

Rheological Studies on Pretreated Feed and Melter Feed from AW - 101 and AN - 107

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April 2000

Prepared for BNFL, Inc. under
Project 29953
Richland, Washington, 99352

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Summary

Rheological and physical properties testing were conducted on actual AN-107 and AW-101 melter feed samples prior to the addition of glass formers. Analyses were repeated on the AW-101 samples following the addition of glass formers. Once a glass formulation is available for the AN-107 samples, glass formers will be added and the analyses will be repeated with these samples. Samples from both feeds were tested at the target sodium values of nominally 6, 8, and 10 M. This data on actual waste is required to validate and qualify results obtained with simulants.

The AW-101 feed samples and the AN-107 feed samples at sodium concentrations of 6M and 8M contained no visible solids following evaporation. The 10 M sodium AN-107 sample contained roughly 1 vol% settled solids following evaporation. After approximately 2 weeks, solids rapidly precipitated from the 10 M Na sample resulting in a settled solids layer occupying roughly 70% of the sample volume.

With the exception of the 10 M Na AN-107 sample, the rheograms of both AN-107 and AW-101 samples prior to glass former addition show Newtonian behavior with a linear relation between shear stress and shear rate with no observed yield stress. No thixotropy was observed comparing the increasing and decreasing shear rate curves or between repeat analysis of the same samples. With the exception of some solids in the 10 M AN-107 sample that effected behavior at low shear rates (below $\sim 100 \text{ s}^{-1}$), the rheology of the AN-107 and AW-101 feeds were indistinguishable. The viscosities of the 6, 8, and 10 M Na feeds at 500 s^{-1} were 8, 12, and 21 cP respectively at 25°C , and 4, 7, and 12 respectively at 50°C . The 10M Na AN-107 sample displayed Bingham behavior with a thixotropic component.

Rheograms of the AW-101 samples after glass former addition show nearly Newtonian behavior with only a 20-40% drop in viscosity between 33s^{-1} and 500s^{-1} . No thixotropy or yield stresses were observed. The viscosities at 500 s^{-1} of the 6, 8, and 10 M Na feeds were 36, 88, and 230 cP respectively at 25°C , and 16, 46, and 130 cP respectively at 50°C .

A mixing and aging study was conducted on the 8 M Na AW-101 feed following glass former addition. Glass formers were added and the slurry was stirred at a rate consistent with that expected in the River Protection Project Waste Treatment Plant flow sheet. The viscosity of the slurry increased from 52 cP at 350 s^{-1} after 1 hour to 67 cP after 1 day. The viscosity then remained essentially constant as indicated by the 64 cP measurement after 1 week. These measurements were all conducted at 25°C . Sample behavior was nearly Newtonian. No thixotropy or yield stresses were observed.

The 8 M Na AW-101 samples with glass formers were then allowed to settle for 1 week. No gas retention or gas releases were observed during the study. After one week two settled solids layers formed in the sample. Both were analyzed for shear stress versus shear rate at 25°C and displayed near Bingham behavior with a linear increase in shear stress with shear rate above a yield stress. The yield stress for both layers was 4.6 Pa. Both samples also displayed a thixotropic component with a decreased viscosity and no yield point on the decreasing rate portion of the rheograms.

Table of Contents

1.0	Introduction	1.1
2.0	Experimental Approach	2.1
2.1	Evaporation and Settling Study	2.1
2.2	Mixing and Aging Study.....	2.6
3.0	Experimental Results	3.1
3.1	Density and Settling Study.....	3.1
3.2	Rheology	3.6
3.2.1	Rheology of Evaporated Feed Samples	3.6
3.2.2	Rheology of Melter Feed with Glass Formers.....	3.8
3.3	Mixing and Mixing Study.....	3.8
4.0	Conclusions	4.1
	Appendix A: Figures for AN-107 & AW-101	A.1
	Appendix B: Test Plan (BNFL-TP-29953-046)	B.1

Figures

Figure 2.1. AW-101 Samples and Glass Formers Prior to Mixing. Samples A, B (B-1 and B-2), and C are 6M, 8M, and 10M Sodium Respectively 2.2

Figure 2.2. AN-107 Samples Following 24 hours at 50°C. This Photograph Was Taken Two Weeks After Completion of Evaporation. Note Solids in Sample C That Were Not Observed 2 Days Prior to This Photograph. Samples A, B (B-1 and B-2), and C are 6M, 8M, and 10M Sodium Respectively 2.3

Figure 2.3. Close Up of Solids Precipitated Out of 10M AN-107 sample. While Loosely Packed, Solids Account for Roughly 60% of the Sample Volume..... 2.3

Figure 2.4. AW-101 Samples During 25°C Settling Study Following Glass Former Addition. Samples A, B-1, and C are 6M, 8M, and 10M Sodium Respectively 2.5

Figure 3.1. Volume Percent Settled Solids Versus Time for AW-101 Melter Feed With Glass Formers Using a Semi-Log Scale..... 3.2

Figure 3.2. Volume Percent Settled Solids Versus Time for AW-101 Melter Feed With Glass Formers Using a Linear Scale..... 3.3

Figure 3.3. Settling Rates for AW-101 Melter Feed With Glass Formers..... 3.5

Tables

Table 2.1. Glass Formers Added to AW-101 Samples..... 2.7

Table 3.1. Density of AN-107 and AW-101 Samples at 25°C and 50°C With and Without Addition of Glass Formers in g/ml..... 3.1

Table 3.2. Volume Percent Settles Solids for the AW-101 Samples with Glass Formers..... 3.4

Table 3.3. Viscosity of Evaporated Feed Samples in cP. Values are the Average of Two Duplicate Analyses..... 3.7

Table 3.4. Yield Power Law Fit for the 10M AN-107 Samples at 25°C..... 3.7

Table 3.5. Viscosity of AW-101 Melter Feed Samples with Glass Formers in cP. Values are the Average of Two Duplicate Analyses..... 3.8

Table 3.6. Viscosity of 8M Na AW-101 Melter Feed Samples with Glass Formers During Mixing and Aging Study. Analyses Conducted at 25°C Using a Haake M5 Viscometer with an SVI Concentric Cylinder Geometry. Values are in cP 3.9

Table 3.7. Yield Power Law Fit for the 8M AW-101 Settled Solids following Glass Former Addition. Analyses Were Conducted at 25°C..... 3.10

1.0 Introduction

The scope of the present work was to obtain physical and rheological data on actual LAW melter feed samples. The physical and rheological properties of the LAW melter feed are important considerations in the selection of the melter feed preparation flowsheet and processing equipment such as mixers, pumps, piping and sampling. Measurements on actual waste are required to validate and qualify results obtained with simulants.

Actual samples from tank AW-101 and AN-107 were used in this testing. Multiple samples from each tank were received from Handford's 222-S laboratory. Using this material, a composite was prepared for each of the tanks. Entrained solids were removed from the AW-101 composite by ultrafiltration. The cesium and technetium were then removed by ion exchange. Entrained solids, Sr and transuranics (TRU) were removed from the AN-107 material during the Sr/TRU removal process. Cesium was then removed from the AN-107 material by ion exchange.

Solids concentration, settling rate, density and shear stress versus shear rate were measured on the samples after evaporation to three sodium concentrations (nominally 6, 8 and 10 M) at ambient temperature and at 50°C. The same measurements were conducted on the three mixtures from tank 241-AW-101 after the addition of glass formers. The 8M Na AW-101 slurry was mixed for 1 week at a shear rate consistent with that expected in the facility. During this mixing, shear stress versus shear rate was measured after 1 hour, 1 day and 1 week. A shear stress versus shear rate analysis of this slurry was conducted again after 1 week with no mixing. Tests involving AN-107 slurry with glass formers will be conducted once a glass formulation is developed.

This report describes the experimental approach and results of the testing. Specifications for this work were provided to Battelle by BNFL under Task Specification Number TS-W375LV-TE00001. This report also provides a means of transmitting to BNFL the completed test plan and analytical data¹.

¹ Results presented in this report are based on work conducted under Technical Procedure 29953-010, and Test Plan 29953-46.

2.0 Experimental Approach

2.1 Evaporation and Settling Study

The actual waste samples used in this testing were prepared under conditions similar to those anticipated in the River Protection Project Waste Treatment Plant flow sheet. Both the AW-101 and AN-107 wastes were received for this task following cesium ion exchange. The process flow sheet includes technetium ion exchange for both of these wastes prior to evaporation. However, the technetium ion exchange process does not significantly alter the waste composition. Therefore, samples were not subject to technetium ion exchange prior to this scope of work.

The density of the waste samples were measured at ambient temperature ($\sim 23^{\circ}\text{C}$). This measurement was conducted by placing a subsample in a graduated glass cylinder of known mass. The density was then calculated by dividing the mass by the volume. The measured densities were 1.246 g/ml and 1.236 g/ml for the AW-101 and AN-107 samples respectively.

Following the density measurements, each waste was partitioned into three subsamples. Each of the subsamples was then evaporated to one of the target sodium concentrations (6M, 8M, or 10M). The evaporation was conducted in a vacuum oven at $\sim 50^{\circ}\text{C}$ under approximately 23 inches of Hg vacuum. Three subsamples of each waste were weighed into glass beakers. Using the density of the initial slurries, target weights required to produce the desired sodium concentrations were calculated. Water was then evaporated from the samples. For estimating purposes, it was assumed that for every gram of water evaporated the volume of the samples decreased by one milliliter. Figures 2.1 and 2.2 show the AW-101 and AN-107 samples, respectively, after evaporation.

Following the evaporation step, the samples were placed in glass-graduated cylinders, and the density of each was measured at ambient temperature ($\sim 23^{\circ}\text{C}$). This data was then used to calculate the actual sodium concentrations of the evaporated samples. The AW-101 samples were calculated to have Na concentrations of 5.9 M, 7.7 M, and 9.4 M. The AN-107 samples were calculated to have Na concentrations of 5.9 M, 7.9 M, and 9.7 M. The 9.7 M Na AN-107 sample contained approximately 1 vol% settled solids at 23°C . These settled solids were clear to white in color and consisted of particles approximately 1 mm in size. These solids settled within a few seconds after agitation. None of the other evaporated samples contained visible solids. The densities of the samples were measured at 50°C by placing the sealed graduated cylinders containing the samples in an oven at 50° overnight. The samples were removed from the oven and the volumes were immediately recorded. The samples were allowed to cool before they were reweighed.

Following the density measurements at 50°C, additional solids were observed in the AN-107 10 M Na sample. This observation was made approximately 2 weeks after completion of the evaporation. A photograph of these settled solids is presented in Figure 2.3. It is estimated the sample contains roughly 70 vol% loosely settled solids. Settling studies at 25°C and 50°C are currently being repeated on this sample and will be reported in a revision to this report.



Figure 2.1. AW-101 Samples and Glass Formers Prior to Mixing. Samples A, B (B-1 and B-2), and C are 6M, 8M, and 10M Sodium Respectively



Figure 2.2. AN-107 Samples Following 24 hours at 50°C. This Photograph Was Taken Two Weeks After Completion of Evaporation. Note Solids in Sample C That Were Not Observed 2 Days Prior to This Photograph. Samples A, B (B-1 and B-2), and C are 6M, 8M, and 10M Sodium Respectively



Figure 2.3. Close Up of Solids Precipitated Out of 10M AN-107 Sample. While Loosely Packed, Solids Account for Roughly 60% of the Sample Volume

Following the 50°C density measurement on the AW-101 samples, but before the 50°C density measurement on the AN-107 samples, the evaporated samples were stirred using an overhead mixer. While stirring, subsamples were removed for shear stress versus shear rate analyses. Samples were analyzed for shear stress versus shear rate in duplicate at 25°C and 50°C using the Bohlin CS viscometer modified for glovebox operations. Concentric cylinders with a 25-mm-diameter inner cylinder and a “Small Sample Cell” outer cylinder were used as the measuring geometries. The gap for this geometry set is 0.75 mm.

As per our procedure (29953-010), the Test Plan (29953-046), and manufacturers recommendations, we performed a single point calibration check on the instrument every 30 days. This was done with either the 50 cP or 95 cP standard. This calibration check needs to be within 10% of the certified value for standards above 10cP. Rheograms for standards can be found in Appendix A, Figures 1, 15, and 48. The measured viscosity of both the 50 and 95 cP standards were not constant over the entire shear rate range, but remained within the required 10% criteria between approximately 10 and 550 s⁻¹. Since the measured viscosity was not constant over the range, reporting a particular error is not appropriate. The viscosity is the ratio of shear stress to shear rate and the viscosity of the standards were within this acceptance criteria over the 10 s⁻¹ to ~550 s⁻¹ range. Therefore, this single standard is an effective check of the instruments torque calibration over 3 orders of magnitude. Manufacturer recommendations are only for this single check even if some samples display viscosities above the stress range of the calibration checked provided they are not beyond the maximum torque of the instrument. None of the samples exceeded the maximum torque of the instrument.

Shear stress versus shear rate rheograms were obtained by measuring the shear stress produced at a specific shear rate. The increasing shear rate curve was generated by gradually increasing the shear rate from approximately 0.1 s⁻¹ to the maximum achievable shear rate for the given sample, nominally 700 s⁻¹. The decreasing shear rate curve was generated by reducing the shear rate back down to 0.1 s⁻¹. While the instrument is rated to 1100 s⁻¹ with this geometry set, these high shear rates are not easily attained with these slurries. At high shear rates, the system tends to overspin producing poor quality data. The shear rate analysis was conducted again with the same sample still in the instrument. A difference between the first and second run would indicate potentially unusual behavior in the samples including (but not limited to) settling of the solids within the instrument, the sample being effected by shearing in the instrument, or water loss through evaporation. In all cases, the first and second runs were virtually identical.

The 8M Na AW-101 sample was divided into 2 equal aliquots. Glass formers were then added to the 6M Na sample, one of the 8M Na AW-101 samples, and the 10M Na AW-101 sample. The second 8M Na AW-101 sample was retained for mixing and aging studies that will be described in Section 2.2 of this report. The AN-107 sample is currently being retained until a glass formulation is provided to Battelle. Glass former quantities were based on the formulation provided by VSL. The VSL formulation was provided for a 4.59 M sodium feed on a mass per liter basis. The quantity of glass formers were first adjusted on a per liter basis to the targeted sodium concentrations (6, 8, and 10 M). The masses to be added to the 6M Na sample were multiplied by 1.31 (6/4.59=1.31), the masses added to the 8M Na samples were multiplied by 1.74, and the masses added to the 10 M sample were multiplied by 2.18. These adjusted masses on a per liter basis were then multiplied by the

volume of sample to calculate how much material to add to each sample. Table 2.1 lists the quantity and type of glass formers added to each of the AW-101 samples.

Prior to addition, the dry glass formers were weighed into a flask and hand mixed using a spatula. The glass former mixture was then slowly added to the samples while the samples were stirred using an overhead mixer. Following the glass former addition, the samples were stirred for an additional hour.

After stirring for one hour, the stir blade was removed and the volume of settled solids was monitored for three days at ambient temperature. After three days, the mass and volume of the bulk samples were recorded and used to calculate the densities of the bulk slurries. Figure 2.4 shows the AW-101 samples during this settling study. The samples were then placed in an oven at 50°C overnight before repeating the settling study at 50°C.

Following the 50°C settling study, shear stress versus shear rate analyses were performed on the AW-101 samples at 25°C and 50°C as described previously for the evaporated samples.



Figure 2.4. AW-101 Samples During 25°C Settling Study Following Glass Former Addition. Samples A, B-1, and C are 6M, 8M, and 10M Sodium Respectively

2.2 Mixing and Aging Study

The second 8M Na AW-101 sample was subjected to a mixing and aging study as specified in the BNFL Task Specification. The 8M Na sample was placed in a 250 ml round bottom flask with a side tube. The sample was stirred using an overhead mixer while the glass formers were added. The flask was then sealed using a Teflon stirrer bearing. The sample with glass formers was continually stirred for 1 week using a 1-inch (2.54 cm) diameter blade at 480 rpm. The volume of the sample with glass formers was 6.1 in³ (100 ml). The 480 rpm mixing rate provides the same energy per volume as anticipated in the River Protection Project Waste Treatment Plant. The following equation (provided by BNFL) was used to calculate the proper rotational rate:

$$N^3 = 1.85 \times 10^7 V/D_i^5 \quad [1]$$

For this equation, N is the impeller rotational rate in rpm, V is the sample volume in cubic inches and D_i is the impeller diameter in inches.

Samples of the 8M Na slurry were removed from the mixing vessel after 1 hour, 1 day and 1 week. These samples were immediately analyzed for shear stress versus shear rate at 25°C. These analyses were conducted as described in Section 2.1 for both the evaporated samples and samples with glass formers.

The 8M Na samples were then transferred to a 100 ml glass graduated cylinder. The sample was left to settle undisturbed for 1 week. During the settling time, the sample was monitored for any gas retention and releases. Visual observations were supplemented with time lapse video. No gas bubbles were observed in this sample or any other AW-101 samples during this study.

After one week of settling, the standing liquid was removed, and the settled solids were immediately analyzed for shear stress versus shear rate at 25°C. Given the high solids content of the sample, the Bohlin CS rheometer could not be used. Instead, a Haake M5 measuring head modified for hot cell operations was used with an SV I measuring geometry. The SV I is a concentric cylinder geometry with a gap of 1.45 mm and a maximum shear rate range of 350 s⁻¹. A 95.5 cP Brookfield viscosity standard was used to check the calibration of the instrument before samples were analyzed. The calibration check was within 10% of the certified value. Upon close inspection, the settled layer in the 8M Na sample was found to consist of a loosely settled layer on top of a firmer higher solids content layer. Both layers were analyzed separately. The shear rate was gradually increased from approximately 0.1 to 350 s⁻¹ generating the increasing shear rate curve, and then back down to 0.1 s⁻¹ generating the decreasing curve.

Table 2.1. Glass Formers Added to AW-101 Samples

		Formulation g/L				Start Vol = 142 mL		Start Vol = 146/2 = 73 mL		Start Vol = 146 mL	
		4.59M	6M	8M	10M	Target Wt	Actual Wt	Target Wt	Actual Wt	Target Wt	Actual Wt
Additive	Grade					6 M	6 M	8 M	8 M (B-1)	8 M	10 M
Kyanite (Al ₂ SiO ₅)	Raw Kyanite, 325 Mesh	41.74	54.56	72.75	90.94	7.748	7.74	5.311	5.33	5.35	13.33
Orthoboric Acid (H ₃ BO ₃)	Technical Grade	127.8	166.99	222.66	278.32	23.713	23.70	16.254	16.31	16.32	40.67
Wollastonite (CaSiO ₃)	Powder untreated, NYAN 325 Mesh	32.44	42.41	56.54	70.68	6.022	6.07	4.127	4.21	4.19	10.35
Red Iron Oxide (FeO ₃)	Red Iron Oxide, 325 Mesh (5001)	37.35	48.82	65.10	81.37	6.933	6.97	4.752	4.78	4.79	11.88
Olivine (Mg ₂ SiO ₄ with some Fe ₂ SiO ₄)	325 Mesh (#180)	22.1	28.89	38.52	48.15	4.102	4.11	2.812	2.81	2.83	7.04
Ground Silica Sand (SiO ₂)	Sil-co-Sil 75, 200 Mesh	262.8	343.58	458.11	572.64	48.789	48.71	33.442	33.45	33.49	83.63
Rutile (TiO ₂)	Premium Grade, Airfloated	14.32	18.72	24.96	31.20	2.658	2.64	1.822	1.85	1.82	4.55
Zinc Oxide (ZnO)	KADOX-920	21.21	27.73	36.97	46.21	3.937	3.94	2.699	2.71	2.71	6.76
Zircon Sand (ZrSiO ₄)	Flour 325 Mesh	31.94	41.75	55.67	69.59	5.929	5.96	4.064	4.12	4.06	10.15
Sugar	Granular Sugar	51.34	67.11	89.48	111.85	9.530	9.55	6.532	6.61	6.57	16.43

3.0 Experimental Results

This section details the results of tests conducted on actual AW-101 and AN-107 samples following evaporation to nominal sodium concentrations of 6M, 8M, and 10M. The AW-101 samples were evaporated to 5.9M, 7.7M, and 9.4M. The AN-107 samples were evaporated to 5.9M, 7.9M, and 9.7M. In this section, samples will be referred to according to the nominal sodium concentrations (6 M, 8M, and 10M).

3.1 Density and Settling Study

The densities of the evaporated samples with and without glass formers are provided in Table 3.1. As expected, the densities of the samples prior to glass former addition increase with increasing sample concentration and decreases slightly with temperature. No temperature trend is seen in the AW-101 samples with glass formers. The densities for the AW-101 samples with glass formers increase from 1.59 g/ml to 1.80 g/ml for the 6M and 8M Na samples respectively; however, the densities of the 8M and 10M Na samples are similar at roughly 1.80 g/ml.

Table 3.1. Density of AN-107 and AW-101 Samples at 25°C and 50°C With and Without Addition of Glass Formers in g/ml (Error is estimated at $\pm 2\%$ of the Measured Value).

Sample	Without Glass Formers		With Glass Formers	
	25°C	50°C	25°C	50°C
6M AN-107	1.28	1.26	NA	NA
8M AN-107	1.37	1.33	NA	NA
10M AN-107	1.44	1.38	NA	NA
6M AW-101	1.31	NM (1.27)	1.59	1.59
8M AW-101	1.37	NM (1.33)	1.80	1.81
10M AW-101	1.44	NM (1.38)	1.77	1.80

NM = not measured due to elimination of 50°C settling study on AW-101 evaporated samples. Values in parentheses are estimated based on sodium concentration.

NA = not available. These measurements will be completed following determination of the AN-107 glass formulation.

As described in Section 2.1, the AW-101 samples were agitated and then allowed to settle at 25°C and 50°C over a 3-day period. The solids settled leaving a clear supernatant above with a distinct interface between the settling solids and the clarified supernatant. Figure 3.1 and 3.2 plots the vol% settled solids (interface height/sample height x 100%) versus time for the samples. Figure 3.1 uses a semi-log scale while Figure 3.2 is linear. With the exception of the 10M Na sample at 25°C, all samples finished settling within the first 24 hours. The 10M Na sample at 25°C reached a constant value after approximately 45 hours.

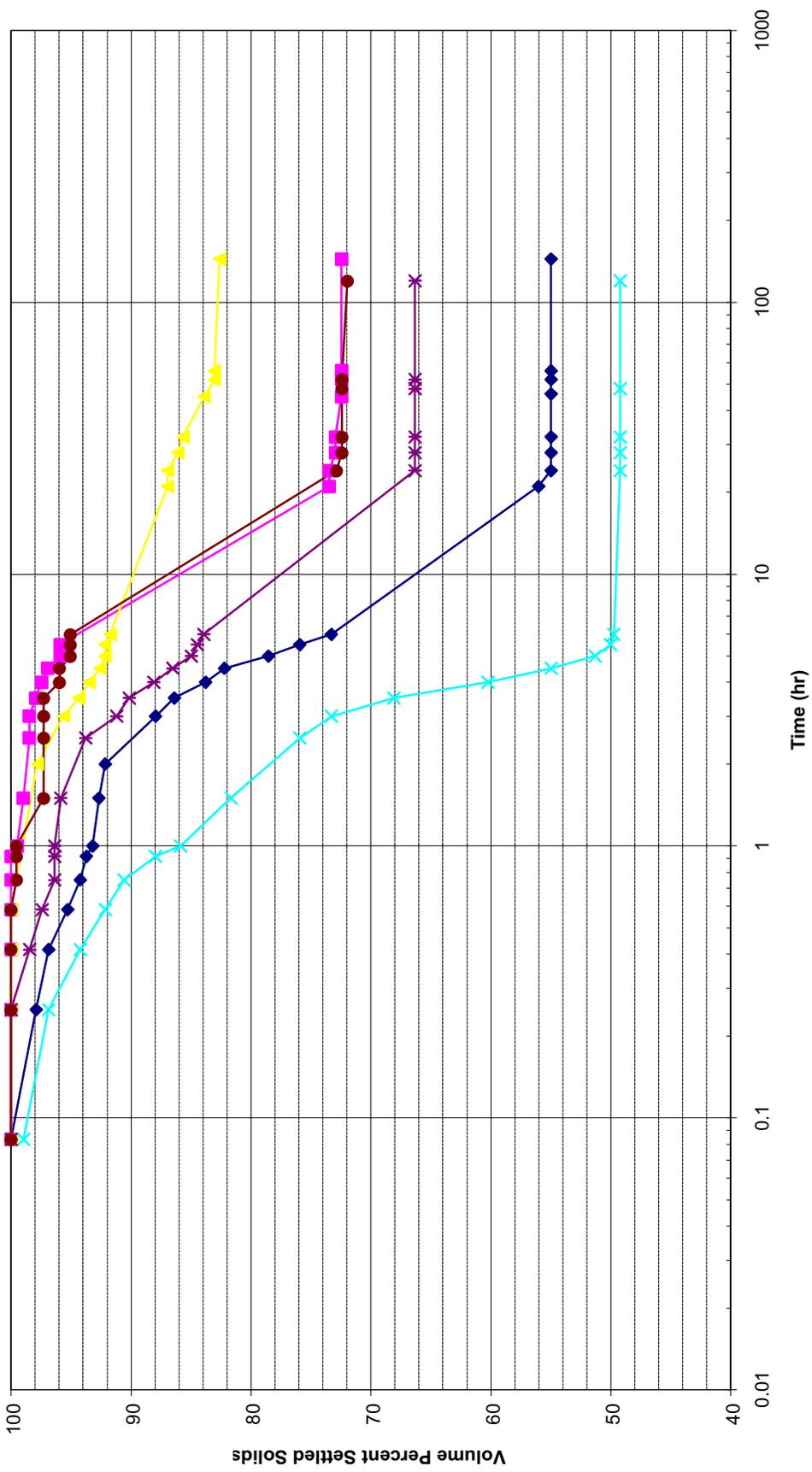


Figure 3.1. Volume Percent Settled Solids Versus Time for AW-101 Melter Feed With Glass Formers Using a Semi-Log Scale

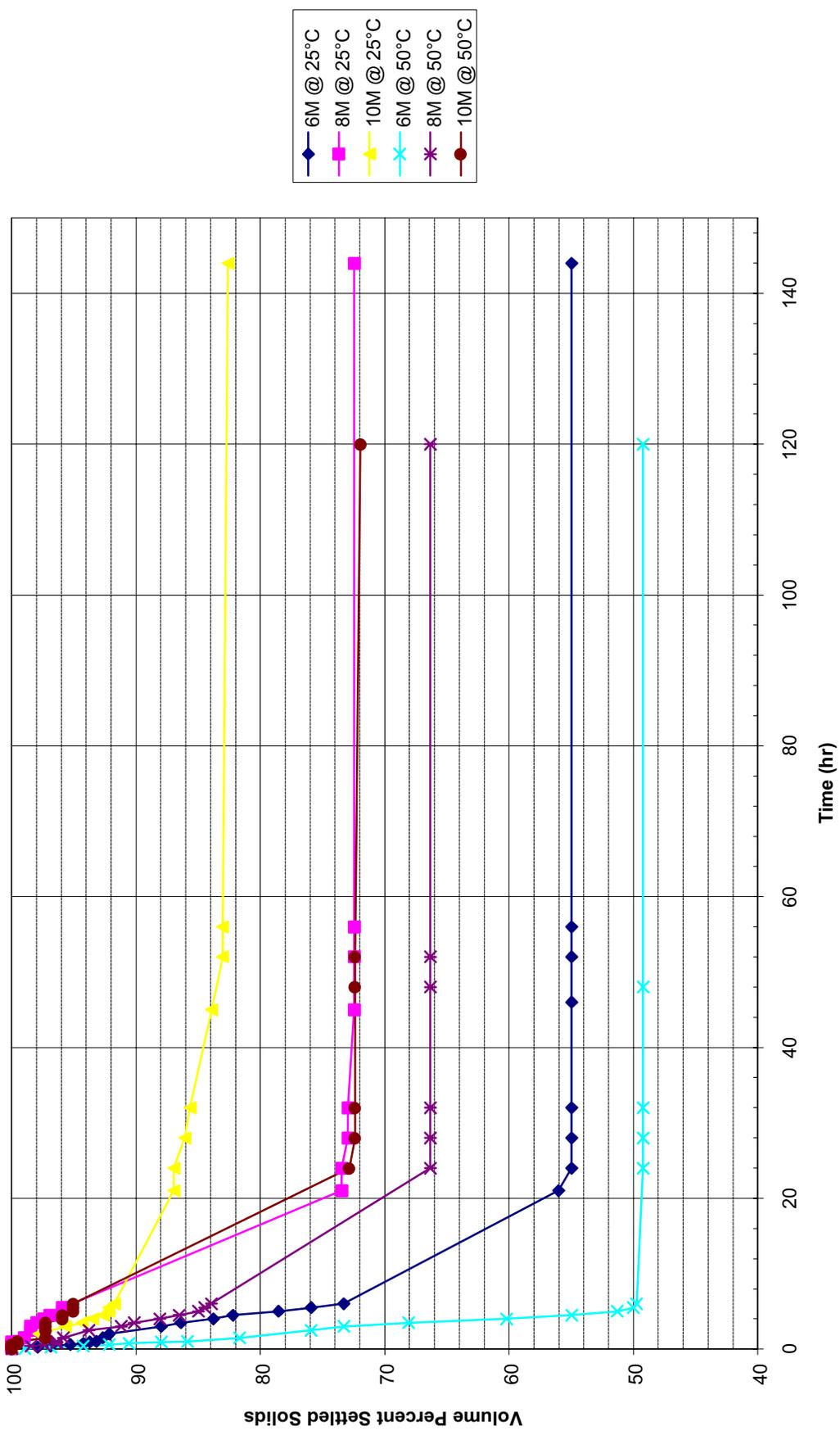


Figure 3.2. Volume Percent Settled Solids Versus Time for AW-101 Melter Feed With Glass Formers Using a Linear Scale

Table 3.2 lists the vol% settled solids for the samples. As expected, the data shows the volume percent settled solids increased with sodium concentration and decreased with temperature. At 25°C, the volume percent settled solids increased from 55 to 72 to 83 vol% for the 6, 8, and 10M Na sample respectively. At 50°C, volume percent settled solids increased from 49 to 66 to 72 vol% for the 6, 8, and 10M Na sample respectively. This trend is the result of adding larger mass ratios of insoluble glass formers to the higher concentration samples. The decrease in vol% settled solids with increasing temperature could be the result of one or more soluble species in the precipitate at the lower temperature. This trend could also be the result of a lower viscosity and yield stress leading for greater compaction of the solids at the higher temperature.

Table 3.2. Volume Percent Settles Solids for the AW-101 Samples with Glass Formers

Sample	25°C	50°C
6M	55	49
8M	72	66
10M	83	72

Figure 3.3 plots the settling rate as a function of time. The settling rate was calculated by dividing the change in settled solids height by time. Minor parallax errors can be magnified by this calculation and result in significant scatter. To minimize scatter the data plotted in Figure 3.3 have been smoothed using a four point moving average.

Three settling mechanisms are generally observed for this type of sample matrix: free settling, hindered settling, and compression settling. Free settling is settling of discrete particles or flocculated particles without interaction from other particles or the vessel wall. Free settling is denoted by a linear decrease in vol% settled solids over time as the particles fall with fixed velocities. A linear decrease in settling velocity would be seen as a constant or flat portion of a settling rate versus time plot. For flocculating systems, the velocity generally increases as the mass of the particles increase, although particle shape changes can also decrease the settling velocity. Hindered settling occurs when the when particle-particle and particle-wall interactions effect the settling velocities. In an ideal system, hindered settling is denoted by the break from a linear decrease in vol% settled solids with time (i.e. a decrease in the settling rate). Compression settling is the final portion of the vol% settling curve as the system approaches the stable value. Two or even all three of these mechanisms usually occur simultaneously.

All samples in Figure 3.3 show an initially constant settling rate. This region is probably a combination of free as well as some hindered settling. Free settling of the smaller particles is dominating the behavior of the observed solids interface. However, the majority of the solids are at a much higher concentration at the bottom of the column where hindered settling is the dominant mechanism. For the 6M Na and 8M Na samples, the initial settling rates increase with temperature and decrease with increasing sodium concentration (i.e. 6M Na samples settle faster than 8M Na, and 50°C settle faster than 25°C). The initial settling rate of the 6M Na sample at 50°C was the highest (~0.02 cm/min) followed by the same sample at 25°C (~0.005-0.015 cm/min). The initial settling rate of the 8 M sample at 50°C was between 0.004-0.007 cm/min and at 25°C between 0.001-0.002 cm/min.

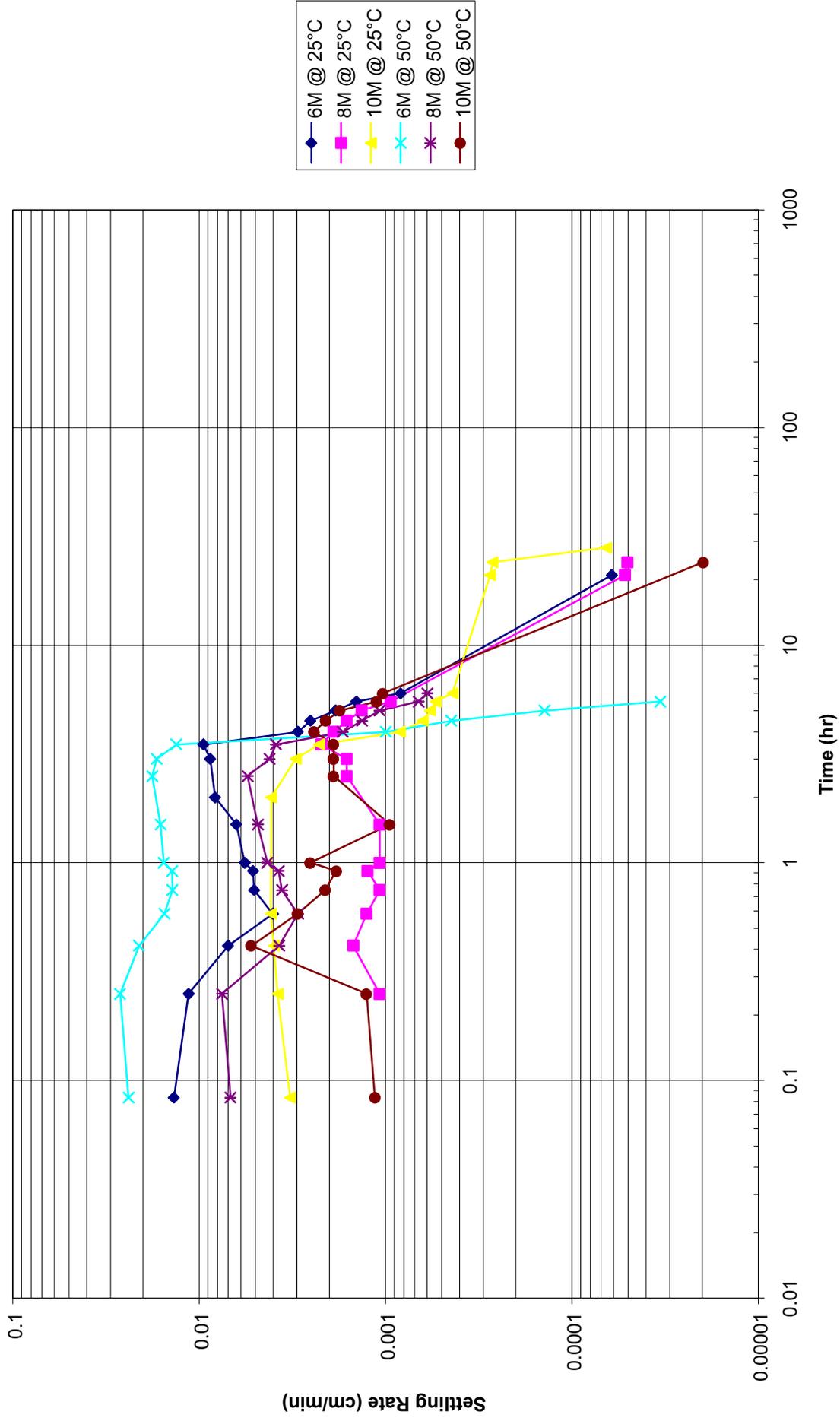


Figure 3.3. Settling Rates for AW-101 Melter Feed With Glass Formers

This increase in settling rate with increasing temperature and decrease in sodium concentration is probably the result of several factors including a lower supernatant viscosity as well as a decrease in the solids content. Viscosity results to be presented later in this report show that the viscosity of the samples decrease with increasing temperature and increase with higher sodium concentration. The vol% settled solids data in Table 3.2 shows the solids content of the samples decreases with increasing temperature and increases with increasing sodium concentration.

