	6	0	0	21	19
1	0	37	0	4	0	0	0	12	6

d90	4424	19	500	47	19	3700	562	2176	2207
d50	1780	4	386	13	13	2186	295	18	27
d10	409	0	180	2	6	1255	166	1	2



Simulant Component	Tungsten or Tungsten Alloy	Cerium Oxide (CeO ₂)	Class F Fly Ash	Ferric Hydroxide	Steel G40 & G80	Simulant
Wt. %, dry	6	68	11	1	14	100.00
Particle Density, g/cm ³	16.9	7.13	2.35	2.85	7.86	5.99
Particle Size, microns	Cumulative Percent Finer Than					
600	100	100	100	100	100	100
500	85	100	100	100	85	97
250	0	100	100	100	29	84
100	0	100	97	100	0	80
40	0	97	81	100	0	76
20	0	91	65	100	0	70
10	0	73	46	100	0	56
5	0	57	30	100	0	43
1	0	37	8	93	0	27
0.1	0	0	0	0	0	0

d90	533	19	96	0.97	533	
d50	397	4	12	0.58	344	
d10	279	0.34	1.4	0.20	101	



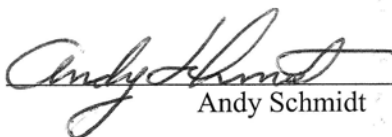
Sand represents quartz, zeolite, aluminosilicates, and other materials of similar density and particle size in the sludge. Preferably the sand should be actual Hanford blow sand obtained from drifts on site, but similar commercial sands can be used. Aggregate represents larger materials such as small rocks, concrete pieces, large corrosion products, etc. The aluminum hydroxide and ferric oxide-hydroxides represent the same compounds in the sludge that resulted from corrosion of the steel racks, aluminum cladding, etc. The dense metal is a substitute for uranium metal; tungsten metal is preferred, but other high-density ($> \text{s.g.} = 14$) metals or alloys can be used, based on the specific test objectives.

Cerium oxide is a substitute for either all uranium oxides and hydroxides, or for just the less dense hydrated species ($\text{UO}_3 \cdot x\text{H}_2\text{O}$). The steel grit is a substitute for UO_2 and U_4O_9 . Bi_2O_3 , bismuth metal, or other materials having a higher specific gravity than steel and available in larger particle sizes than CeO_2 may be substituted to represent UO_2 and U_4O_9 . None of these other materials are included in the recipes above because UO_2 is not a limiting species – the uranium in actual sludge is more dense and larger in particle size than the oxides, and sand is equally or more abrasive than UO_2 .

The particles size specifications are the desired, but due to the large range and uncertainty in the actual sludge properties and variability in commercial products, variation in the specifications of up to about 50% are acceptable for individual ingredients, and about 20% for the overall particle sizes. The recipes above do not include ion exchange beads, but these can be added if important for certain tests. Other variations may be appropriate as determined on a case-by-case basis.

A words of caution: When a simulated waste is made with the above recipe, the settled sludge density is often considerably higher that measured in actual sludge - it compacts substantially more. This is probably because the FeOOH and CeO_2 (which are dry powders) do not hydrate or attract and carry around water molecules like the wet iron oxide-hydroxides and $\text{UO}_3 \cdot x\text{H}_2\text{O}$ compounds do in the real sludge, making it less compressible.

Concurrence:

 8/10/08
Andy Schmidt

Chain of Custody (COC) Form

Chain of Custody No.	Project No./Title	Analyses	Project Point of Contact	Phone Number
CHPRC No: STP-TEST-002	CHPRC K Basin Sludge Simulant Samples to be Characterized by PNNL under Project 53451 Sludge Treatment Project Process Testing Project 53451, Evolving Technical Initiate Activities		Jim Criddle	876-1350
			Scope of Work Document(s): N/A	
Date	Time	Sample Identification	Matrix	Comments
2-12-09	0930	KW Container Simulant (KW-B) P500 KW Container Simulant (KW-B) M500	Dry particles	This is the KW simulant dry solids, segregated by sieving through 500 micron sieve. Two bottles result.
		# of Containers		
		Total of 2	See attached table	
Samples Preserved?	No			
Date	Time	Relinquished by	Time	Received by
2/12/09	4:17p	<i>[Signature]</i>	4:17 P	<i>[Signature]</i>
Date	Time	Relinquished by	Time	Received by
Date	Time	Relinquished by	Time	Received by

* If yes, then note preservation in Comments section.

Characterization Approach for Simulant Confirmation

Attachment to CoC-1

Property/Parameter	Measurement/Calculation Approach	Frequency
Settled Density	Prep simulant with excess water, allow sludge to settle for 24 hr, measure volume and mass.	Routine ^(a)
Wt % Solids (in settled sludge)	Dry aliquot (known mass and volume) of as-settled sludge at 105°C, measure mass, and record dry bulk volume.	Routine ^(a)
Volume Fraction Water (in settled sludge)	Calculated from settled density and wt% solids. Assumption: all mass loss during drying at 105°C is from loss of water	Routine ^(a)
Volume Fraction Solids (in settled sludge)	Calculated from Volume Fraction Water	Routine ^(a)
Settling Rate	Graduated cylinder and stop watch	Routine ^(a)
Average Particle Density	Calculated from Wt% solids and Vol fraction solids	Routine ^(a)
	Calculated from vendor/handbook data on simulant components and simulant make up	Routine ^(a)
	Representative dry sample, density via pycnometry	As needed ^(b) , can be used to confirm individual components
Particle Size Distribution	Calculated from vendor data on components and simulant make up	Routine ^(a)
	< 500 µm - Particle size analyzer	Routine ^(a)
	Sieve full sample using the following Sieves: 2000µm and 500 µm – dry mass of resulting three fraction	Routine ^(a)
Viscosity vs Shear Rate Shear Stress vs. Shear Rate (Rheograms)	< 500 µm fraction: Concentrations: as settled, 75%, 50% and 25% volume ratio of as settled + water.. Rheology of Each concentration will be measured at 55°F and 72°F	Routine ^{(a)(c)}
Shear strength	< 500 µm fraction: Settled sludge (48 to 72 hour gel time, vane rheometer	Routine ^(a)
<p>(a) Routine – Recommend base characterization for all simulants. Significant data from actual sludge exists for comparison.</p> <p>(b) As needed – based on testing objectives for specific simulant, (note part of scope).</p> <p>(c) There is a limited set of rheograms from actual sludge to compare to rheograms from simulants</p>		

KW Simulant Sample Batch - Segregated (KW-B)

Total target mass = 600.00

Component	As-Supplied Size fraction*	Simulant size distribution	Wt% of total	target mass (g)	measured mass (g)
Cerium Oxide	n/a	100.00%	30.9%	185.40	185.4
Steel Grit - G120	n/a	100.00%	4.2%	25.20	25.2
Al(OH)3	n/a	100.00%	7.8%	46.80	46.8
Fe(OOH) (Shepherd)	n/a	100.00%	21.9%	131.40	131.4
Sand	n/a	100.00%	14.7%	88.20	88.2
Aggregate	-1/4 +8	35.00%	16.9%	35.49	35.5
	-8 +12	54.00%		54.76	54.8
	-12 +20	5.00%		5.07	5.1
	-10 +20	6.00%		6.08	6.1
Tungsten	-12 +20	26.96%	3.6%	5.82	5.8
	-20 +40	16.55%		3.57	3.6
	-40 +60	8.33%		1.80	1.8
	-60 +200	0.38%		0.08	0.1
	4830 micron shot	8.61%		1.86	2.0
	4570 micron shot	3.10%		0.67	0.8
	4060 micron shot	1.93%		0.42	0.5
	3810 micron shot	5.90%		1.27	0.9
	3560 micron shot	3.94%		0.85	1.0
	3300 micron shot	4.80%		1.04	0.9
	3050 micron shot	4.36%		0.94	1.0
	2790 micron shot	4.36%		0.94	0.9
	2540 micron shot	3.51%		0.76	0.7
	2290 micron shot	2.99%		0.65	0.7
2160 micron shot	2.22%	0.48	0.5		
2030 micron shot	2.07%	0.45	0.4		
			100.0%	600.00	600.0

*sieve size unless as noted.

William H Combs 2/12/09

Greater than 500 microns	148.8
Less than 500 microns	449.2

Component	Supplier Item number	Lot Number	Supplier Information
Cerium Oxide	5310	5310-08-0708-12P	Molycorp Minerals, LLC. Mountain Pass, CA
Steel Grit G120	LG-120	MT120111308	AGSCO Corp. 160 W. Hintz Road, Wheeling, Illinois, 60090
Al(OH)3	ATH OC1000 4001291	Batch 0843213932	Almatis Inc. 501 West Park Road, Leetsdale, PA, 15056
Fe(OOH) - (Shepherd)	2615	Batch 1048410	Shepherd Chemical Co. 4900 Beech St., Norwood, Ohio, 45212
Sand	LM#30	none	Lane Mountain Company PO Box 127, Valley, WA, 99181
Aggregate -1/4 +8	Gravel 1/4" X 1/8"	111008	AGSCO Corp. 160 W. Hintz Road, Wheeling, Illinois, 60090
Aggregate -8 +12	Silica Sand #8-12	062608	AGSCO Corp. 160 W. Hintz Road, Wheeling, Illinois, 60090
Aggregate -12 +20	Silica Sand #12-20	092908	AGSCO Corp. 160 W. Hintz Road, Wheeling, Illinois, 60090
Aggregate -10 +20	Silica Sand #10-20	05908	AGSCO Corp. 160 W. Hintz Road, Wheeling, Illinois, 60090
Tungsten Granulated Powder -12 +20	P896-3	GT657	ATI Alldyne 7300 Highway 20 West, Huntsville, AL 35806
Tungsten Granulated Powder -20 +40	P897-3	GT648	ATI Alldyne 7300 Highway 20 West, Huntsville, AL 35806
Tungsten Granulated Powder -40 +60	P897-4	GT662	ATI Alldyne 7300 Highway 20 West, Huntsville, AL 35806
Tungsten Crystalline Powder -60 +200	P614	WSP-5719	ATI Alldyne 7300 Highway 20 West, Huntsville, AL 35806
Tungsten Shot (all sizes)	Identified by size	PTHP1208WS	Tungsten Heavy Powder, Inc. 9090 Kenamar Dr., Ste A, San Diego, CA, 92121

Chain of Custody (COC) Form

Chain of Custody No.	Project No./Title	Analyses	Project Point of Contact	Phone Number
CHPRC No: STP-TEST-003	CHPRC K Basin Sludge Simulant Samples to be Characterized by PNNL under Project 53451 Sludge Treatment Project Process Testing Project 53451, Evolving Technical Initiate Activities		Jim Criddle	376-1350
Scope of Work Document(s): N/A				
Date	Sample Identification	# of Containers	Matrix	Comments
2-12-09	Settler Tank Simulant (SI-A) Dry Solids Settler Tank Simulant (SI-A) Fe(OH) ₃ Slurry	Total of 2	Dry particles	This is the Settler Tank simulant. It has been packaged in 2 bottles, keeping the Fe(OH) ₃ slurry separate from the dry solids. The slurry must be weighed before adding to the solids, in accordance with the attached sheets.
Samples Preserved? No				
Date	Relinquished by	Date	Time	Received by
2/12/09		2-12-09	4:17p	
Date	Relinquished by	Date	Time	Received by
Date	Relinquished by	Date	Time	Received by

* If yes, then note preservation in Comments section.
COCForm.doc

Characterization Approach for Simulant Confirmation

Attachment to CoC-1

Property/Parameter	Measurement/Calculation Approach	Frequency
Settled Density	Prep simulant with excess water, allow sludge to settle for 24 hr, measure volume and mass.	Routine ^(a)
Wt % Solids (in settled sludge)	Dry aliquot (known mass and volume) of as-settled sludge at 105°C, measure mass, and record dry bulk volume.	Routine ^(a)
Volume Fraction Water (in settled sludge)	Calculated from settled density and wt% solids. Assumption: all mass loss during drying at 105°C is from loss of water	Routine ^(a)
Volume Fraction Solids (in settled sludge)	Calculated from Volume Fraction Water	Routine ^(a)
Settling Rate	Graduated cylinder and stop watch	Routine ^(a)
Average Particle Density	Calculated from Wt% solids and Vol fraction solids	Routine ^(a)
	Calculated from vendor/handbook data on simulant components and simulant make up	Routine ^(a)
	Representative dry sample, density via pycnometry	As needed ^(b) , can be used to confirm individual components
Particle Size Distribution	Calculated from vendor data on components and simulant make up	Routine ^(a)
	< 500 µm - Particle size analyzer	Routine ^(a)
	Sieve full sample using the following Sieves: 2000µm and 500 µm – dry mass of resulting three fraction	Routine ^(a)
Viscosity vs Shear Rate Shear Stress vs. Shear Rate (Rheograms)	< 500 µm fraction: Concentrations: as settled, 75%, 50% and 25% volume ratio of as settled + water.. Rheology of Each concentration will be measured at 55°F and 72°F	Routine ^{(a)(c)}
Shear strength	< 500 µm fraction: Settled sludge (48 to 72 hour gel time, vane rheometer	Routine ^(a)
<p>(a) Routine – Recommend base characterization for all simulants. Significant data from actual sludge exists for comparison.</p> <p>(b) As needed – based on testing objectives for specific simulant, (note part of scope).</p> <p>(c) There is a limited set of rheograms from actual sludge to compare to rheograms from simulants</p>		

Settler Simulant Sample Batch (ST-A)

Total target mass (dry basis)= 600.00

Component	As-Supplied Size fraction*	Simulant size distribution	Wt% of total	target mass (g)	measured mass (g)
Cerium Oxide	n/a	100.00%	68.0%	408.00	408.0
Steel Grit - G40	n/a	100.00%	10.0%	60.00	60.0
Steel Grit - G80	n/a	100.00%	4.0%	24.00	24.0
Fe(OH) ₃ (Noah) **	n/a	100.00%	1.0%	6.00 **	see below **
Fly Ash	n/a	100.00%	11.0%	66.00	66.0
Tungsten	-30 +40	53.63%	6.0%	19.31	19.3
	-40 +60	43.59%		15.69	15.7
	-60 +200	2.77%		1.00	1.0

*sieve size unless as noted.

100.0% 600.00 **

measured mass of dry solids =

592.8

** This material comes in a slurry and will be kept separate from the dry solids. It is advertised as 13%wt solids. To achieve the required 6 grams of solids, add (6.0 / .13 = 46.2 grams) to the provided Dry solids to make the sample batch. NOTE... the supplied container will contain extra material to assist you in obtaining the correct amount... the container will have 70 grams. Discard unused portion.

70.0 gm

William H Combs 2/12/09

Component	Supplier Item number	Lot Number	Supplier Information
Cerium Oxide	5310	5310-08-0708-12P	Molycorp Minerals, LLC. Mountain Pass, CA
Steel Grit G40	LG-40	MT40111308	AGSCO Corp. 160 W. Hintz Road, Wheeling, Illinois, 60090
Steel Grit G80	LG-80	MT80111308	AGSCO Corp. 160 W. Hintz Road, Wheeling, Illinois, 60090
Fe(OH)3 - (Noah)	11177	0196090/1.1	Noah chemical Div. 1 Noah Park, San Antonio, TX 78249
Fly Ash	Centralia, WA Class F	1952CT	Headwaters Resources 16817 155 th Pl SE, Renton, WA 98058
Tungsten Granulated Powder -30 +40 (sieved from -20 +40)	P897-3	GT648	ATI Alldyne 7300 Highway 20 West, Huntsville, AL 35806
Tungsten Granulated Powder -40 +60	P897-4	GT662	ATI Alldyne 7300 Highway 20 West, Huntsville, AL 35806
Tungsten Crystalline Powder -60 +200	P614	WSP-5719	ATI Alldyne 7300 Highway 20 West, Huntsville, AL 35806

Chain of Custody (COC) Form

Chain of Custody No.	Project No./Title	Analyses	Project Point of Contact	Phone Number
CHPRC No: STP-TEST-001	CHPRC K Basin Sludge Simulant Samples to be Characterized by PNNL under Project 53451 Sludge Treatment Project Process Testing Project 53451, Evolving Technical Initiate Activities		Jim Criddle	376-1850
			Scope of Work Document(s): N/A	
Date	Time	Sample Identification	Matrix	Comments
2-12-09	0930	KW Container Simulant (KW-A) Complete	Dry particles	This is a complete sample of KW simulant dry solids.
Samples Preserved? No				
Date	Time	Relinquished by	Time	Received by
2/12/09	4:17p	<i>Doug Hill</i>	4:17p	<i>MP Luna</i>
Date	Time	Relinquished by	Time	Received by
Date	Time	Relinquished by	Time	Received by

* If yes, then note preservation in Comments section.

Characterization Approach for Simulant Confirmation

Attachment to CoC-1

Property/Parameter	Measurement/Calculation Approach	Frequency
Settled Density	Prep simulant with excess water, allow sludge to settle for 24 hr, measure volume and mass.	Routine ^(a)
Wt % Solids (in settled sludge)	Dry aliquot (known mass and volume) of as-settled sludge at 105°C, measure mass, and record dry bulk volume.	Routine ^(a)
Volume Fraction Water (in settled sludge)	Calculated from settled density and wt% solids. Assumption: all mass loss during drying at 105°C is from loss of water	Routine ^(a)
Volume Fraction Solids (in settled sludge)	Calculated from Volume Fraction Water	Routine ^(a)
Settling Rate	Graduated cylinder and stop watch	Routine ^(a)
Average Particle Density	Calculated from Wt% solids and Vol fraction solids	Routine ^(a)
	Calculated from vendor/handbook data on simulant components and simulant make up	Routine ^(a)
	Representative dry sample, density via pycnometry	As needed ^(b) , can be used to confirm individual components
Particle Size Distribution	Calculated from vendor data on components and simulant make up	Routine ^(a)
	< 500 µm - Particle size analyzer	Routine ^(a)
	Sieve full sample using the following Sieves: 2000µm and 500 µm – dry mass of resulting three fraction	Routine ^(a)
Viscosity vs Shear Rate Shear Stress vs. Shear Rate (Rheograms)	< 500 µm fraction: Concentrations: as settled, 75%, 50% and 25% volume ratio of as settled + water.. Rheology of Each concentration will be measured at 55°F and 72°F	Routine ^{(a)(c)}
Shear strength	< 500 µm fraction: Settled sludge (48 to 72 hour gel time, vane rheometer	Routine ^(a)
<p>(a) Routine – Recommend base characterization for all simulants. Significant data from actual sludge exists for comparison.</p> <p>(b) As needed – based on testing objectives for specific simulant, (note part of scope).</p> <p>(c) There is a limited set of rheograms from actual sludge to compare to rheograms from simulants</p>		

KW Simulant Sample Batch - Not segregated (KW-A)

Total target mass = 600.00

Component	As-Supplied Size fraction*	Simulant size distribution	Wt% of total	target mass (g)	measured mass (g)
Cerium Oxide	n/a	100.00%	30.9%	185.40	185.4
Steel Grit - G120	n/a	100.00%	4.2%	25.20	25.2
Al(OH) ₃	n/a	100.00%	7.8%	46.80	46.8
Fe(OOH) (Shepherd)	n/a	100.00%	21.9%	131.40	131.4
Sand	n/a	100.00%	14.7%	88.20	88.2
Aggregate	-1/4 +8	35.00%	16.9%	35.49	35.5
	-8 +12	54.00%		54.76	54.8
	-12 +20	5.00%		5.07	5.1
	-10 +20	6.00%		6.08	6.1
Tungsten	-12 +20	26.96%	3.6%	5.82	5.8
	-20 +40	16.55%		3.57	3.6
	-40 +60	8.33%		1.80	1.8
	-60 +200	0.38%		0.08	0.1
	4830 micron shot	8.61%		1.86	2.0
	4570 micron shot	3.10%		0.67	0.8
	4060 micron shot	1.93%		0.42	0.5
	3810 micron shot	5.90%		1.27	0.9
	3560 micron shot	3.94%		0.85	1.0
	3300 micron shot	4.80%		1.04	0.9
	3050 micron shot	4.36%		0.94	1.0
	2790 micron shot	4.36%		0.94	0.9
	2540 micron shot	3.51%		0.76	0.7
	2290 micron shot	2.99%		0.65	0.7
2160 micron shot	2.22%	0.48	0.5		
2030 micron shot	2.07%	0.45	0.4		
			100.0%	600.00	600.0

*sieve size unless as noted.

William H Combs 2/12/09

Component	Supplier Item number	Lot Number	Supplier Information
Cerium Oxide	5310	5310-08-0708-12P	Molycorp Minerals, LLC. Mountain Pass, CA
Steel Grit G120	LG-120	MT120111308	AGSCO Corp. 160 W. Hintz Road, Wheeling, Illinois, 60090
Al(OH)3	ATH OC1000 4001291	Batch 0843213932	Almatis Inc. 501 West Park Road, Leetsdale, PA, 15056
Fe(OOH) - (Shepherd)	2615	Batch 1048410	Shepherd Chemical Co. 4900 Beech St., Norwood, Ohio, 45212
Sand	LM#30	none	Lane Mountain Company PO Box 127, Valley, WA, 99181
Aggregate -1/4 +8	Gravel 1/4" X 1/8"	111008	AGSCO Corp. 160 W. Hintz Road, Wheeling, Illinois, 60090
Aggregate -8 +12	Silica Sand #8-12	062608	AGSCO Corp. 160 W. Hintz Road, Wheeling, Illinois, 60090
Aggregate -12 +20	Silica Sand #12-20	092908	AGSCO Corp. 160 W. Hintz Road, Wheeling, Illinois, 60090
Aggregate -10 +20	Silica Sand #10-20	05908	AGSCO Corp. 160 W. Hintz Road, Wheeling, Illinois, 60090
Tungsten Granulated Powder -12 +20	P896-3	GT657	ATI Alldyne 7300 Highway 20 West, Huntsville, AL 35806
Tungsten Granulated Powder -20 +40	P897-3	GT648	ATI Alldyne 7300 Highway 20 West, Huntsville, AL 35806
Tungsten Granulated Powder -40 +60	P897-4	GT662	ATI Alldyne 7300 Highway 20 West, Huntsville, AL 35806
Tungsten Crystalline Powder -60 +200	P614	WSP-5719	ATI Alldyne 7300 Highway 20 West, Huntsville, AL 35806
Tungsten Shot (all sizes)	Identified by size	PTHP1208WS	Tungsten Heavy Powder, Inc. 9090 Kenamar Dr., Ste A, San Diego, CA, 92121

Appendix B

Particle-Size Distribution Data

Appendix B: Particle-Size Distribution Data

Particle-size distributions (PSDs), (Volume), obtained for both the settler simulant, ST-A, and the KW-B M500 simulant are given in this section. The average PSD obtained with 75% sonication has been used throughout this report and discussed in Section 3.2.. The effects of both sonication and refractive index (RI) have been illustrated and are included along with the number average PSD for both simulants. The KW-B M500 simulant was analyzed as received, a dry powder, and as the settled simulant; both sets of data are given here. A summary of the volume and number averages for the final PSD for each simulant is given below.

PSD Run ID	Plot Type	RI	Sonication	No of Runs	D10, μm	D50 μm	D90 μm
			Power	Averaged			
Settler Simulant-75	Volume	2.94	75	14	2.6	13.6	334
Settler Simulant-75	Number	2.94	75	14	0.35	0.53	1.2
KW-B M500-75	Volume	2.94	75	5	2.83	24.61	344
KW-B M500-75	Number	2.94	75	5	0.30	0.44	0.95



Result Analysis Report

Sample Name:
Settler Simulant_4 aliquots

SOP Name:

Measured:
Friday, March 27, 2009 2:14:38 PM

Settler Simulant ST-A
average PSD from obtained
from 4 sub samples

Measured by:
d3m514

Analysed:
Friday, March 27, 2009 2:14:40 PM

Result Source:
Averaged

Particle Name:
Ferric Oxide Hydroxide
Particle RI:
2.940
Dispersant Name:
Water

Accessory Name:
Hydro 2000S (A)
Absorption:
0
Dispersant RI:
1.330

Analysis model:
General purpose
Size range:
0.020 to 2000.000 um
Weighted Residual:
1.081 %

Sensitivity:
Normal
Obscuration:
19.05 %
Result Emulation:
Off

Concentration:
0.0163 % Vol

Span :
24.471

Uniformity:
4.98

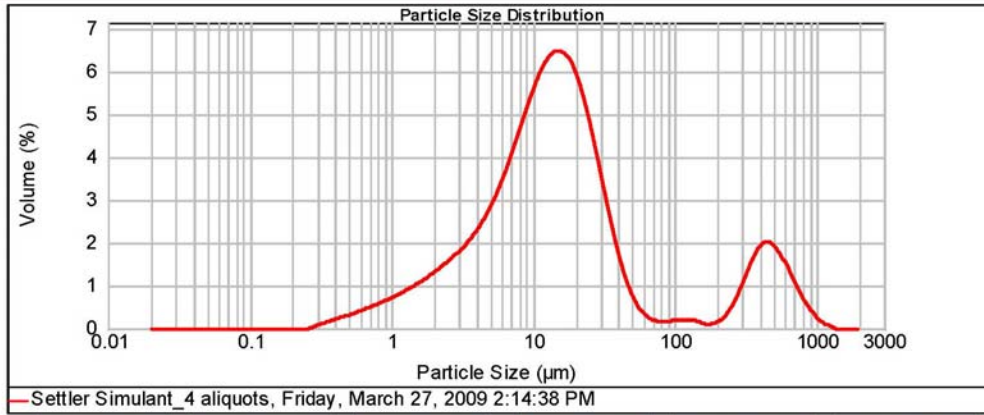
Result units:
Volume

Specific Surface Area:
1.03 m²/g

Surface Weighted Mean D[3,2]:
5.845 um

Vol. Weighted Mean D[4,3]:
74.014 um

d(0.1): 2.597 um d(0.5): 13.537 um d(0.9): 333.872 um



Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.58	7.096	3.28	50.238	0.45	355.656	1.35
0.022	0.00	0.159	0.00	1.125	0.64	7.962	3.65	56.368	0.27	399.052	1.51
0.025	0.00	0.178	0.00	1.262	0.71	8.934	4.02	63.246	0.17	447.744	1.52
0.028	0.00	0.200	0.00	1.416	0.78	10.024	4.36	70.963	0.13	502.377	1.39
0.032	0.00	0.224	0.00	1.589	0.86	11.247	4.63	79.621	0.12	563.677	1.16
0.036	0.00	0.252	0.01	1.783	0.94	12.619	4.82	89.337	0.13	632.456	0.90
0.040	0.00	0.283	0.05	2.000	1.03	14.159	4.89	100.237	0.15	709.627	0.64
0.045	0.00	0.317	0.10	2.244	1.13	15.887	4.82	112.468	0.16	796.214	0.43
0.050	0.00	0.356	0.14	2.518	1.24	17.825	4.62	126.191	0.16	893.367	0.27
0.056	0.00	0.399	0.18	2.825	1.36	20.000	4.28	141.589	0.11	1002.374	0.13
0.063	0.00	0.448	0.22	3.170	1.50	22.440	3.82	158.866	0.08	1124.683	0.07
0.071	0.00	0.502	0.27	3.557	1.65	25.179	3.28	178.250	0.09	1261.915	0.00
0.080	0.00	0.564	0.31	3.991	1.84	28.251	2.70	200.000	0.16	1415.892	0.00
0.089	0.00	0.632	0.36	4.477	2.05	31.698	2.11	224.404	0.29	1588.656	0.00
0.100	0.00	0.710	0.41	5.024	2.30	35.566	1.57	251.785	0.51	1782.502	0.00
0.112	0.00	0.796	0.46	5.637	2.59	39.905	1.09	282.508	0.78	2000.000	0.00
0.126	0.00	0.893	0.52	6.325	2.92	44.774	0.72	316.979	1.09		
0.142	0.00	1.002	0.58	7.096	3.28	50.238	0.45	355.656	1.35		

Operator notes: Average of 14 measurements from 090327.mea



MASTERSIZER 2000

Result Analysis Report

Sample Name:
wet settler simulant (75%) sonic Averaged

SOP Name:

Measured:
Wednesday, March 04, 2009 11:15:21 AM

Settler Simulant ST-A, Sub-samples used to calculate average PSD

Measured by:
D3M514

Analysed:
Wednesday, April 29, 2009 5:33:39 PM

Result Source:
Averaged

Particle Name:
Ferric Oxide Hydroxide
Particle RI:
2.940
Dispersant Name:
Water

Accessory Name:
Hydro 2000S (A)
Absorption:
0
Dispersant RI:
1.330

Analysis model:
General purpose
Size range:
0.020 to 2000.000 μm
Weighted Residual:
0.997 %