The initial settling rate of the 10M Na sample at 25°C (~0.004 cm/min) is higher than the same sample at 50°C (~0.001-0.003 cm/min) and is higher than the 8M Na sample at 25°C. The reason for this reverse in the trend observed for the 6 and 8M Na samples is unclear from the available data. It is probably the result of several factors that could include more effective flocculation of fine particulates at higher solids content of the 10M Na sample, competing with a higher concentration of fine soluble particulates that can not be dissolved at the higher temperature with the higher solids loading. This is speculative and more information would be needed if this is to be resolved.

After approximately 4 hours of settling, the settling rate for all of the samples drop dramatically as seen in Figure 3.3. This drop indicates the end of any free settling, and a transition to hindered and compression settling. This region of the settling curve is probably a mixture of compression settling and hindered settling.

The transition from the combination of both hindered and compressive settling to only compression settling as the dominant mechanism is seen at different times for the samples. From Figure 3.1, the 6 M Na sample at 50°C shows the very slow linear decrease in settled solids volume expected for the compression settling at 6 hours, and the 6M Na at 25°C show the linear decrease starting after 24 hours. With the exception of the 10M Na sample at 25°C, compression settling becomes the dominant mechanism at ~24 hours. Compression settling does not appear to be the dominant settling mechanism for the 10M Na sample at 25°C until after ~60 hour. This late onset for compression settling of the 10M Na sample at 25°C suggests that while the sample is very close to a stable value, the sample may not have reached its final settled height.

3.2 Rheology

The results of the shear stress versus shear rate analyses are presented in this section. The full set of rheograms, both samples and calibration checks, are included in Appendix A.

3.2.1 Rheology of Evaporated Feed Samples

Shear stress versus shear rate analyses were conducted on the AW-101 evaporated feed samples approximately one month after the evaporation step. The AN-107 sample analyses were conducted approximately 1 week after evaporation. The tabular result for the viscosities at 33s⁻¹ and 500s⁻¹ are listed in Table 3.3. These results are the average of 2 duplicate samples. With the exception of the 10M Na AN-107 sample at 25°C, the evaporated samples for both AN-107 and AW-101 displayed a nearly linear relationship between shear stress and shear rate over the shear rate range examined with no detectable

yield stress. This is referred to as Newtonian behavior. Since the viscosity is the ratio of the shear stress to the shear rate, the viscosity was nearly constant over the shear rate range examined. As expected, the viscosity of all samples from both tanks increase with sodium concentration and decrease with temperature.

Table 3.3. Viscosity of Evaporated Feed Samples in cP. Values are the Average of Two Duplicate Analyses

Sample	25°C		50°C	
	33s ⁻¹	500s ⁻¹	33s ⁻¹	500s ⁻¹
6M AN-107	9	8	6	4
8M AN-107	14	13	10	8
10M AN-107	56	21	14	11
6M AW-101	8	8	6	4
8M AW-101	13	12	7	6
10M AW-101	22	21	14	12

It should be noted that the results for the AN-107 samples are similar to the AW-101 samples. The only significant difference in the two data sets is the viscosity of the 10M Na samples at 33s⁻¹. Under these conditions, the AN-107 and AW-101 samples have viscosities of 57 and 22 cP respectively. The higher AN-107 viscosity is probably the result of visible solids in the AN-107 sample while the AW-101 sample was a clear liquid with no solids. The increasing shear rate curve for the AN-107 10M Na samples were models using the following yield power law:

$$\text{Tau} = a + bD^c \quad [2]$$

where Tau is the shear stress in Pa, and D is the shear rate in s⁻¹. The constants a, b, and c were calculated by a best fit regression to the data. The results of this fit are presented in Table 3.4. In the equation, the constant a is the yield stress which is an average of 1.2 Pa for the two duplicates. The divergence of c from unity is a measure of the samples shear thinning (c<1) or thickening (c>1). The duplicate suggests shear thickening, however, this sample also displays a thixotropic component, so c is greater than 1 for the duplicate as a result of the model trying to fit the initial thinning of the material. Therefore, this material is best described as a Bingham plastic with a thixotropic component.

Table 3.4. Yield Power Law Fit for the 10M AN-107 Samples at 25°C

Sample	a (Pa)	b (Pa · s)	c (unitless)
1	0.62	0.0187	1.00
2	1.7	0.00824	1.13

3.2.2 Rheology of Melter Feed with Glass Formers

Shear stress versus shear rate analyses were conducted on the AW-101 melter feed samples approximately two weeks after glass former addition. These analyses were conducted after the ambient and 50°C settling studies. The tabular result for the viscosities at 33s⁻¹ and 500s⁻¹ are listed in Table 3.5.

While a the viscosity of the sample decreased slightly with shear rate, approximately 20-40% between 33s⁻¹ and 500s⁻¹, this is still a nearly linear relationship between shear stress and shear rate over the shear rate range. In addition, no yield stress was observed. Therefore, the samples with glass formers are still roughly Newtonian in behavior.

As seen in Table 3.5, the viscosities of the samples increase with sodium concentration and decrease with temperature. The sodium trend is expected since the amounts of glass formers added were proportional to the sodium concentration. The glass formers were primarily insoluble in these solutions. Therefore, the higher sodium samples have higher insoluble solids content as well as higher dissolved solids content. Both higher dissolved and insoluble solids result in an increased viscosity. The temperature trend is also expected as the viscosity of most liquids and slurries decrease with increasing temperature.

Table 3.5. Viscosity of AW-101 Melter Feed Samples with Glass Formers in cP. Values are the Average of Two Duplicate Analyses

Sample	25°C		50°C	
	33s ⁻¹	500s ⁻¹	33s ⁻¹	500s ⁻¹
6M AW-101	46	36	26	16
8M AW-101	110	88	60	46
10M AW-101	260	230	160	130

3.3 Mixing and Mixing Study

As described in Section 2.2, an 8M Na AW-101 subsample was used for the mixing and aging study. Glass formers were added and the slurry stirred for 1 week. Subsamples were removed after 1 hour, 1 day and 1 week and immediately analyzed for shear stress versus shear rate at 25°C using a Bohlin CS rheometer with a concentric cylinder geometry. The results are presented in tabular form in Table 3.6 at 33s⁻¹ and 350s⁻¹. The rheograms show an increase in viscosity for the duplicate runs, therefore; only results for the initial runs are presented. The increase in viscosity is probably the result of sample evaporation between analyses.

The viscosity of the sample decreased only slightly with shear rate, approximately 10-15% between 33s⁻¹ and 500s⁻¹. This is a linear relationship between shear stress and shear rate over the shear rate range. In addition, no yield stress was observed. Therefore, the samples during the mixing study exhibit Newtonian behavior.

Table 3.6. Viscosity of 8M Na AW-101 Melter Feed Samples with Glass Formers During Mixing and Aging Study. Analyses Conducted at 25°C Using a Haake M5 Viscometer with an SVI Concentric Cylinder Geometry. Values are in cP

Sample ^a	Yield Stress (Pa)	Increasing Curve @ 33s ⁻¹	Decreasing Curve @ 33s ⁻¹	350 s ⁻¹
After 1 Hour of Mixing	NO	59	NA	52
After 1 Day of Mixing	NO	77	NA	67
After 1 Week of Mixing	NO	76	NA	64
Loosely Settled Solids	4.8	270	180	130
Tightly Settled Solids	5.3	355	260	220

^a Due to sample drying between analyses, mixing results are for initial samples only.

Settled solids results are the average of two duplicates.

NO, Yield stresses were not observed during the mixing study for the 8M Na slurry

NA, Viscosity of increasing and decreasing rate curves are similar for the 8M Na slurry

The results in Table 3.6 show a 30% increase in viscosity over the first day of mixing, but no change after the first day. The viscosity at 350s⁻¹ after 1 hour was 52 cP and increased to 67 cP after 1 day. However, after 1 week the viscosity did not increase again and was measured at 64 cP.

The sample was then transferred to a 100 ml graduated cylinder and allowed to settle for one week. No gas retention or releases were observed during this work. After one week, the standing liquid was removed and the settled solids were analyzed for shear stress versus shear rate at 25°C. During sample collection, it was noted that the settled solids had formed in two distinct layers. The upper layer appeared to be more soupy and containing finer solids. This upper layer was referred to as the loosely settled solids layer. The lower layer was pastier and appeared to contain more solids that had less interstitial liquid. Both layers were analyzed separately.

As seen in Table 3.6, the viscosity of the loosely settled solids decreased from 270 cP at 33 s⁻¹ to 130 cP at 350 s⁻¹. The tightly settled solids decreased from 355 to 220 cP of the same range. This is still a roughly linear relationship between shear stress and shear rate. To quantify the sample behavior, the increasing shear rate curves were modeled using a yield power law as described in Section 3.2.2. The result of this fit is presented in Table 3.7. Both samples displayed average yield stresses of 4.6 Pa. The average value of c was 0.976 for both samples indicating a nearly linear relationship between shear stress and shear rate. This is defined as Bingham behavior. These samples display a thixotropic component as seen in the lack of a yield point on the decreasing rate portion of the rheogram, and the slightly lower viscosity.

Table 3.7. Yield Power Law Fit for the 8M AW-101 Settled Solids following Glass Former Addition. Analyses Were Conducted at 25°C

Sample	a (Pa)	b (Pa · s)	c (unitless)
Loosely Settled 1	5.2	0.183	0.929
Loosely Settled 2	4.0	0.110	1.019
Tightly Settled 1	5.7	0.223	0.983
Tightly Settled 2	3.5	0.240	0.969

4.0 Conclusions

The following conclusions were made based on the rheological and physical properties of the AN-107 and AW-101 evaporator and melter feeds. These conclusions have been divided into categories for clarity.

Evaporation

- Evaporation of the pretreated AW-101 feed to sodium concentrations of 6, 8, and 10 M, resulted in clear yellow solutions with no visible solids.
- Evaporation of the pretreated AN-107 feed to 6 and 8 M Na resulted in darkened brown solution with no visible solids.
- The 10 M Na AN-107 sample contained only ~1vol% solids following evaporation. After approximately 2 weeks, solids rapidly precipitated from solution forming a settled solids layer representing roughly 70 percent of the sample volume.

Rheology of Evaporated Feeds

- With the exception of the 10 M Na AN-107 sample at shear rates below $\sim 100 \text{ s}^{-1}$, the rheology of the AN-107 and AW-101 feeds were indistinguishable at similar sodium concentrations. The viscosities of the 6, 8, and 10 M Na feeds at 500 s^{-1} were 8, 12, and 21 cP respectively at 25°C , and 4, 7, and 12 respectively at 50°C .
- The 6, 8, and 10 M Na AW-101 feeds as well 6, and 8 M Na AN-107 feeds exhibited Newtonian behavior with no thixotropy.
- The 10 M Na AN-107 feed at 25°C exhibited Bingham behavior with yield stress of approximately 1 Pa, and a thixotropic component.

AW-101 Melter Feed Settling and Rheology

- For the 6 M Na and 8 M Na samples, the initial settling rates increase with temperature and decrease with increasing sodium concentration (i.e. 6 M Na samples settle faster than 8 M Na, and 50°C settle faster than same sample at 25°C).
- The initial settling rate of the 10 M Na sample at 25°C is higher than the same sample at 50°C and is higher than the 8 M Na sample at 25°C . The reason for this reverse in the trend observed for the 6 and 8 M Na samples is unclear from the available data. It is probably the result of several factors that could include more effective flocculation of fine particulates at higher solids content of the 10 M Na sample, competing with a higher concentration of fine soluble particulates that cannot be dissolved at the higher

temperature with the higher solids loading. This is speculative and more information would be needed if this is to be resolved.

- The AW-101 melter feed samples show nearly Newtonian behavior with no thixotropy or yield stresses observed. The viscosities at 500 s^{-1} of the 6, 8, and 10 M feeds were 36, 88, and 230 cP respectively at 25°C , and 16, 46, and 130 cP respectively at 50°C .

8M Na AW-101 Melter Feed Mixing and Aging

- The viscosity (at 500 s^{-1} and 25°C) of the slurry increased from 52 cP after 1 hour of mixing to 67 cP after 1 day. The viscosity then remained essentially constant at 65 cP after 1 week of mixing. For comparison, the viscosity of the 8 M Na melter feed sample that was used in settling studies was 88 cP at 500 s^{-1} and 25°C . This other sample was analyzed approximately 3 weeks after glass former addition with only occasional stirring. This suggests that a combination of additional aging and lack of mixing could result in an additional increase in viscosity.
- Rheograms of slurry samples analyzed after 1 hour, 1 day, and 1 week show nearly Newtonian behavior with no thixotropy or yield stresses.
- No gas retention or releases were observed during 1 week of settling.
- After 1 week of settling, 2 settled solids layers formed. The upper layer appeared to have a lower solids content while the lower layer had a thicker consistency.
- Rheograms of the two separate settled solid layers exhibit Bingham behavior with a similar yield point of 4.6 Pa. Both layers displayed a thixotropic component.

Appendix A: Figures for AN-107 & AW-101

Appendix B: Test Plan (BNFL-TP-29953-046)

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Appendix A: Figures for AN-107 & AW-101

1922-1923

Figure 1. 95.5 cP Brookfield Lot 111199 at 25°C 3/16/00

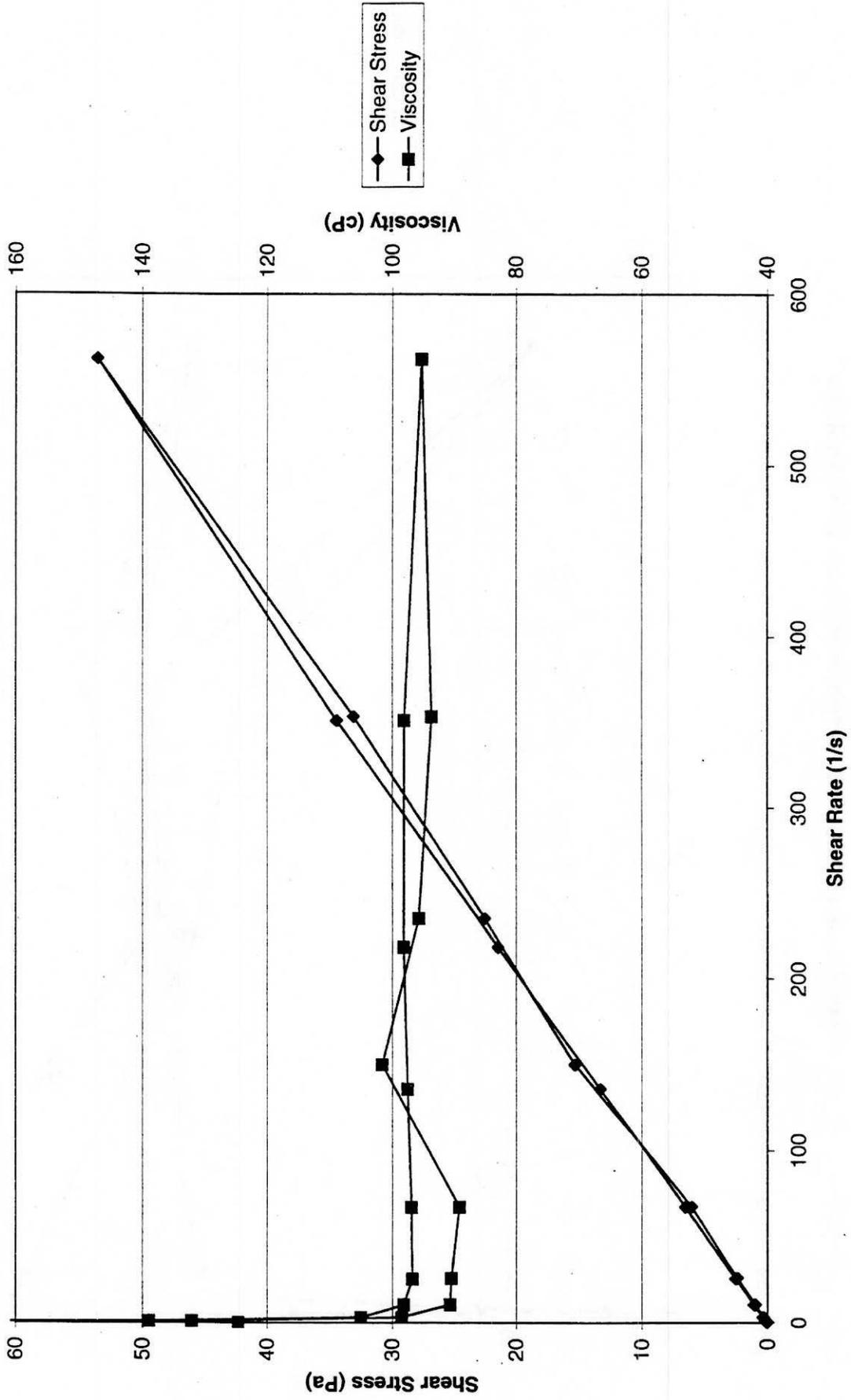


Figure 2. AN-107 6M Na Evaporated Feed: 25°C Analysis 1

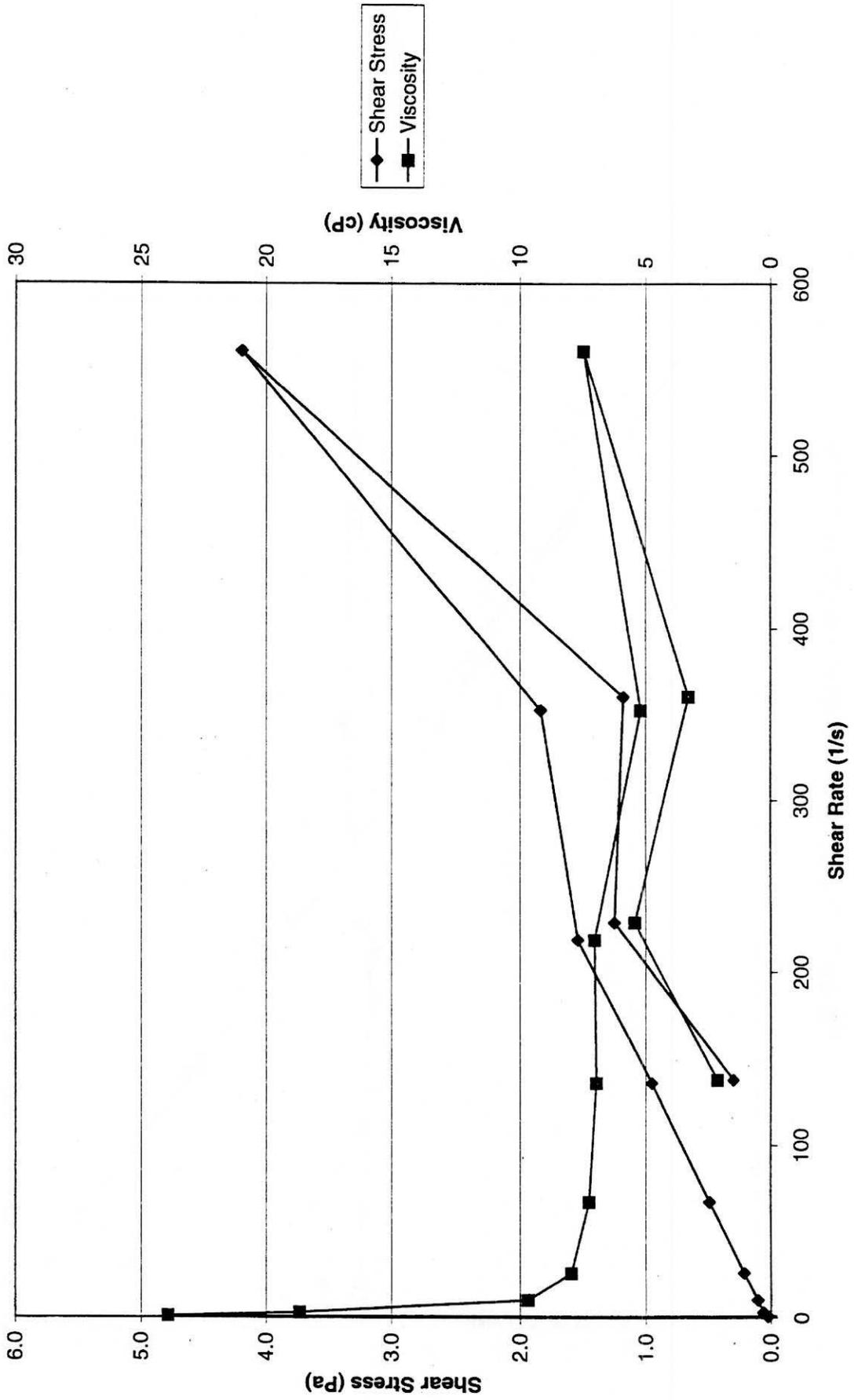


Figure 3. AN-107 6M Na Evaporated Feed: 25°C Analysis 2

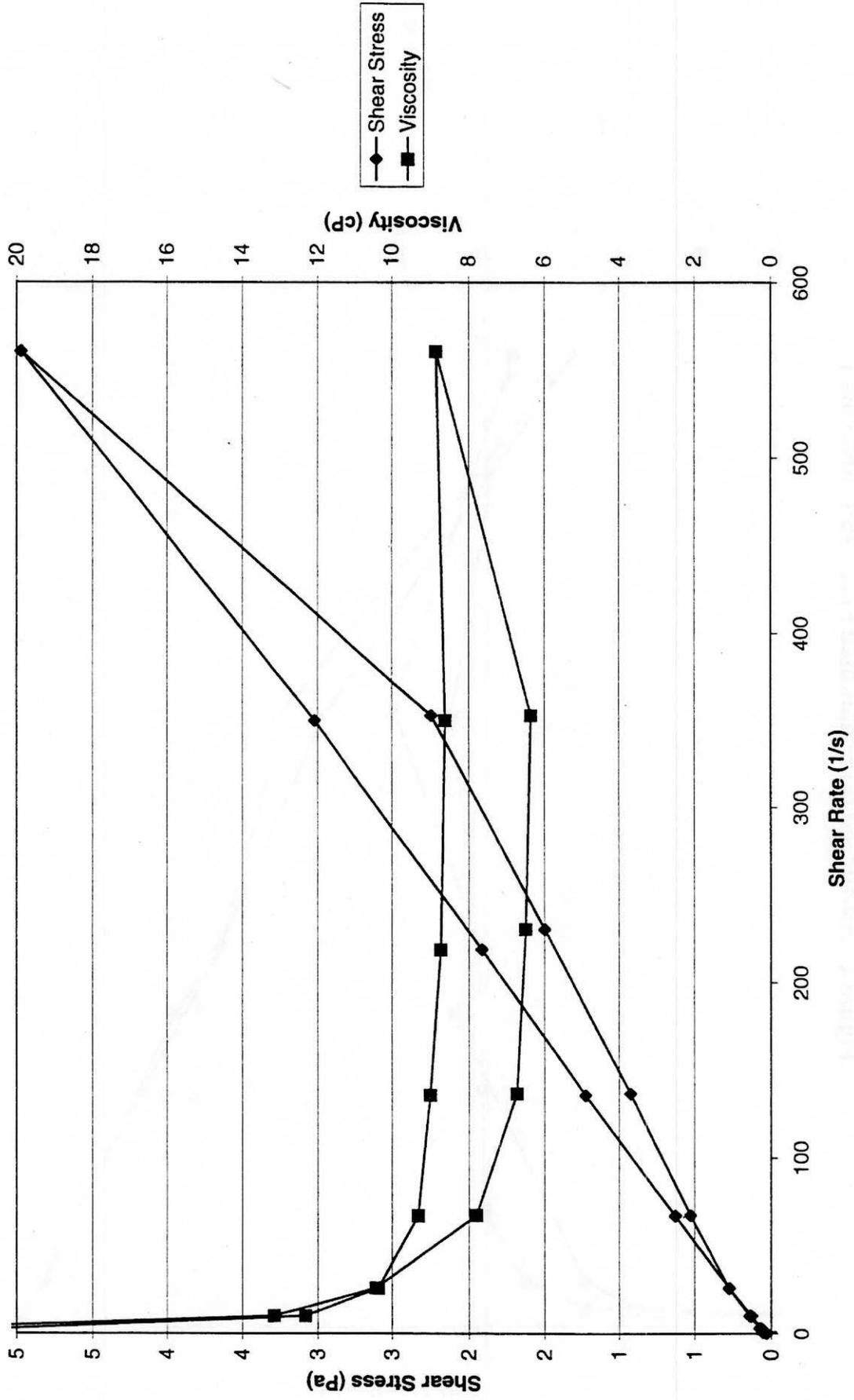


Figure 4. AN-107 8M Na Evaporated Feed: 25°C Analysis 1

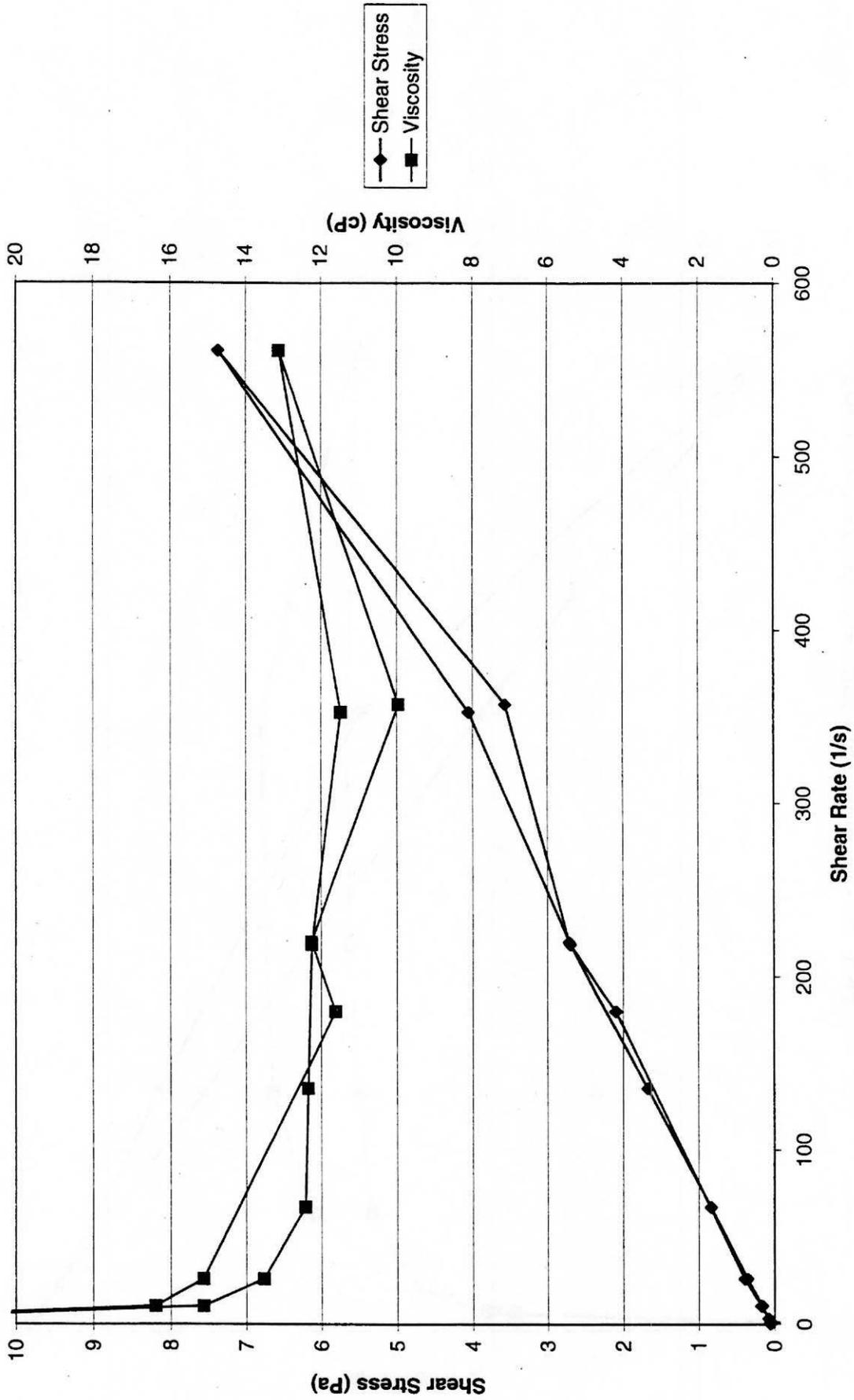


Figure 5. AN-107 8M Na Evaporated Feed: 25°C Analysis 2

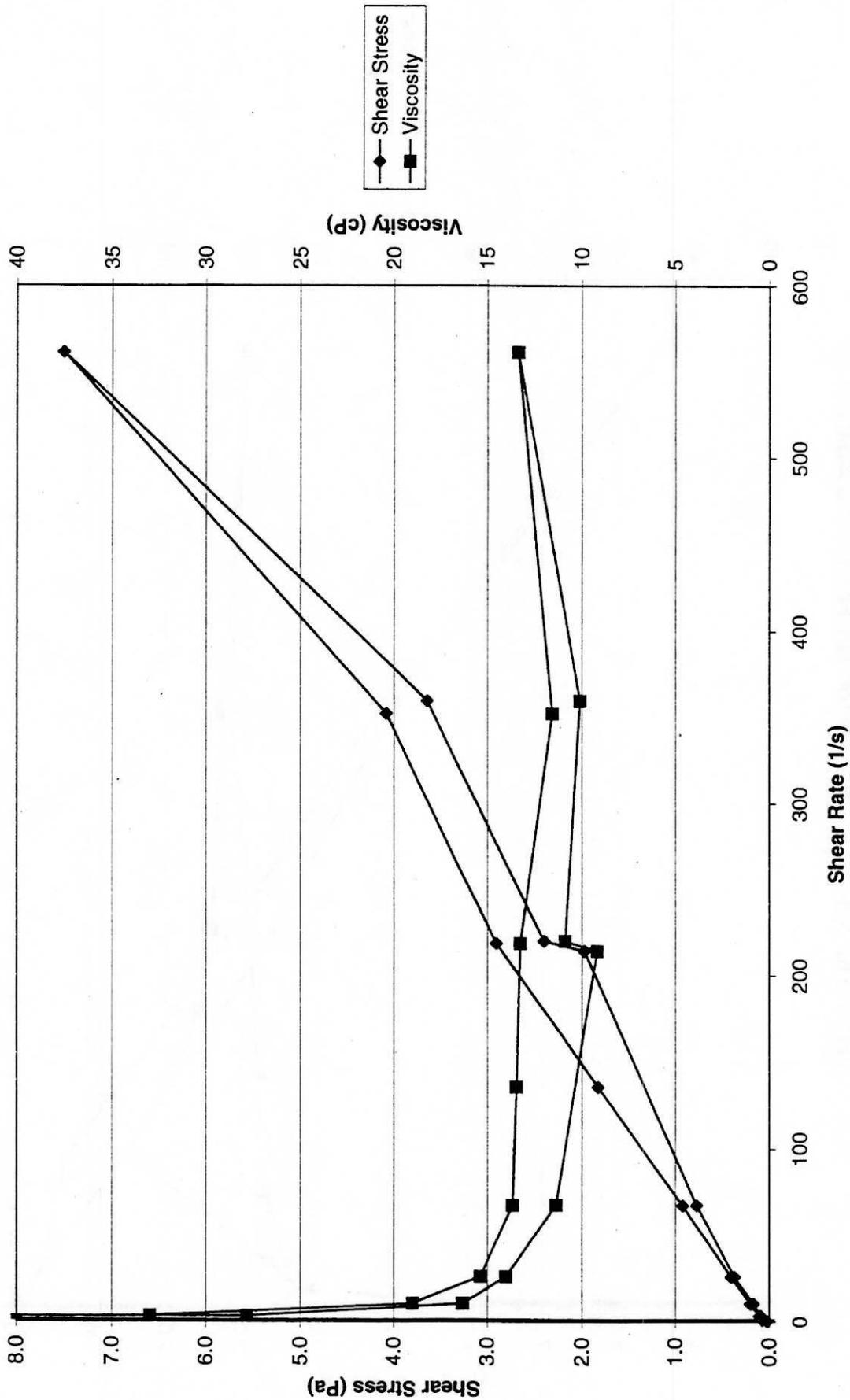


Figure 6. AN-107 8M Na Evaporated Feed: 25°C Analysis 3

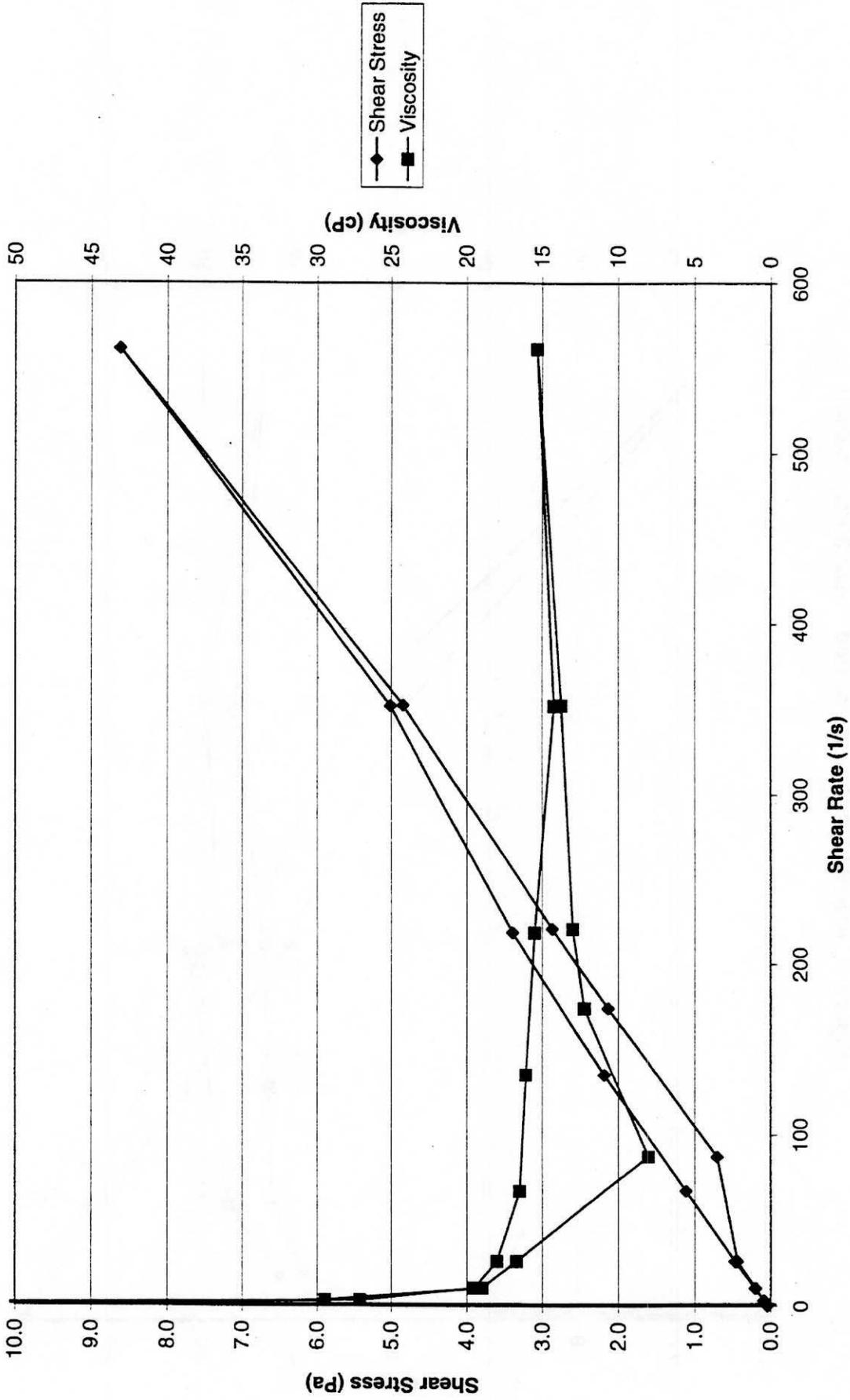


Figure 7. AN-107 10M Na Evaporated Feed: 25°C Analysis 1

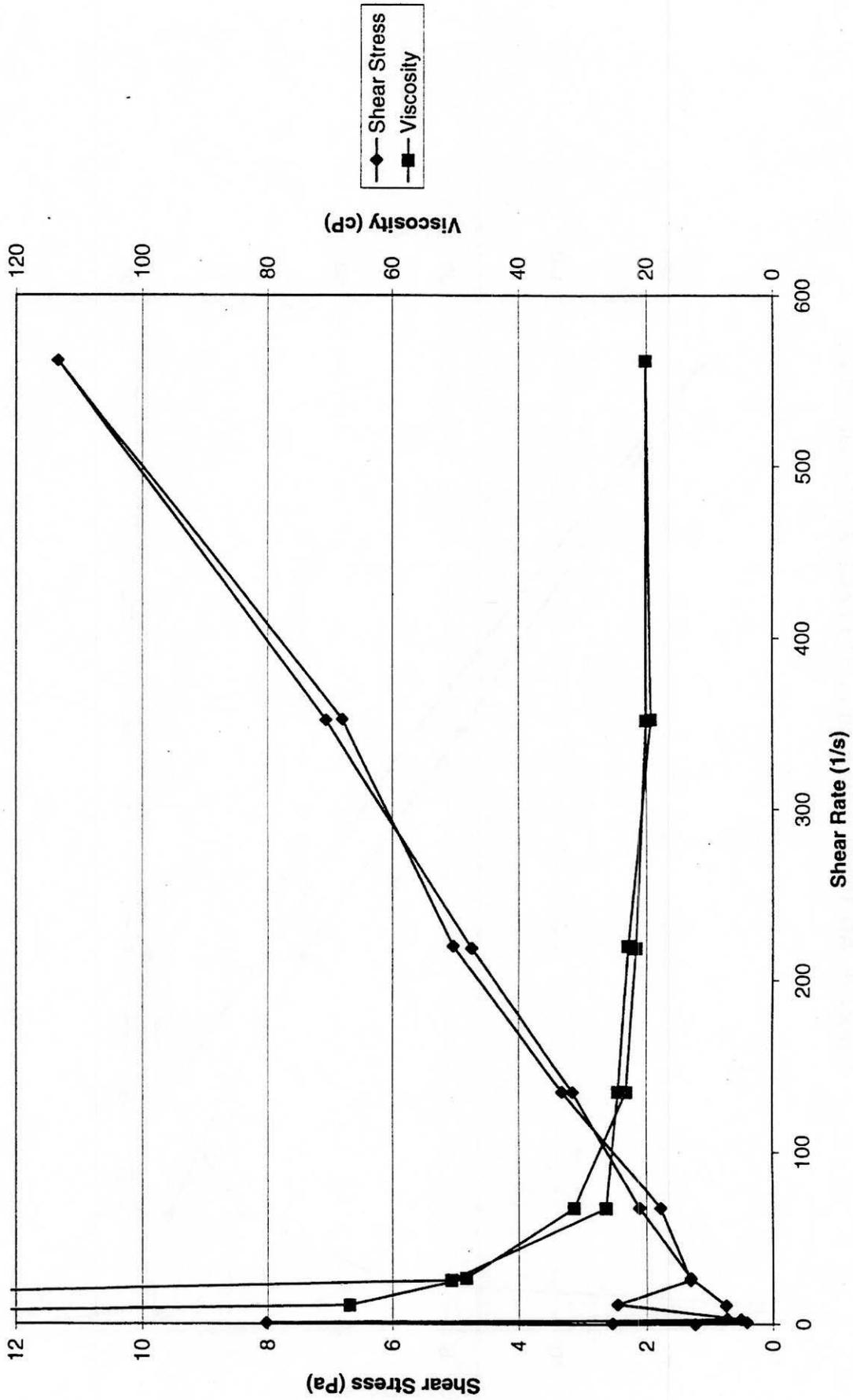


Figure 8. AN-107 10M Na Evaporated Feed: 25°C Analysis 2

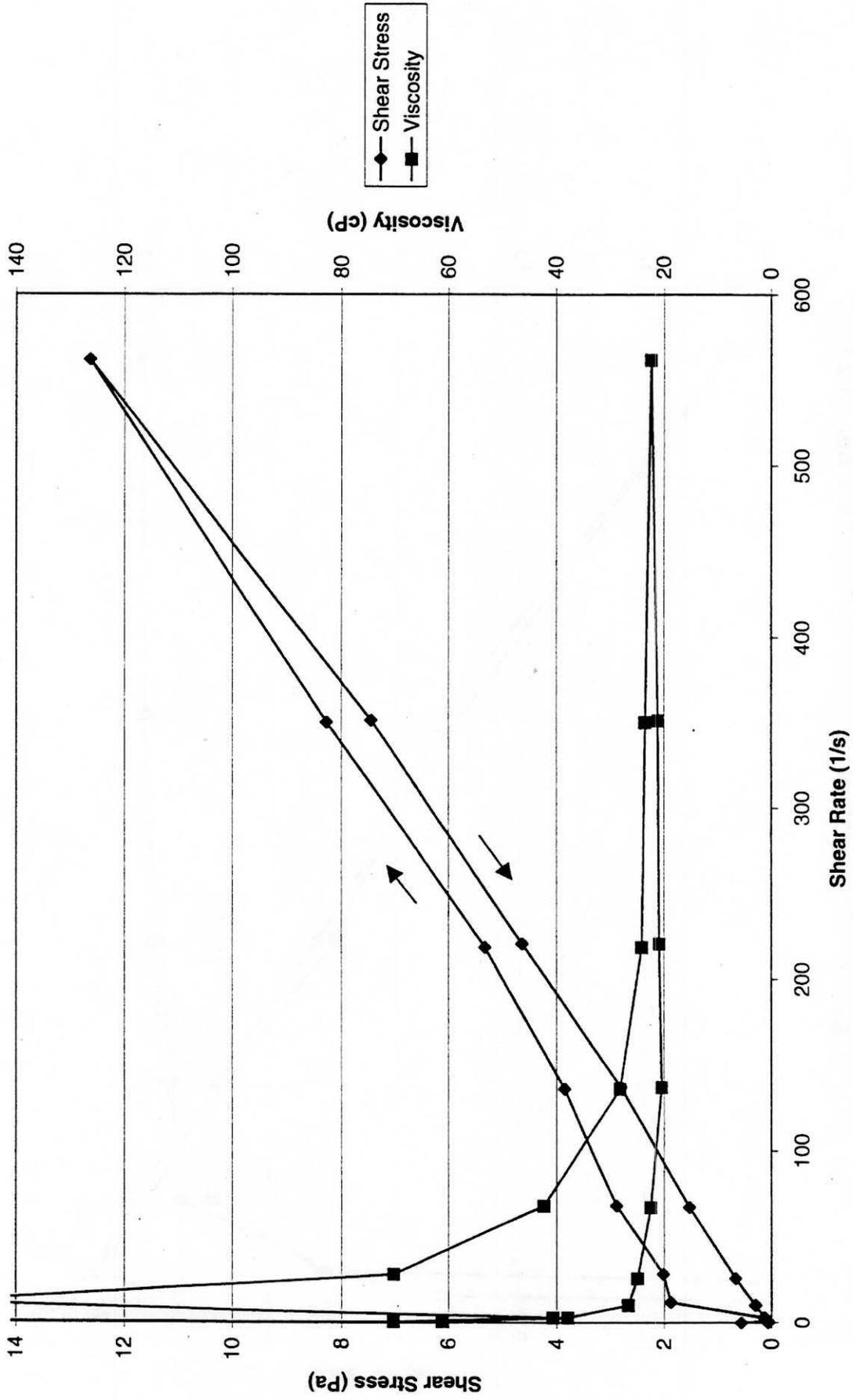


Figure 9. AN-107 6M Na Evaporated Feed: 50°C Analysis 1

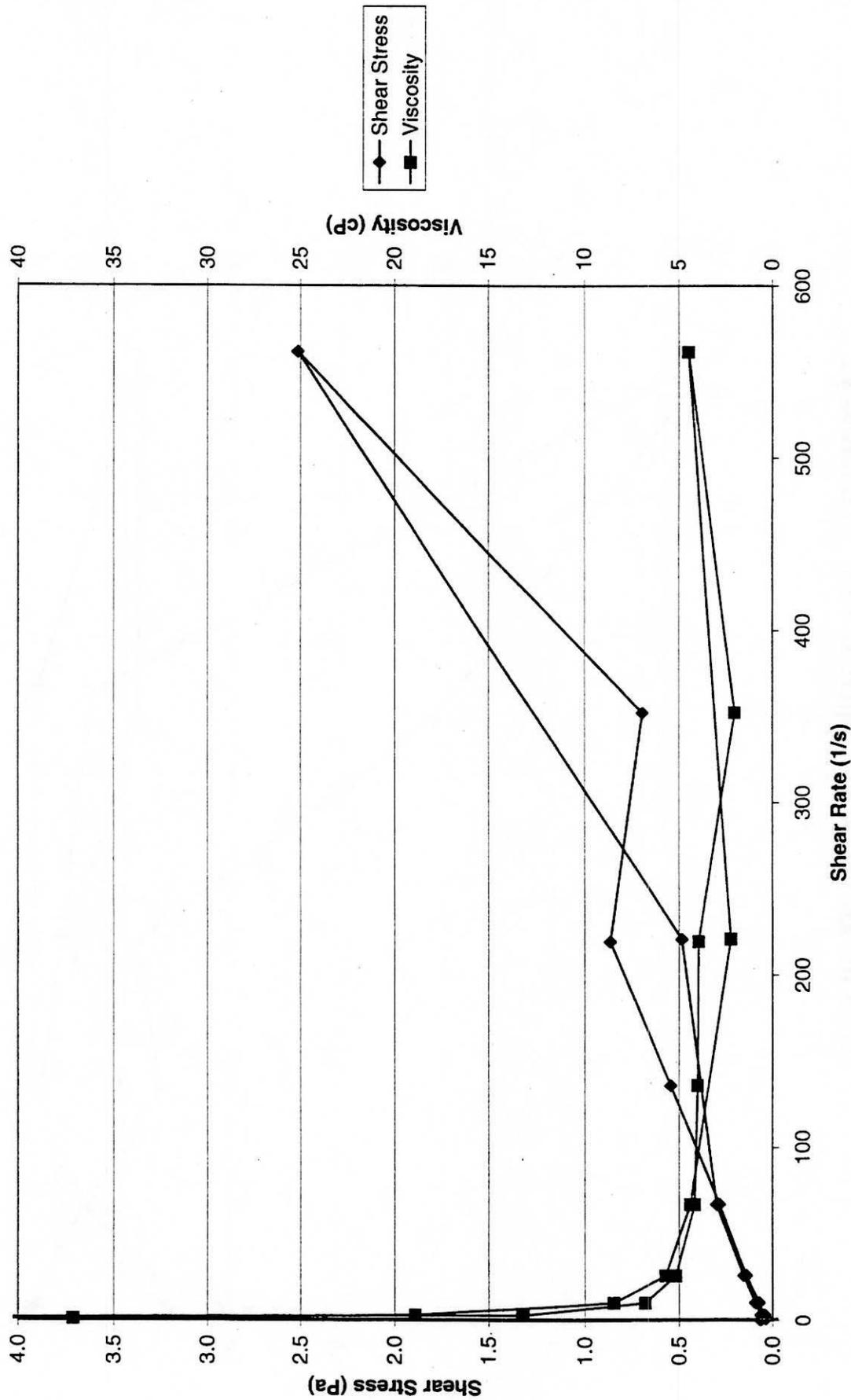


Figure 10. AN-107 6M Na Evaporated Feed: 50°C Analysis 2

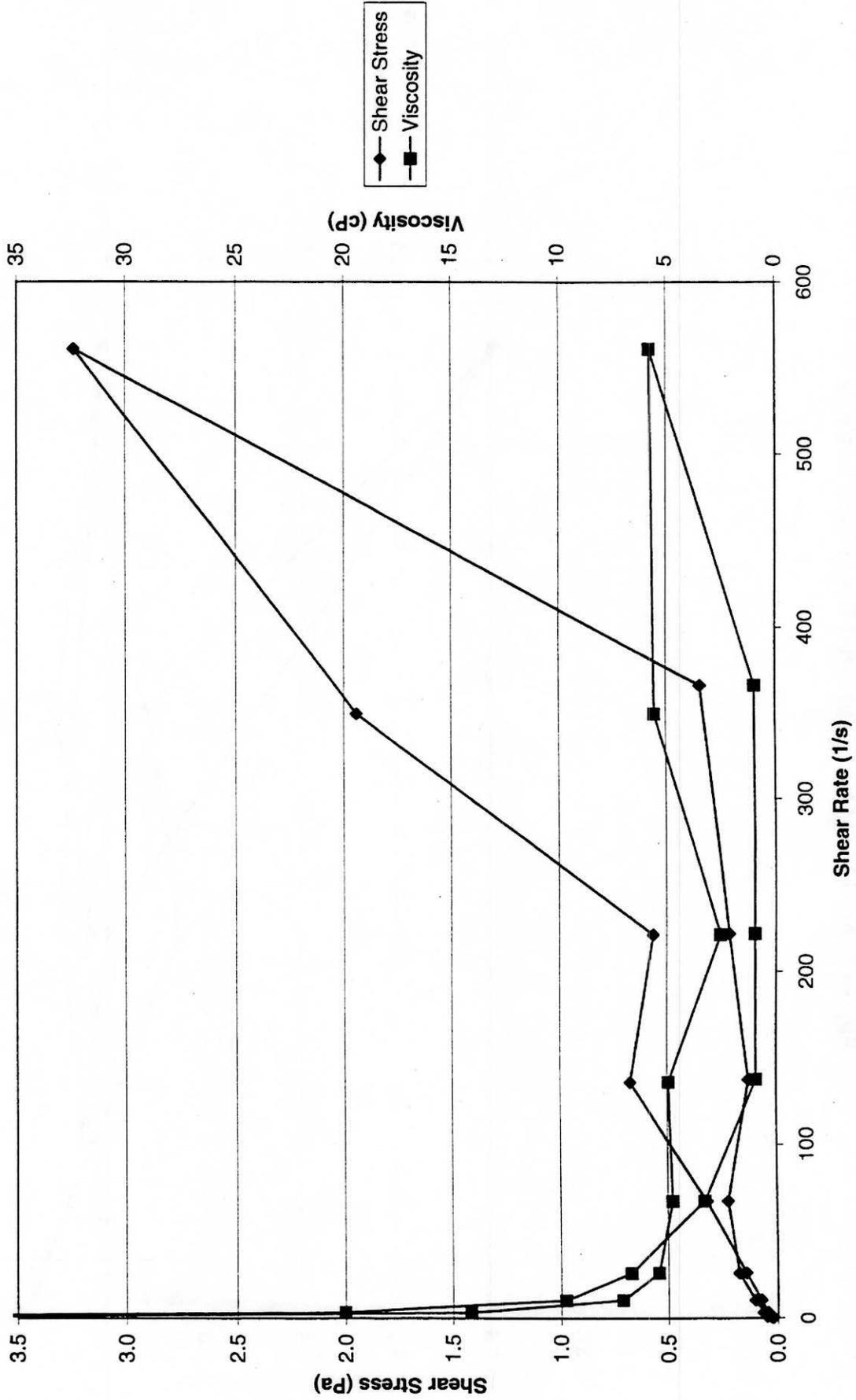


Figure 11. AN-107 8M Na Evaporated Feed: 50°C Analysis 1

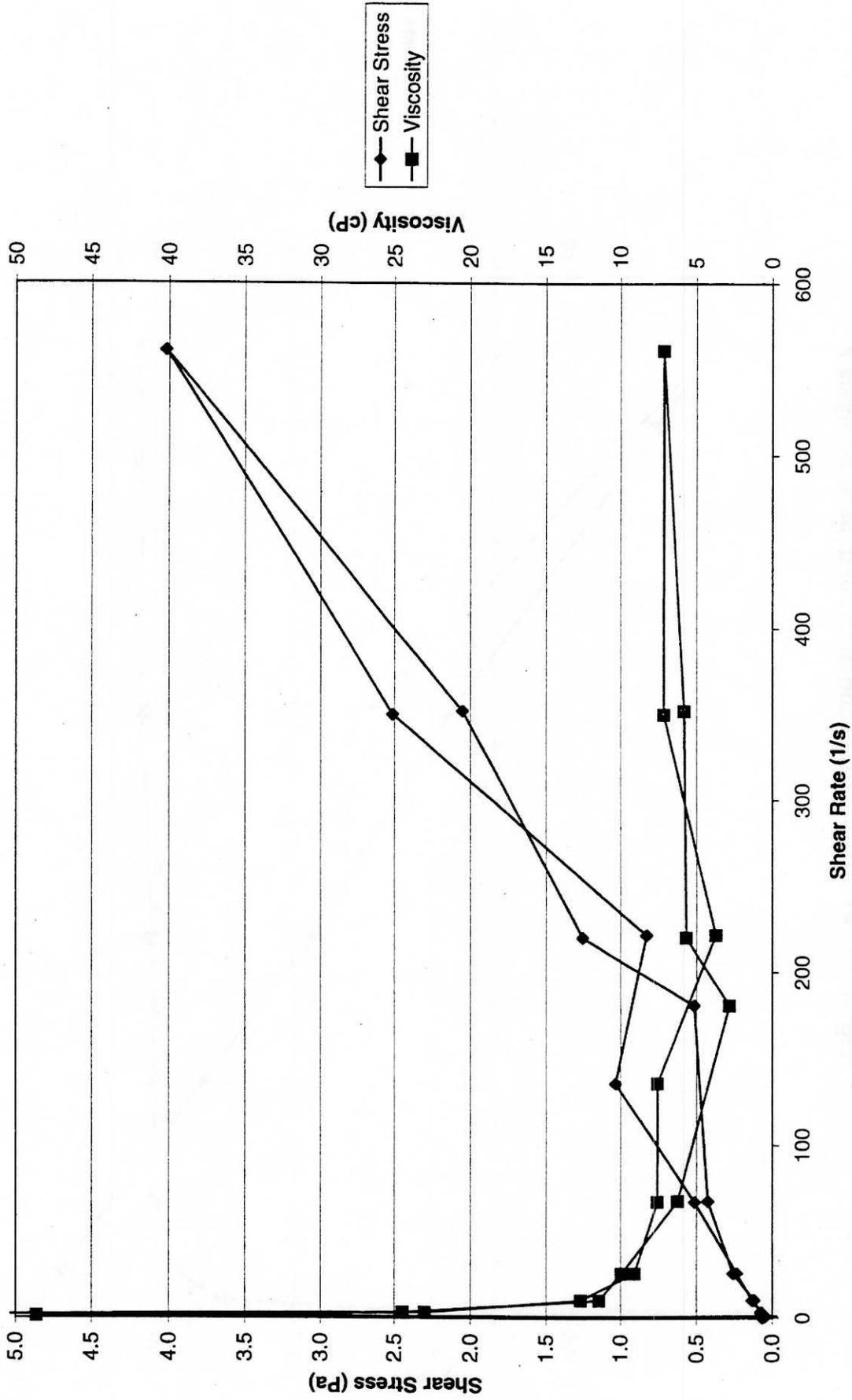


Figure 12. AN-107 8M Na Evaporated Feed: 50°C Analysis 2

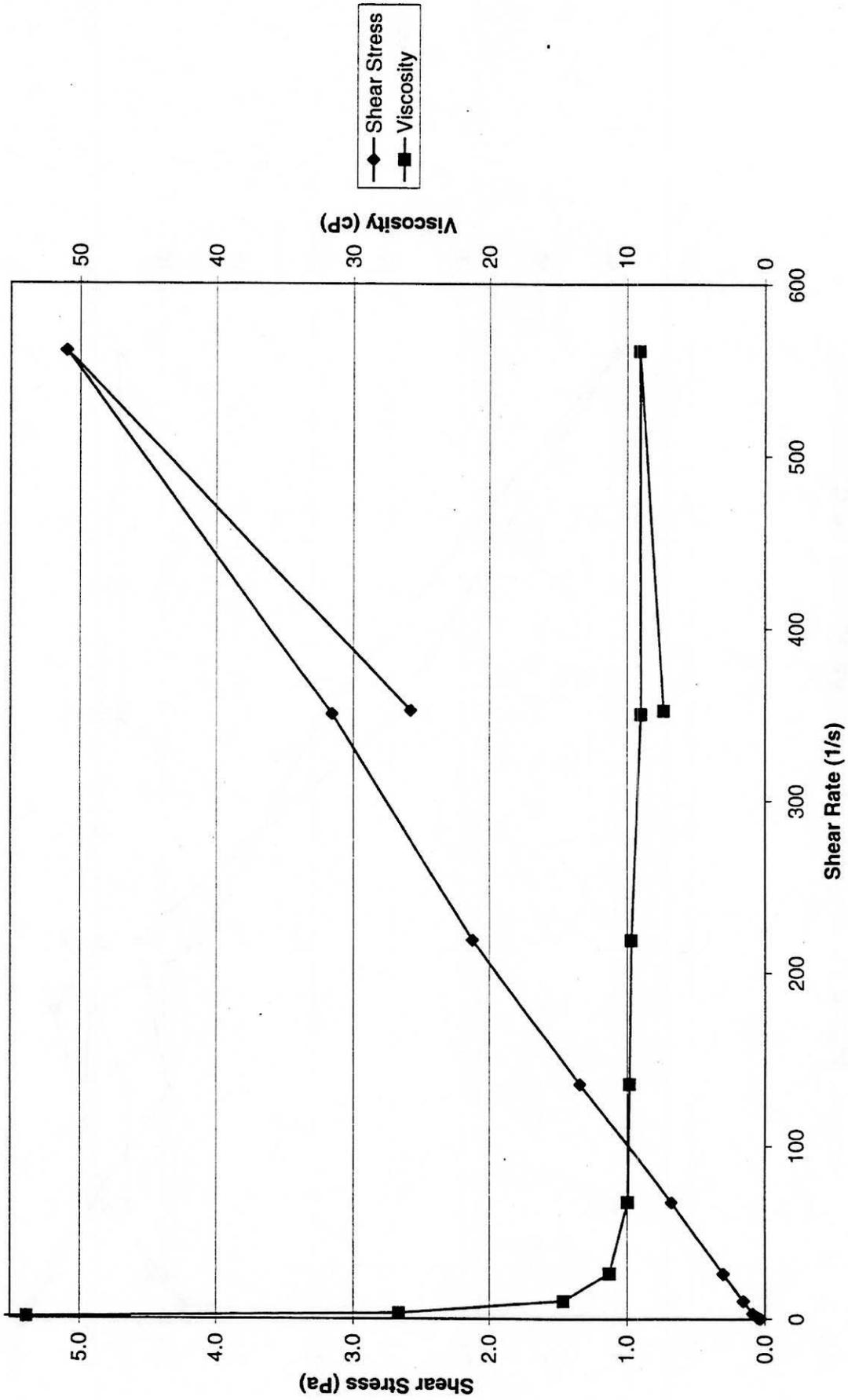