Sensitivity:
Normal
Obscuration:
18.07 %
Result Emulation:
Off

Concentration:
0.0114 %Vol

Span :
2.277

Uniformity:
0.756

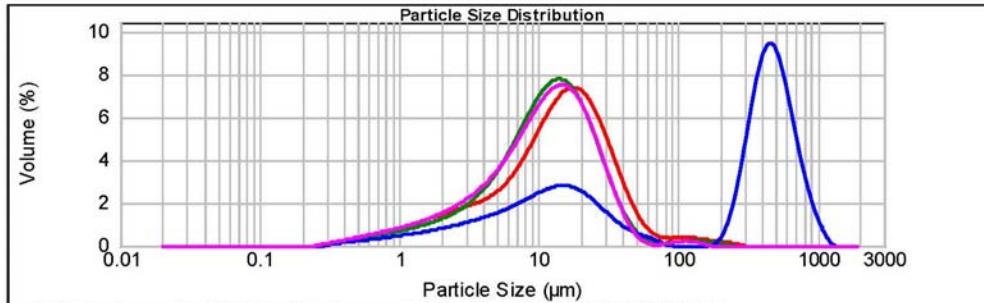
Result units:
Volume

Specific Surface Area:
1.23 m^2/g

Surface Weighted Mean D[3,2]:
4.877 μm

Vol. Weighted Mean D[4,3]:
13.801 μm

d(0.1): 2.215 μm d(0.5): 11.010 μm d(0.9): 27.281 μm



— ST-A Pycnometer (75% Sonic) - Average, Friday, March 27, 2009 2:14:38 PM
 — ST-A Shear Strength Bottle (75% Sonic) - Average, Friday, March 27, 2009 2:34:35 PM
 — ST-A Original Bottle (75% Sonic) - Average, Friday, March 27, 2009 2:55:06 PM
 — wet settler simulant (75%) sonic Averaged Result, Wednesday, March 04, 2009 11:15:21 AM

Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.67	7.096	3.96	50.238	0.24	355.656	0.00
0.022	0.00	0.159	0.00	1.125	0.74	7.962	4.38	56.368	0.09	399.052	0.00
0.025	0.00	0.178	0.00	1.262	0.82	8.934	4.79	63.246	0.04	447.744	0.00
0.028	0.00	0.200	0.00	1.416	0.90	10.024	5.16	70.963	0.05	502.377	0.00
0.032	0.00	0.224	0.00	1.589	0.99	11.247	5.45	79.621	0.09	563.677	0.00
0.036	0.00	0.252	0.03	1.783	1.09	12.619	5.62	89.337	0.13	632.456	0.00
0.040	0.00	0.283	0.08	2.000	1.21	14.159	5.66	100.237	0.16	709.627	0.00
0.045	0.00	0.317	0.13	2.244	1.34	15.887	5.53	112.468	0.16	796.214	0.00
0.050	0.00	0.356	0.18	2.518	1.48	17.825	5.24	126.191	0.16	893.367	0.00
0.056	0.00	0.399	0.23	2.825	1.63	20.000	4.79	141.589	0.12	1002.374	0.00
0.063	0.00	0.448	0.28	3.170	1.81	22.440	4.20	158.866	0.07	1124.683	0.00
0.071	0.00	0.502	0.33	3.557	2.01	25.179	3.53	178.250	0.00	1261.915	0.00
0.080	0.00	0.564	0.38	3.991	2.24	28.251	2.81	200.000	0.00	1415.892	0.00
0.089	0.00	0.632	0.44	4.477	2.51	31.696	2.11	224.404	0.00	1588.656	0.00
0.100	0.00	0.710	0.49	5.024	2.82	35.566	1.47	251.785	0.00	1782.502	0.00
0.112	0.00	0.796	0.55	5.637	3.16	39.905	0.93	282.508	0.00	2000.000	0.00
0.126	0.00	0.893	0.61	6.325	3.55	44.774	0.52	316.979	0.00		
0.142	0.00	1.002	0.67	7.096	3.96	50.238	0.24	355.656	0.00		

Operator notes: Average of 5 measurements



MASTERSIZER 2000

Result Analysis Report

Sample Name:
Wet Settler Simulant (75% Sonic) -

Sample Source & type:

Settler Simulant ST-A,
Effects of RI.

SOP Name:

Measured by:
D3M514

Result Source:
Averaged

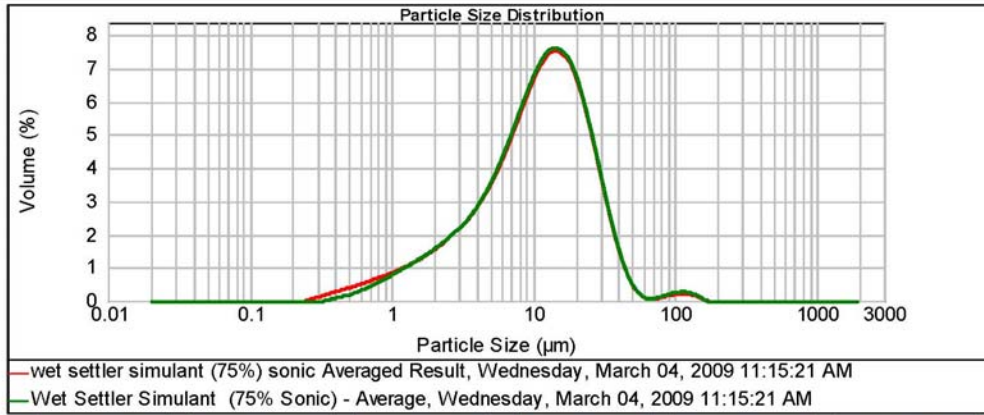
Measured:
Wednesday, March 04, 2009 11:15:21 AM

Analysed:
Wednesday, March 04, 2009 11:15:22 AM

Particle Name: cerium oxide	Accessory Name: Hydro 2000S (A)	Analysis model: General purpose	Sensitivity: Normal
Particle RI: 2.200	Absorption: 0	Size range: 0.020 to 2000.000 um	Obscuration: 18.07 %
Dispersant Name: Water	Dispersant RI: 1.330	Weighted Residual: 1.022 %	Result Emulation: Off

Concentration: 0.0133 %Vol	Span : 2.243	Uniformity: 0.759	Result units: Volume
Specific Surface Area: 1.06 m ² /g	Surface Weighted Mean D[3,2]: 5.649 um	Vol. Weighted Mean D[4,3]: 14.220 um	

d(0.1): 2.498 um d(0.5): 11.204 um d(0.9): 27.634 um



Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.63	7.096	4.04	50.238	0.25	355.656	0.00
0.022	0.00	0.159	0.00	1.125	0.72	7.962	4.47	56.368	0.10	399.052	0.00
0.025	0.00	0.178	0.00	1.262	0.81	8.934	4.88	63.246	0.05	447.744	0.00
0.028	0.00	0.200	0.00	1.416	0.91	10.024	5.24	70.963	0.07	502.377	0.00
0.032	0.00	0.224	0.00	1.589	1.01	11.247	5.52	79.621	0.12	563.677	0.00
0.036	0.00	0.252	0.00	1.783	1.11	12.619	5.69	89.337	0.17	632.456	0.00
0.040	0.00	0.283	0.00	2.000	1.23	14.159	5.72	100.237	0.20	709.627	0.00
0.045	0.00	0.317	0.00	2.244	1.35	15.887	5.59	112.468	0.21	796.214	0.00
0.050	0.00	0.356	0.00	2.518	1.35	17.825	5.29	126.191	0.18	893.367	0.00
0.056	0.00	0.399	0.04	2.825	1.48	20.000	4.84	141.589	0.09	1002.374	0.00
0.063	0.00	0.448	0.08	3.170	1.64	22.440	4.24	158.866	0.08	1124.683	0.00
0.071	0.00	0.502	0.17	3.557	1.82	25.179	3.57	178.250	0.00	1261.915	0.00
0.080	0.00	0.564	0.23	3.991	2.02	28.251	2.85	200.000	0.00	1415.892	0.00
0.089	0.00	0.632	0.30	4.477	2.24	31.696	2.14	224.404	0.00	1588.656	0.00
0.100	0.00	0.710	0.38	5.024	2.46	35.566	1.49	251.785	0.00	1782.502	0.00
0.112	0.00	0.796	0.46	5.637	2.68	39.905	0.94	282.508	0.00	2000.000	0.00
0.126	0.00	0.893	0.54	6.325	2.92	44.774	0.53	316.979	0.00		
0.142	0.00	1.002	0.63	7.096	3.22	50.238	0.53	355.656	0.00		

Operator notes: Refractory Index
Cerium Oxide 2.2



MASTERSIZER 2000

Result Analysis Report

Sample Name:
ST-A Original Bottle (Post Sonic) -

SOP Name:

Measured:
Friday, March 27, 2009 2:57:33 PM

Sample Source & type:

Measured by:
d3m514

Analysed:
Friday, March 27, 2009 2:57:34 PM

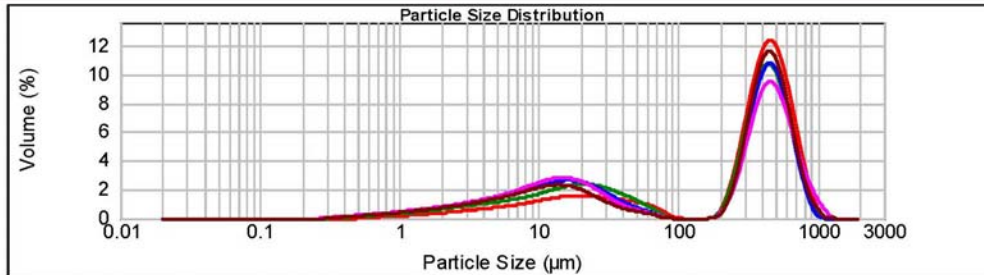
**Settler Simulant ST-A,
Effects of Sonication**

Result Source:
Averaged

Particle Name: Ferric Oxide Hydroxide	Accessory Name: Hydro 2000S (A)	Analysis model: General purpose	Sensitivity: Normal
Particle RI: 2.940	Absorption: 0	Size range: 0.020 to 2000.000 um	Obscuration: 23.13 %
Dispersant Name: Water	Dispersant RI: 1.330	Weighted Residual: 1.995 %	Result Emulation: Off

Concentration: 0.0355 % Vol	Span : 1.793	Uniformity: 0.626	Result units: Volume
Specific Surface Area: 0.529 m ² /g	Surface Weighted Mean D[3,2]: 11.345 um	Vol. Weighted Mean D[4,3]: 305.146 um	

d(0.1): 4.663 um d(0.5): 345.228 um d(0.9): 623.715 um



- ST-A Original Bottle (Pre Sonic) - Average, Friday, March 27, 2009 2:44:18 PM
 - ST-A Original Bottle (25% Sonic) - Average, Friday, March 27, 2009 2:46:54 PM
 - ST-A Original Bottle (50% Sonic) - Average, Friday, March 27, 2009 2:52:23 PM
 - ST-A Original Bottle (75% Sonic) - Average, Friday, March 27, 2009 2:55:06 PM
 - ST-A Original Bottle (Post Sonic) - Average, Friday, March 27, 2009 2:57:33 PM

Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.34	7.096	1.37	50.238	0.34	355.656	7.79
0.022	0.00	0.159	0.00	1.125	0.37	7.962	1.48	56.368	0.28	399.052	8.67
0.025	0.00	0.178	0.00	1.262	0.41	8.934	1.58	63.246	0.20	447.744	8.62
0.028	0.00	0.200	0.00	1.416	0.45	10.024	1.66	70.963	0.08	502.377	7.67
0.032	0.00	0.224	0.00	1.589	0.50	11.247	1.73	79.621	0.01	563.677	6.10
0.036	0.00	0.252	0.00	1.783	0.54	12.619	1.76	89.337	0.00	632.456	4.32
0.040	0.00	0.283	0.01	2.000	0.59	14.159	1.74	100.237	0.00	709.627	2.66
0.045	0.00	0.317	0.06	2.244	0.64	15.887	1.68	112.468	0.00	796.214	1.42
0.050	0.00	0.356	0.08	2.518	0.68	17.825	1.68	126.191	0.00	893.367	0.88
0.056	0.00	0.399	0.08	2.825	0.70	20.000	1.58	141.589	0.00	1002.374	0.23
0.063	0.00	0.448	0.10	3.170	0.75	22.440	1.43	158.866	0.00	1124.683	0.05
0.071	0.00	0.502	0.15	3.557	0.81	25.179	1.26	178.250	0.09	1261.915	0.00
0.080	0.00	0.564	0.18	3.991	0.94	28.251	0.89	200.000	0.49	1415.892	0.00
0.089	0.00	0.632	0.21	4.477	1.02	31.696	0.73	224.404	1.33	1588.656	0.00
0.100	0.00	0.710	0.24	5.024	1.09	35.566	0.59	251.785	2.67	1782.502	0.00
0.112	0.00	0.796	0.27	5.637	1.18	39.905	0.49	282.508	4.38	2000.000	0.00
0.126	0.00	0.893	0.30	6.325	1.26	44.774	0.41	316.979	6.25		
0.142	0.00	1.002	0.30	7.096	1.28	50.238	0.41	355.656	6.25		

Operator notes:



MASTERSIZER 2000

Result Analysis Report

Sample Name:
Settler Simulant (ST-A)_75% son_
Sample Source & type:
**Settler Simulant, ST-A
Number Averaged.**

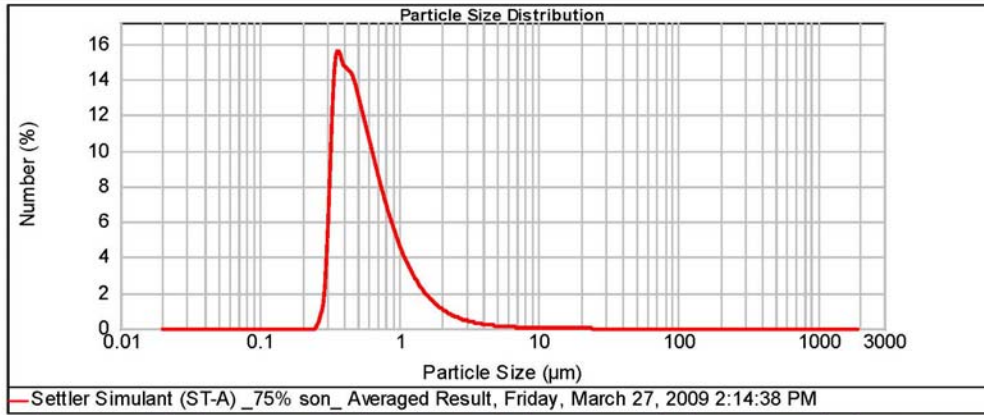
SOP Name:
Measured by:
d3m514
Result Source:
Averaged