Figure 13. AN-107 10M Na Evaporated Feed: 50°C Analysis 1

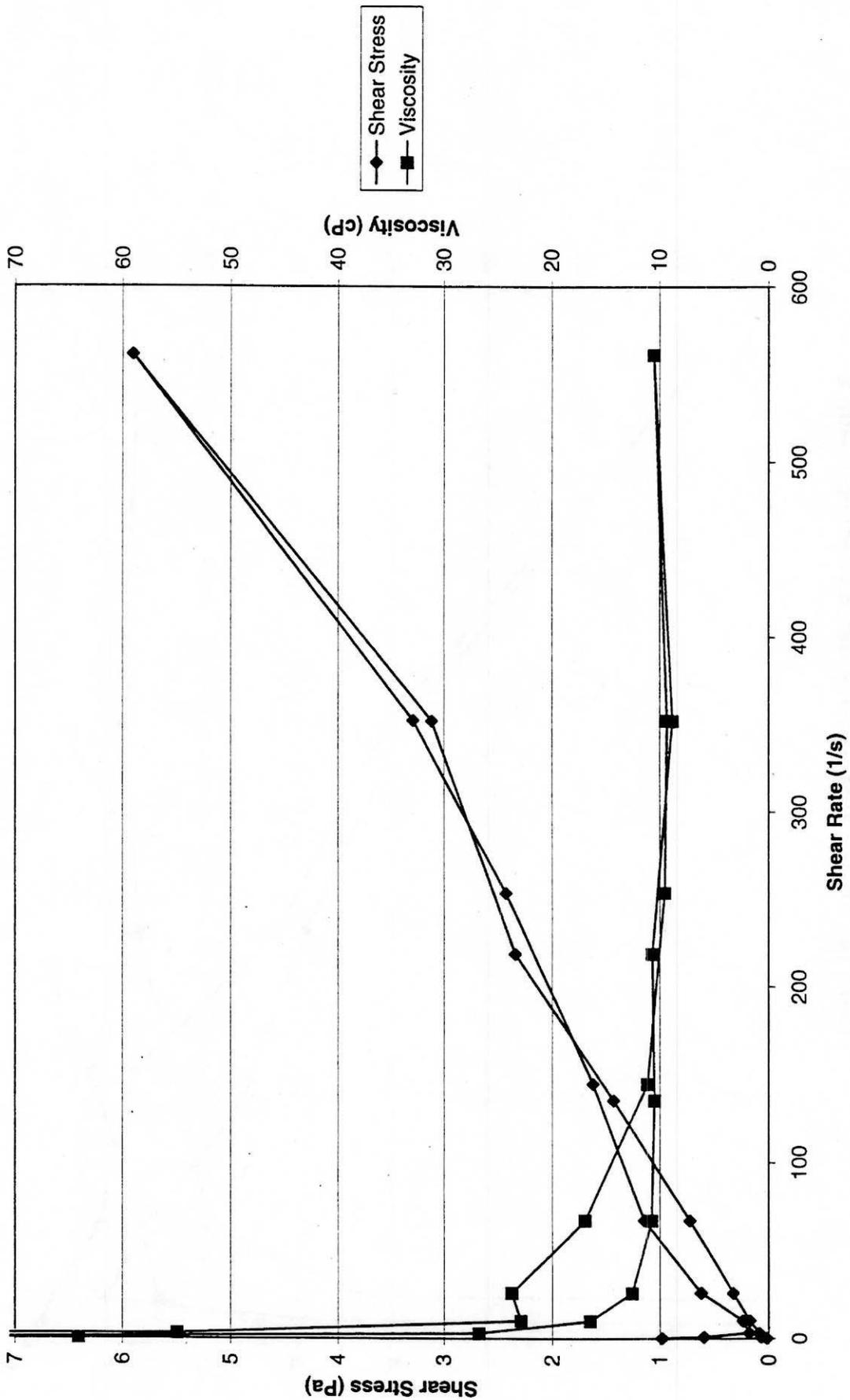


Figure 14. AN-107 10M Na Evaporated Feed: 50°C Run 2

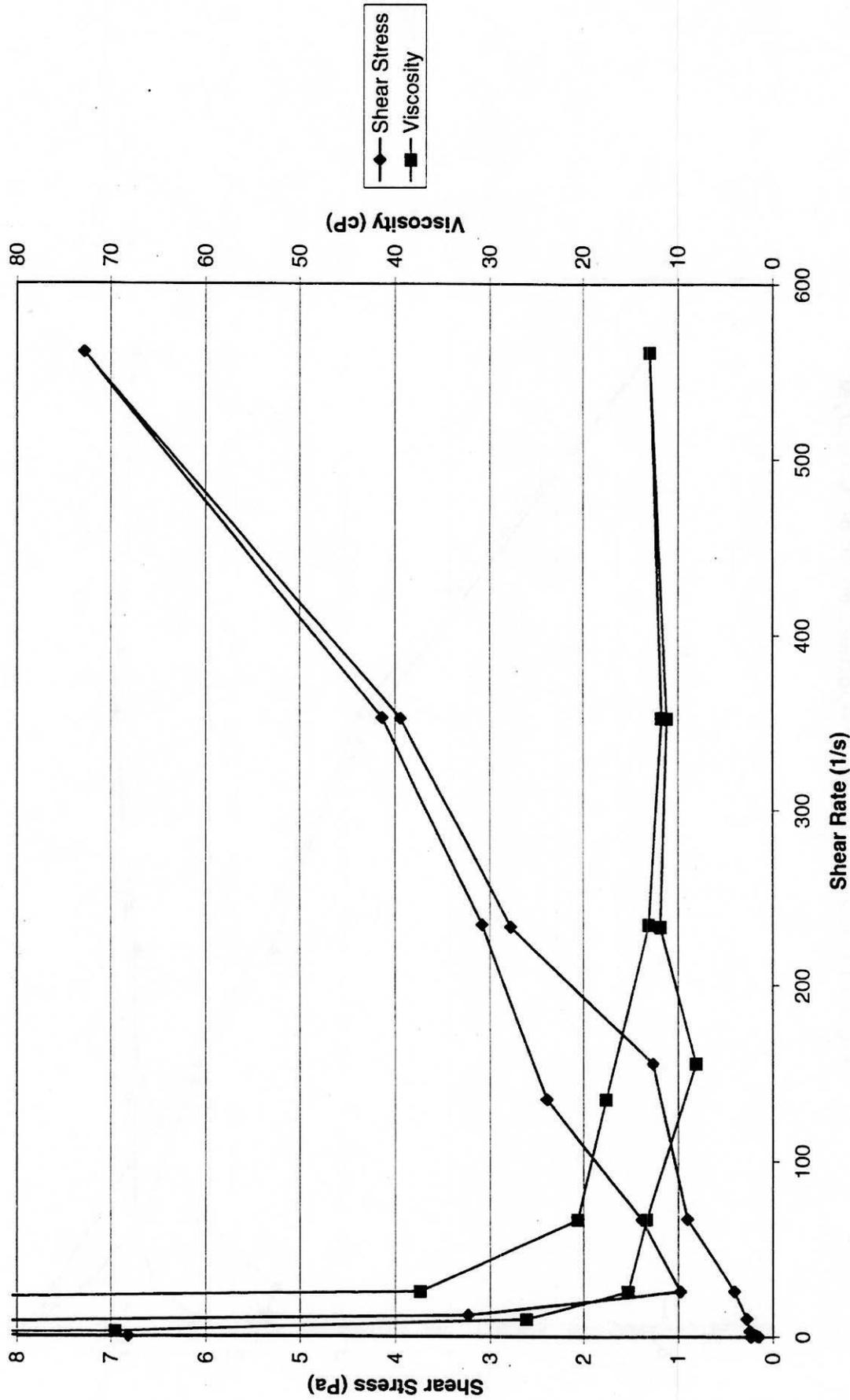


Figure 15. 47.2 cP Standard Brookfield PNNL Barcode 179430 Analyzed 02/02/00

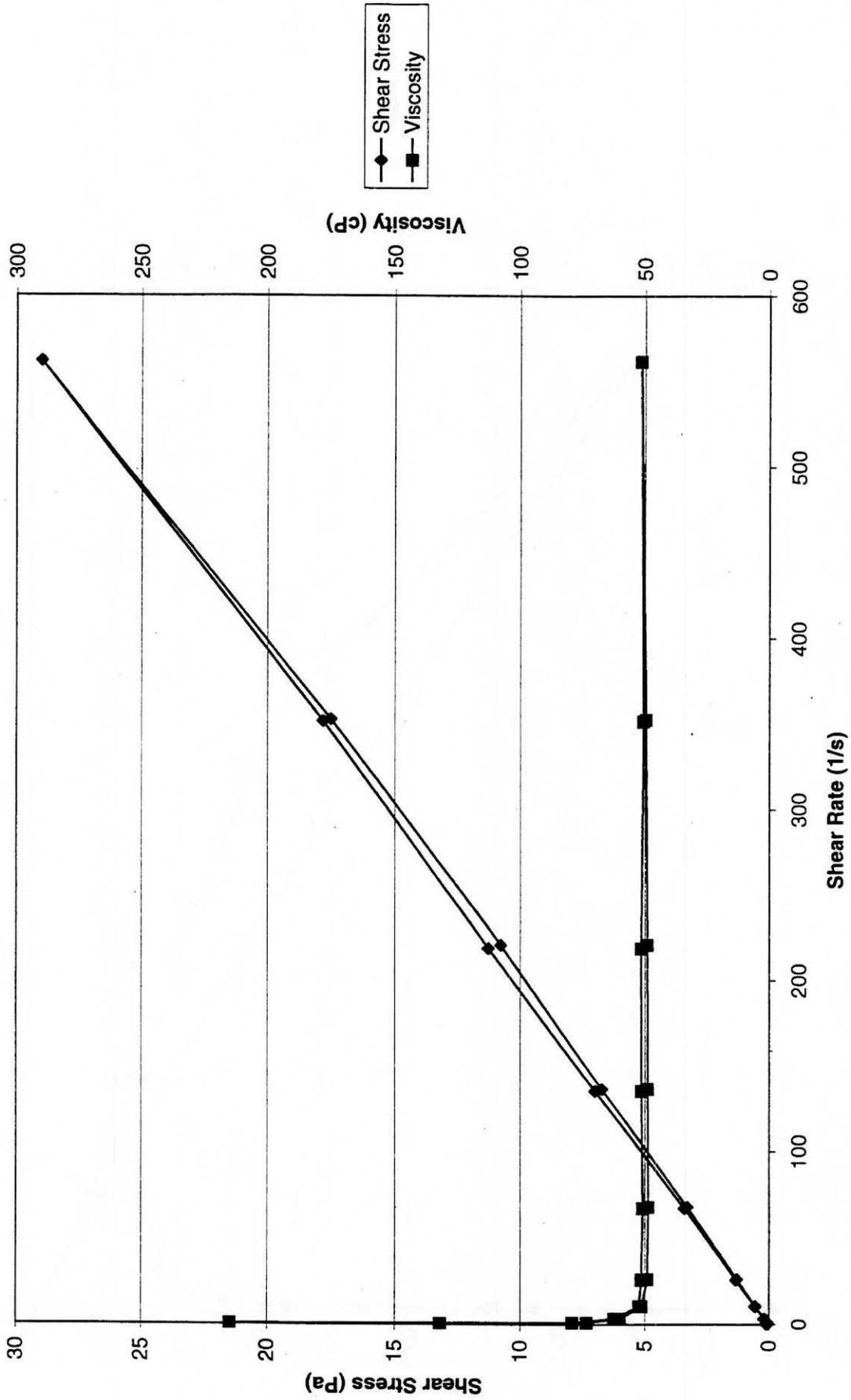


Figure 16. AW-101 6M Na Evaporated Feed: 25°C Analysis 1

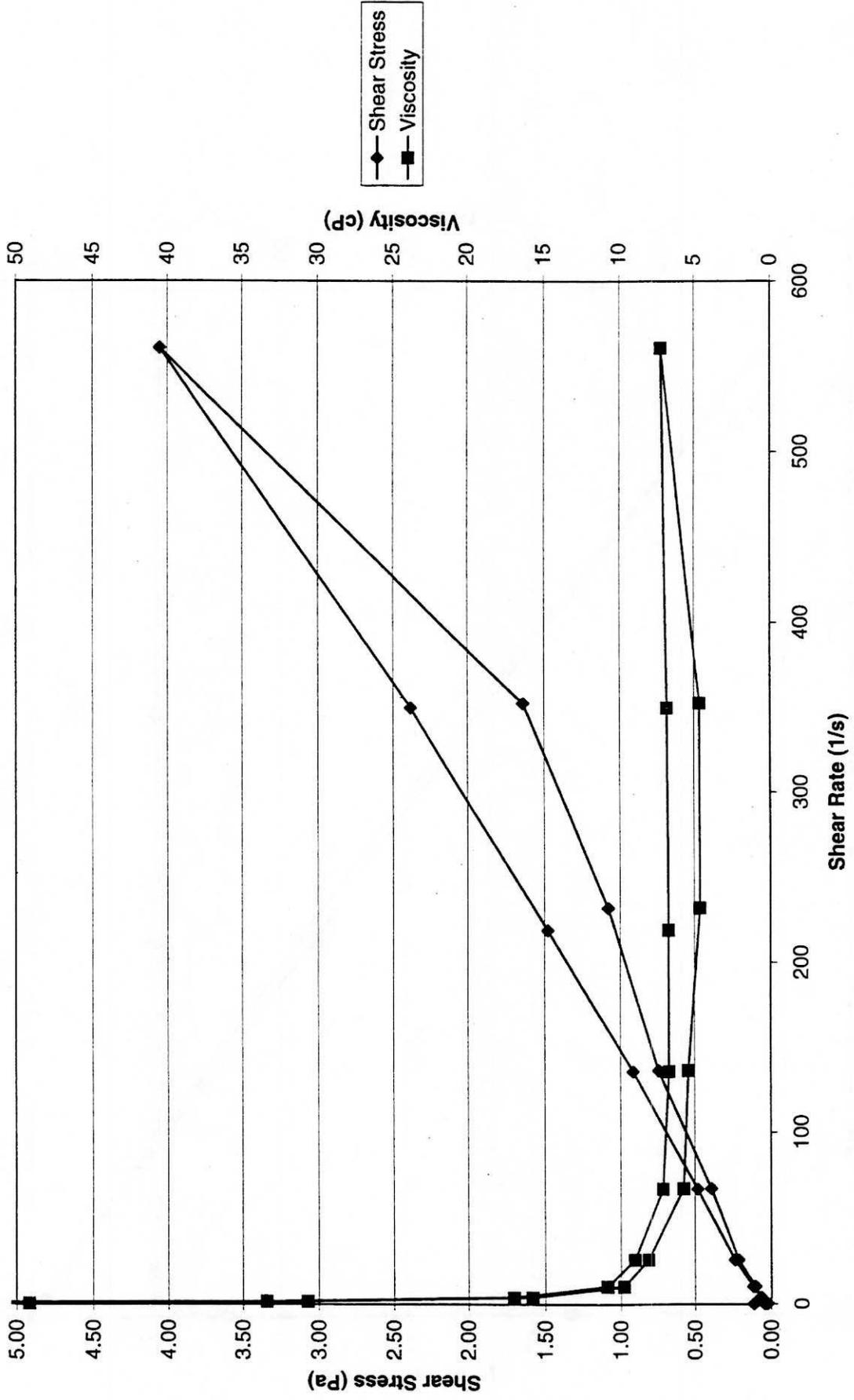


Figure 17. AW-101 6M Na Evaporated Feed: 25°C Analysis 2

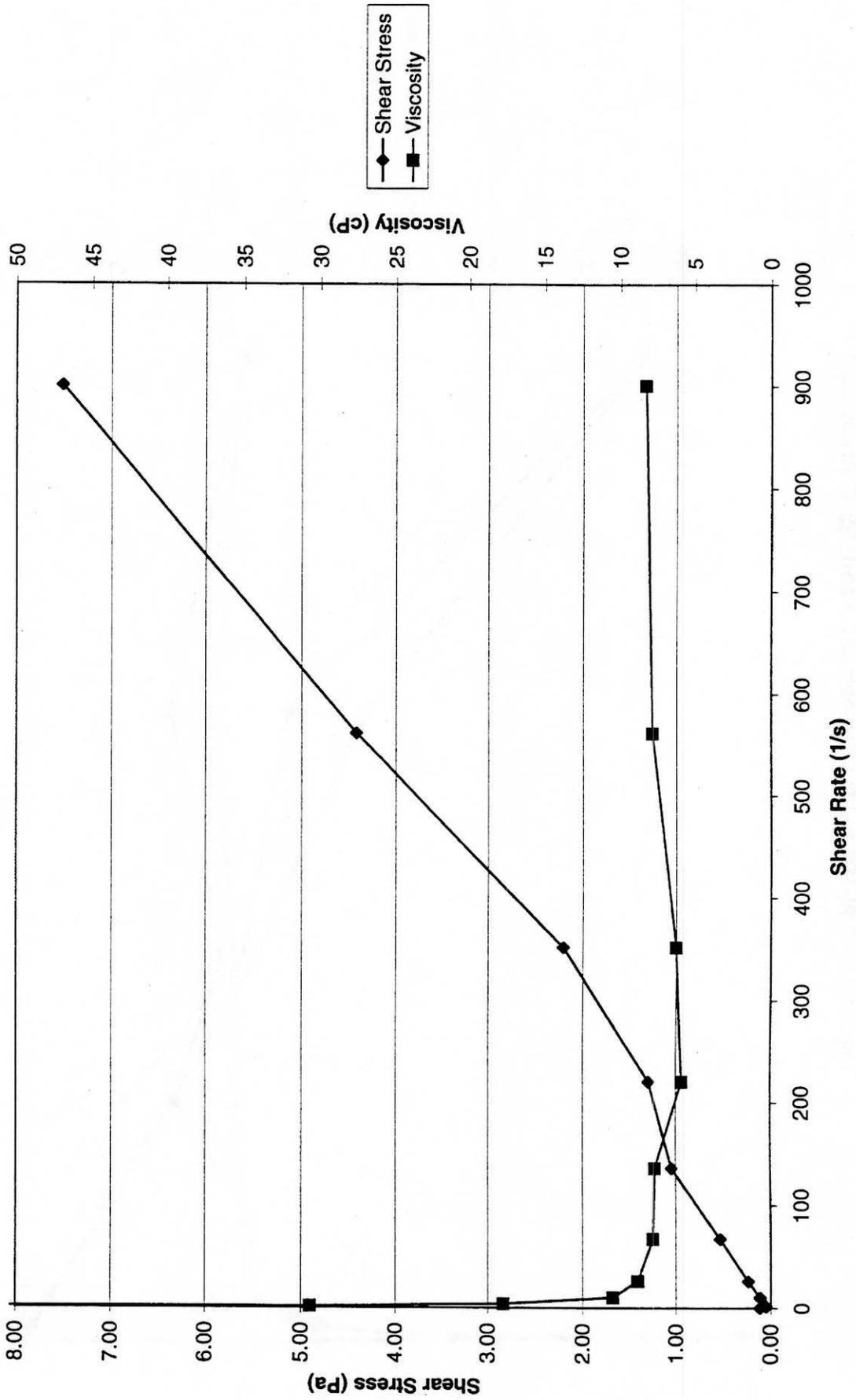


Figure 18. AW-101 8M Na Evaporated Feed: 25°C Analysis 1

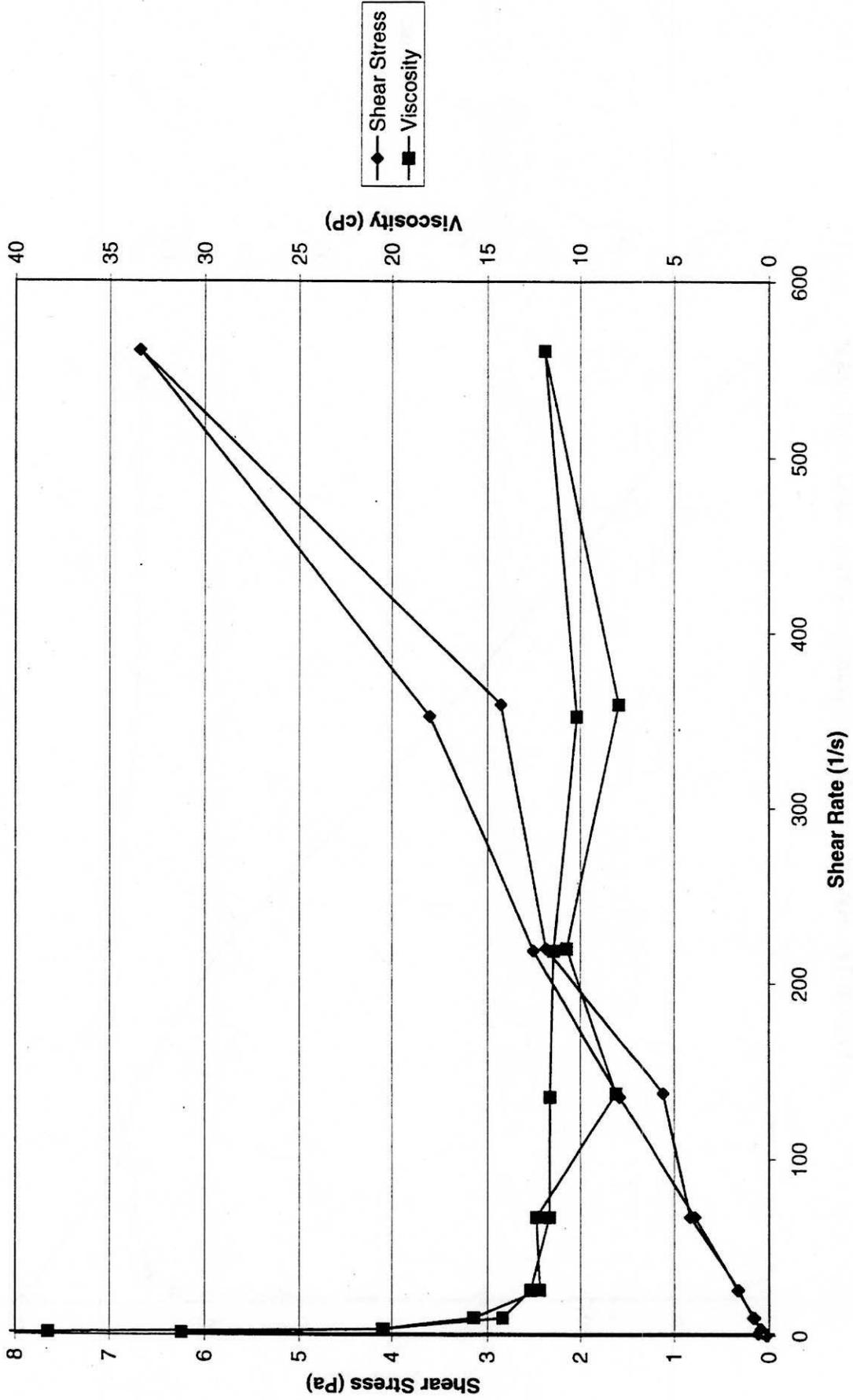


Figure 19. AW-101 8M Na Evaporated Feed: 25°C Analysis 2

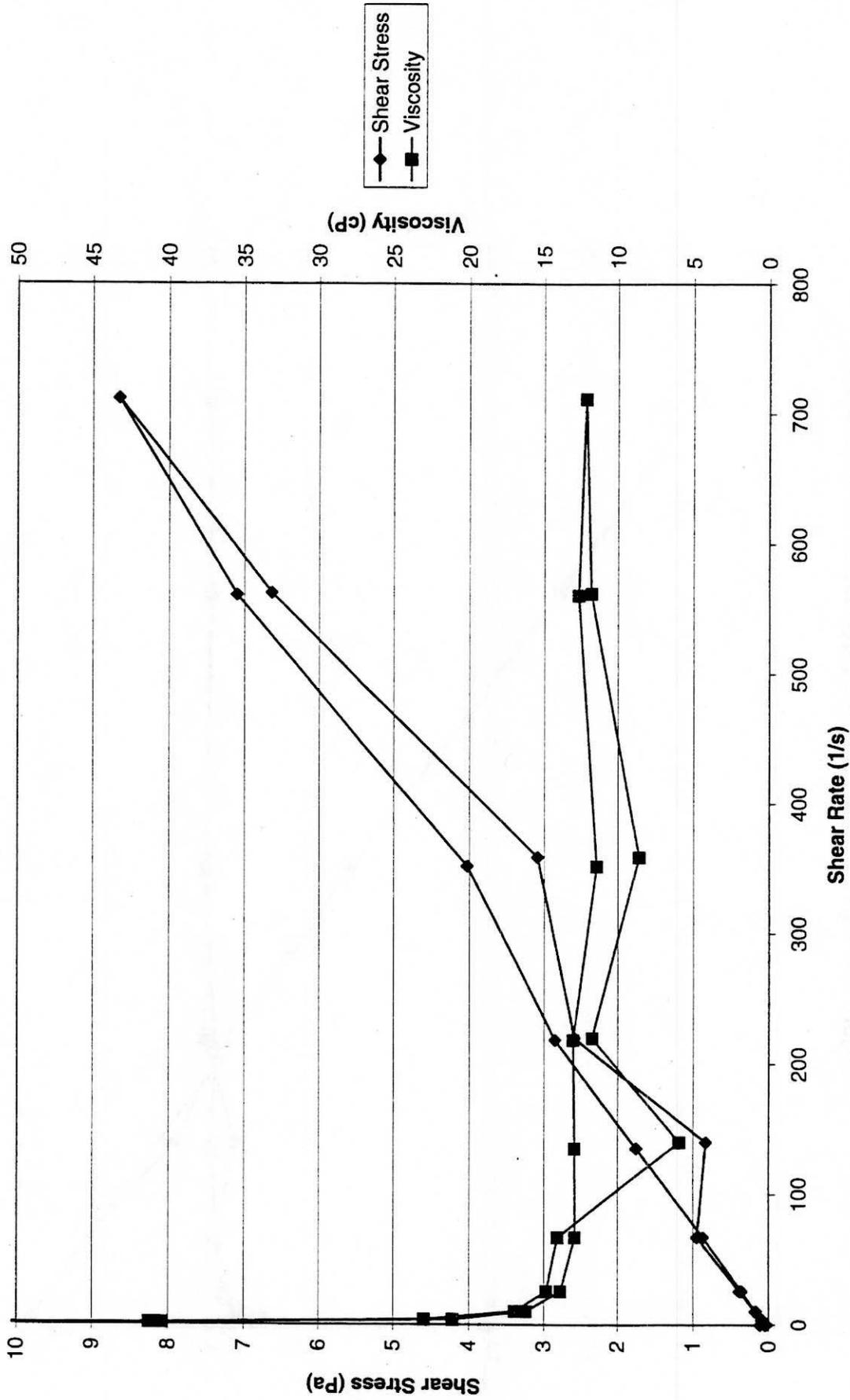


Figure 20. AW-101 10M Na Evaporated Feed: 25°C Analysis 1

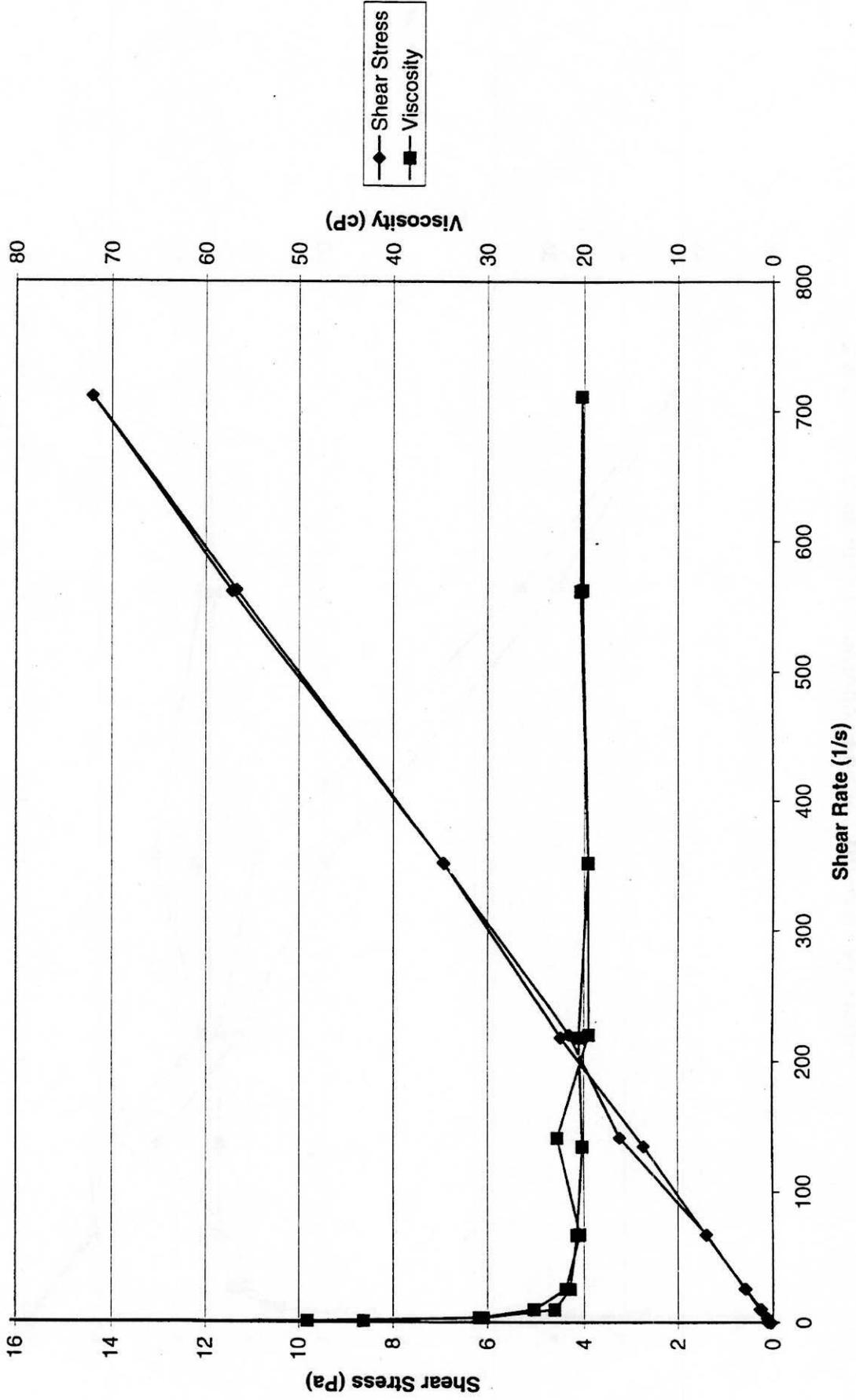


Figure 21. AW-101 10M Na Evaporated Feed: 25°C Analysis 2

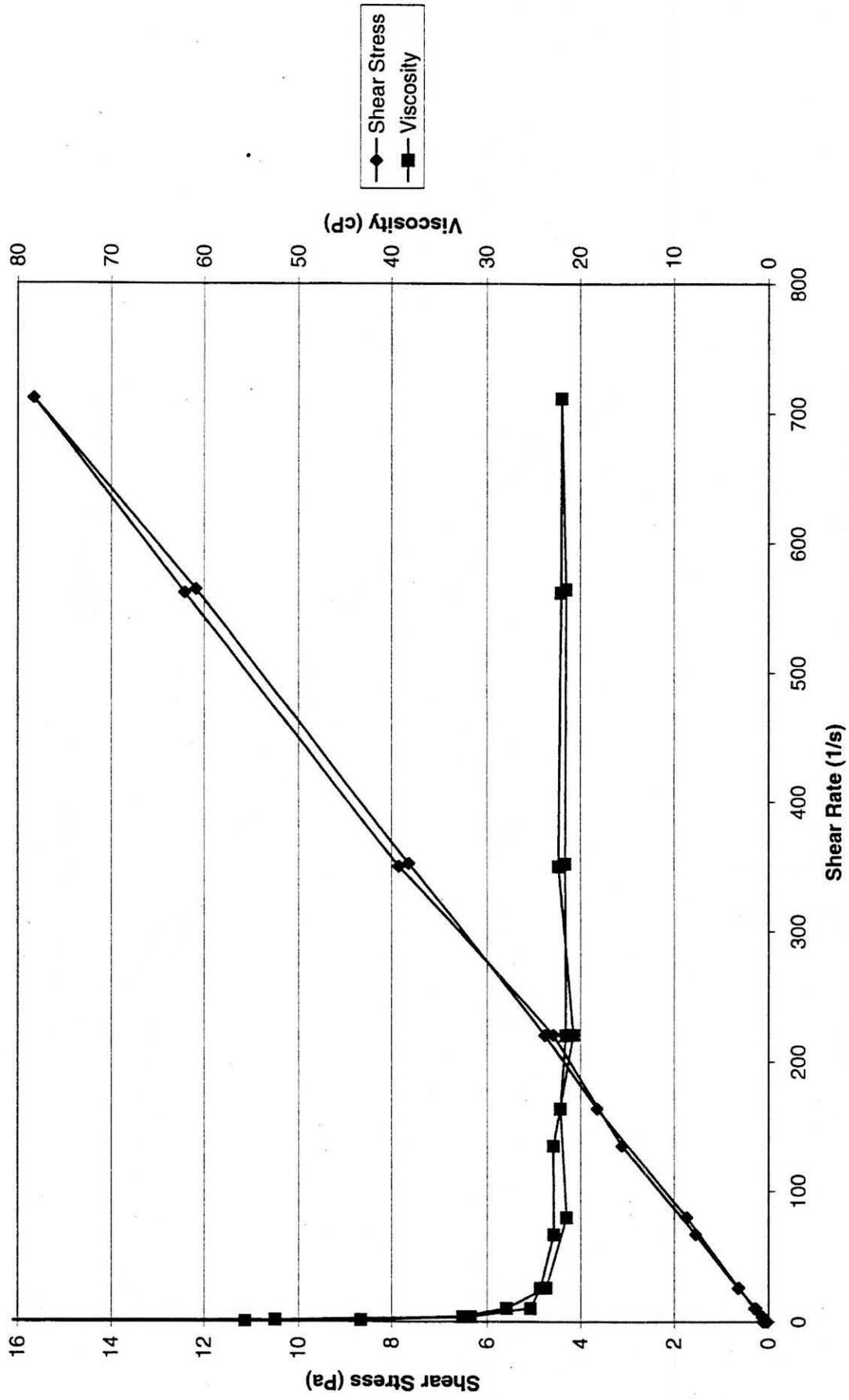


Figure 22. AW-101 6M Na Evaporated Feed: 50°C Analysis 1

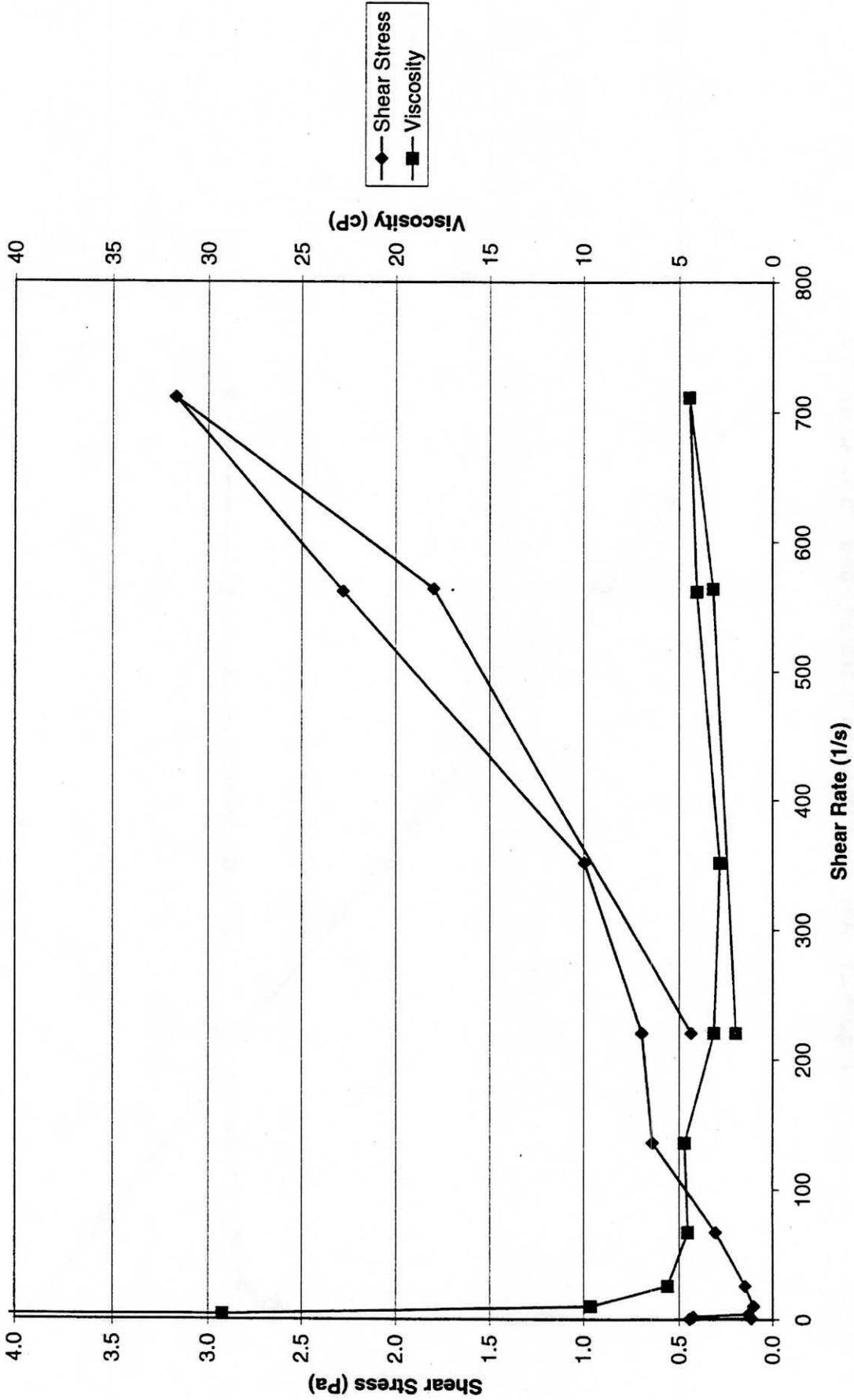


Figure 23. AW-101 6M Na Evaporated Feed: 50°C Analysis 2

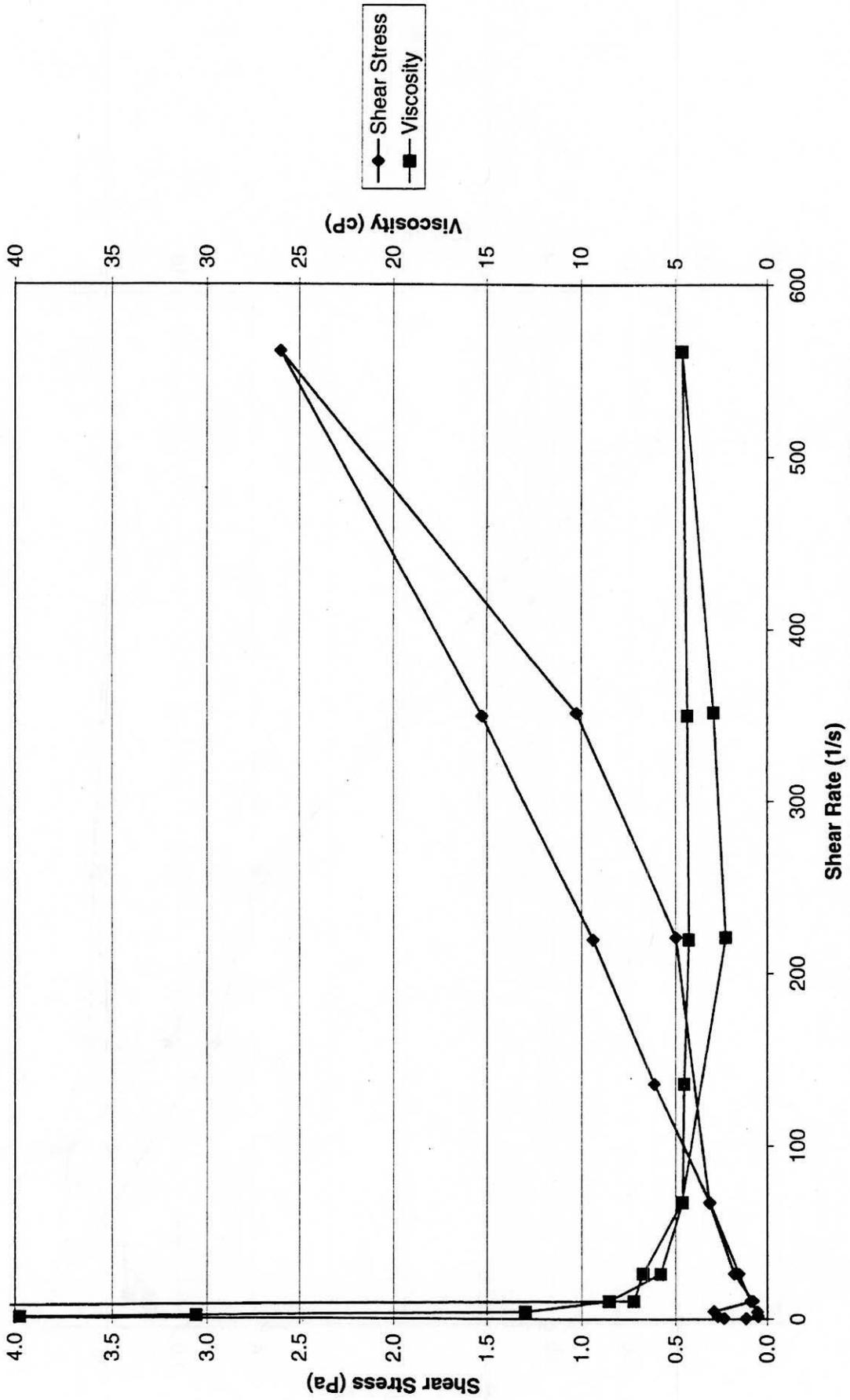


Figure 24. AW-101 8M Na Evaporated Feed: 50°C Analysis 1

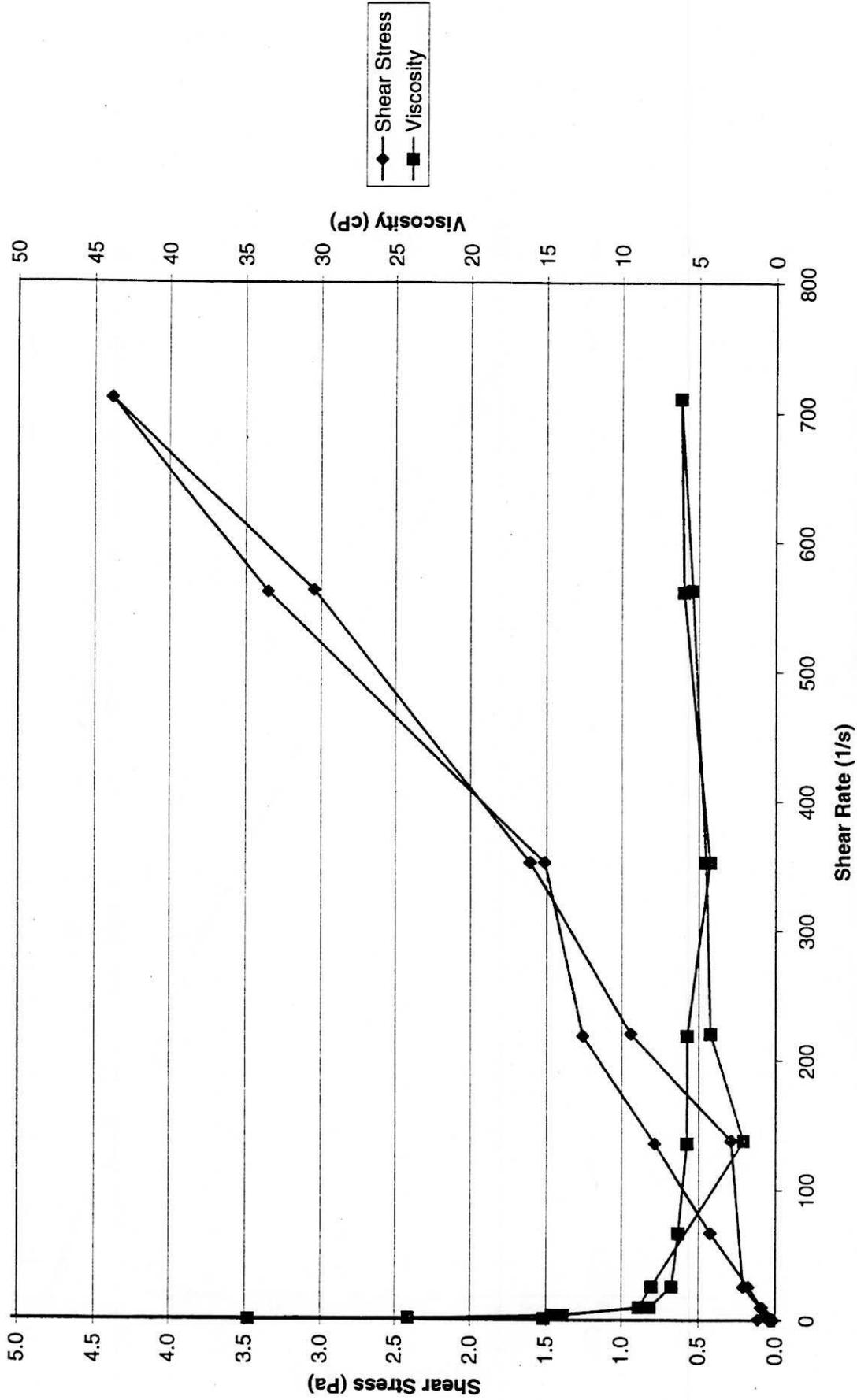


Figure 25. AW-101 8M Na Evaporated Feed: 50°C Analysis 2