Measured:
Friday, March 27, 2009 2:14:38 PM
Analysed:
Monday, April 27, 2009 12:36:14 AM

Particle Name: Ferric Oxide Hydroxide	Accessory Name: Hydro 2000S (A)	Analysis model: General purpose	Sensitivity: Normal
Particle RI: 2.940	Absorption: 0	Size range: 0.020 to 2000.000 um	Obscuration: 19.05 %
Dispersant Name: Water	Dispersant RI: 1.330	Weighted Residual: 1.090 %	Result Emulation: Off

Concentration: 0.0170 %Vol	Span : 1.631	Uniformity: 0.596	Result units: Number
Specific Surface Area: 1.21 m ² /g	Surface Weighted Mean D[3,2]: 4.955 um	Vol. Weighted Mean D[4,3]: 11.263 um	

d(0.1): 0.345 um d(0.5): 0.525 um d(0.9): 1.201 um



Size (µm)	Number In %	Size (µm)	Number In %	Size (µm)	Number In %	Size (µm)	Number In %	Size (µm)	Number In %	Size (µm)	Number In %
0.020	0.00	0.142	0.00	1.002	3.14	7.096	0.05	50.238	0.00	355.656	0.00
0.022	0.00	0.159	0.00	1.125	2.51	7.962	0.04	56.368	0.00	399.052	0.00
0.025	0.00	0.178	0.00	1.262	1.98	8.934	0.03	63.246	0.00	447.744	0.00
0.028	0.00	0.200	0.00	1.416	1.55	10.024	0.02	70.963	0.00	502.377	0.00
0.032	0.00	0.224	0.00	1.589	1.21	11.247	0.02	79.621	0.00	563.677	0.00
0.036	0.00	0.252	0.34	1.783	0.94	12.619	0.01	89.337	0.00	632.456	0.00
0.040	0.00	0.283	2.38	2.000	0.73	14.159	0.01	100.237	0.00	709.627	0.00
0.045	0.00	0.317	10.58	2.244	0.57	15.887	0.01	112.468	0.00	796.214	0.00
0.050	0.00	0.356	11.53	2.518	0.44	17.825	0.01	126.191	0.00	893.367	0.00
0.056	0.00	0.399	10.98	2.825	0.34	20.000	0.01	141.589	0.00	1002.374	0.00
0.063	0.00	0.448	10.48	3.170	0.27	22.440	0.01	158.866	0.00	1124.683	0.00
0.071	0.00	0.502	9.35	3.557	0.21	25.179	0.00	178.250	0.00	1261.915	0.00
0.080	0.00	0.564	8.17	3.991	0.16	28.251	0.00	200.000	0.00	1415.892	0.00
0.089	0.00	0.632	6.98	4.477	0.13	31.698	0.00	224.404	0.00	1588.656	0.00
0.100	0.00	0.710	5.84	5.024	0.10	35.586	0.00	251.785	0.00	1782.502	0.00
0.112	0.00	0.796	4.81	5.637	0.08	39.905	0.00	282.508	0.00	2000.000	0.00
0.126	0.00	0.893	3.91	6.325	0.07	44.774	0.00	316.979	0.00		
0.142	0.00	1.002		7.096		50.238	0.00	355.656	0.00		

Operator notes: Average of 14 measurements from 4 different samples at 75 % sonication



MASTERSIZER 2000

Result Analysis Report

Sample Name:
Settled KW-B M500 (75% Sonic) - Average

SOP Name:

Measured:
Monday, March 02, 2009 3:37:08 PM

Sample Source & type:

Measured by:
D3M514

Analysed:
Monday, March 02, 2009 3:37:09 PM

KW_B M500 Data used in Report

Result Source:
Averaged

Particle Name:
Ferric Oxide Hydroxide

Accessory Name:
Hydro 2000S (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
2.940

Absorption:
0

Size range:
0.020 to 2000.000 μm

Obscuration:
20.37 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.391 %

Result Emulation:
Off

Concentration:
0.0179 %Vol

Span :
13.864

Uniformity:
3.62

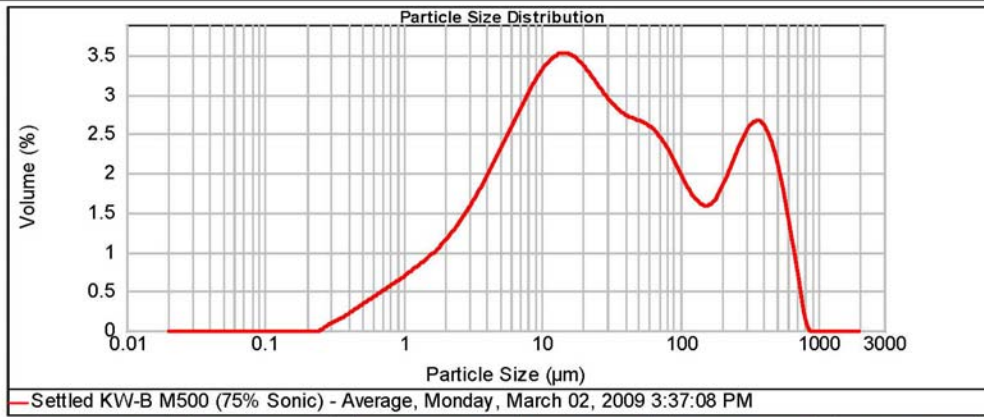
Result units:
Volume

Specific Surface Area:
0.887 m^2/g

Surface Weighted Mean D[3,2]:
6.766 μm

Vol. Weighted Mean D[4,3]:
98.370 μm

d(0.1): 2.823 μm d(0.5): 24.608 μm d(0.9): 343.992 μm



Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.54	7.096	2.21	50.238	2.00	355.656	2.01
0.022	0.00	0.159	0.00	1.125	0.59	7.962	2.34	56.368	1.96	399.052	1.93
0.025	0.00	0.178	0.00	1.262	0.64	8.934	2.45	63.246	1.90	447.744	1.76
0.028	0.00	0.200	0.00	1.416	0.69	10.024	2.54	70.963	1.81	502.377	1.49
0.032	0.00	0.224	0.00	1.589	0.75	11.247	2.61	79.621	1.70	563.677	1.13
0.036	0.00	0.252	0.02	1.783	0.82	12.619	2.65	89.337	1.57	632.456	0.76
0.040	0.00	0.283	0.07	2.000	0.89	14.159	2.66	100.237	1.44	709.627	0.34
0.045	0.00	0.317	0.10	2.244	0.98	15.887	2.63	112.468	1.32	796.214	0.01
0.050	0.00	0.356	0.14	2.518	1.07	17.825	2.58	126.191	1.23	893.367	0.00
0.056	0.00	0.399	0.18	2.825	1.18	20.000	2.50	141.589	1.19	1002.374	0.00
0.063	0.00	0.448	0.23	3.170	1.29	22.440	2.41	158.866	1.21	1124.683	0.00
0.071	0.00	0.502	0.27	3.557	1.41	25.179	2.32	178.250	1.29	1261.915	0.00
0.080	0.00	0.564	0.32	3.991	1.53	28.251	2.23	200.000	1.42	1415.892	0.00
0.089	0.00	0.632	0.36	4.477	1.67	31.696	2.15	224.404	1.58	1588.656	0.00
0.100	0.00	0.710	0.41	5.024	1.80	35.566	2.09	251.785	1.75	1782.502	0.00
0.112	0.00	0.796	0.45	5.637	1.94	39.905	2.05	282.508	1.89	2000.000	0.00
0.126	0.00	0.893	0.49	6.325	2.08	44.774	2.02	316.979	1.99		
0.142	0.00	1.002	0.54	7.096	2.21	50.238	2.00	355.656	1.99		

Operator notes: Used FeOH Refractive Index 2.94



Result Analysis Report

Sample Name:
Settled KW-B M500 (75% Sonic) - Average

SOP Name:

Measured:
Monday, March 02, 2009 3:37:08 PM

Sample Source & type:

Measured by:
D3M514

Analysed:
Monday, March 02, 2009 3:37:09 PM

KW_B M500 Dry sample and settled sample

Result Source:
Averaged

Particle Name:
Ferric Oxide Hydroxide

Accessory Name:
Hydro 2000S (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
2.940

Absorption:
0

Size range:
0.020 to 2000.000 um

Obscuration:
20.37 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.391 %

Result Emulation:
Off

Concentration:
0.0179 %Vol

Span :
13.864

Uniformity:
3.62

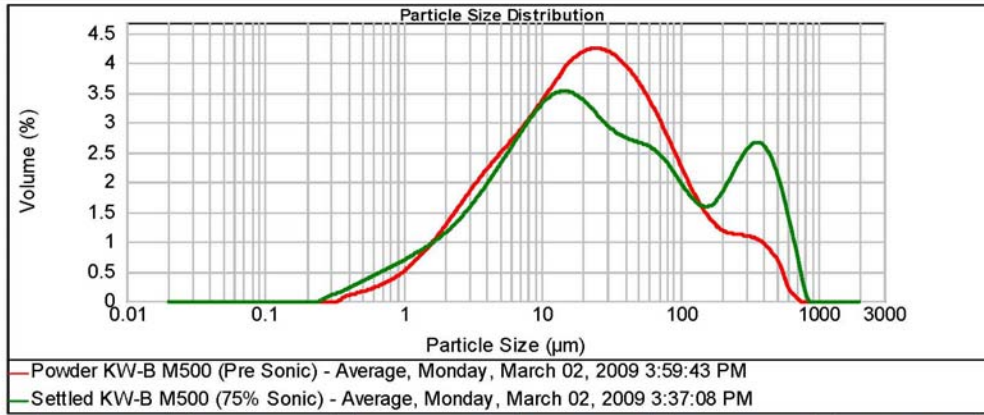
Result units:
Volume

Specific Surface Area:
0.887 m²/g

Surface Weighted Mean D[3,2]:
6.766 um

Vol. Weighted Mean D[4,3]:
98.370 um

d(0.1): 2.823 um d(0.5): 24.608 um d(0.9): 343.992 um



Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.54	7.096	2.21	50.238	2.00	355.656	2.01
0.022	0.00	0.159	0.00	1.125	0.59	7.962	2.34	56.368	1.96	399.052	1.93
0.025	0.00	0.178	0.00	1.262	0.64	8.934	2.45	63.246	1.90	447.744	1.76
0.028	0.00	0.200	0.00	1.416	0.69	10.024	2.54	70.963	1.81	502.377	1.49
0.032	0.00	0.224	0.00	1.589	0.75	11.247	2.61	79.621	1.70	563.677	1.13
0.036	0.00	0.252	0.02	1.783	0.82	12.619	2.65	89.337	1.57	632.456	0.76
0.040	0.00	0.283	0.07	2.000	0.89	14.159	2.66	100.237	1.44	709.627	0.34
0.045	0.00	0.317	0.10	2.244	0.98	15.887	2.63	112.468	1.32	796.214	0.01
0.050	0.00	0.356	0.14	2.518	1.07	17.825	2.58	126.191	1.19	893.367	0.00
0.056	0.00	0.399	0.18	2.825	1.18	20.000	2.50	141.589	1.07	1002.374	0.00
0.063	0.00	0.448	0.23	3.170	1.29	22.440	2.41	158.866	0.96	1124.683	0.00
0.071	0.00	0.502	0.27	3.557	1.41	25.179	2.32	178.250	0.86	1261.915	0.00
0.080	0.00	0.564	0.32	3.991	1.53	28.251	2.23	200.000	0.77	1415.892	0.00
0.089	0.00	0.632	0.36	4.477	1.67	31.696	2.15	224.404	0.69	1588.656	0.00
0.100	0.00	0.710	0.41	5.024	1.80	35.566	2.09	251.785	0.62	1782.502	0.00
0.112	0.00	0.796	0.45	5.637	1.94	39.905	2.05	282.508	0.56	2000.000	0.00
0.126	0.00	0.893	0.49	6.325	2.08	44.774	2.02	316.979	0.51		
0.142	0.00	1.002	0.54	7.096	2.21	50.238	2.00	355.656	1.99		

Operator notes: Used FeOH Refractive Index 2.94



Result Analysis Report

Sample Name:
Settled KW-B M500 (75% Sonic) - Average

SOP Name:

Measured:
Monday, March 02, 2009 3:37:08 PM

Sample Source & type:

Measured by:
D3M514

Analysed:
Monday, March 02, 2009 3:37:09 PM

KW_B M500 Effects of RI.

Result Source:
Averaged

Particle Name:
Ferric Oxide Hydroxide

Accessory Name:
Hydro 2000S (A)

Analysis model:
General purpose

Sensitivity:
Normal

Particle RI:
2.940

Absorption:
0

Size range:
0.020 to 2000.000 um

Obscuration:
20.37 %

Dispersant Name:
Water

Dispersant RI:
1.330

Weighted Residual:
0.391 %

Result Emulation:
Off

Concentration:
0.0179 %Vol

Span :
13.864

Uniformity:
3.62

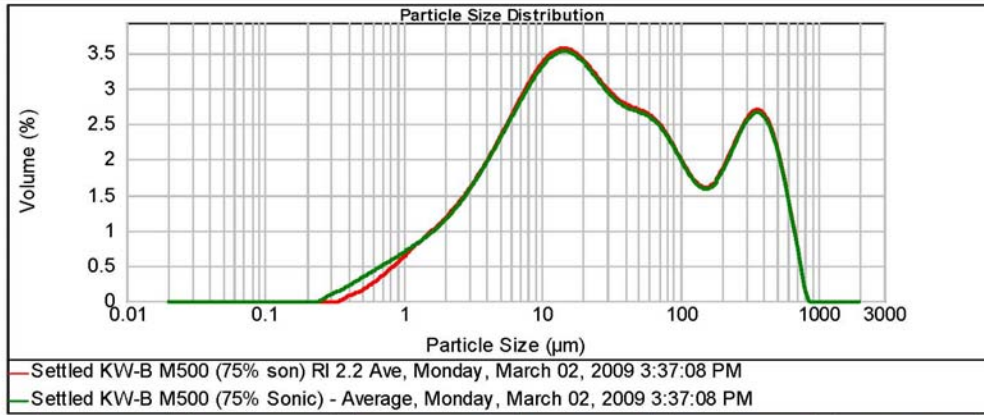
Result units:
Volume

Specific Surface Area:
0.887 m²/g

Surface Weighted Mean D[3,2]:
6.766 um

Vol. Weighted Mean D[4,3]:
98.370 um

d(0.1): 2.823 um d(0.5): 24.608 um d(0.9): 343.992 um



Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.54	7.096	2.21	50.238	2.00	355.656	2.01
0.022	0.00	0.159	0.00	1.125	0.59	7.962	2.34	56.368	1.96	399.052	1.93
0.025	0.00	0.178	0.00	1.262	0.64	8.934	2.45	63.246	1.90	447.744	1.76
0.028	0.00	0.200	0.00	1.416	0.69	10.024	2.54	70.963	1.81	502.377	1.49
0.032	0.00	0.224	0.00	1.589	0.75	11.247	2.61	79.621	1.70	563.677	1.13
0.036	0.00	0.252	0.02	1.783	0.82	12.619	2.65	89.337	1.57	632.456	0.76
0.040	0.00	0.283	0.07	2.000	0.89	14.159	2.66	100.237	1.44	709.627	0.34
0.045	0.00	0.317	0.10	2.244	0.98	15.887	2.63	112.468	1.32	796.214	0.01
0.050	0.00	0.356	0.14	2.518	1.07	17.825	2.58	126.191	1.19	893.367	0.00
0.056	0.00	0.399	0.18	2.825	1.18	20.000	2.50	141.589	1.07	1002.374	0.00
0.063	0.00	0.448	0.23	3.170	1.29	22.440	2.41	158.866	0.96	1124.683	0.00
0.071	0.00	0.502	0.27	3.557	1.41	25.179	2.32	178.250	0.86	1261.915	0.00
0.080	0.00	0.564	0.32	3.991	1.53	28.251	2.23	200.000	0.77	1415.892	0.00
0.089	0.00	0.632	0.36	4.477	1.67	31.696	2.15	224.404	0.69	1588.656	0.00
0.100	0.00	0.710	0.41	5.024	1.80	35.566	2.09	251.785	0.62	1782.502	0.00
0.112	0.00	0.796	0.45	5.637	1.94	39.905	2.05	282.508	0.56	2000.000	0.00
0.126	0.00	0.893	0.49	6.325	2.08	44.774	2.02	316.979	0.51		
0.142	0.00	1.002	0.54	7.096	2.21	50.238	2.00	355.656	1.99		

Operator notes: Used FeOH Refractive Index 2.94



Result Analysis Report

Sample Name:
Settled KW-B M500 (Post Sonic) - Average

Sample Source & type:

KW_B M500 Effects of sonication

SOP Name:

Measured by:
D3M514

Result Source:
Averaged

Measured:
Monday, March 02, 2009 3:41:04 PM

Analysed:
Monday, March 02, 2009 3:41:05 PM

Particle Name:
Ferric Oxide Hydroxide

Particle RI:
2.940

Dispersant Name:
Water

Accessory Name:
Hydro 2000S (A)

Absorption:
0

Dispersant RI:
1.330

Analysis model:
General purpose

Size range:
0.020 to 2000.000 um

Weighted Residual:
0.395 %

Sensitivity:
Normal

Obscuration:
20.77 %

Result Emulation:
Off

Concentration:
0.0194 %Vol

Span :
14.900

Uniformity:
3.98

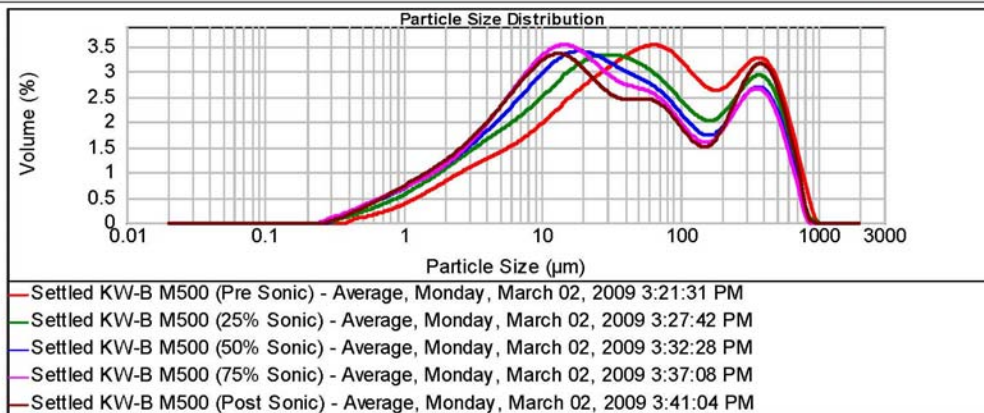
Result units:
Volume

Specific Surface Area:
0.856 m²/g

Surface Weighted Mean D[3,2]:
7.005 um

Vol. Weighted Mean D[4,3]:
113.555 um

d(0.1): 2.753 um d(0.5): 26.182 um d(0.9): 392.864 um



Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.020	0.00	0.142	0.00	1.002	0.57	7.096	2.17	50.238	1.85	355.656	2.38
0.022	0.00	0.159	0.00	1.125	0.63	7.962	2.29	56.368	1.84	399.052	2.34
0.025	0.00	0.178	0.00	1.262	0.69	8.934	2.39	63.246	1.81	447.744	2.18
0.028	0.00	0.200	0.00	1.416	0.75	10.024	2.46	70.963	1.74	502.377	1.90
0.032	0.00	0.224	0.00	1.589	0.82	11.247	2.51	79.621	1.64	563.677	1.50
0.036	0.00	0.252	0.00	1.783	0.89	12.619	2.52	89.337	1.51	632.456	1.08
0.040	0.00	0.283	0.02	2.000	0.96	14.159	2.50	100.237	1.37	709.627	0.59
0.045	0.00	0.317	0.06	2.244	1.04	15.887	2.45	112.468	1.25	796.214	0.07
0.050	0.00	0.356	0.10	2.518	1.13	17.825	2.36	126.191	1.16	893.367	0.00
0.056	0.00	0.399	0.15	2.825	1.22	20.000	2.26	141.589	1.13	1002.374	0.00
0.063	0.00	0.448	0.19	3.170	1.32	22.440	2.15	158.866	1.17	1124.683	0.00
0.071	0.00	0.502	0.24	3.557	1.43	25.179	2.05	178.250	1.28	1261.915	0.00
0.080	0.00	0.564	0.30	3.991	1.55	28.251	1.96	200.000	1.46	1415.892	0.00
0.089	0.00	0.632	0.35	4.477	1.67	31.696	1.89	224.404	1.68	1588.656	0.00
0.100	0.00	0.710	0.40	5.024	1.79	35.566	1.85	251.785	1.92	1782.502	0.00
0.112	0.00	0.796	0.46	5.637	1.92	39.905	1.84	282.508	2.13	2000.000	0.00
0.126	0.00	0.893	0.51	6.325	2.05	44.774	1.84	316.979	2.30		
0.142	0.00	1.002	0.57	7.096	2.17	50.238	1.84	355.656	2.30		

Operator notes: Used FeOH Refractive Index 2.94



MASTERSIZER 2000

Result Analysis Report

Sample Name:
KW-B M500 75% son Number Averaged

SOP Name:

Measured:
Monday, March 02, 2009 3:37:08 PM

Sample Source & type:

Measured by:
D3M514

Analysed:
Monday, April 27, 2009 2:33:27 AM

KW_B M500 Number Averaged.

Result Source:
Averaged

Particle Name:
Ferric Oxide Hydroxide
Particle RI:
2.940
Dispersant Name:
Water

Accessory Name:
Hydro 2000S (A)
Absorption:
0
Dispersant RI:
1.330

Analysis model:
General purpose
Size range:
0.020 to 2000.000 um
Weighted Residual:
0.391 %

Sensitivity:
Normal
Obscuration:
20.37 %
Result Emulation:
Off

Concentration:
0.0179 %Vol

Span :
1.467

Uniformity:
0.533

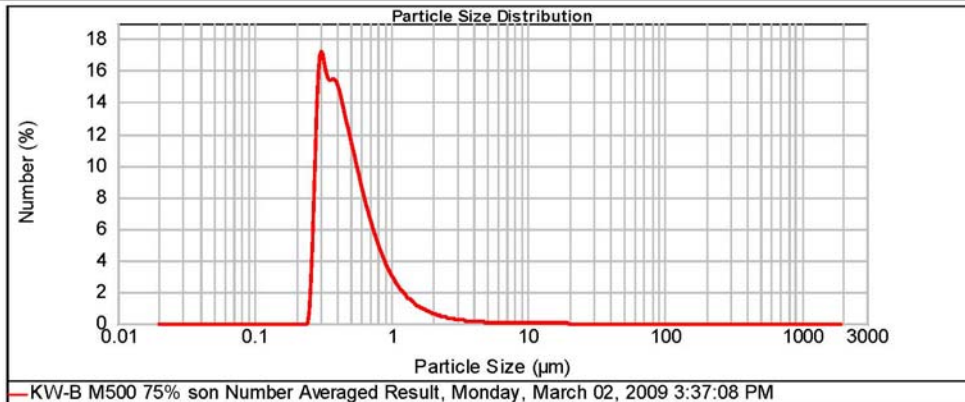
Result units:
Number

Specific Surface Area:
1.73 m²/g

Surface Weighted Mean D[3,2]:
3.467 um

Vol. Weighted Mean D[4,3]:
8.525 um

d(0.1): 0.299 um d(0.5): 0.441 um d(0.9): 0.946 um



Size (µm)	Number In %	Size (µm)	Number In %	Size (µm)	Number In %	Size (µm)	Number In %	Size (µm)	Number In %	Size (µm)	Number In %
0.020	0.00	0.142	0.00	1.002	2.02	7.096	0.02	50.238	0.00	355.656	0.00
0.022	0.00	0.159	0.00	1.125	1.55	7.962	0.02	56.368	0.00	399.052	0.00
0.025	0.00	0.178	0.00	1.262	1.19	8.934	0.01	63.246	0.00	447.744	0.00
0.028	0.00	0.200	0.00	1.416	0.92	10.024	0.01	70.963	0.00	502.377	0.00
0.032	0.00	0.224	0.00	1.589	0.70	11.247	0.01	79.621	0.00	563.677	0.00
0.036	0.00	0.252	0.00	1.783	0.54	12.619	0.01	89.337	0.00	632.456	0.00
0.040	0.00	0.283	3.87	2.000	0.42	14.159	0.00	100.237	0.00	709.627	0.00
0.045	0.00	0.317	13.14	2.244	0.33	15.887	0.00	112.468	0.00	796.214	0.00
0.050	0.00	0.356	11.73	2.518	0.25	17.825	0.01	126.191	0.00	893.367	0.00
0.056	0.00	0.399	11.68	2.825	0.20	20.000	0.00	141.589	0.00	1002.374	0.00
0.063	0.00	0.448	10.93	3.170	0.15	22.440	0.00	158.866	0.00	1124.683	0.00
0.071	0.00	0.502	9.47	3.557	0.12	25.179	0.00	178.250	0.00	1261.915	0.00
0.080	0.00	0.564	8.09	3.991	0.09	28.251	0.00	200.000	0.00	1415.892	0.00
0.089	0.00	0.632	6.67	4.477	0.07	31.698	0.00	224.404	0.00	1588.656	0.00
0.100	0.00	0.710	5.40	5.024	0.05	35.566	0.00	251.785	0.00	1782.502	0.00
0.112	0.00	0.798	4.29	5.637	0.04	39.905	0.00	282.508	0.00	2000.000	0.00
0.126	0.00	0.893	3.36	6.325	0.03	44.774	0.00	316.979	0.00		
0.142	0.00	1.002	2.61	7.096	0.03	50.238	0.00	355.656	0.00		

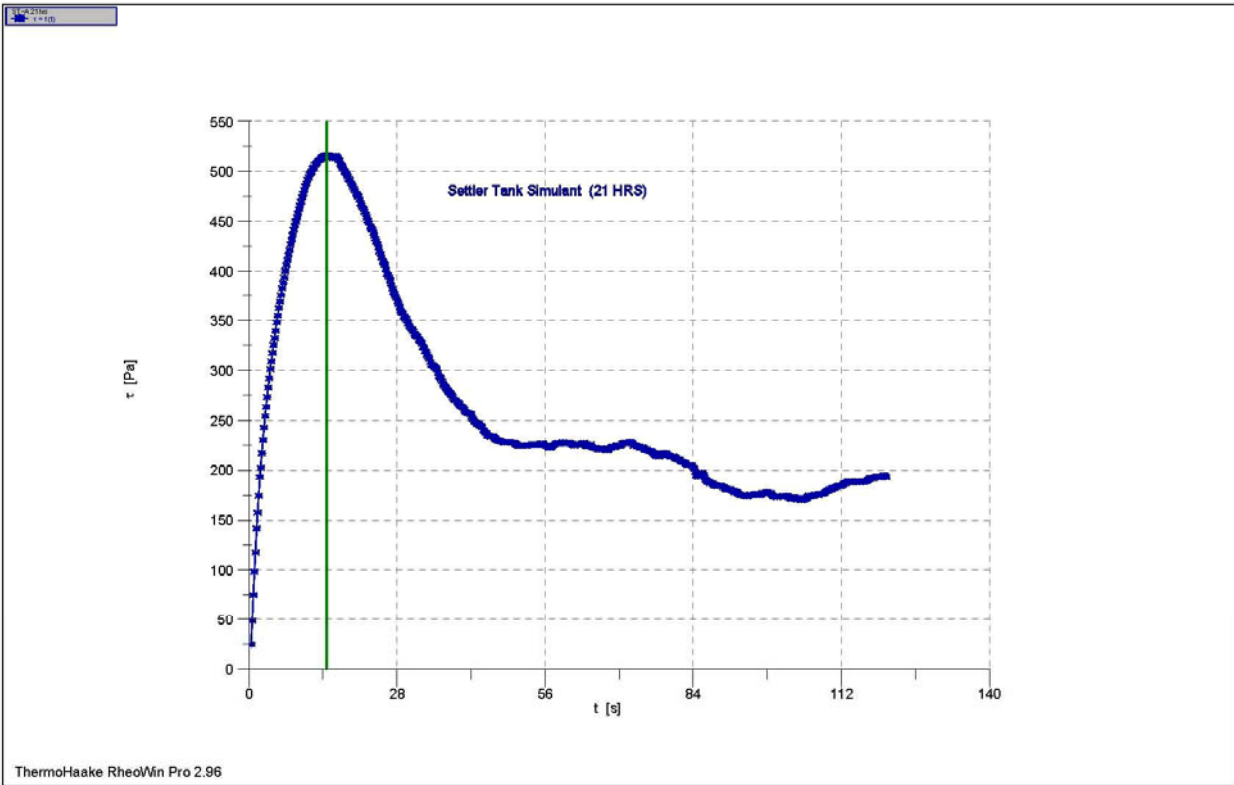
Operator notes: Average of 5 measurements