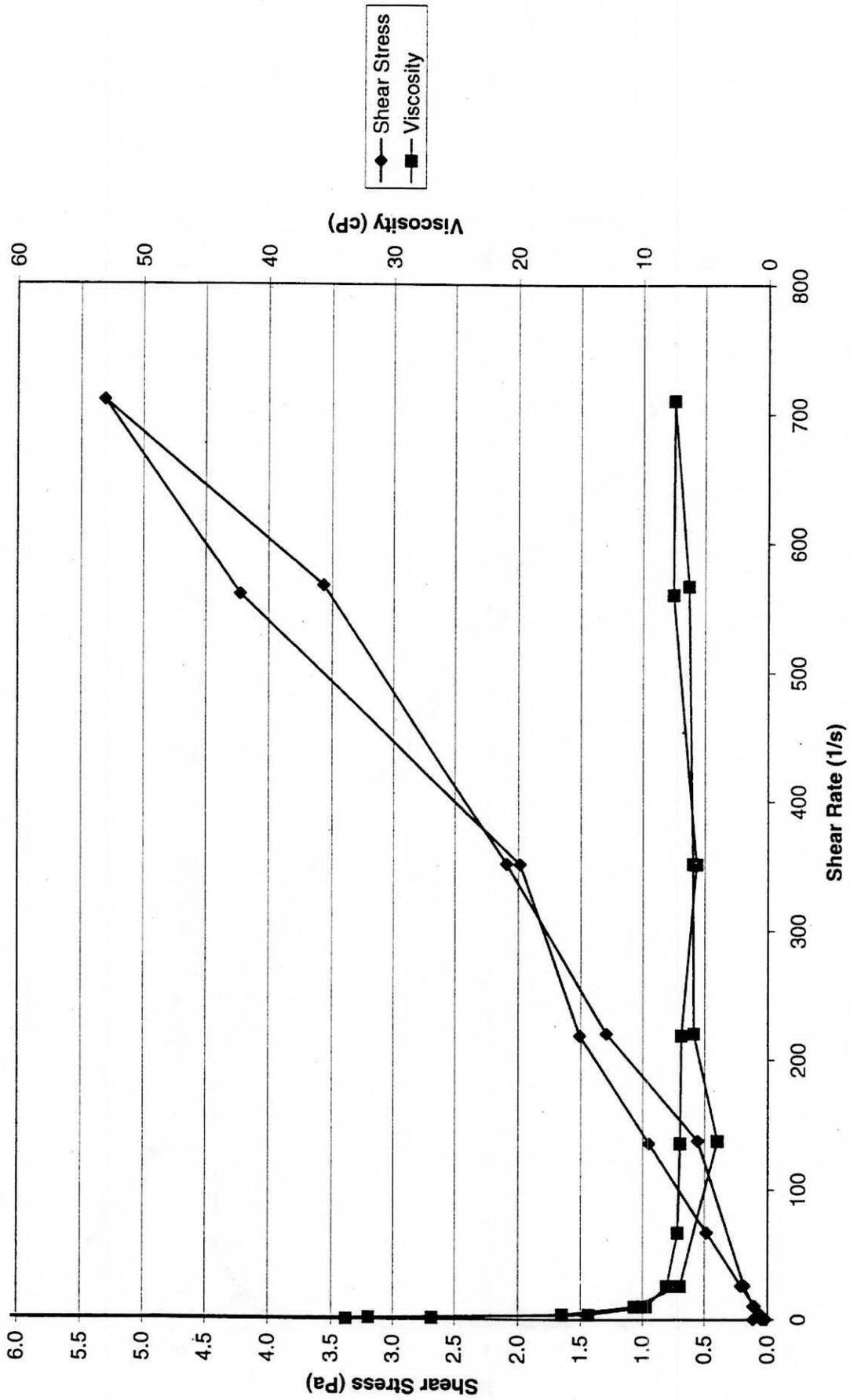


Figure 26. AW-101 10M Na Evaporated Feed: 50°C Analysis 1

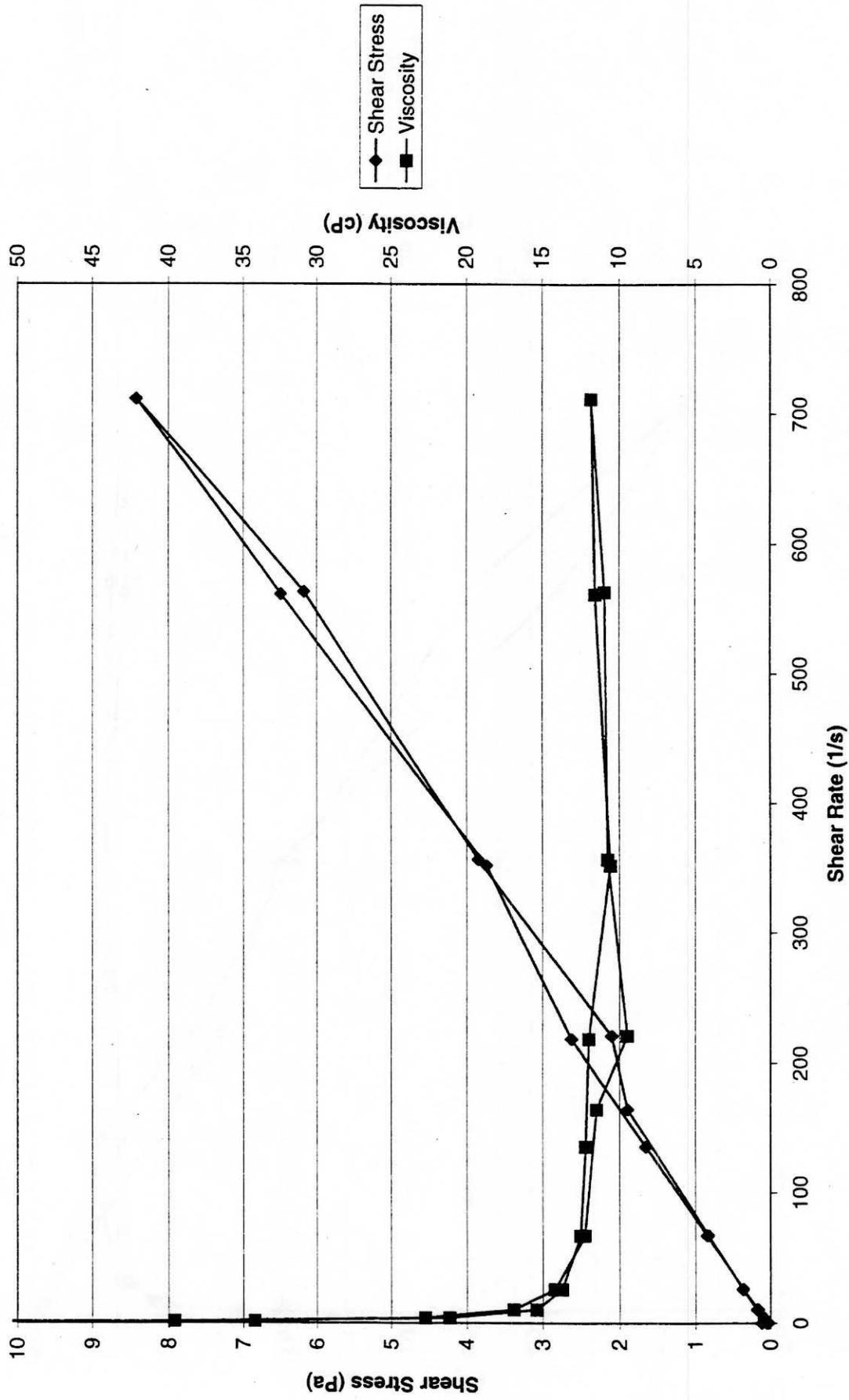


Figure 27. AW-101 10M Na Evaporated Feed: 50°C Analysis 2

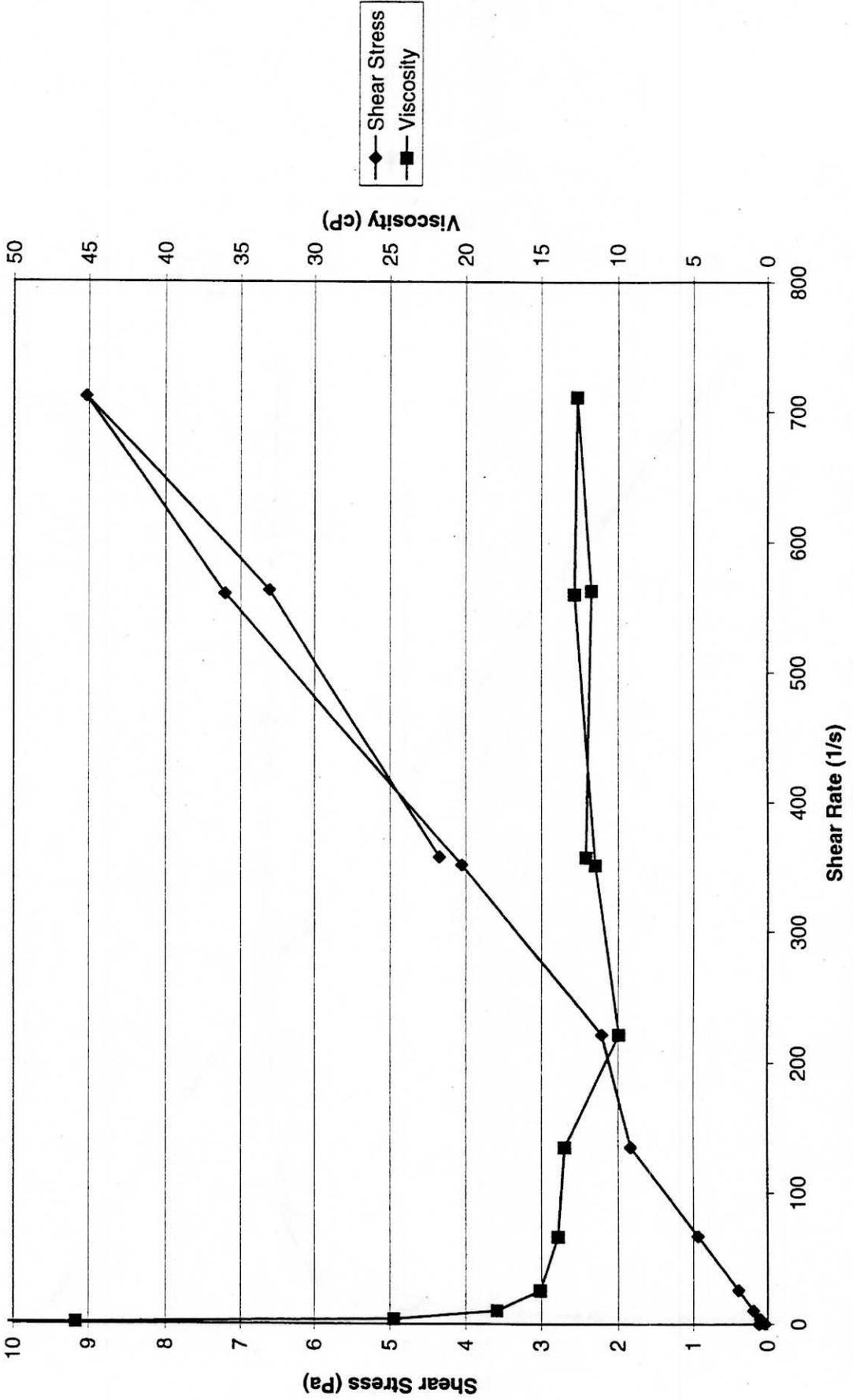


Figure 28. AW-101 6M Na Melter Feed With Glass Formers: 25°C Analysis 1

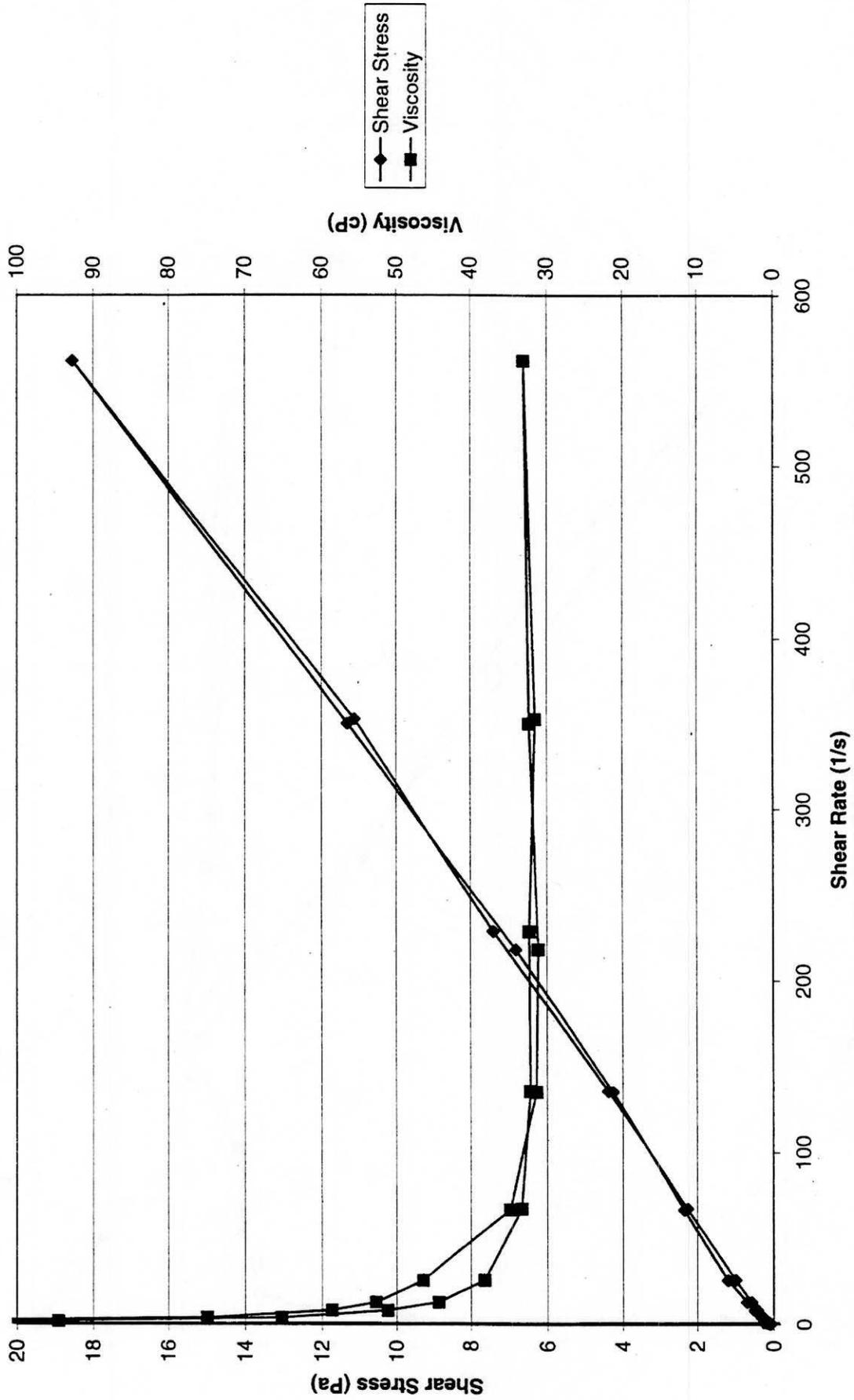


Figure 29. AW-101 6M Na Melter Feed With Glass Formers: 25°C Analysis 2

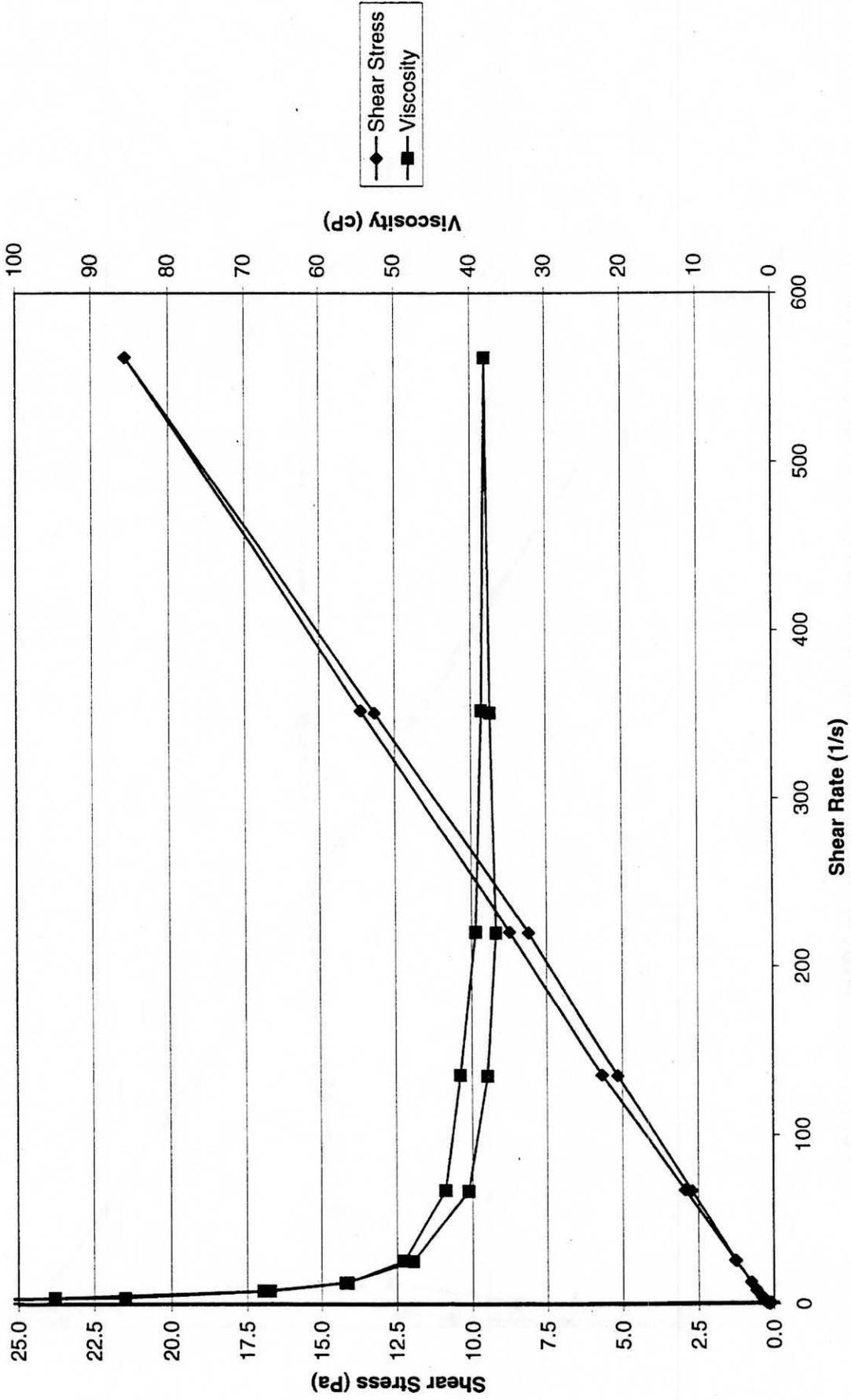


Figure 30. AW-101 8M Na Melter Feed With Glass Formers: 25°C Analysis 1

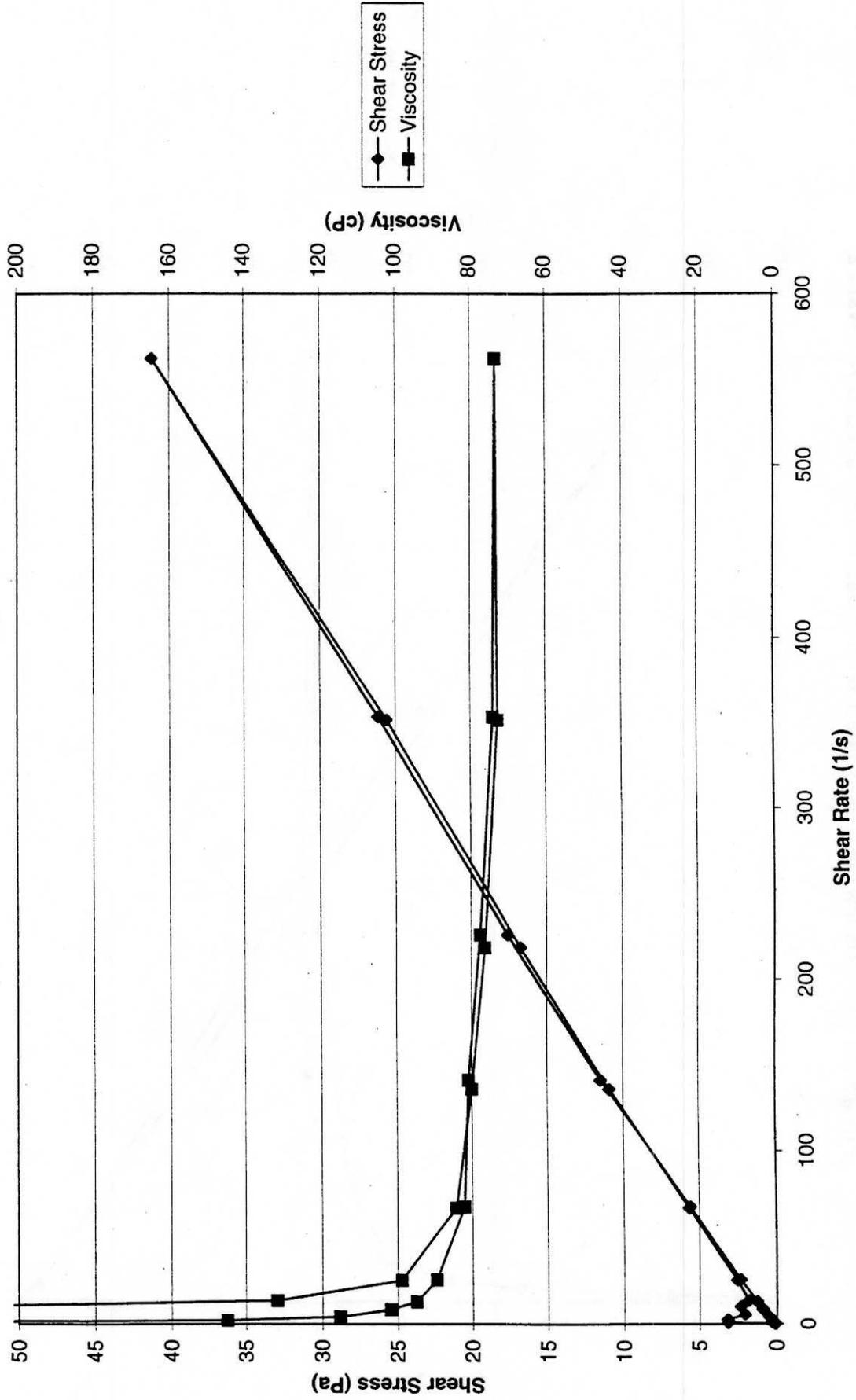


Figure 31. AW-101 8M Na Melter Feed With Glass Formers: 25°C Analysis 2

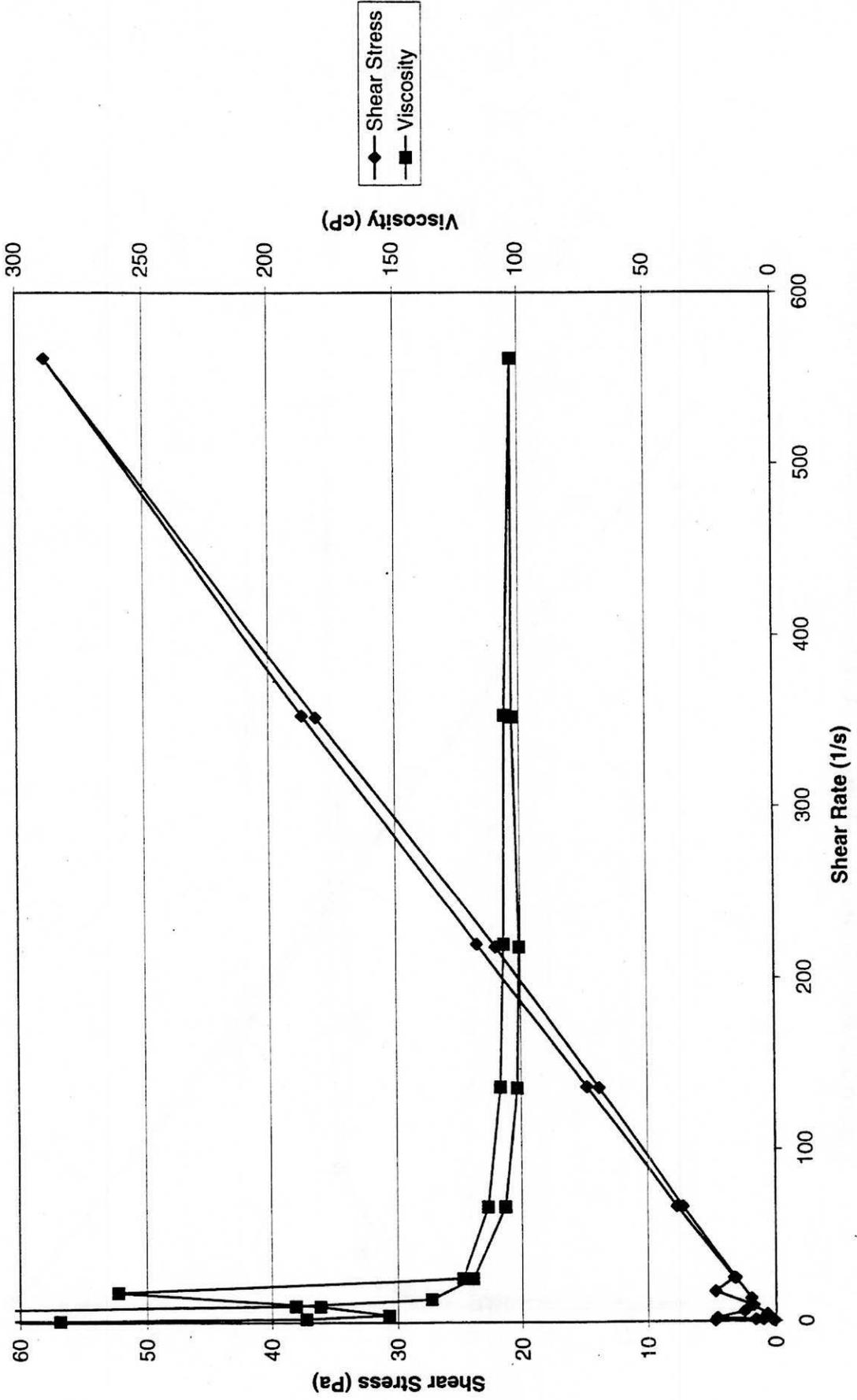


Figure 32. AW-101 10M Na Melter Feed With Glass Formers: 25°C Analysis 1

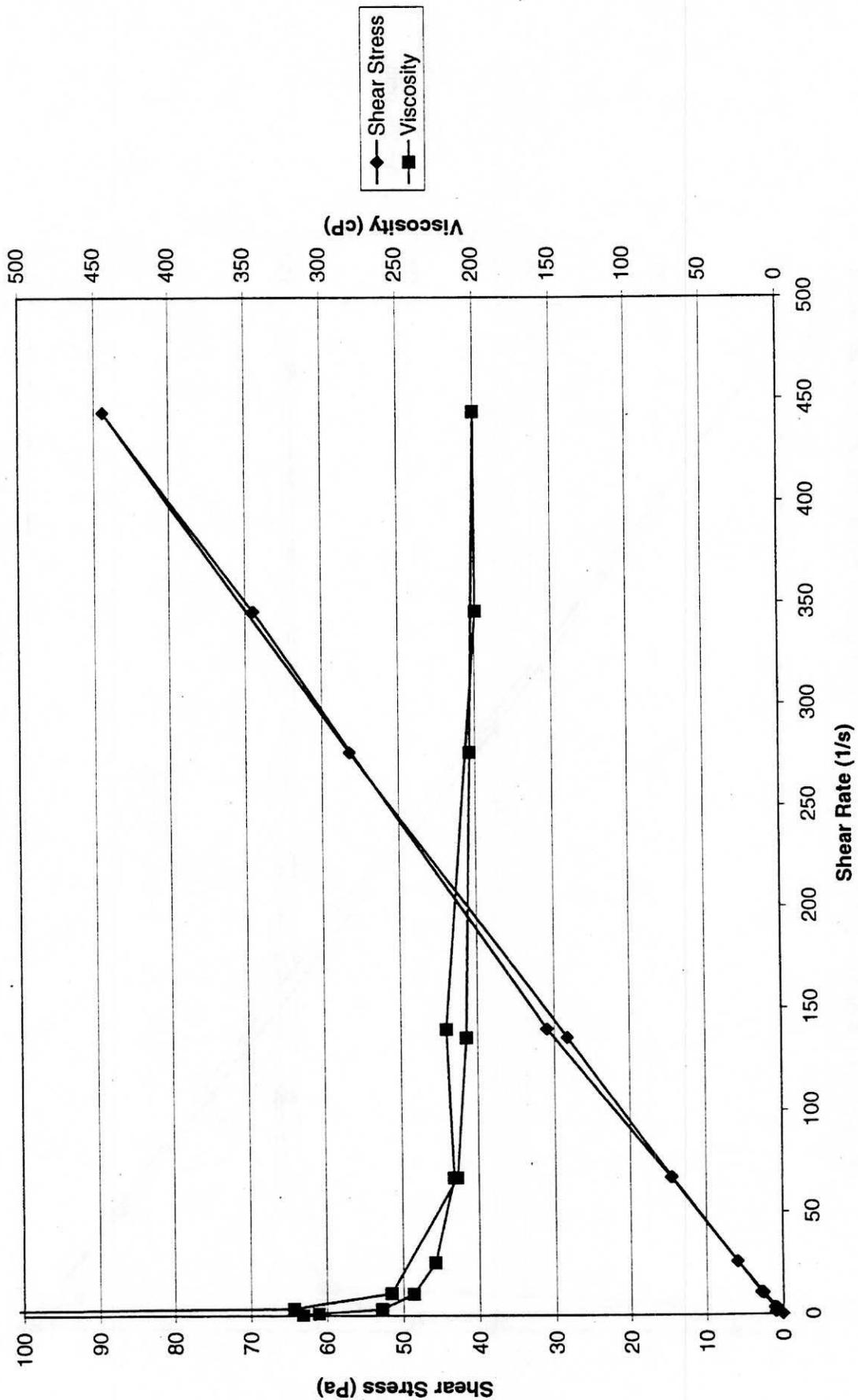


Figure 33. AW-101 10M Na Melter Feed With Glass Formers: 25°C Analysis 2

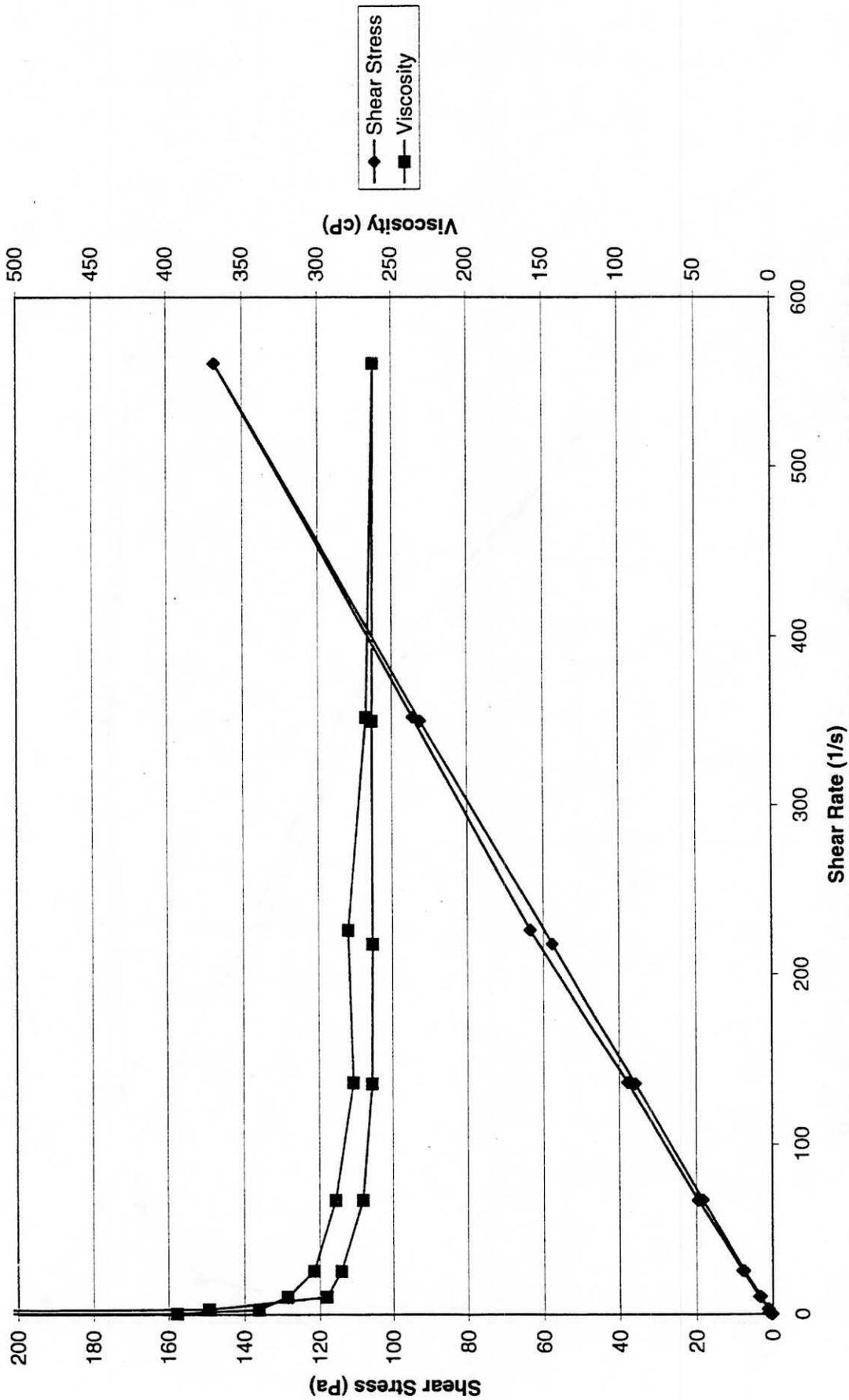


Figure 34. AW-101 6M Na Melter Feed With Glass Formers: 50°C Analysis 1

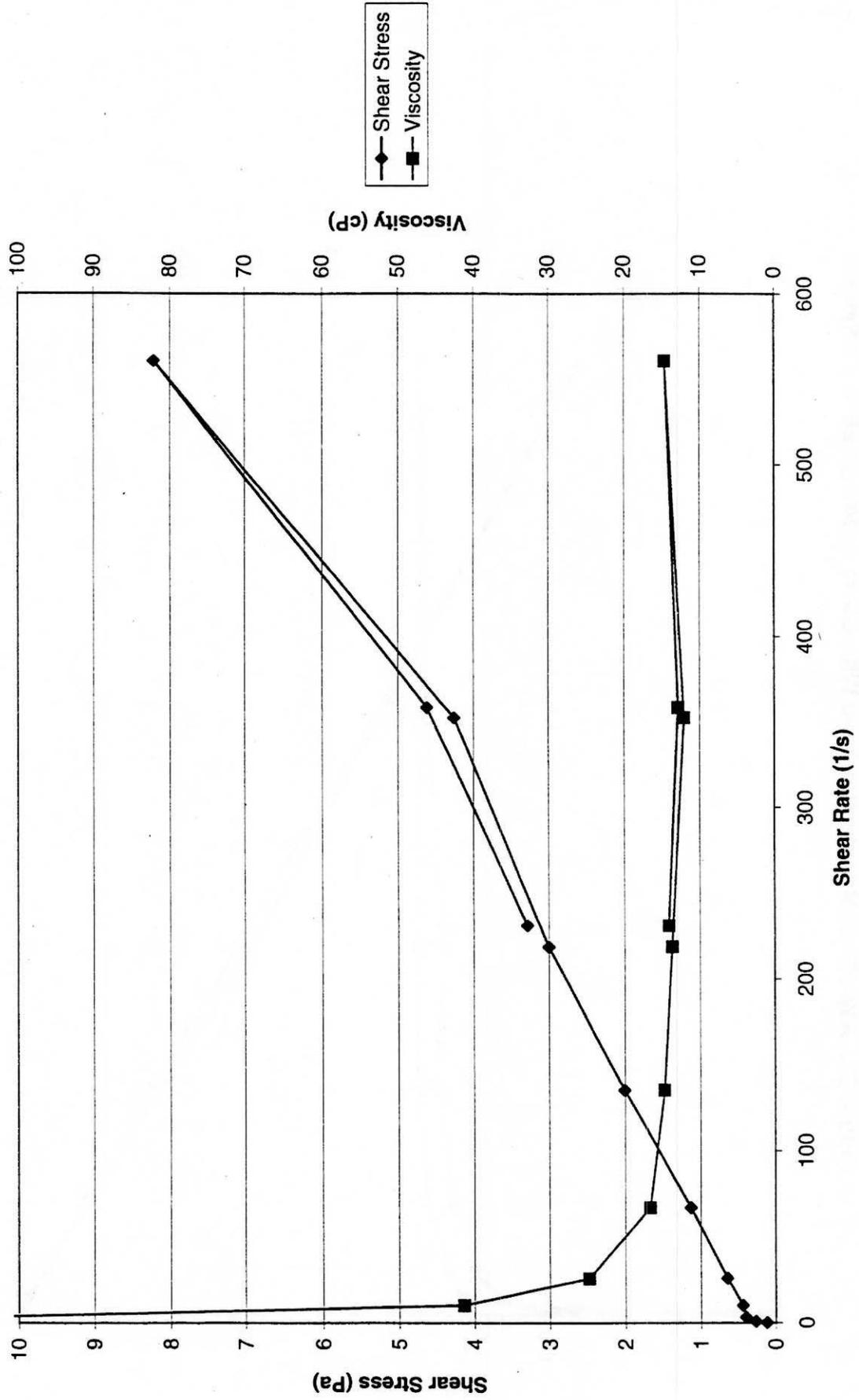


Figure 35. AW-101 6M Na Melter Feed With Glass Formers: 50°C Analysis 2

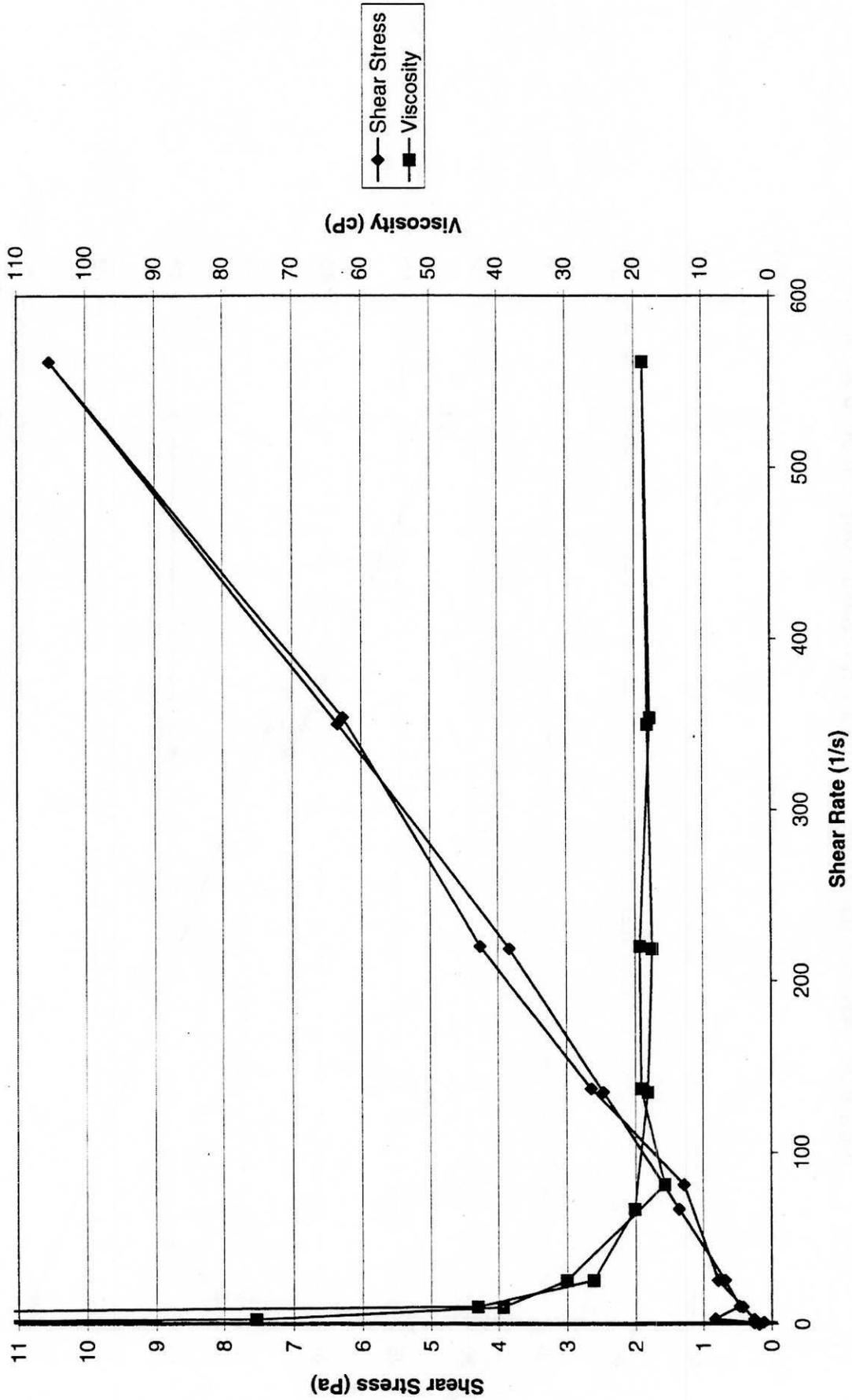


Figure 36. AW-101 8M Na Melter Feed With Glass Formers: 50°C Analysis 1