Appendix C

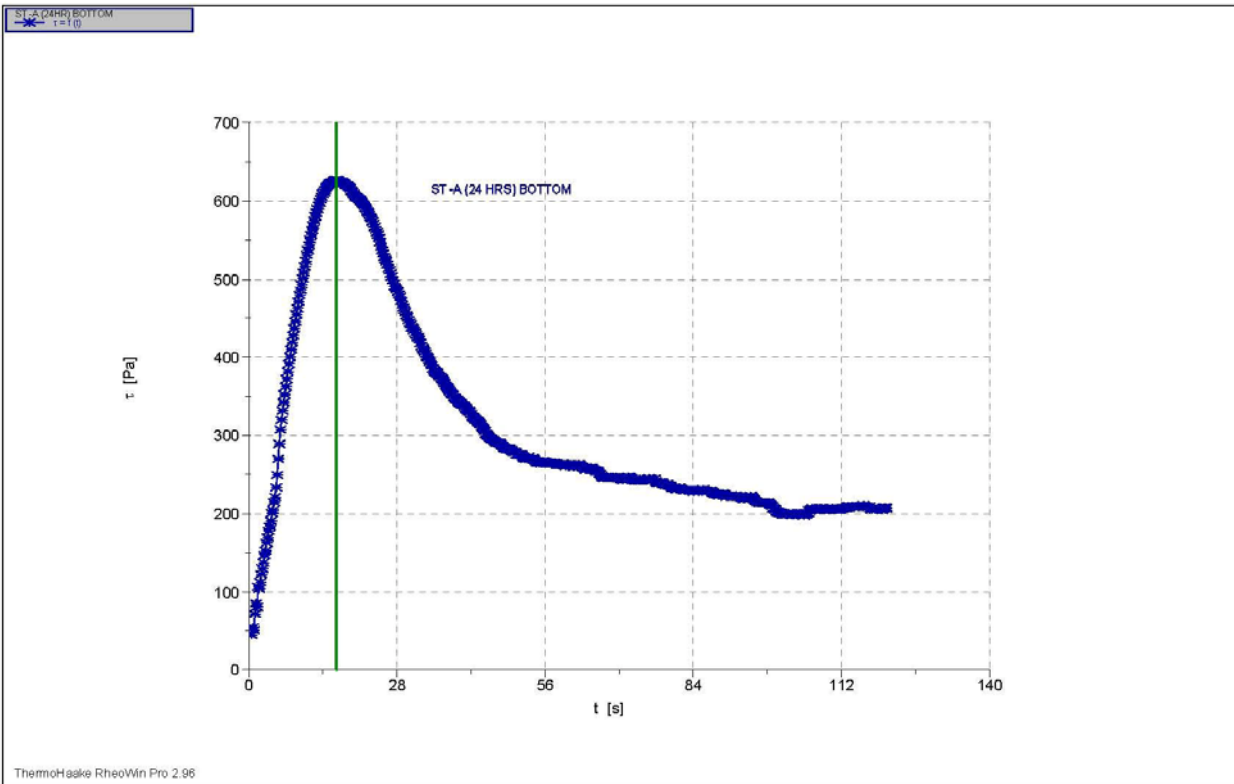
Shear Strength vs. Time and Rheograms

Appendix C: Rheology Plots

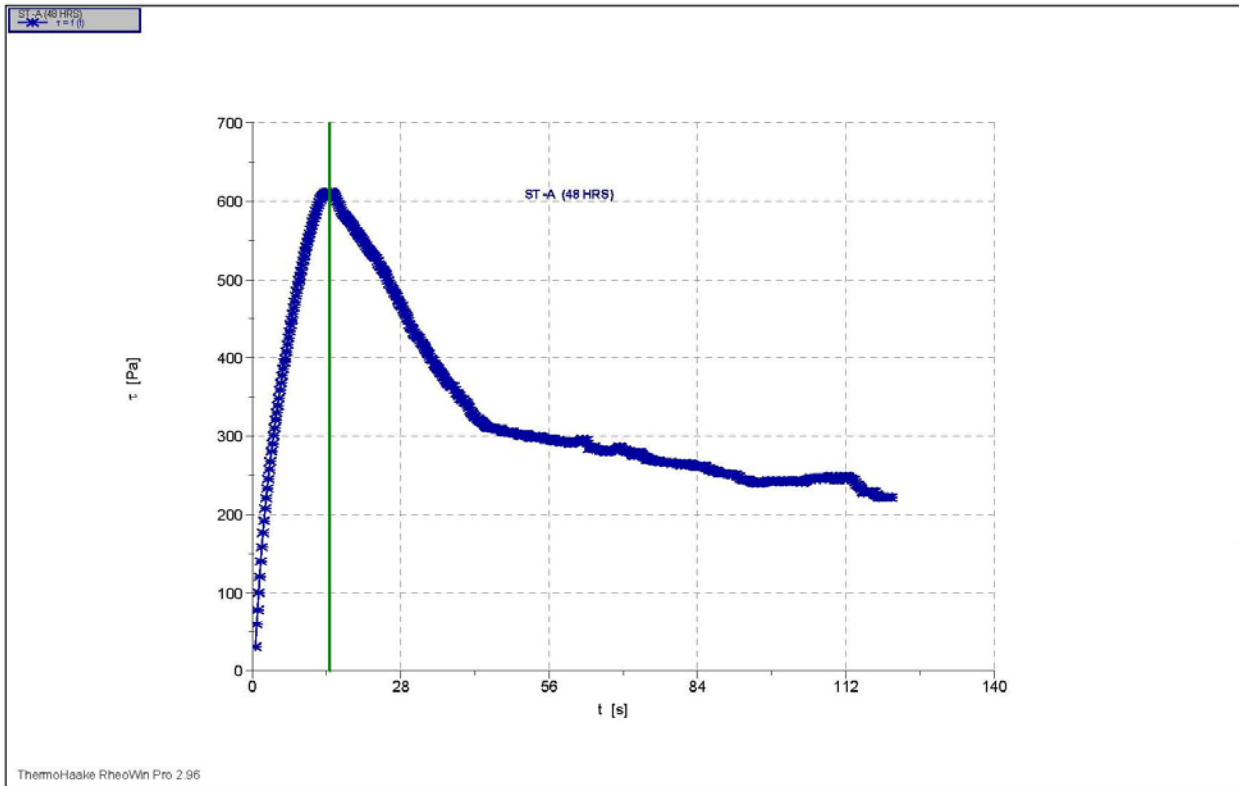
Shear-strength values were obtained from stress/time profiles that are given in this appendix. The flow curves used to obtain yield stress and viscosity values for both simulants are also included along with their corresponding viscosity plots (triplicate analysis). All data have been discussed and summarized in Section 3.3.



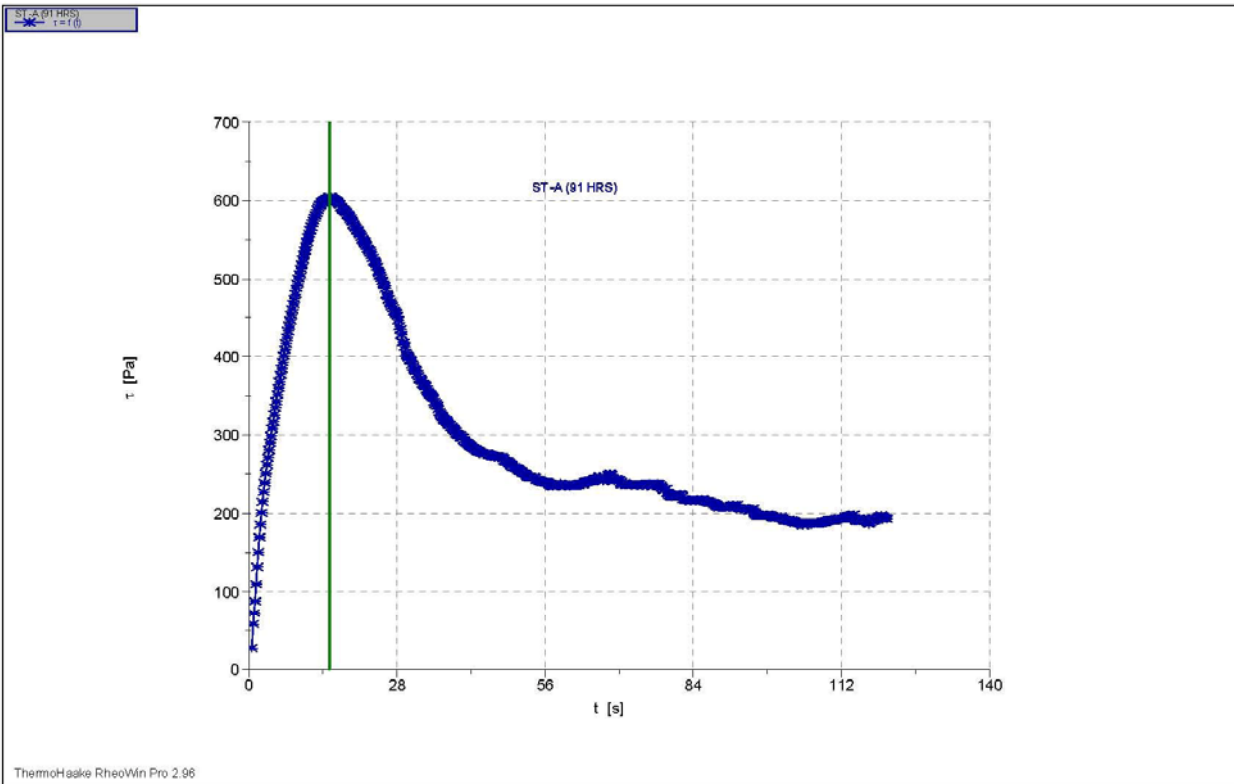
1: C:\Rheology Results\53019-M12\Shear Strength 1.6 X 1.6 cm Vane\ST-A 21hrs.rwd
Company / Operator: PNNL / MARIA LUNA
Date / Time / Version: 03.03.2009 / 13:04:34 PM / RheoWin Pro 296
Substance / Sample no: 090303 Settler Tank Simulant 21 hrs / ST-A 21hrs
Curve discussion: Greatest value t [s] 14.67 τ [Pa] 516.4



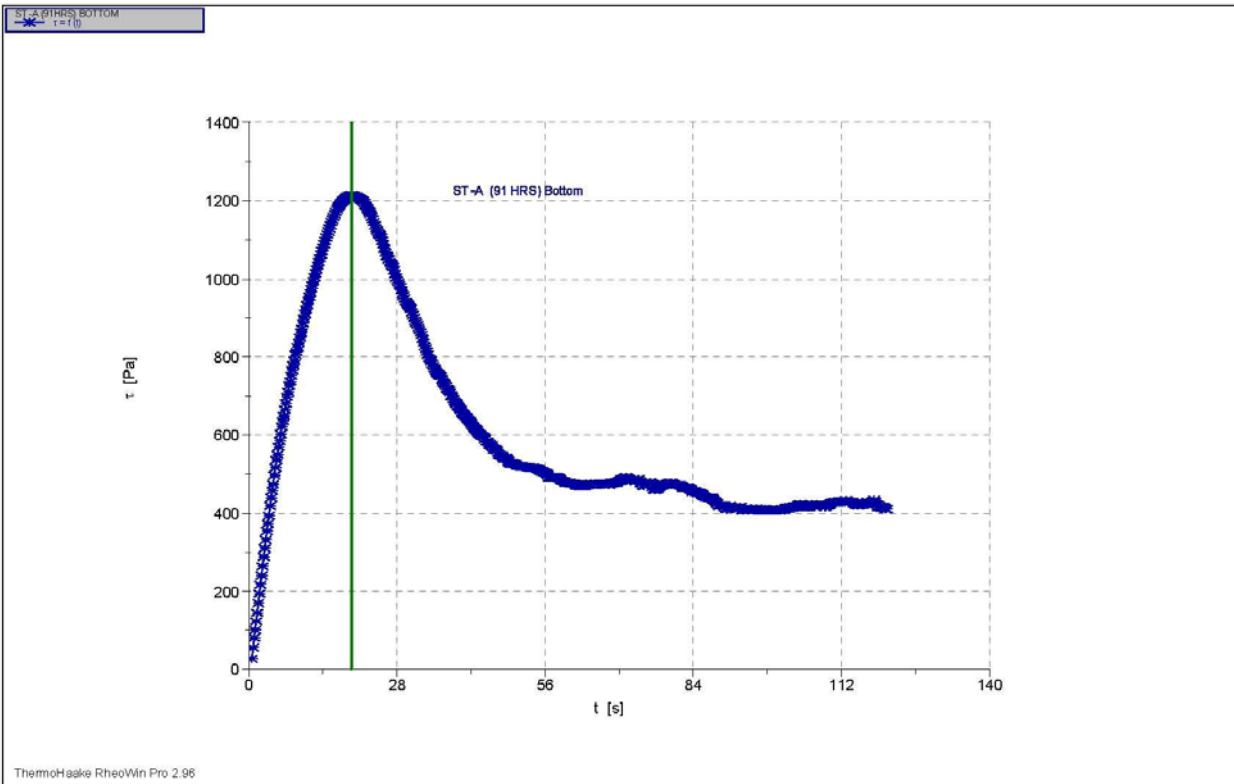
1: C:\Rheology Results\K-Basin\Shear Strength (vane)\ST-A (24HR) BOTTOM.rwd
Company / Operator: PNNL / MARIA LUNA
Date / Time / Version: 11.03.2009 / 15:24:28 PM / RheoWin Pro 296
Substance / Sample no: ST-A (24HR) / ST-A (24HR)
Curve discussion: Greatest value t [s] 16.56 τ [Pa] 627.3



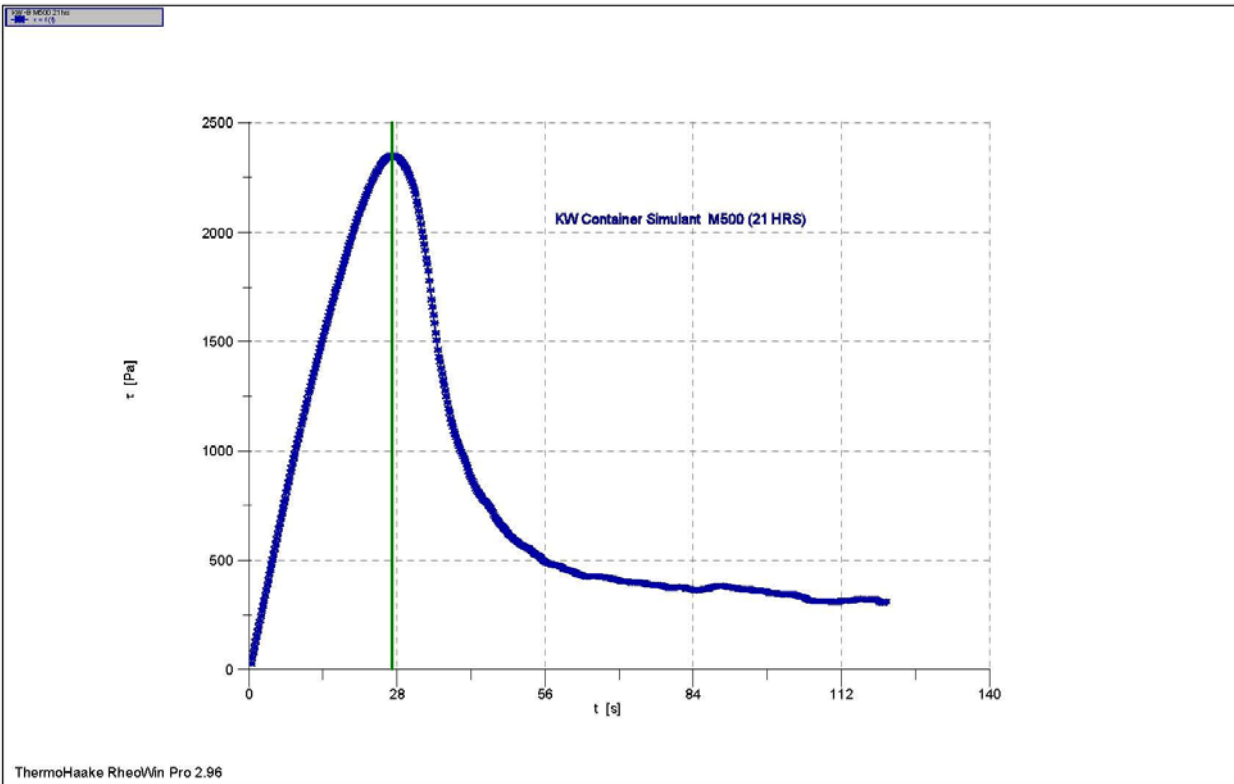
1: C:\Rheology Results\53019 -M12\Shear Strength 1.6 X 1.6 cm Vane\ST-A (48 HRS).rwd
Company / Operator: PNNL / MARIA LUNA
Date / Time / Version: 05.03.2009 / 15:58:51 PM / RheoWin Pro 296
Substance / Sample no: ST-A (48 HRS) / ST-A (48 HRS)
Curve discussion: Greatest value t [s] 14.60 τ [Pa] 612.4



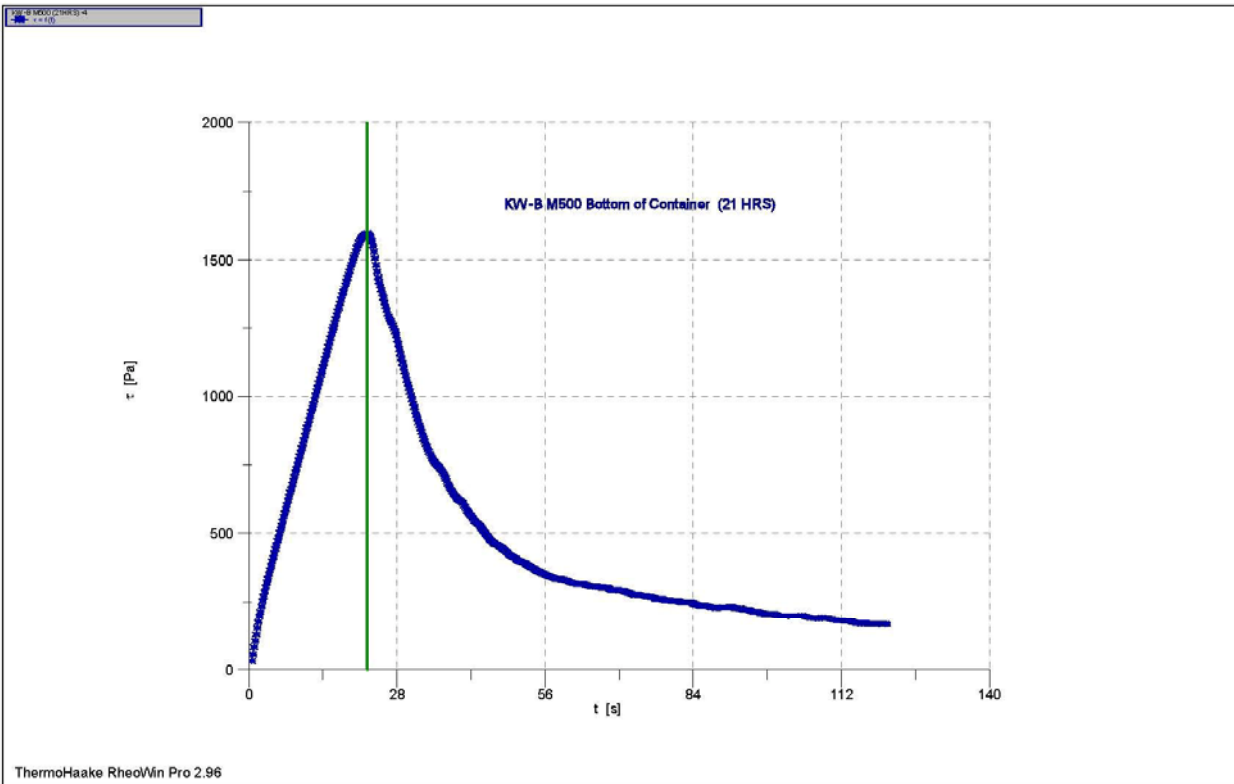
1: C:\Rheology Results\53019 -M12\Shear Strength 1.6 X 1.6 cm Vane\ST-A (91 HRS).rwd
Company / Operator: PNNL / MARIA LUNA
Date / Time / Version: 09.03.2009 / 12:47:34 PM / RheoWin Pro 296
Substance / Sample no: ST-A (91 HRS) / ST-A (91 HRS)
Curve discussion: Greatest value t [s] 15.22 τ [Pa] 604.3



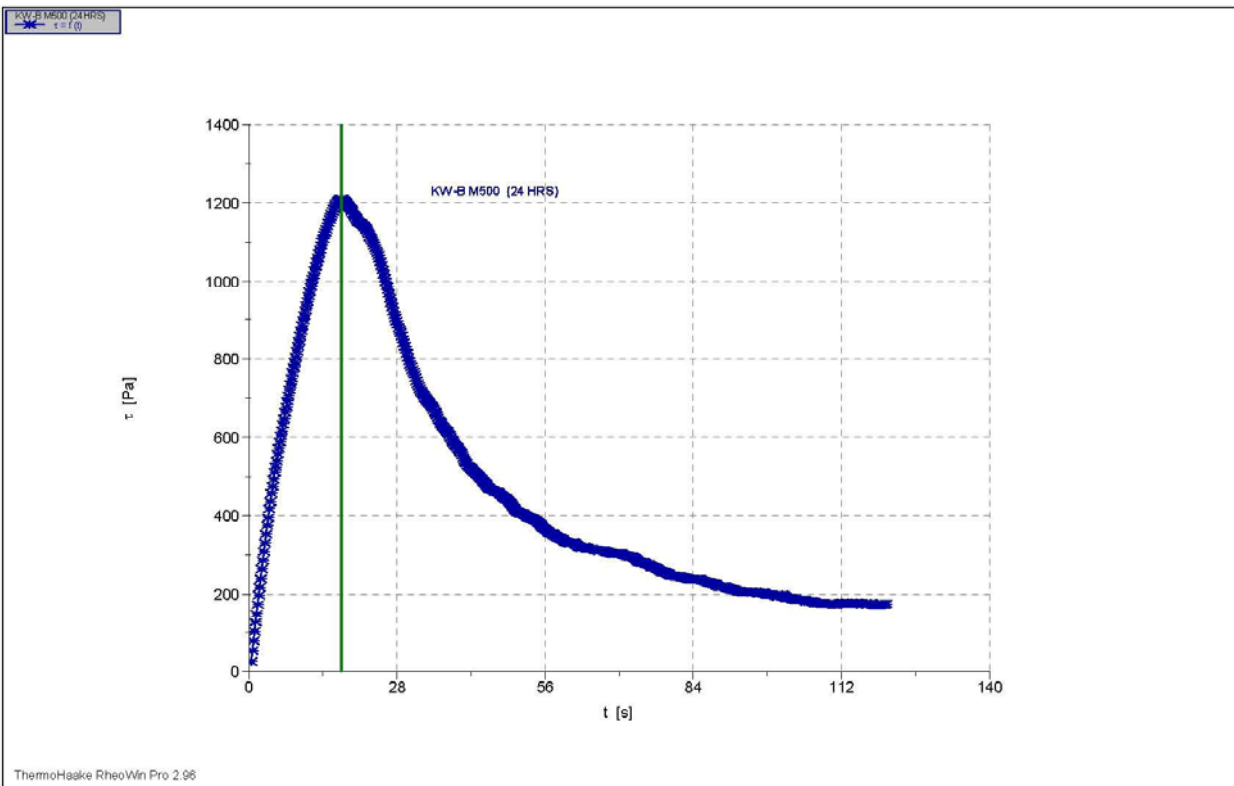
1: C:\Rheology Results\K-Basin\Shear Strength (vane)\ST-A (91HRS) BOTTOM.rwd
Company / Operator: PNNL / MARIA LUNA
Date / Time / Version: 09.03.2009 / 15:11:11 PM / RheoWin Pro 296
Substance / Sample no: ST-A (91HRS) BOTTOM / ST-A (91HRS) BOTTOM
Curve discussion: Greatest value t [s] 19.40 τ [Pa] 1214.



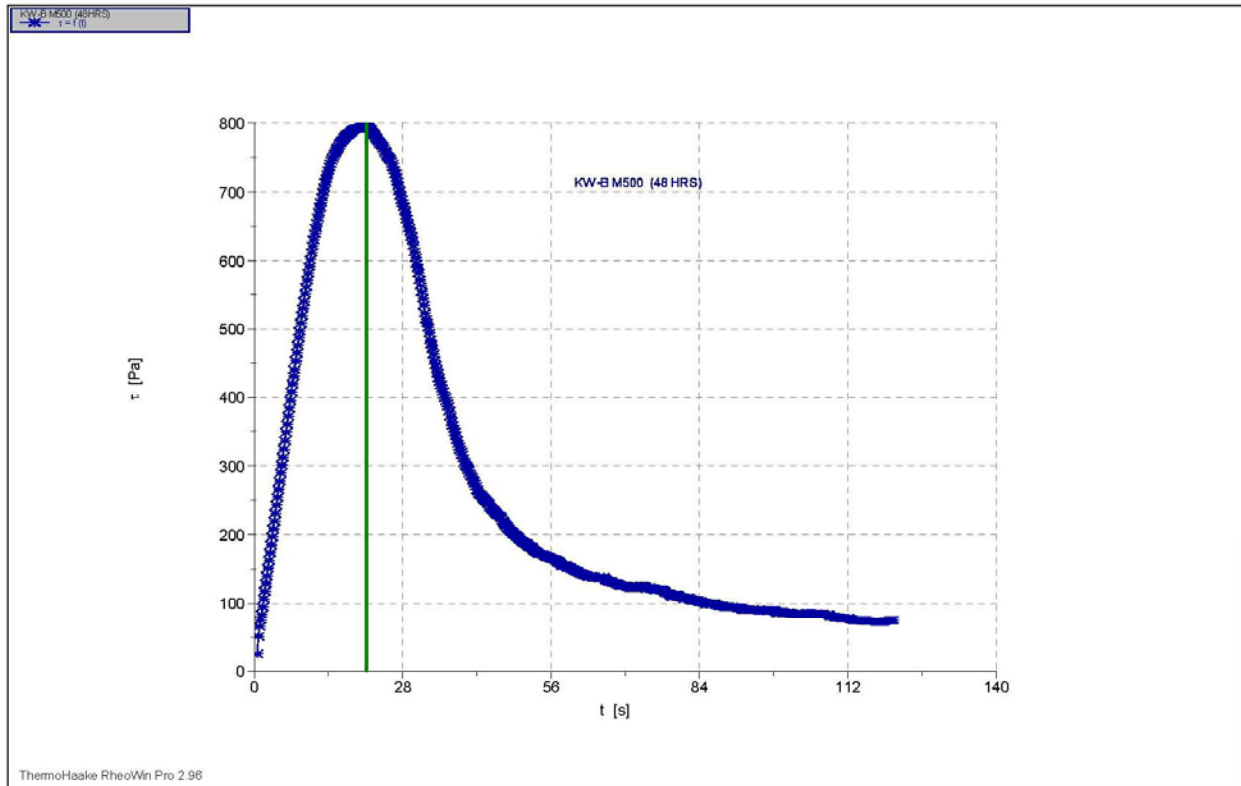
1: C:\Rheology Results\53019 -M12\Shear Strength 1.6 X 1.6 cm Vane\KW-B M500 21hrs.rwd
Company / Operator: PNNL / MARIA LUNA
Date / Time / Version: 03.03.2009 / 13:09:22 PM / RheoWin Pro 296
Substance / Sample no: 090303 KW Container Simulant 21 hrs / KW-B M500 21hrs
Curve discussion: Greatest value t [s] 27.08 τ [Pa] 2350.