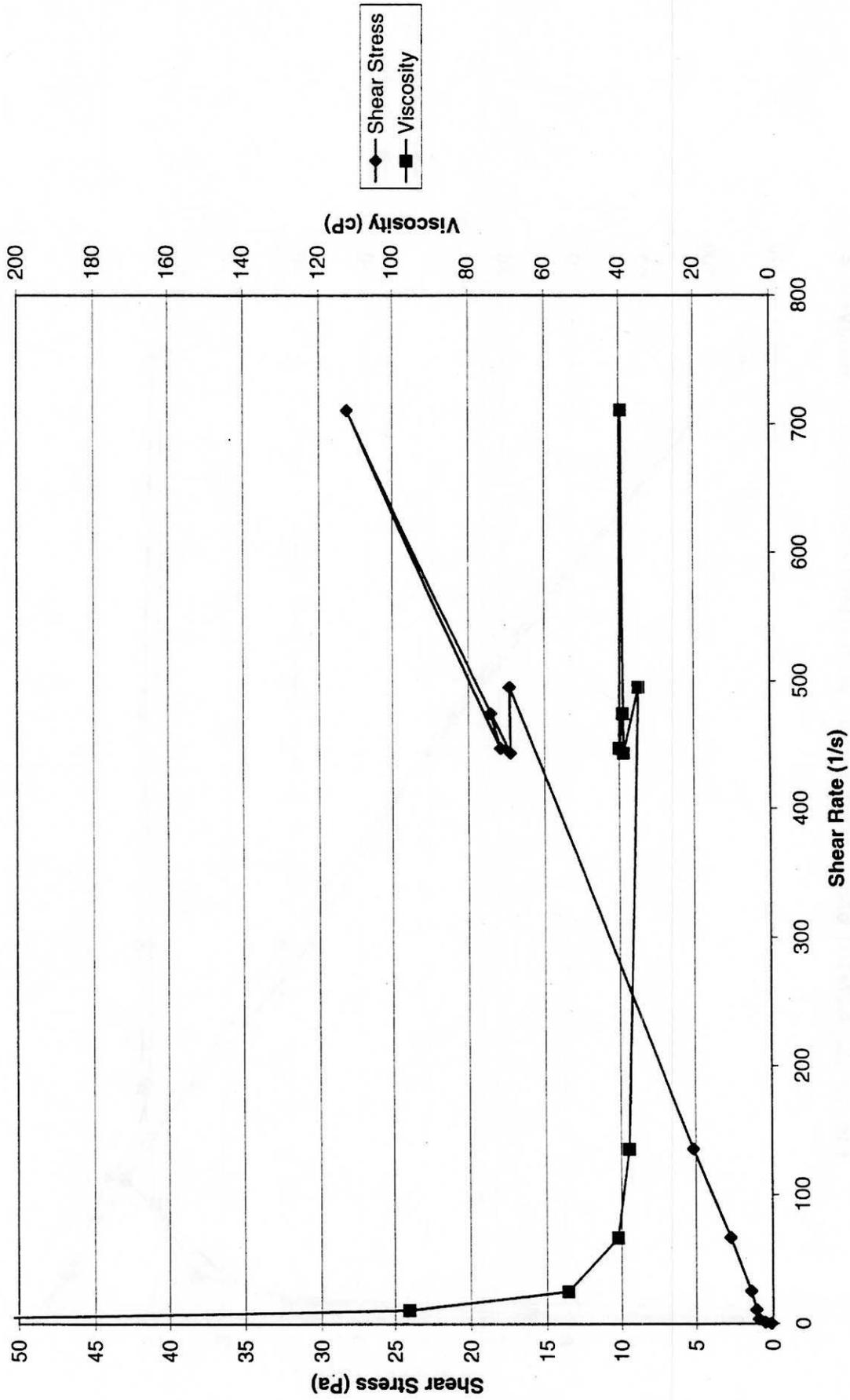


Figure 37. AW-101 8M Na Melter Feed With Glass Formers: 50°C Analysis 2

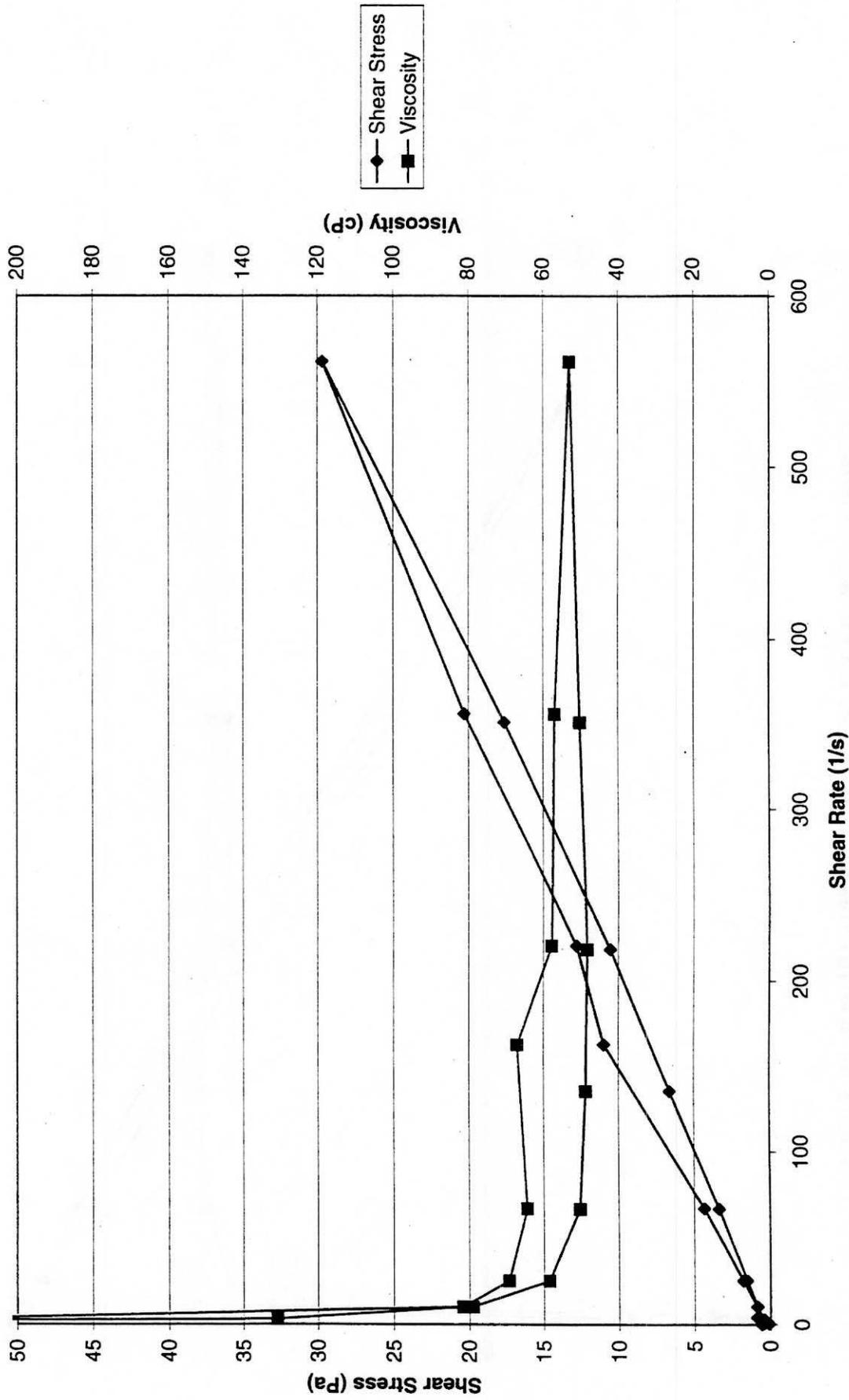


Figure 38. AW-101 10M Na Melter Feed With Glass Formers: 50°C Analysis 1

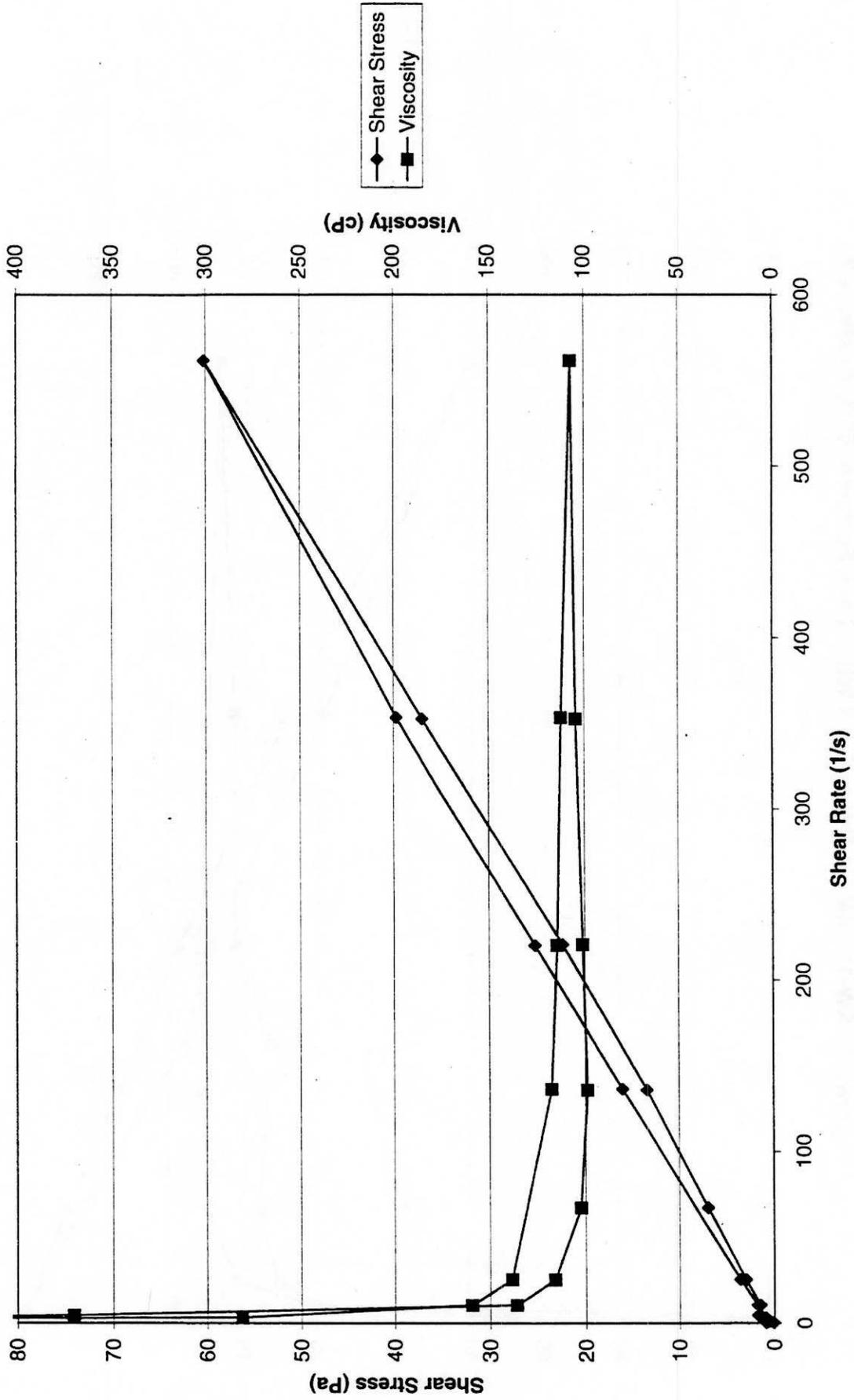


Figure 39. AW-101 10M Na Melter Feed With Glass Formers: 50°C Analysis 2

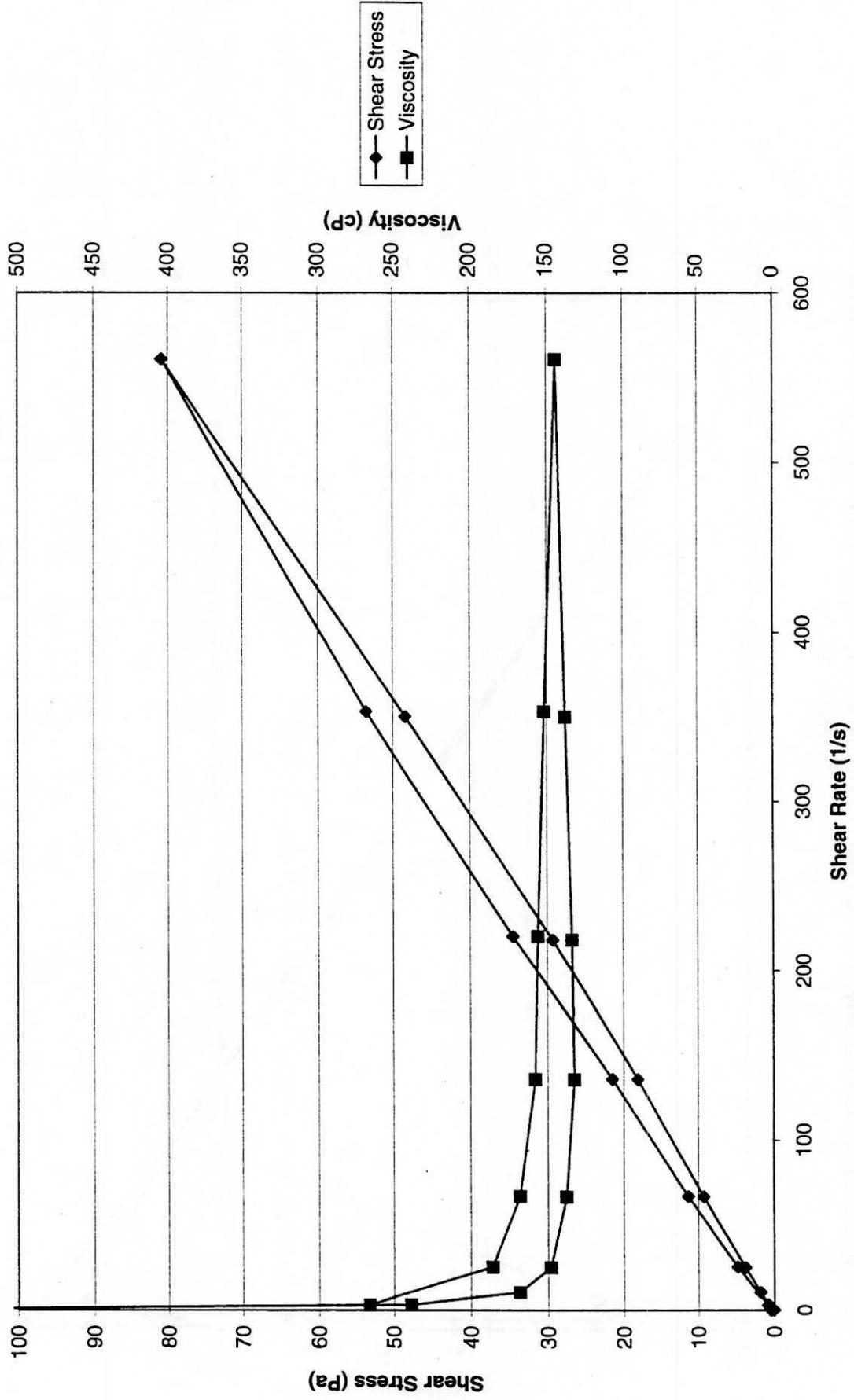


Figure 40. Mixing Study, AW-101 8M Na Melter Feed 1 Hour After Glass Former Addition: 25°C
Analysis 1

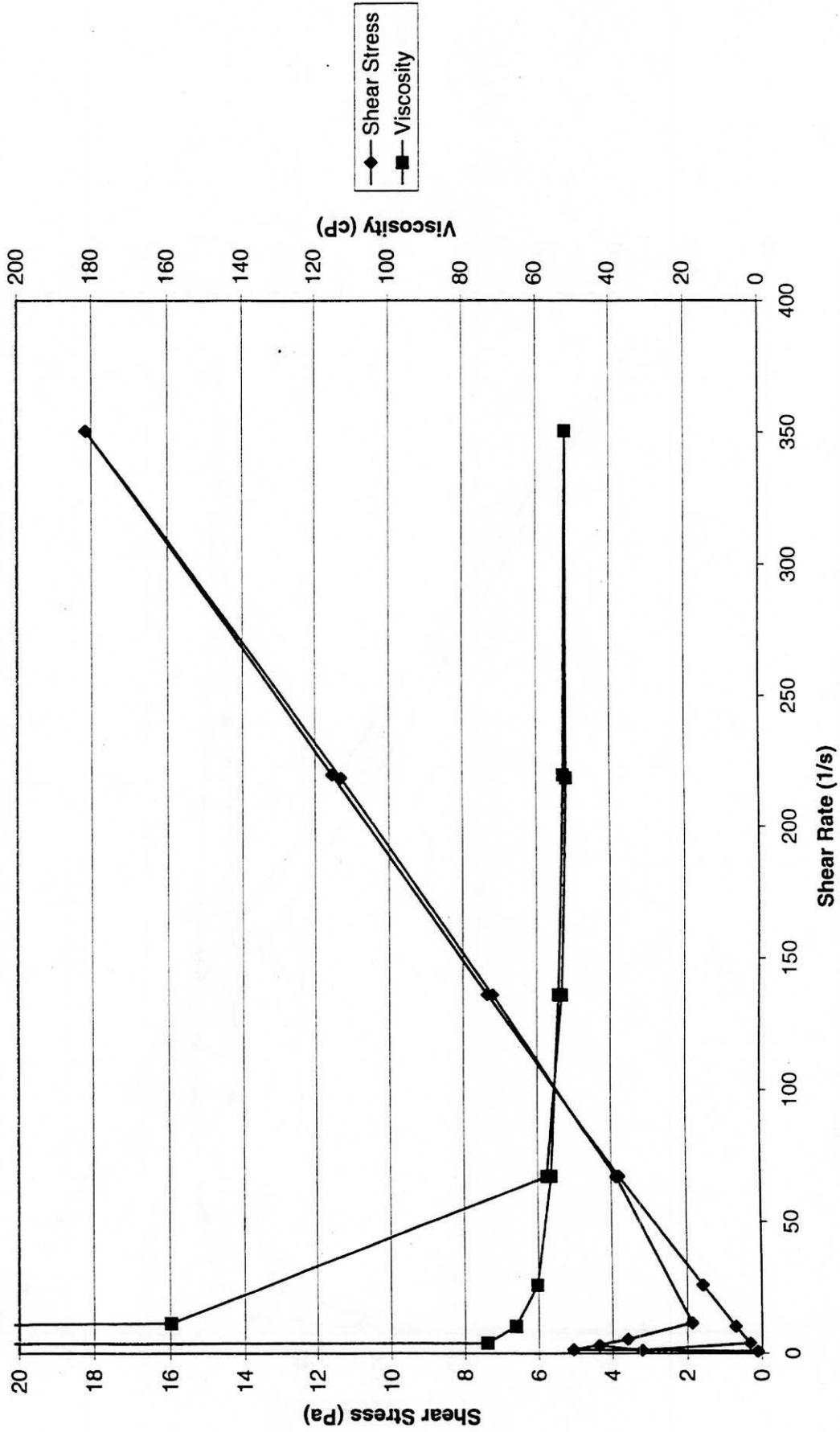


Figure 41. Mixing Study, AW-101 8M Na Melter Feed 1 Hour After Glass Former Addition: 25°C
Analysis 2

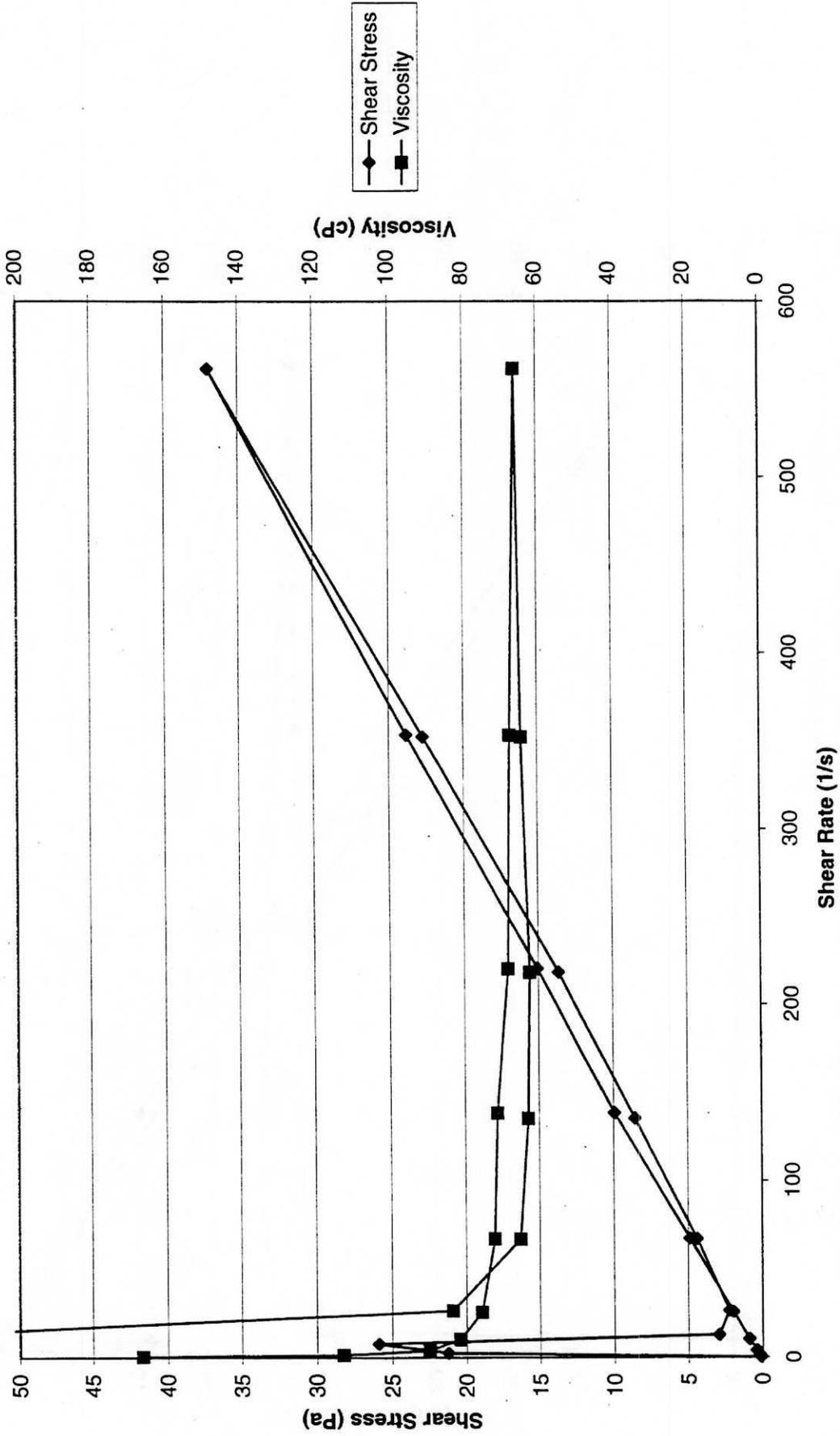


Figure 42. Mixing Study, AW-101 8M Na Melter Feed 1 Hour After Glass Former Addition: 25°C
Analysis 3

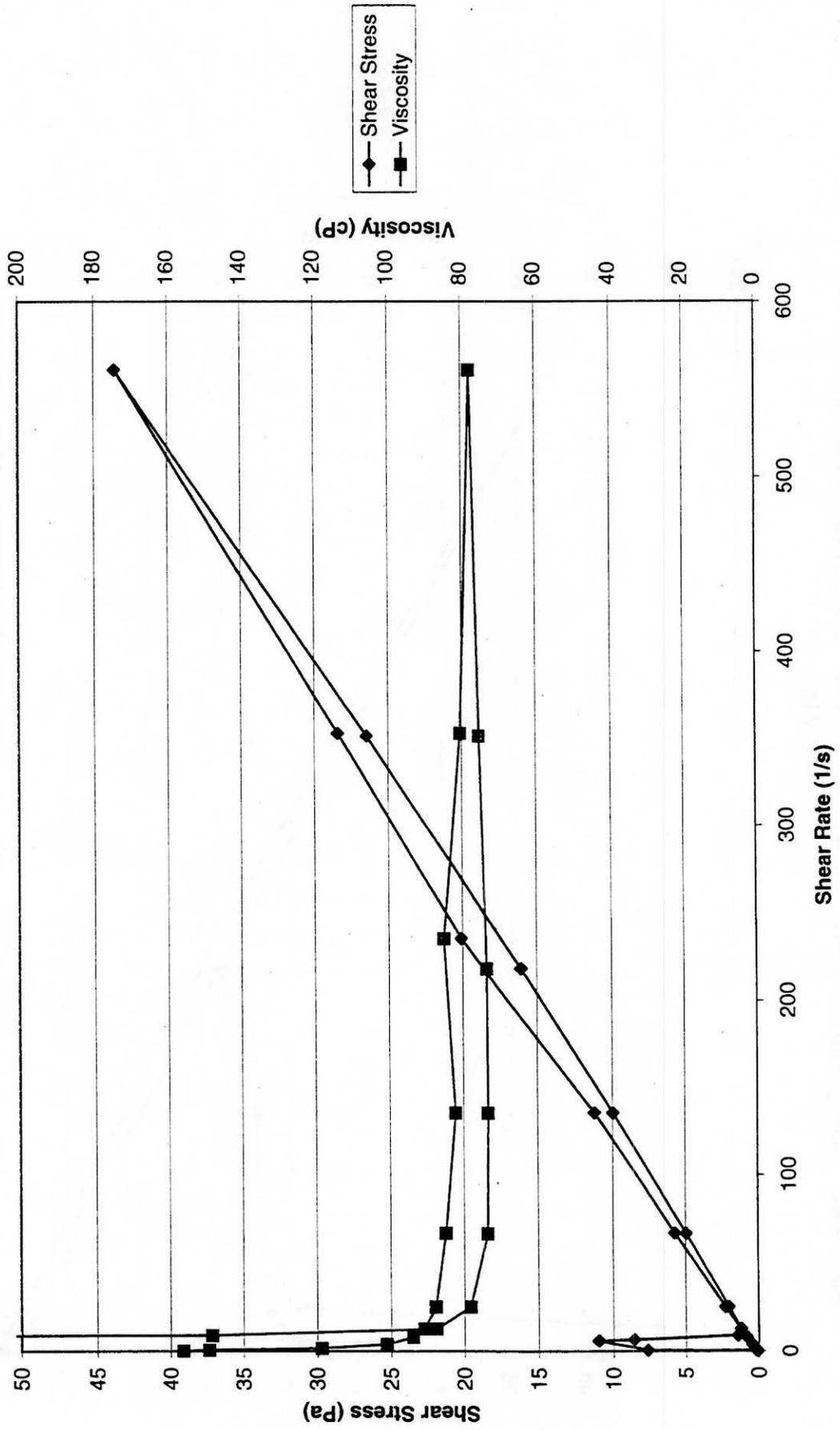


Figure 43. Mixing Study, AW-101 8M Na Melter Feed 1 Day After Glass Former Addition: 25°C
Analysis 1

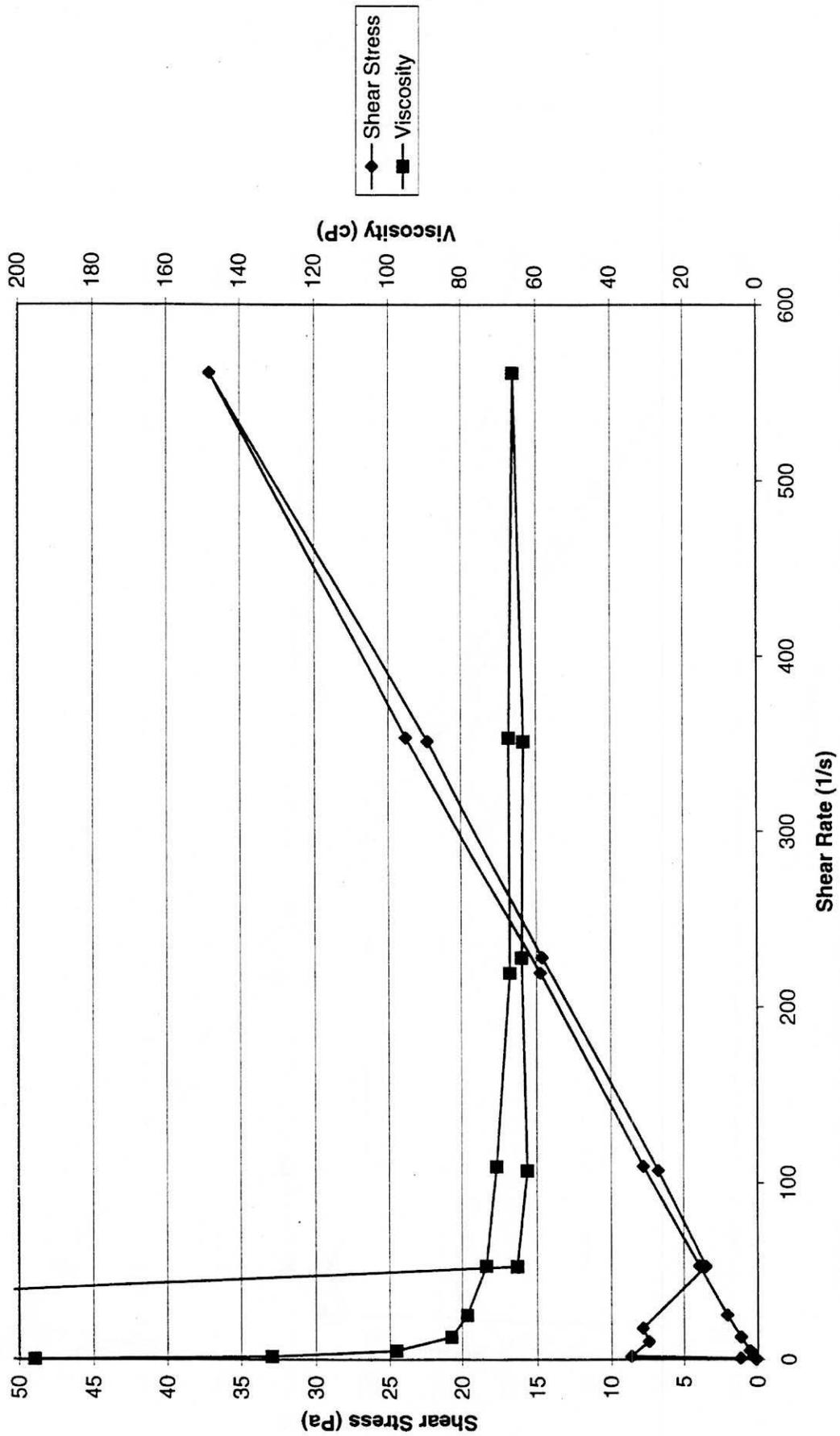


Figure 44. Mixing Study, AW-101 8M Na Melter Feed 1 Day After Glass Former Addition: 25°C
Analysis 2

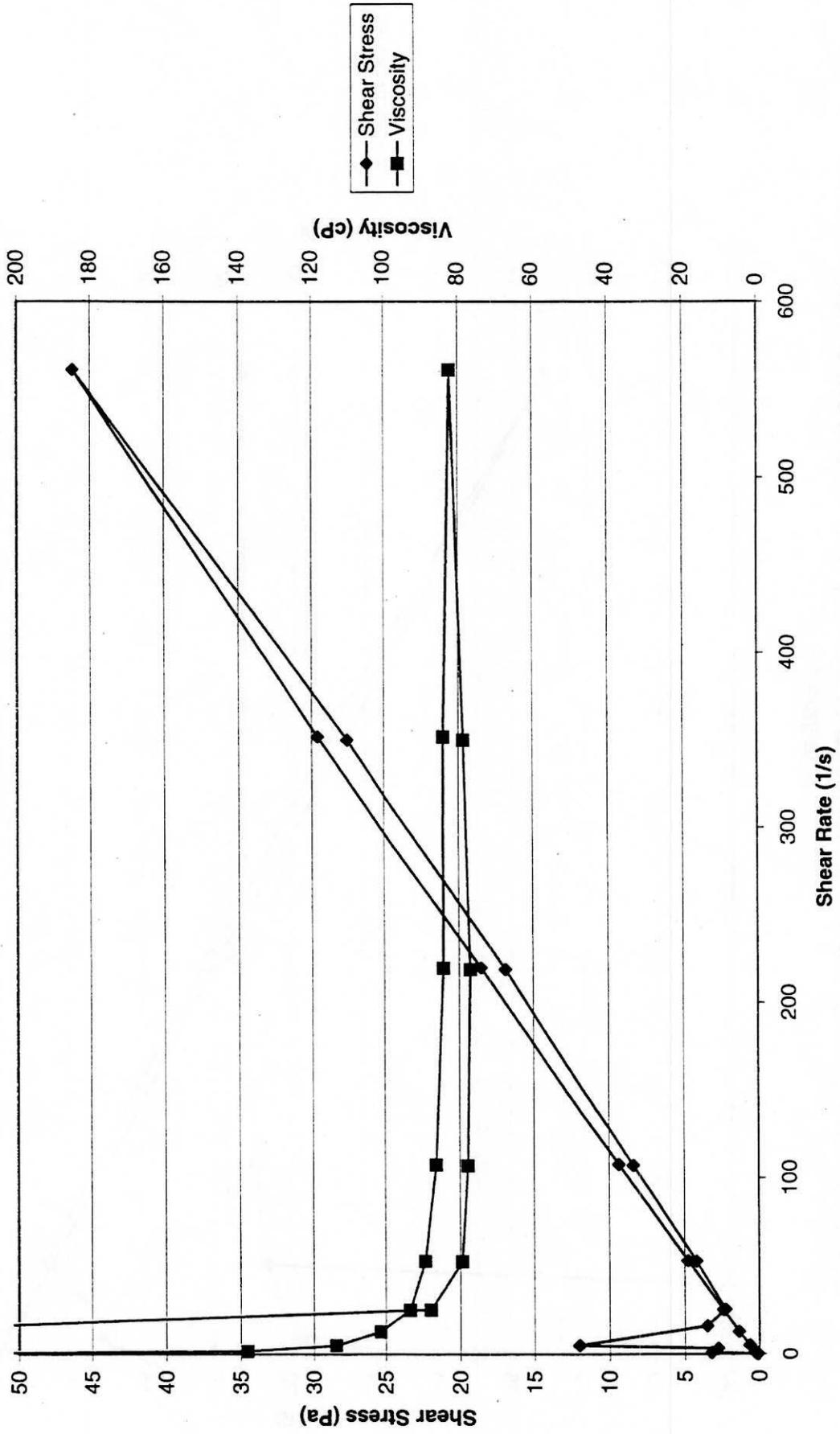


Figure 45. Mixing Study, AW-101 8M Na Melter Feed 1 Week After Glass Former Addition:
25°C Analysis 1

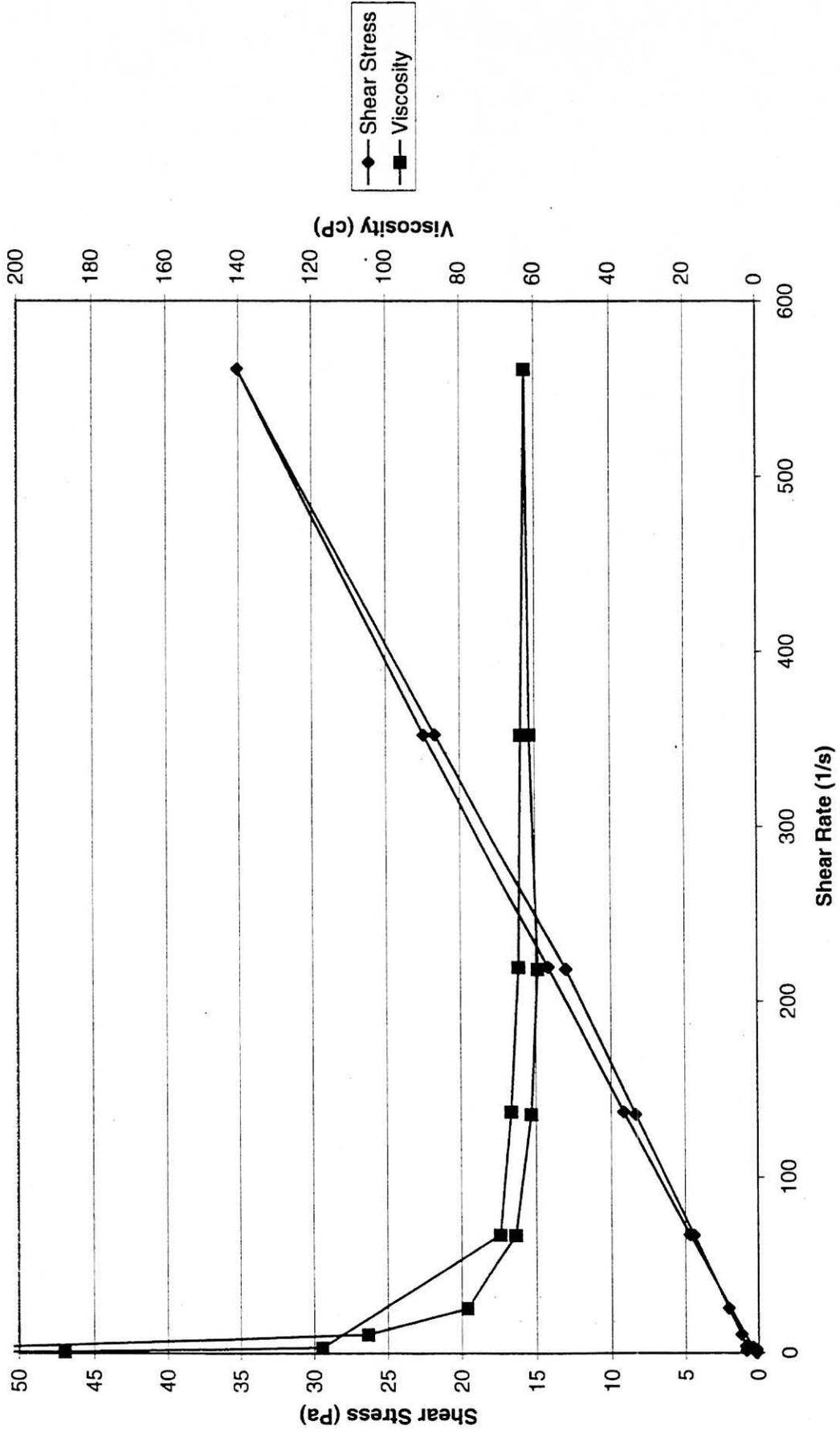


Figure 46. Mixing Study, AW-101 8M Na Melter Feed 1 Week After Glass Former Addition:
25°C Analysis 2

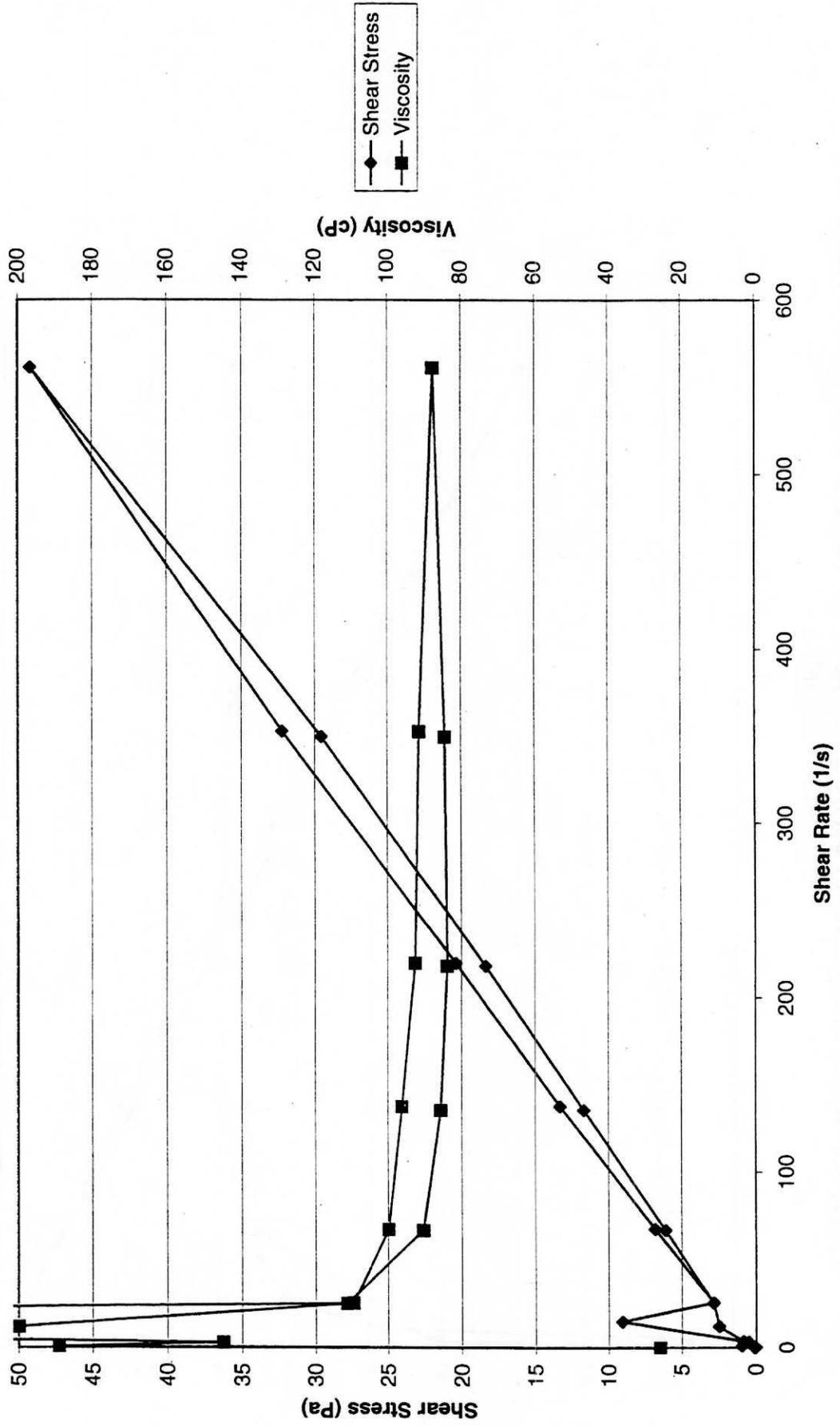


Figure 47. 95 cP Standard Brookfield Lot 111199, 25°C on 3/8/00

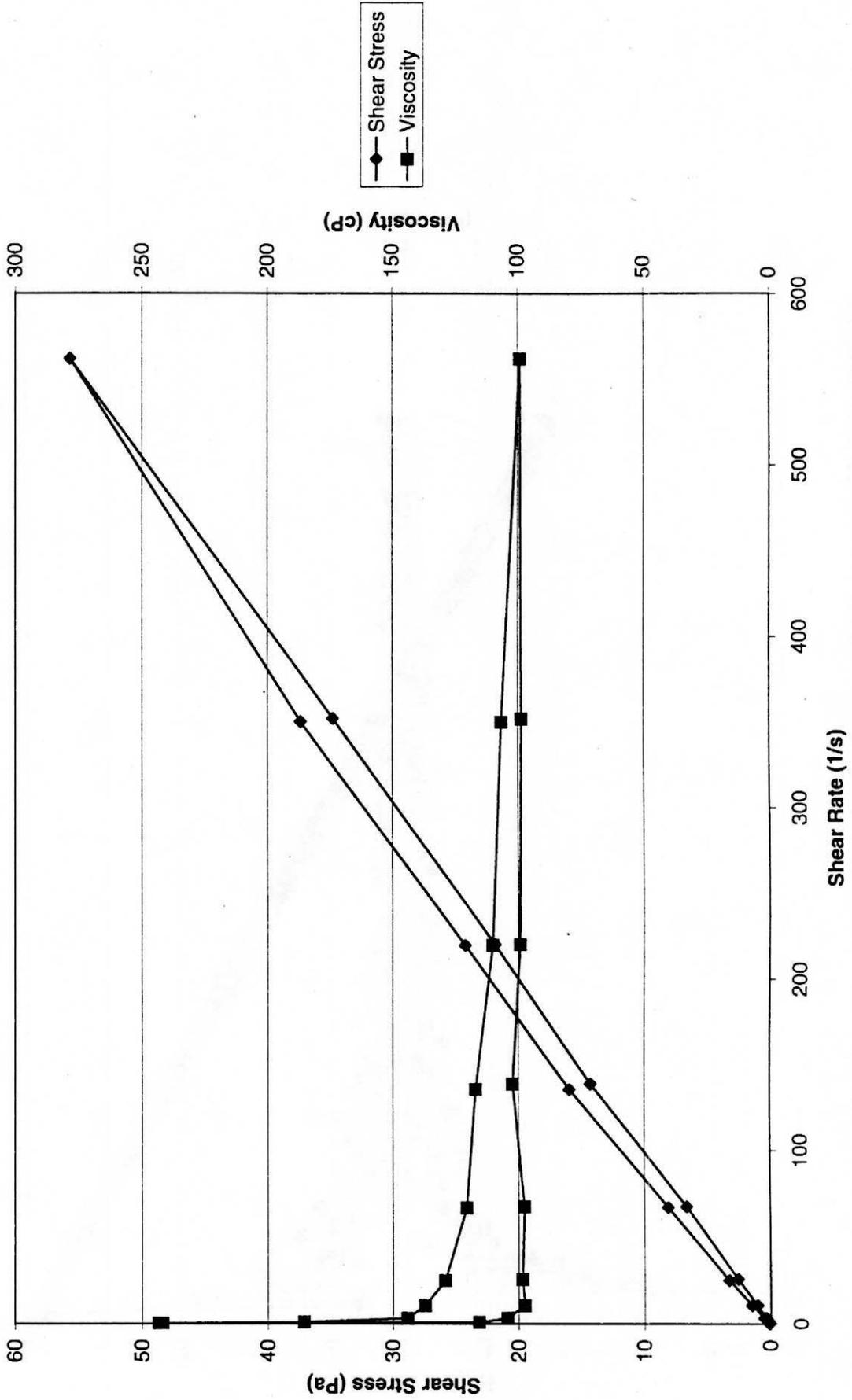


Figure 48. 95.5 cP Standard Brookfield lot 111199, 25°C on 3/15/00

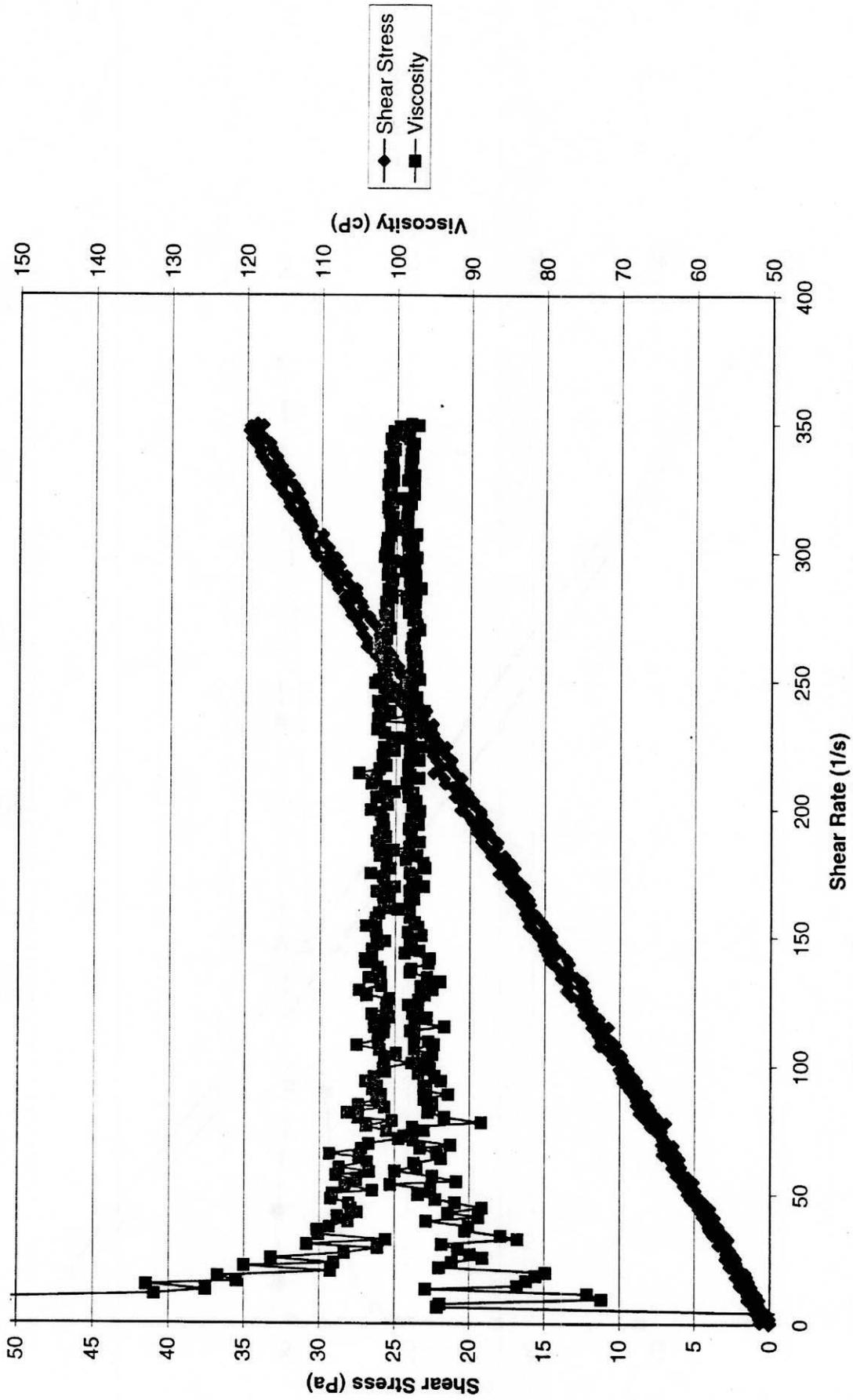


Figure 49. Aging Study: 8M Na Melter Feed 1 Week After Glass Former Addition: 25°C
Loosely Settled Solids, Analysis 1

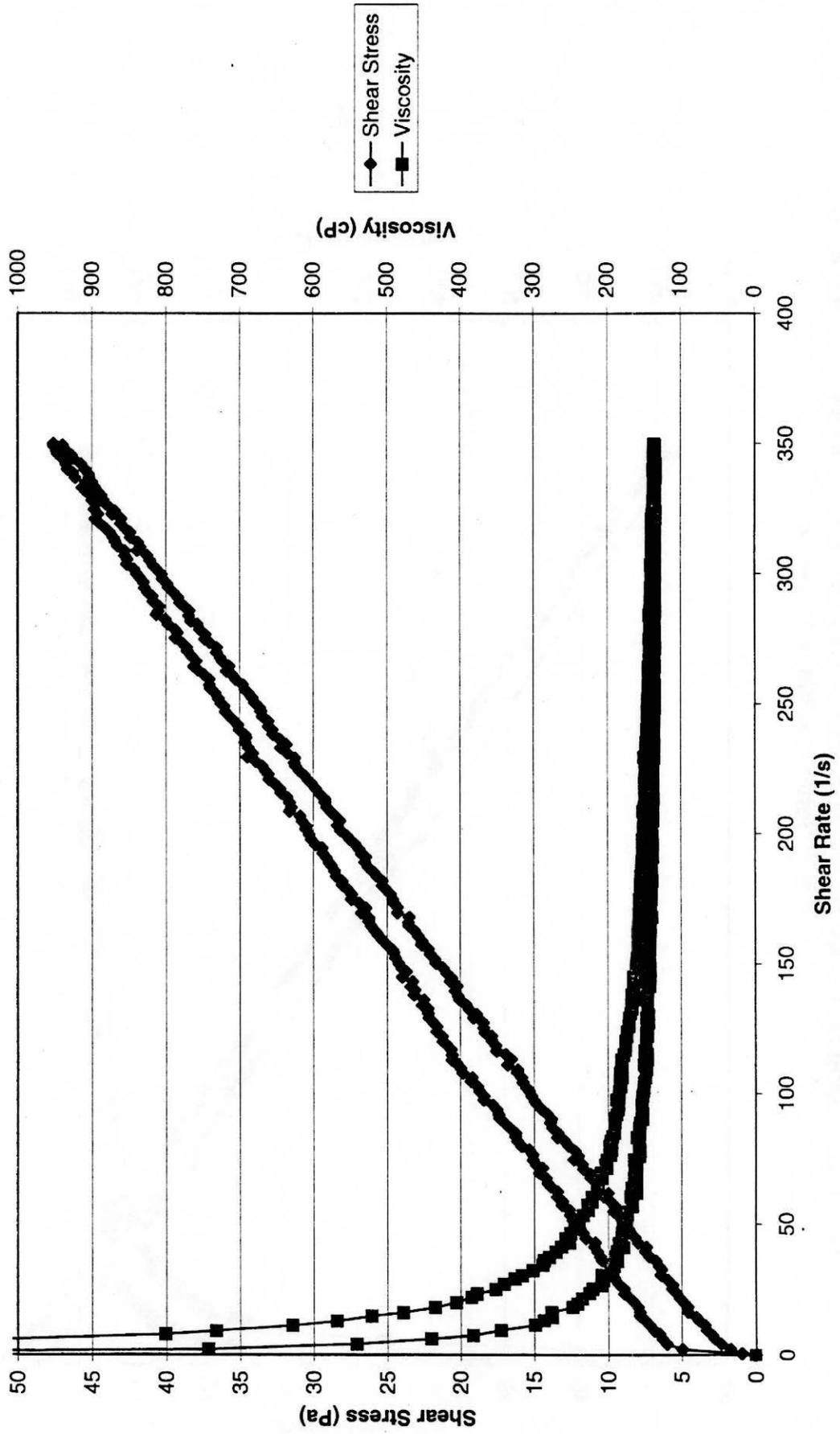


Figure 50. Aging Study: 8M Na Melter Feed 1 Week After Glass Former Addition: 25°C
Loosely Settled Solids, Analysis 2

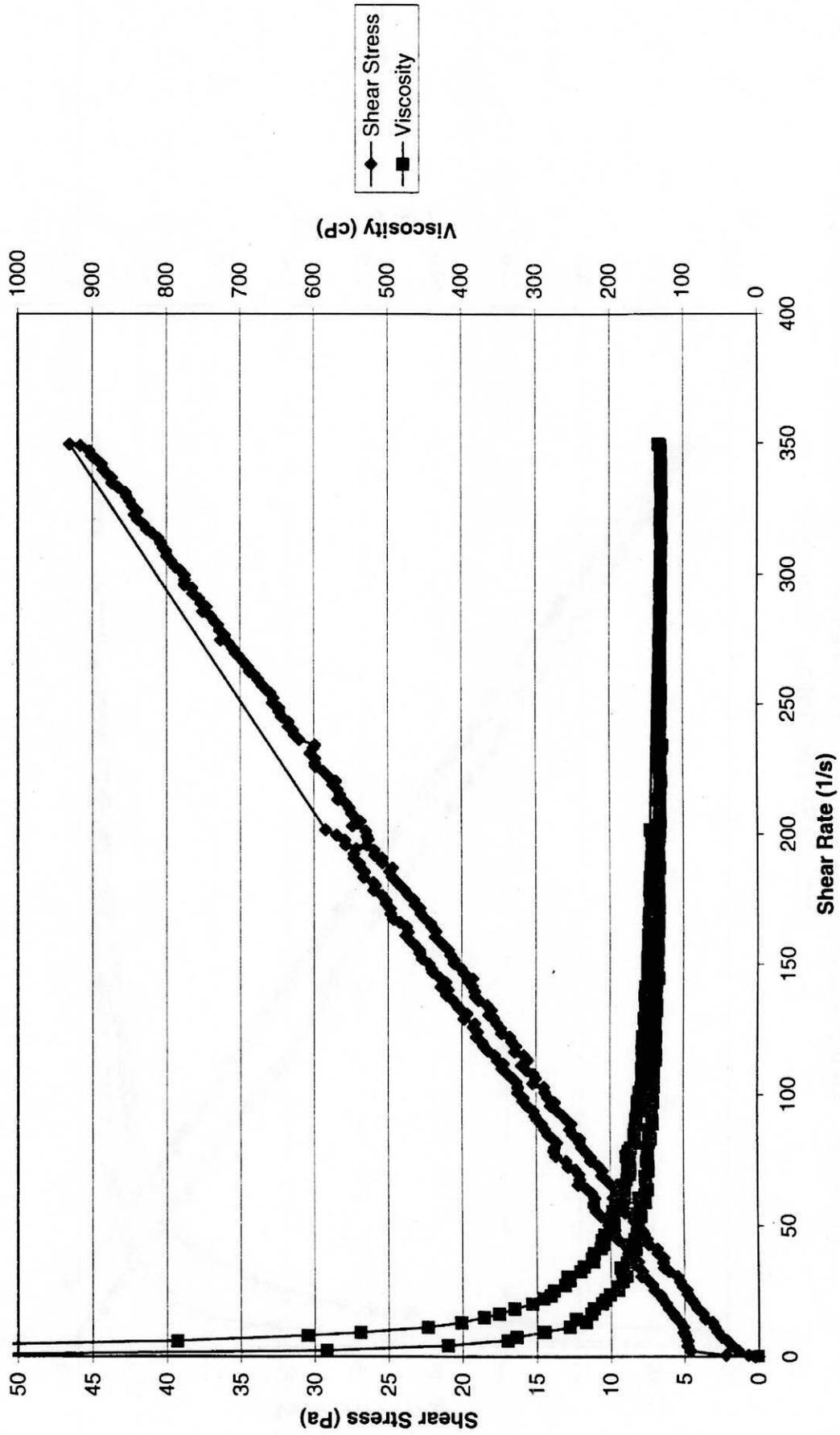


Figure 51. Aging Study: 8M Na Melter Feed 1 Week After Glass Former Addition: 25°C
Tightly Settled Solids, Analysis 1

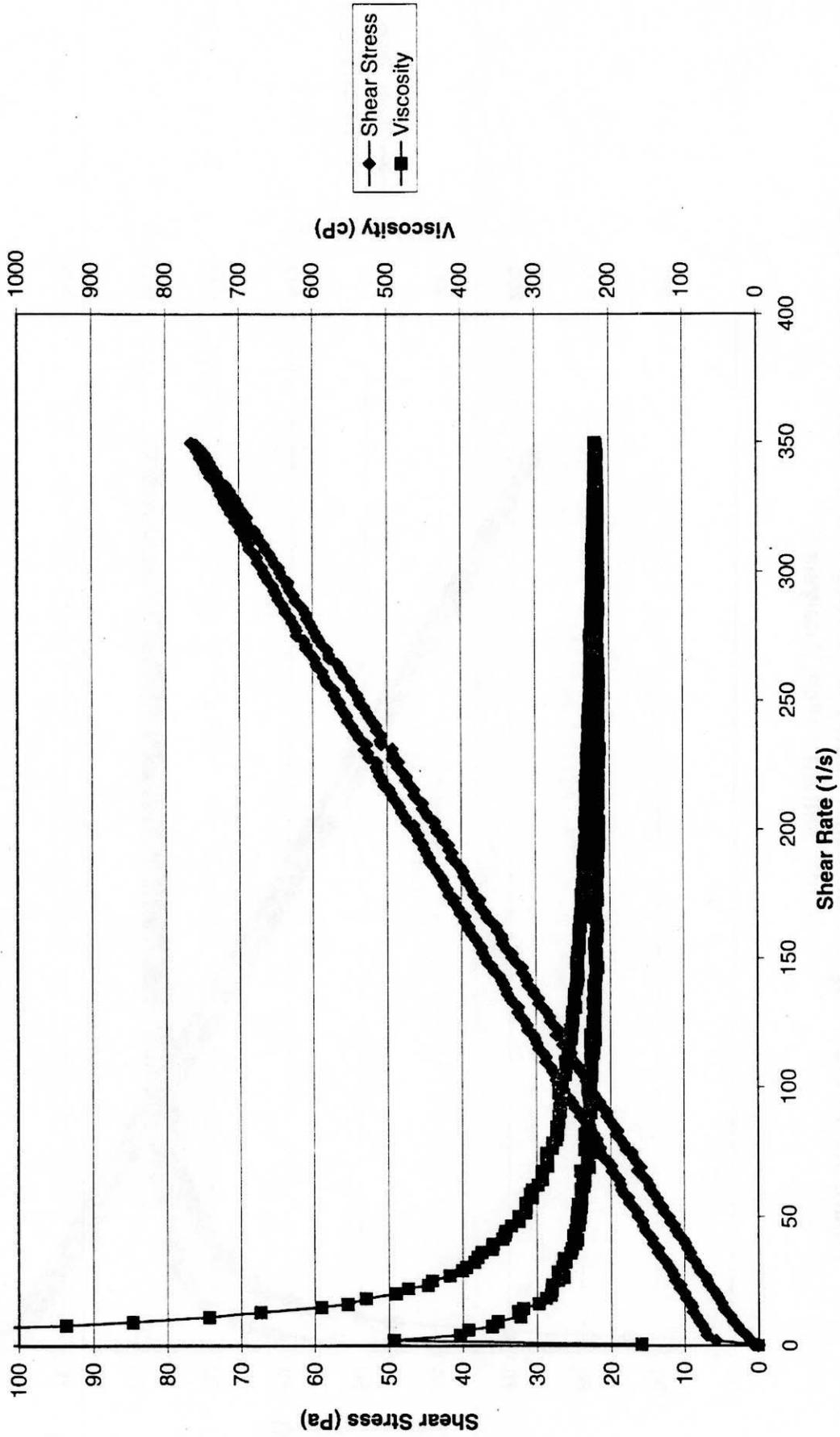
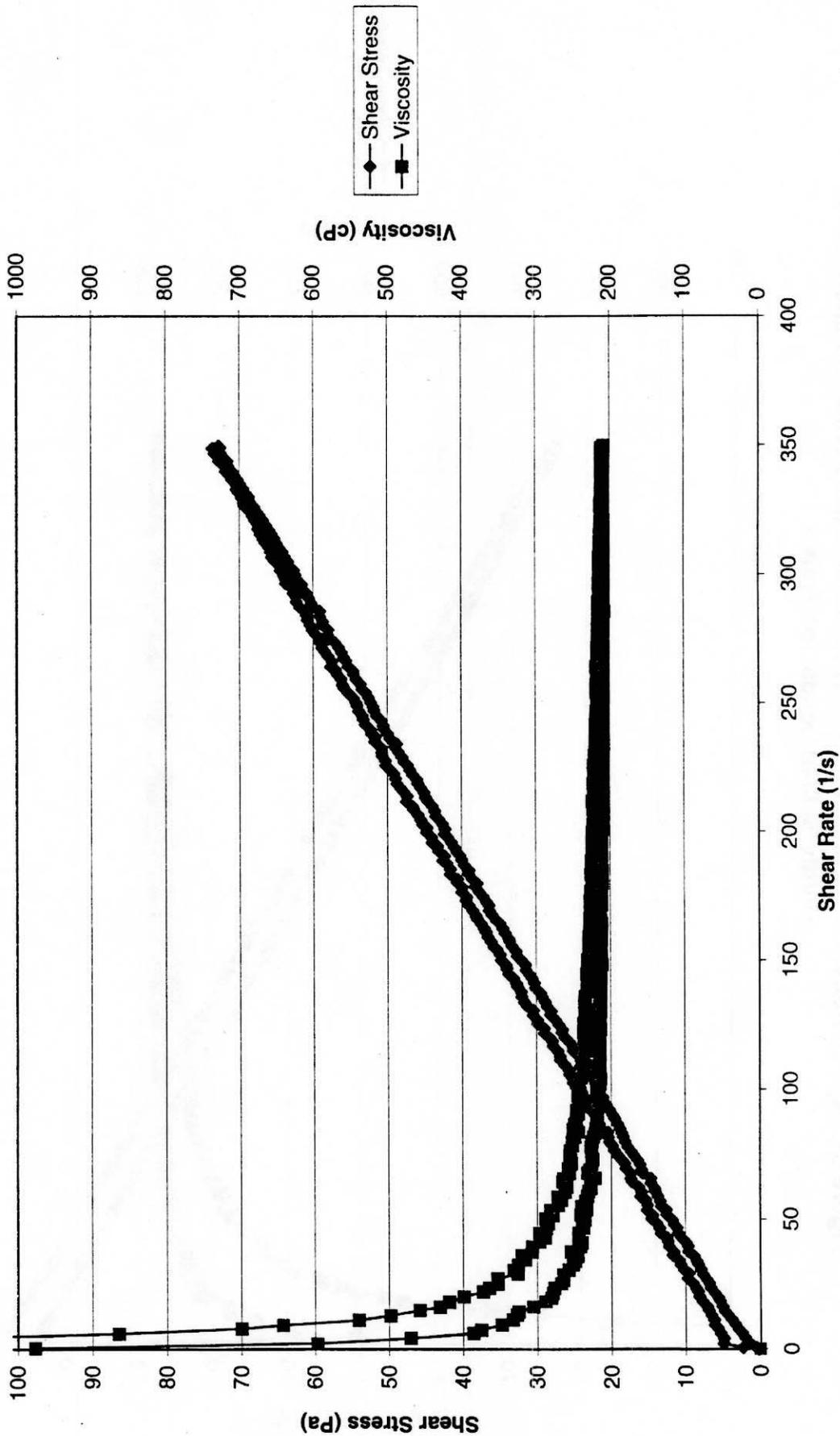


Figure 52. Aging Study: 8M Na Melter Feed 1 Week After Glass Former Addition: 25°C
Tightly Settled Solids, Analysis 2



Appendix B: Test Plan (BNFL-TP-29953-046)

Faint text in the middle of the page, possibly a title or subtitle.



PNNL Test Plan

Document No.: BNFL-TP-29953-46
 Rev. No.: 0
 Document Control: Only the original signed copy is controlled

Title: LAW Melter Feed Rheological and Physical Properties Measurements

Work Location: Radiochemical Processing Laboratory

Page 1 of 54

Author: Paul Bredt

Effective Date: Upon Final Approval
 Supersedes Date: New

Use Category Identification: Reference

Identified Hazards:

- Radiological
- Hazardous Materials
- Physical Hazards
- Hazardous Environment
- Other:

Required Reviewers:

- Technical Reviewer
- Building Manager
- Radiological Control
- ES&H
- Quality Engineer
- Project Manager
- RPL Manager
- SFO Manager

Are One-Time Modifications Allowed to this Procedure? Yes No

NOTE: If Yes, then modifications are not anticipated to impact safety. For documentation requirements of a modification see SBMS or the controlling Project QA Plan as appropriate.

On-The Job Training Required? Yes or No

FOR REVISIONS:

Is retraining to this procedure required? Yes No

Does the OJT package associated with this procedure require revision to reflect procedure changes?
 Yes No N/A

Approval	Signature	Date
Author	<i>Paul Bredt</i>	9/24/99
Technical Reviewer	<i>Harry L. Smith</i>	9/24/99
RPL Manager	<i>LC Casazza</i>	9/24/99
Project Manager	<i>Eugene V. Money</i>	9/24/99
Building Manager	<i>Paul H.</i>	9-24-99
RPL QE	<i>Paul Bredt</i>	9-24-99
BNFL	<i>St. Ann</i>	10/4/99

LAW Melter Feed Rheological and Physical Properties Measurements

Scope

This test plan defines work to be conducted on AN-107 and AW-101 slurry samples following pretreatment. Samples are to be evaporated under vacuum (40-80 torr) at $\sim 50^{\circ}\text{C}$ to three sodium concentrations as specified by the client prior to initiation of work (probably 6, 8, and 10 M). Following evaporation, solids concentration, settling rate, density and shear stress versus shear rate will be measured on the samples at ambient temperature ($\sim 25^{\circ}\text{C}$) and 50°C . Glass formers, as specified by the client prior to initiation of work, will then be added to the samples. Following glass former addition, solids concentration, solids settling rate, density and shear stress versus shear rate will then be measured on the samples at ambient and 50°C . One of the samples will then be actively mixed for one week in a vessel with an impeller to vessel diameter ratio of 77:120. Mixer speed will be specified by the client prior to initiation of work. During this week of mixing, shear stress versus shear rate will be measured after 1 hour, 1 day and 1 week. The slurry will then be left undisturbed for 1 week while observations are made of any gas retention and release behavior by focusing a remote video camera on the solid-liquid interface. The viscosity of this slurry will be further measured after 1 week with no mixing.

Test instructions will be issued to provide specific details to RPG staff regarding the implementation of Technical Procedure 29953-010, "Measurement of Physical and Rheological Properties of Solutions, Slurries and Sludges". Client expectations for successful achievement of project data needs have already been established via the "LAW Melter Feed Rheology" test specifications, numbered TS-W375LV-TE00001, provided to Battelle by BNFL.

Justification of Use Category

This procedure is reference use. Reference use was selected as the use category since this analysis is not a complex process and there are no safety impacts to the order of events. In addition, we may wish to modify the order of analyses or eliminate some analyses depending on the needs at the time of the operation.

Applicability

This test plan applies to RPL staff performing work on BNFL Privatization samples under Project 29953.

Work with actual samples is to be performed in radiological fume hoods and glove boxes in the RPL by staff under the direction of a cognizant scientist.