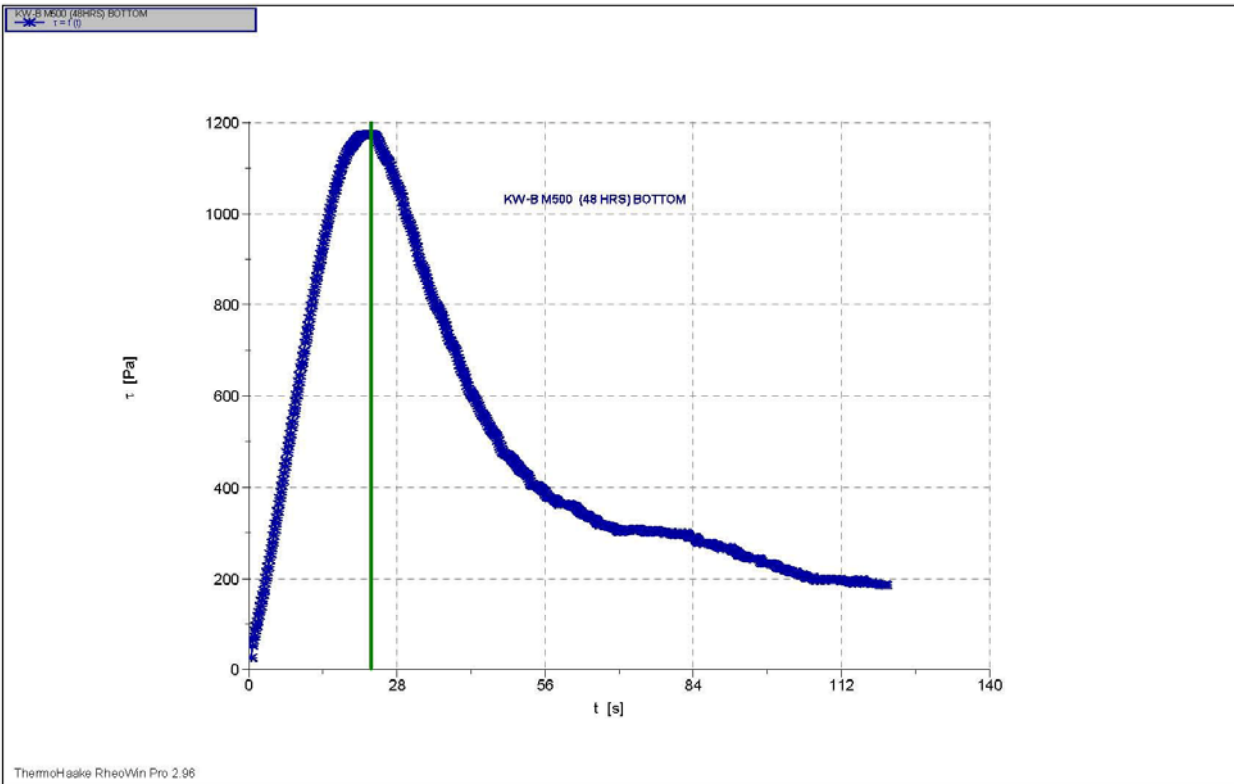
1: C:\Rheology Results\53019 -M12\Shear Strength 1.6 X 1.6 cm Vane\KW-B M500 (21HRS) -4.rwd
Company / Operator: PNNL / MARIA LUNA
Date / Time / Version: 05.03.2009 / 10:44:15 AM / RheoWin Pro 296
Substance / Sample no: KW-B M500 (21HRS) -4 / KW-B M500 (21HRS) -4
Curve discussion: Greatest value t [s] 22.38 τ [Pa] 1597.



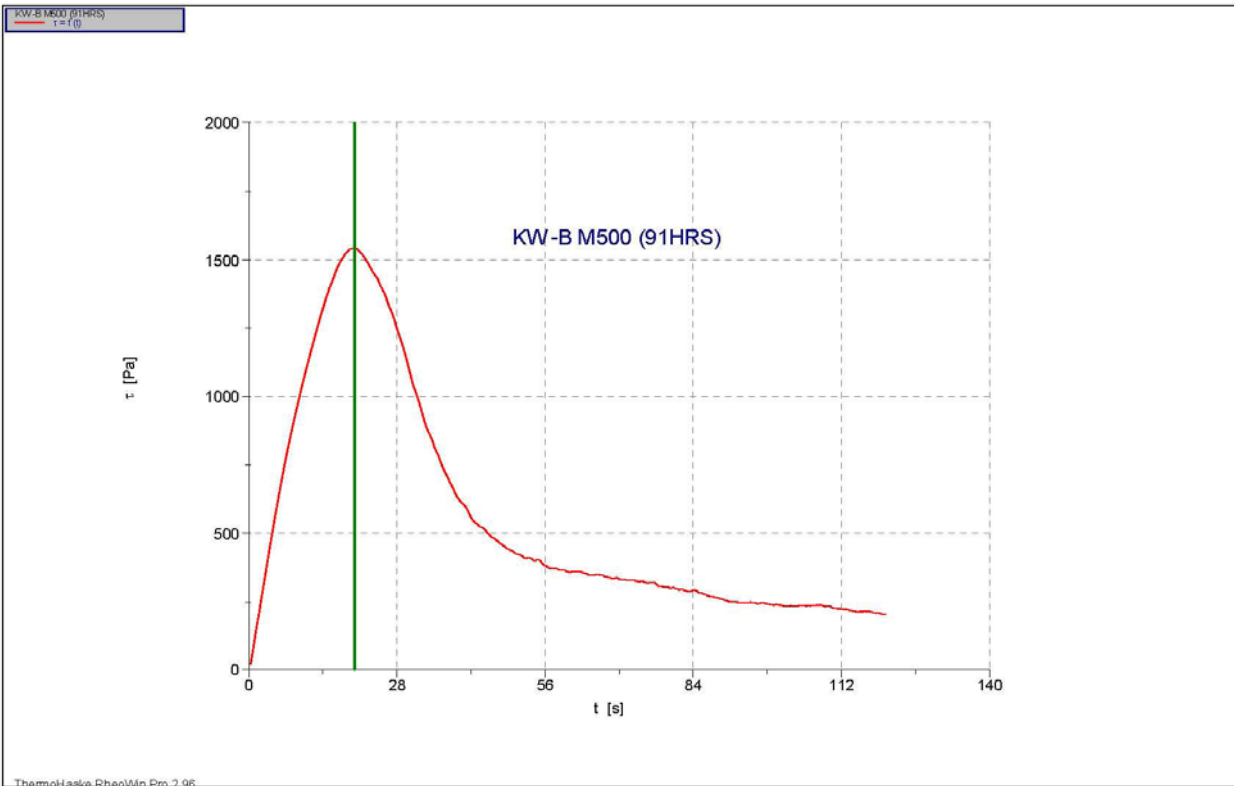
1: C:\Rheology Results\K-Basin\Shear Strength (vane)\KW-B M500 (24HRS).rwd
Company / Operator: PNNL / MARIA LUNA
Date / Time / Version: 06.03.2009 / 11:59:28 AM / RheoWin Pro 296
Substance / Sample no: KW-B M500 (24HRS) / KW-B M500 (24HRS)
Curve discussion: Greatest value t [s] 17.49 τ [Pa] 1209.



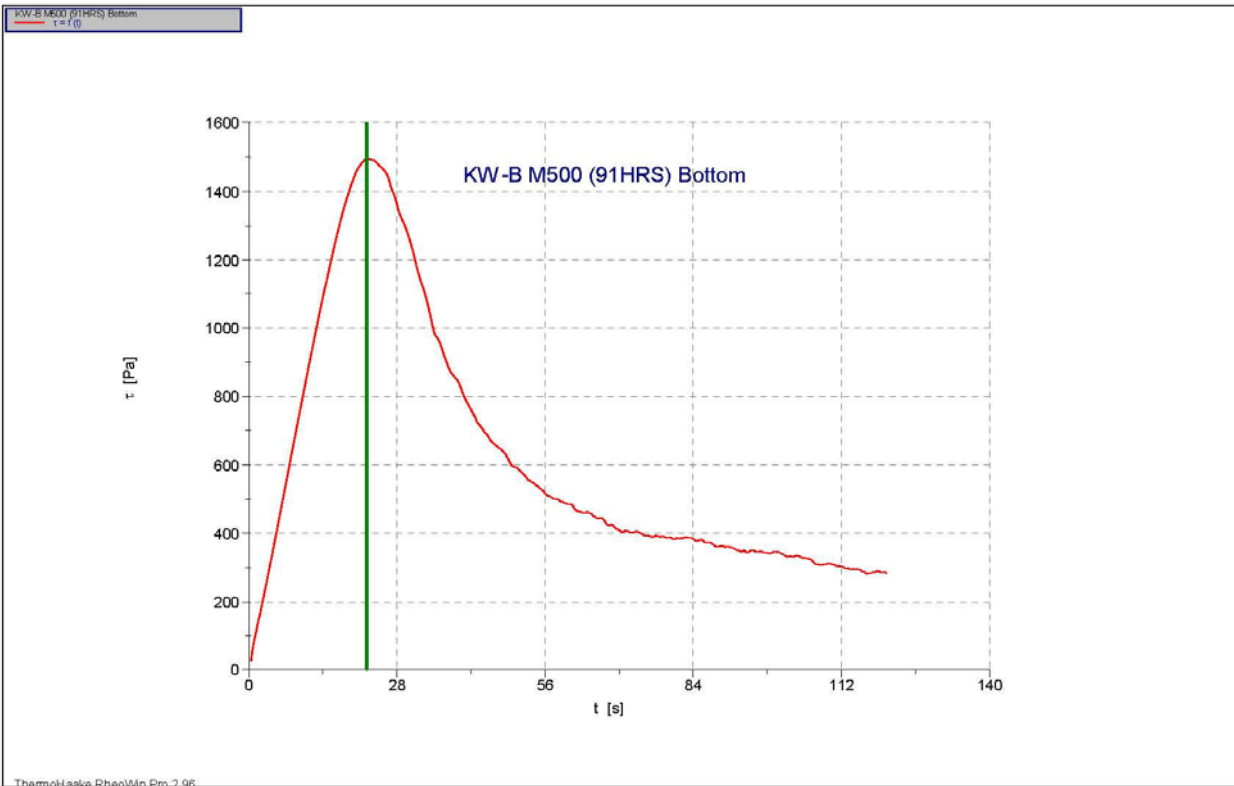
1: C:\Rheology Results\K-Basin\Shear Strength (vane)\KW-B M500 (48HRS).rwd
Company / Operator: PNNL / MARIA LUNA
Date / Time / Version: 11.03.2009 / 15:34:44 PM / RheoWin Pro 296
Substance / Sample no: KW-B M500 (48HRS) / KW-B M500 (48HRS)
Curve discussion: Greatest value t [s] 21.19 τ [Pa] 797.4



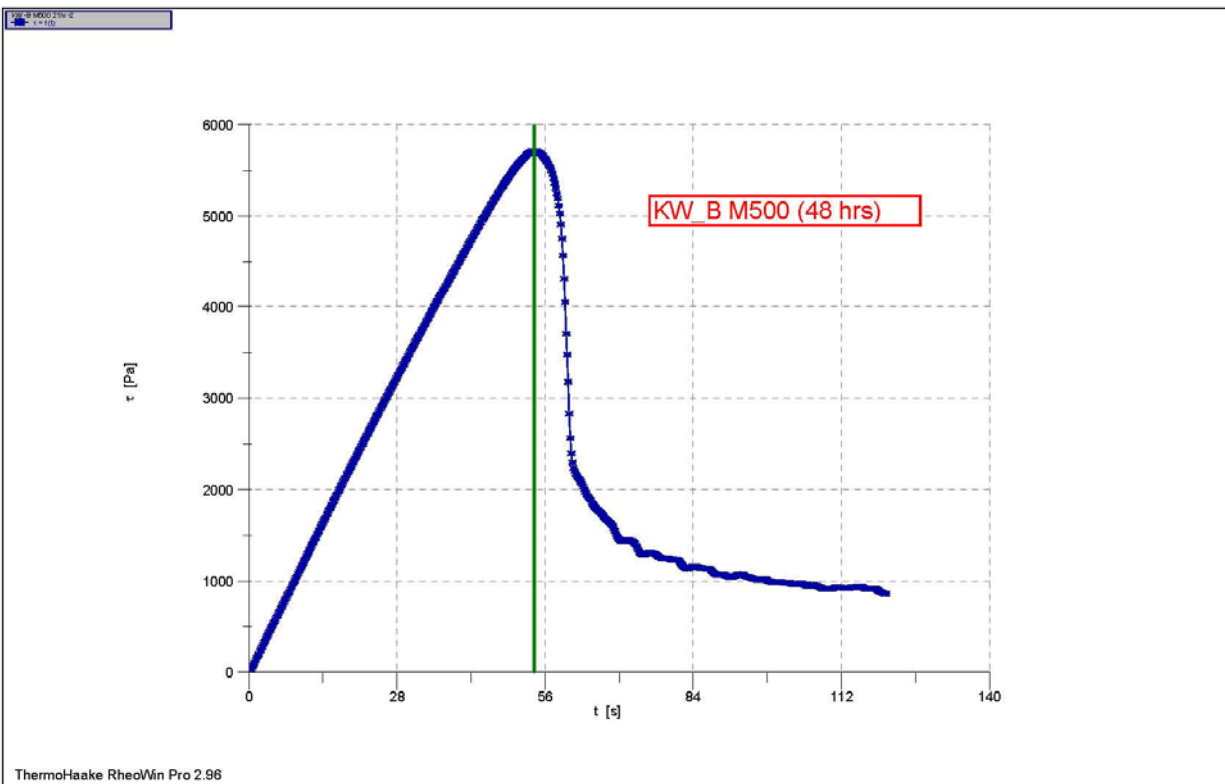
1: C:\Rheology Results\K-Basin\Shear Strength (vane)\KW-B M500 (48HRS) BOTTOM.rwd
Company / Operator: PNNL / MARIA LUNA
Date / Time / Version: 11.03.2009 / 15:38:25 PM / RheoWin Pro 296
Substance / Sample no: KW-B M500 (48HRS) BOTTOM / KW-B M500 (48HRS) BOTTOM
Curve discussion: Greatest value t [s] 23.12 τ [Pa] 1174.



1: C:\Rheology Results\K-Basin\Shear Strength (vane)\KW-B M500 (91HRS).rwd
Company / Operator: PNNL / MARIA LUNA
Date / Time / Version: 16.03.2009 / 8:58:04 AM / RheoWin Pro 296
Substance / Sample no: KW-B M500 (91HRS) / KW-B M500 (91HRS)
Curve discussion: Greatest value t [s] 19.99 τ [Pa] 1543.



1: C:\Rheology Results\K-Basin\Shear Strength (vane)\KW-B M500 (91HRS) Bottom.rwd
Company / Operator: PNNL / MARIA LUNA
Date / Time / Version: 16.03.2009 / 9:02:05 AM / RheoWin Pro 296
Substance / Sample no: KW-B M500 (91HRS) Bottom / KW-B M500 (91HRS) Bottom
Curve discussion: Greatest value t [s] 22.28 τ [Pa] 1495.



1: C:\Rheology Results\53019 -M12\Shear Strength 1.6 X 1.6 cm Vane\KW-B M500 21hr-2.rwd
Company / Operator: PNNL / MARIA LUNA
Date / Time / Version: 04.03.2009 / 13:11:58 PM / RheoWin Pro 296
Substance / Sample no: KW-B M500 21hr-2 / KW-B M500 21hr-2
Curve discussion: Greatest value t [s] 53.96 τ [Pa] 5715.