Prerequisites

- 1) Keep the sample in a sealed glass container as much as possible to prevent it from drying and reduce the potential for organic contamination.
- 2) Cross-contamination between samples and contamination of samples from outside sources must be minimized at each step. Use new tools and bottles for each sample as much as possible. Those tools which are reused should be washed and rinsed prior to reuse.
- 3) Secondary containment is to be used whenever practical to minimize sample loss in the event of a spilled sample or broken sample bottle.
- 4) This Test Plan requires work in an oven or viscometer at 50°C. Unless otherwise indicated, a value of $\pm 5^\circ\text{C}$ is acceptable.

Quality Control

Quality control has been implemented in Technical Procedure 29953-010, "Measurement of Physical and Rheological Properties of Solutions, Slurries and Sludges". This work is to be conducted under the quality requirements of the Standards-Based Management System (SBMS).

M&TE List:

_____ Balance 1:

Calib ID 510-06-01-007 Calib Exp Date 8-2000
Location Hood #1 Lb 517

_____ Balance 2:

Calib ID _____ Calib Exp Date _____
Location _____ Not needed PRB 3/20/00

_____ Thermocouple:

Thermometer
Calib ID NIST tracking # 300-01 Calib Exp Date _____
Location Hood #1 Lb 517 cert. date serial # 6585

_____ Digital Thermometer:

Calib ID _____ Calib Exp Date _____
Location _____

_____ Vacuum Gauge:

Calib ID _____ Calib Exp Date _____
Location _____

not needed PRB 3/20/00

Work Flow

- 1) Load 3 subsamples of a waste in glass jars and place in vacuum oven.
- 2) Apply a vacuum (40-80 torr positive pressure) to the jars at 50°C (±5°C).
- 3) Monitor sample masses.
- 4) At predetermined mass loss intervals, remove each subsample from the vacuum and cap.
- 5) Transfer each subsample to a graduated cylinder and measure the density.
- 6) Mobilize the material in the graduated cylinders and monitor the settled solids volumes over a 3 day period at ambient temperature (~23°C).
N₂ Solids
- 7) Place the graduated cylinders in an oven at 50°C for 1 day.
- 8) Measure the density of the material in each of the graduated cylinders at 50°C.
- 9) Mobilize the material in the graduated cylinders and monitor the settled solids volumes over a 3 day period at 50°C.
N₂ Solids
- 10) While stirring, remove homogeneous subsamples from each of the three graduated jars in duplicate and analyze for shear stress versus shear rate at ambient temperature (~25°C) and 50°C.
- 11) Add the prescribed quantities of glass formers to each of the three graduated cylinders and stir for one hour using an overhead mixer. - *ADD STIR BAR & COVER SAMPLE & ADD FOR STIR BAR VOLUME IN CALC OF SAMPLE DENSITY. 11/23/99 TJS*
- 12) Measure the density of the material in each of the graduated cylinders at ambient. -
- 13) Mobilize the material in the graduated cylinders and monitor the settled solids volumes over a 3 day period at ambient temperature (~23°C). - *STIR BAR & MAG. MIXER 11/23/99 TJS*
- 14) Place each of the graduated cylinders in an oven at 50°C for 1 day.
- 15) Measure the density of the material in each of the graduated cylinders at 50°C.
- 16) Mobilize the material in the graduated cylinders and monitor the settled solids volumes over a 3 day period at 50°C.
- 17) While stirring, remove homogeneous subsamples from each of the three graduated jars in duplicate and analyze for shear stress versus shear rate at ambient temperature (~23°C) and 50°C.
- 18) Transfer one of the three samples prepared in step 15 to an airtight glass mixing vessel with sampling port. The vessel is to have a impeller to vessel diameter ratio of 77:120.
- 19) Mix this sample for one week at ambient temperature. The impeller speed will be specified by the client prior to initiation of work.
- 20) During the week of mixing, remove subsamples after 1 hour, 1 day, and 1 week through the sampling port. Immediately measure the shear stress as a function of shear rate on these samples. These shear stress versus shear rate analyses are to be conducted at ambient temperature (~23°C).
- 21) After the week of stirring, transfer the material back to the graduated cylinder and leave undisturbed for 1 week at ambient temperature (~23°C).
- 22) During this one week, monitor for gas retention/release behavior using a video camera focused on the settled solid-liquid interface.
- 23) After one week, measure shear stress versus shear rate on the sample at ambient temperature.

PR Brent
10/14/99

Bredt, Paul R

From: Stuart Arm [SArm@bnflinc.com]
Sent: Tuesday, October 12, 1999 8:06 AM
To: Bredt, Paul R
Cc: Smith, Gary; pegg@cua.edu; GMcArthur@bnflinc.com; MBeary@bnflinc.com;
^ProjectDocumentControlMailbox@bnflinc.com
Subject: Target Sodium Concentrations for Processing the AW-101 Sampl

Paul,

Please use the target sodium concentrations of 6, 8 and 10M in the AW-101 sample rheological and physical property measurements.

Thanks,
Stuart.

Work Instructions

AW-101

- 1) Record the current sodium concentration of the pretreated AW-101 feed.

[Na]= 4.6 (C1) Data Source Dean Kurath CSIX Report

- 2) Record the [Na] targets provided by BNFL.

Target [Na]= 6 M (C2a) 1.31 *see attached email from Stuart Arm Ratio*
 Target [Na]= 8 M (C2b) 1.74
 Target [Na]= 10 M (C2c) 2.18

- 3) 200 ml of each evaporated slurry are required for this testing. Use the formula below to calculate the volume of pretreated slurry (V1) required to achieve 200 ml of evaporated slurry.

$V1a = (C2a/C1) \times 200ml$ *For 150ml TARGET Vol*
 $V1a = 6.0/4.6 \times 200 = 260ml$ *150 ml*
 $V1b = 8/4.6 \times 200 = 348$ *261 ml*
 $V1c = 10/4.6 \times 200 = 435$ *324 ml*

Handwritten calculations for 150ml target:
 $6/4.6 \times 150 = 196$
 $8/4.6 \times 150 = 261$
 $10/4.6 \times 150 = 326$
783

- 4) Weight three ²⁵⁰ ml glass graduated cylinders labeled AW-101 EVAP A, AW-101 EVAP B, and AW-101 EVAP C (Fisher cat #08-566-11F or equivalent).

	AW-101 EVAP A	AW-101 EVAP B	AW-101 EVAP C
Grad	212.58	195.0	202.1
Stopper	.593	36.4	37.2
Total Tare	<u>272.0</u> g	<u>231.4</u> g	<u>239.3</u> g

- 5) Measure the distance between the highest and lowest graduation on the graduated cylinders using a ruler.

	<u>250 grad</u>	<u>500ml grad</u>
High	<u>250</u> ml	<u>400</u> mL
Low	<u>10</u> ml	<u>50</u> mL
Distance	<u>23.3</u> cm	<u>19.9</u> cm
	$\frac{23.3}{240} = 0.097 \text{ cm/ml}$	$\frac{19.9 \text{ cm}}{350 \text{ ml}} = 0.0568 = 0.057 \frac{\text{cm}}{\text{ml}}$

	AW-101 EVAP A	AW-101 EVAP B	AW-101 EVAP C
Distance	_____ cm	_____ cm	_____ cm

all cylinders are the same PAB 3/20/00

- 6) Place approximately 500 ml of pretreated AW-101 feed into graduated cylinder AW-101 EVAP A. Record the mass and volume, and then return this material to the primary AW-101 feed container.

AW-101 EVAP A

Total 1043.3 g
Tare 420.2 g ~ 500 grad
Slurry 623.1 g
Volume 500 ml

- 7) Calculate the Density of the AW-101 feed.

Density = 1.2464 g/ml g/ml *should be 1.228 g/ml from Dave Blanchard*

- 8) Using this density, calculate the mass of material required for samples A, B, and C.

$M1a = V1a \times \text{Density}$

$M1a = 196 \text{ ml} \times 1.2464 = 244.3 \text{ g}$
 $M1b = 261 \times 1.2464 = 325.3 \text{ g}$
 $M1c = 326 \times 1.2464 = 406.3 \text{ g}$

for 150 ml of final
 $196 \times 1.228 = 240.7$ *10/24/99*
 $261 \times 1.228 = 320.5$
 $326 \times 1.228 = 400.3$

- 9) Weigh three 250 ml glass jars.

AW-101 EVAP 1	AW-101 EVAP 2	AW-101 EVAP 3
Tare <u>115.7</u> g	Tare <u>2a 114.9</u> g <u>2b 113.9</u>	Tare <u>3a 115.0</u> g <u>3b 115.3</u>

- 10) Transfer the required mass calculated above (M1a, M1b, and M1c) into each of the 250 ml jars (M1a in AW-101 EVAP 1, M1b in AW-101 EVAP 2, and M1c in AW-101 EVAP 3).

AW-101 EVAP 1	AW-101 EVAP 2 (g)	AW-101 EVAP 3
Total <u>360.0</u> g	Total <u>2a 297.1</u> g <u>2b 257.2</u>	Total <u>3a 310.7</u> g <u>3b 326.0</u>
Tare <u>115.7</u> g	Tare <u>2a 114.9</u> g <u>2b 113.9</u>	Tare <u>3a 115.0</u> g <u>3b 115.3</u>
Slurry <u>244.3</u> g	Slurry <u>2a 182.2</u> g <u>2b 143.3</u>	Slurry <u>3a 195.7</u> g <u>3b 210.7</u>
	<u>#2 Slurry Total = 325.4 g</u>	<u>+ = 406.4 g</u>

- 11) Calculate the target mass for each sample assuming all mass loss is the result of water evaporation.

10/22/99
Target $1a = M1a - (V1a - 200)$
 $244 - (196 - 150) = 198.3 \text{ g}$ *158 ml*
Target $1b = 325.3 - (261 - 150) = 214.3 \text{ g}$ *170 ml*
Target $1c = 406.3 - (326 - 150) = 230.3 \text{ g}$ *182 ml*

assuming 150 ml
 $T = M1a - (V1a - 150)$
 $T1c = 241 - (196 - 150) = 195 \text{ g}$

Calc of TARGET
Mass including
Tare wts

$$\#2a = \frac{214.3}{145} \cdot \left(\frac{183.2}{325.4} \right) + 114.9 = 234.9g$$

$$3a = 230.3 \cdot \left(\frac{406.9}{406.9} \right) + 115.3 = 234.7$$

$$3b = 230.3 \cdot \left(\frac{210.7}{406.9} \right) + 115.3 = 234.7$$

PR Bredt
09/24/99

$$1 = 198.5 + 115.7 = 313.7g$$

$$2b = 214.3 \cdot \left(\frac{183.3}{325.4} \right) + 113.9 = 208.4g$$

Test Plan: BNFL-TP-29953-046
Page 7 of 54

12) Place glass jars labeled AW-101 EVAP 1, AW-101 EVAP 2, and AW-101 EVAP 3 in a vacuum oven at 50°C (±5°C) and adjust the vacuum to an indicated pressure of 40-80 torr (this need not be done with a calibrated vacuum gauge).

13) Monitor the mass of each sample on a regular basis to assess the rate of evaporation.

TARGET + tare	314 AW-101 EVAP 1	a 235 AW-101 EVAP 2	b 208 AW-101 EVAP 2	a 226 AW-101 EVAP 3	b 235 AW-101 EVAP 3
Date	10-22-99 8:00 AM	Date	10/22/99	Date	10/22/99
Mass	353.5	Mass	294.6 254.7	Mass	305.3 320.5
Date	10-25-99 4pm	Date		Date	
Mass	358.0	Mass	290.5 247.0	Mass	292 307.8
Date	10-26-99 @ 4 pm	Date		Date	
Mass	341.7	Mass	278.4 237.3	Mass	290.8 305.5
Date	10-27-99 8am	Date	10/27/99 @ 8 am	Date	10/27/99 @ 8 am
Mass	336.7	Mass	261.1 223.7	Mass	260.0 272.3
Date	10-28-99 8am	Date		Date	
Mass	323.8	Mass	242.7 207.6	Mass	228.5 236.8
Date	10-28-99 @ 4 pm	Date		Date	
Mass	312.7	Mass	235.7 207.6	Mass	228.2 236.8
Date	11-10-99 @ 1pm	Date		Date	
Mass	312.4	Mass	234.7 207.7	Mass	225.2 234.2
Date		Date		Date	
Mass		Mass		Mass	
Date	11/30/99 @ 10 AM	Date	11/30/99 @ 10 AM	Date	11/30/99 @ 10 AM
Mass	313.7	Mass	234.9 208.4	Mass	225.9g 234.7g

Switched #1 to Back of oven, & put #3 at b forward. rjs 11/26/99

Stopped & covered samples 11/10/99 @ 1:30 rjs

Per over Night re & set @ 70°C

Rel water up to target 3/99 rjs

14) When the mass of each of the slurries reaches the targets calculated above, remove them from the vacuum and cap the sample. Record the mass. - Samples were covered with parafilm

AW-101 EVAP 1	AW-101 EVAP 2 (a) (b)	AW-101 EVAP 3 (a) (b)
Total 313.7 g	Total 234.9 208.4 g	Total 225.9 234.7 g
Tare 115.7 g	Tare 114.9 113.9 g	Tare 115.0 115.3 g
Slurry 198.0 g	Slurry 119.8 94.5 g	Slurry 110.9 119.4 g

$$+ 214.3g$$

$$+ 230.3g$$

~ 8hr Day
* Runaway @ 22 in H₂O & hot plate just barely on temp ~ 70°C

PR Bredt
09/24/99

$V_{Bar} = 49.84857$ Vol 50 mL DI water
- net wt of each 50 mL vol with stir bar

Test Plan: BNFL-TP-29953-046

Page 8 of 54

$$* d = \frac{Wt_{water} - Net\ wt\ with\ bar}{Wt\ bar}$$

- 15) Transfer the material in AW-101 EVAP 1 to graduated cylinder AW-101 EVAP A, AW-101 EVAP 2 to graduated cylinder AW-101 EVAP B, and AW-101 EVAP 3 into graduated cylinder AW-101 EVAP C. Record the mass and volume of material in each of the graduated cylinders.

AW-101 EVAP A	AW-101 EVAP B -1	AW-101 EVAP C	Transferred C to 500 mL Grad
Total <u>468.4</u> g	Total <u>441.9</u> g	Total <u>463.4</u> g	4178.3
Tare <u>272.0</u> g	Tare <u>231.9</u> g	Tare <u>239.3</u> g	269.5
Slurry <u>195.5</u> g	Slurry _____ g	Slurry _____ g	208.8
Volume <u>151</u> ml	Volume <u>159</u> ml	Volume <u>155</u> ml	145

- 16) Preweigh 3 teflon coated magnetic stir bars.

A	B	C
<u>3.87305</u> g	<u>3.89025</u> g	<u>3.85755</u> g

- 17) Pre weigh 3 10 ml volumetric flasks. - Neck size of 10 mL volumetric & 25 mL volumetrics are too small for the teflon stir bar of choice. rgs 11/27/99

	A	B	C
Weight at mark	<u>34.96276</u> g	_____ g	_____ g
With just water	<u>84.81133</u> g	_____ g	_____ g

- 18) Place one stir bar in each of the volumetric flasks and fill to the mark with DI water. Record the mass of the volumetric flask. Calculate the volume of the stir bars by the displacement of water.

Remove 1 stir bar & replace. Repeat for 2nd & 3rd stir bar - Adjust minus values accordingly.

A	B	C
Total <u>87.45152</u> g	Total <u>87.50927</u> g	Total <u>87.45952</u> g
Flask <u>(34.96276)</u> g	Flask <u>(34.96276)</u> g	Flask <u>(34.96276)</u> g
Bar <u>(3.87305)</u> g	Bar <u>(3.89025)</u> g	Bar <u>(3.85755)</u> g
Water <u>48.61571</u> g	Water <u>48.65626</u> g	Water <u>48.63921</u> g
$d = 3.1415$ g/mL	3.2678	3.1897

- 19) Place stir bar A in AW-101 EVAP A, stir bar B in AW-101 EVAP B, and stir bar C in AW-101 EVAP C. Since no solids are present I will add stir bar later

- 20) Mobilize the material in each of the graduated cylinders using a magnetic stir plate. After ~2 minutes, turn off the stir plate and record the time and date for each of the three samples.

AW-101 EVAP A Time _____ Date _____
 AW-101 EVAP B Time _____ Date _____
 AW-101 EVAP C Time _____ Date _____

Net

$$50\text{ mL water} = 84.81133 - 34.96276 = 49.84857\text{ mL of g}$$

no solids
RAB
11/23/99

21) After turning off the stirrer, record the ambient temperature volume of settled solids and liquids after 5 minutes, then every 10 minutes for the first hour, and then every half hour until the end of the work day. As well, record these volumes every 4 hours during the second and third day.

AW-101 EVAP A without glass formers at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

no solids
11/23/99
PRB

~~AW-101 EVAP B without glass formers at ambient temperature~~

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

No solids
11/23/99
PRB

AW-101 EVAP C without glass formers at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

no solids
11/23/99
PRB

22) Place the graduated cylinders in an oven at 50°C for 1 day. Remove the samples and record the mass and volume.

AW-101 EVAP A		AW-101 EVAP B		AW-101 EVAP C	
Total	_____ g	Total	_____ g	Total	_____ g
Tare	_____ g	Tare	_____ g	Tare	_____ g
Slurry	_____ g	Slurry	_____ g	Slurry	_____ g
Volume	_____ ml	Volume	_____ ml	Volume	_____ ml

Do
TASS

PRB
3/24/00

23) Mobilize the material in each of the graduated cylinders using a magnetic stir plate. After ~2 minutes, turn off the stir plate and record the time and date for each of the three samples.

AW-101 EVAP A	Time _____	Date _____
AW-101 EVAP B	Time _____	Date _____
AW-101 EVAP C	Time _____	Date _____

24) Return the graduated cylinders to the oven at 50°C.

No solids
11/23/99
PRB

25) After turning off the stirrer, record the volume of settled solids and liquids after 5 minutes, then every 10 minutes for the first hour, and then every half hour until the end of the work day. As well, record these volumes every 4 hours during the second and third day.

AW-101 EVAP A without glass formers at 50°C

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

no solids
11/23/99
PRB

~~AW-101 EVAP B without glass formers at 50°C~~

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

No solids
11/23/99
PRB

~~AW-101 EVAP C without glass formers at 50°C~~

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

No Solids
4/23/99
PRB

* Since glove box was @ 19°C

equilibrium needed to be Test Plan: BNFL-TP-29953-046
30 → 40 min before starts
would come into high range limit
Page 16 of 54

26) If not performed in the last 30 days, analyze one standard between 10 and 100 cP for shear stress as a function of shear rate at 25°C from 0 to approximately 1000 s⁻¹. Print out a copy of the rheogram and attach to this test plan.

Viscometer BokhwaCS Location 506 Geometry C-25 short

Viscosity 47.2 cP Lot Barcode # 179430 Manufacturer Brookfield
020100 A 2/1/2000 Expire 2-1-2001

File name 020200 A Date analyzed 2/2/2000
020200 B → OK 95.5cp std File name 020200 C
Finally Too cool
↓ D
E
↑
finally in

27) Remove the graduated cylinders from the oven and allow to cool overnight.

11/23/99 rxs No solids hence
28) ~~While stirring the samples on a magnetic stir plate~~ Remove subsamples from each of the graduated cylinders and analyze for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C and 50°C from 0 to approximately 1000 s⁻¹. Print out a copy of the rheograms and attach to this test plan.
~ 15ml each in 20ml vials

AW-101 EVAP A	File name <u>020200 F</u>	Date analyzed <u>02-02-2000</u>	rg B
AW-101 EVAP A Duplicate	File name <u>020200 G</u>	Date analyzed _____	
AW-101 EVAP B	File name <u>f H</u>	Date analyzed _____	
AW-101 EVAP B Duplicate	File name <u>f I</u>	Date analyzed _____	
AW-101 EVAP B ⁰²⁰ _{11/23/99}	File name <u>f J</u>	Date analyzed _____	
AW-101 EVAP B ⁰²⁰ Duplicate	File name <u>f K</u>	Date analyzed _____	

29) If possible, return this material to the respective graduated cylinders.

50°C {	Aw-101 A	File name <u>020200 Q</u>	Date <u>2-2-2000</u>	rg B
	Dup	↓ <u>R</u>		
	B	<u>020200 N</u>		
	Dup	↓ <u>P</u>		
	C	<u>020200 L</u>		
	Dup	↓ <u>M</u>		

rg Swoboden 2/2/2000

Glass Formulation used for AW-101 testing is proprietary to GTS Duratek and has been intentionally removed from this report.

31) Cap the graduated cylinders and record the mass and volume.

	AW-101 EVAP A	AW-101 EVAP B-1	AW-101 EVAP C	
for me over elution y>	<p>→ 575.9</p> <p>Total <u>457.5</u> g</p> <p>Tare <u>272.0</u> g</p> <p>Slurry <u>303.9</u> g</p> <p>Volume <u>142</u> ml</p> <p>→ 191</p>	<p>3379 407.9 g</p> <p>Total <u>432.8</u> g</p> <p>Tare <u>231.9</u> g</p> <p>Slurry <u>176.0</u> g</p> <p>Volume <u>73</u> ml</p> <p>98 nas</p>	<p>675.6 g</p> <p>Total <u>478.3</u> g</p> <p>Tare <u>269.5</u> g</p> <p>Slurry <u>406.1</u> g</p> <p>Volume <u>145</u> ml</p> <p>230</p>	<p>← were before glass formers ng 3/20/00</p>

120/99

32) Mobilize the material in each of the graduated cylinders using a magnetic stir plate. After ~2 minutes, turn off the stir plate and record the time and date for each of the three samples.

AW-101 EVAP A	Time <u>09:50 Am</u> 2:16:00	Date <u>2-16-00</u>
AW-101 EVAP B	Time <u>10:30</u>	Date <u>↓</u>
AW-101 EVAP C	Time <u>10:00</u>	Date <u>↓</u>

used over head mixer

Calibrated using	NIST Traceable	Photo tachometer
Resant Stirrer 10X	↓	L 710985
100rpm =	101.8	
1000 rpm =	1001	
1250 =	1246	

2/15/00 *[Signature]*

33) After turning off the stirrer, record the ambient temperature, volume of settled solids and liquids after 5 minutes, then every 10 minutes for the first hour, and then every half hour until the end of the work day. As well, record these volumes every 4 hours during the second and third day.

Ambient Temp 23.5°C

AW-101 EVAP A with glass formers at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min	2/16/00	09:50	186	191	
15 min			191	187	
25 min			191	185	
35 min			191	182	
45 min			191	180	
55 min			191	179	
1 hour			191	178	
1.5 hour			191	177	
2 hour			191	176	
2.5 hour					
3 hour			190	168	
3.5 hour			190	165	
4 hour		13:30	190	160	
4.5 hour			190	157	
5 hour			190	150	
5.5 hour			190	145	
6 hour			190	140	
6.5 hour			190	107	went home
n 21 7 hour	2/17/00	07:30	190	107	
24 hour		10:00	190	105	
28 hour		2 pm	190	105	
32 hour		4 pm	190	105	
46 48 hour	2/16/00	08:00	190	105	
52 hour		12 pm	190	105	
56 hour		1600m	190	105	

Replaced Stopper

6 days 2/22/00 9 AM 189 105

[Signature]
2/22/00

AW-101 EVAP B with glass formers at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min	2/16/00	10:30	98	98	—
15 min			98	98	
25 min			98	98	
35 min			98	98	
45 min			98.5	98	
55 min			98.5	98	
1 hour			98.5	97.5	
1.5 hour			98.5	97	
2 hour					Lunch time
2.5 hour			99	96.5	
3 hour			99	96.5	
3.5 hour			99	96	
4 hour			99	95.5	
4.5 hour			99	95	
5 hour			↓	94	
5.5 hour			↓	94	
6 hour			99	72	Went home sick!
6.5 hour					
21 hour	2/17/00	07:30	99	72	
24 hour		10:30	99	72	
28 hour		2 pm	99	71.5	
32 hour		4 pm	99	71.5	
45 48 hour	2/18/00	8 Am	99	71	
52 hour		12 pm	99	71	
56 hour		1600	99	71	

— Replaced stopper here!

Went home sick!

6 days 2/22/00 9 AM 98 71.5

rgb 2/22/00

AW-101 EVAP C with glass formers at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min	2/16/00	10:00	230	230	—
15 min			230	230	—
25 min			230	230	—
35 min			230	230	—
45 min					
55 min					
1 hour					
1.5 hour					
2 hour			230	225	
2.5 hour			—	—	Lunch time
3 hour			230	220	
3.5 hour			230	217	
4 hour		13:30	230	215	
4.5 hour			230	213	
5 hour			↓	212	
5.5 hour			↓	212	
6 hour			↓	211	
6.5 hour			224	200	Went home rrs
21 hr 7 hour	2/17/00	@ 7:30	229	200	
24 hour	↓	@ 10 am	228	200	
28 hour	↓	2 pm	228	198	
32 hour		4 pm	228	197	
46 48 hour	2/18/00	8 am	228	193	
52 hour	↓	12 pm	227	191	
56 hour	↓	1600	227	191	

← Forgot, so I replaced stopper,

21 hr

46

~ 6 days 2/22/00 9 am 227 190

[Signature] 2/22/00

34) Place the graduated cylinders in an oven at 50°C for 1 day. Remove the samples and record the mass and volume. *Start 2/22/00 @ 9 AM rgs*

AW-101 EVAP A		AW-101 EVAP B - 1		AW-101 EVAP C	
Total	<u>575.8</u> g	Total	<u>407.7</u> g	Total	<u>675.3</u> g
Tare	<u>272.0</u> g	Tare	<u>231.9</u> g	Tare	<u>269.5</u> g <i>— see pg 8</i>
Slurry	<u>303.8</u> g	Slurry	<u>175.8</u> g	Slurry	<u>405.8</u> g
Volume	<u>191</u> ml	Volume	<u>97</u> ml	Volume	<u>225</u> ml

35) Mobilize the material in each of the graduated cylinders using a ~~magnetic stir plate~~ *overhead mixer. rgs*. After ~2 minutes, turn off the ~~stir plate~~ *mixer* and record the time and date for each of the three samples. *

AW-101 EVAP A	Time <u>0945</u>	Date <u>2-23-00</u>
AW-101 EVAP B	Time <u>1005</u>	Date <u>2-23-00</u>
AW-101 EVAP C	Time <u>0950</u>	Date <u>2-23-00</u>

36) Return the graduated cylinders to the oven at 50°C.

* Small amt of sample is lost on the mixer rod & blade on each sample <.5 mL is projected loss.

** - Rubber stoppers blew out in oven on B & C samples

rgs
2/23/00

37) After turning off the stirrer, record the volume of settled solids and liquids after 5 minutes, then every 10 minutes for the first hour, and then every half hour until the end of the work day. As well, record these volumes every 4 hours during the second and third day.

AW-101 EVAP A with glass formers at 50°C

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min	2/23/00	09:50	191	189	
15 min		10:05	191	185	
25 min		10:20	191	180	
35 min		10:30	190	176	
45 min		10:40	190	173	
55 min		10:50	190	168	
1 hour		11:00	190	164	
1.5 hour		11:40	190	156	
2 hour					
2.5 hour		12:45	190	145	
3 hour		1:15	191	140	
3.5 hour		1:30		130	
4 hour		2 pm		115	
4.5 hour		2:30		105	
5 hour		3 pm	192	98	
5.5 hour		3:30		95.5	
6 hour		4 pm		95	
6.5 hour					
7 hour					
24 hour	2/24/00	09:00	192	94	
28 hour		13:00	192	94	
32 hour	2/25/00	09:00	192	94	
48 hour	2/25/00	13:00	198	94	
52 hour					
56 hour					

Solid Line
< @ 10:15 183

went home rgs

After Weekends 2/28/00 10 Am 191 94

[Signature]
2/28/00

AW-101 EVAP B with glass formers at 50°C

open & look 11/27/99

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min	2/23/00	10:10	96.5	96.5	—
15 min		20	↓	96.5	
25 min		30	96.5	95	
35 min		40	↓	94	
45 min		50	↓	93	
55 min		11:00	96.5	93	
1 hour		11:10	↓	93	
1.5 hour		11:40	↓	92.5	
2 hour					
2.5 hour		12:45	96.5	90.5	
3 hour		1:15	↓	88	
3.5 hour		1:30	↓	87	
4 hour		2 pm	↓	85	
4.5 hour		2:30	↓	83.5	
5 hour		3 pm	96.5	82	
5.5 hour		3:30	↓	81.5	
6 hour		4 pm	↓	81	
6.5 hour					
7 hour					
24 hour	2/24/00	9 am	96.5	64	
28 hour	↓	1300	↓	64	
32 hour		1600	↓	64	
48 hour	2/25/00	0900	96.5	64	
52 hour	2/25/00	1300	96.5	64	
56 hour					

unch

went home rps

No change rgs 2/28/00

After Weeknd 2/28/00 10 AM 96.5 64

AW-101 EVAP C with glass formers at 50°C

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min	2/23/00	0955	221	221	
15 min		10:10	222	222	
25 min		10:20		222	
35 min		1:30	221	221	
45 min		40	220	220	
55 min		50	220	220	
1 hour		11:00	220	220	
1.5 hour		11:40	218	215	
2 hour					
2.5 hour		12:45	218	215	
3 hour		1:15			
3.5 hour		1:30			
4 hour		2:00	218	212	
4.5 hour		2:30			
5 hour		3 pm	218	210	Estimate
5.5 hour		3:30			
6 hour		4 pm			
6.5 hour					
7 hour					
24 hour	2/24/00	0900	220	161	
28 hour		1300	220	160	
32 hour		1600	220	160	
48 hour	2/25/00	0900	220	160	
52 hour	2/25/00	1300	220	160	
56 hour					

After weekend 2/28/00 10 am 220 159

Hard to tell if clean or not!
rgs

Went home

rgs whoah
2/28/00

38) If not performed in the last 30 days, analyze one standard between 10 and 100 cP for shear stress as a function of shear rate at 25°C from 0 to approximately 1000 s⁻¹. Print out a copy of the rheogram and attach to this test plan.

Viscometer Bohlin CS Location 506 Geometry C-25-short
 Viscosity 47.2 cP Lot Barcode 179430 Manufacturer Brookfield
 Date analyzed 2/2/2000 Express 2-1-2001

* File name 020200A 020200B 020200C 020200D 020200E
020200B - OK within limits locally → 95.5cP std File NAMES
 ↓
 C
 D
 E

39) Remove the graduated cylinders from the oven and allow to cool overnight.

40) While stirring the samples on a magnetic stir plate remove subsamples from each of the graduated cylinders and analyze for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C and 50°C from 0 to approximately 1000 s⁻¹. Print out a copy of the rheograms and attach to this test plan.
25°C — See next page for 50°C data

AW-101 EVAP A	File name <u>030100D</u>	Date analyzed <u>03-01-00</u>	} 4 runs Overstressed @ 185 on the 1st 2 runs, but worked but locked BAO Will Run on 3-2-00 see next page data.
AW-101 EVAP A Duplicate	File name <u>E</u>	Date analyzed <u>F</u>	
AW-101 EVAP B	File name <u>G</u>	Date analyzed <u>H</u>	
AW-101 EVAP B Duplicate	File name <u>I</u>	Date analyzed <u>J, K</u>	
AW-101 EVAP C	File name <u>L</u>	Date analyzed <u>L</u>	
AW-101 EVAP C Duplicate	File name <u>L</u>	Date analyzed <u>L</u>	

41) If possible, return this material to the respective graduated cylinders.

42) Assemble a mixing vessel using the following parts or equivalent. Attach impeller to a stirring motor capable of maintaining a constant rotational rate from 100-1400 rpm.

Part	Vendor	Catalog number
500 ml O-ring Sealed Kettle, 3.75 inch OD, 5 3/8 inch flange	Labglass	LG-8071-100
Clamp	Labglass	LG-7316-106
O-ring	Labglass	LG-1022-476
Kettle top with three 24/40 necks	Labglass	LG-8072-100
2 3/8 diameter 4 blade impeller	Fisher Scientific	14-505-20G

Modified this design due to sample volume on split only being 73 mL (see adjacent sketch.) vs 2/14/00 for modified mixing vessel drawings.

03-01-00


38) If not performed in the last 30 days, analyze one standard between 10 and 100 cP for shear stress as a function of shear rate at 25°C from 0 to approximately 1000 s⁻¹. Print out a copy of the rheogram and attach to this test plan.

Viscometer Bonlin CS Location 506 Geometry C-25-short

Viscosity 47.2 cP Lot Barcode 179430 Manufacturer Brookfield

File name 020200A Date analyzed 2/2/2000 Expires 2-1-2001

020200B - OK within limits
locally

95.5cP std File Names
020200 C
D
E

39) Remove the graduated cylinders from the oven and allow to cool overnight.

40) While stirring the samples on a magnetic stir plate remove subsamples from each of the graduated cylinders and analyze for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C and 50°C from 0 to approximately 1000 s⁻¹. Print out a copy of the rheograms and attach to this test plan.

50°C Data

AW-101 EVAP A File name 030200L Date analyzed 0302 @ 1:00

AW-101 EVAP A Duplicate File name M Date analyzed 0302 @ 1:30

AW-101 EVAP B File name 030200J Date analyzed 0302 @ 12:05

AW-101 EVAP B Duplicate File name K Date analyzed 12:15

AW-101 EVAP E File name 03-0200H Date analyzed 3-2-00 11:35

AW-101 EVAP E Duplicate File name 030200I Date analyzed 11:45

Rerun started 3-2-00 @ 10:30 → 11:30

Rerun Evap C @ 25
File Name

up to 440 cP
failed

good
F* up to 560 cP

41) If possible, return this material to the respective graduated cylinders.

42) Assemble a mixing vessel using the following parts or equivalent. Attach impeller to a stirring motor capable of maintaining a constant rotational rate from 100-1400 rpm.

Part	Vendor	Catalog number
500 ml O-ring Sealed Kettle, 3.75 inch OD, 5 3/8 inch flange	Labglass	LG-8071-100
Clamp	Labglass	LG-7316-106
O-ring	Labglass	LG-1022-476
Kettle top with three 24/40 necks	Labglass	LG-8072-100
2 3/8 diameter 4 blade impeller	Fisher Scientific	14-505-20G

Modified this design due to sample volume on split only being 73 mL (see adjacent sketch.) rgs 2/14/00 for modified mixing vessel drawings.

3/2/00
[Signature]

* E & F viscosities were > 10% Appat viscosity increased on dup run, hence G was done and it too increased in viscosity. Evaporation in cell is suspected. rgs 3/2/00

* Chose 480 rpm based on impeller being 1" diameter
in actual operating conditions
(see attached graph) 3/1/00

43) Transfer the sample specified by BNFL to the mixing vessel (do not transfer the stir bar). Record which sample was transferred as well as the day and time transferred below.

Sample transferred Evap B-2 Date 3/1/00 Time @ 9 AM

44) Turn on the stirrer and adjust the rotational speed to that specified by BNFL. Record the time, date and speed below.

Speed 190 rpm Date 3/1/00 Time @ 9:05 hr
480 * 3/1/00 rgs
See Attached email rgs

45) After 1 hour of stirring, remove a sample through the sampling port and immediately analyze for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C from 0 to approximately 1000 s⁻¹. Print out a copy of the rheograms and attach to this test plan.

Run 1 File name 030100 A Date analyzed 3-1-00 @ 10 AM

Run 2 File name B Date analyzed 3-1-00 @ 10:30 - higher stress (failed) rgs
C 3-1-00 @ 10:45

46) After 1 day of stirring, remove a sample through the sampling port and immediately analyze for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C from 0 to approximately 1000 s⁻¹. Print out a copy of the rheograms and attach to this test plan. Pulled Sample 3-2-00 @ 9 AM

Run 1 File name 030200 A Date analyzed 030200 @ 9:45 AM (Set up to 730 s⁻¹ - did not finish)

Run 2 File name B Date analyzed @ 10 AM (set @ 560 s⁻¹ - OK run)
C @ 10:15 (" ")

47) After 1 week of stirring, remove a sample through the sampling port and immediately analyze for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C from 0 to approximately 1000 s⁻¹. Print out a copy of the rheograms and attach to this test plan. Pulled sample 3/8/00

Run 1 File name 030800 A Date analyzed 3/8/00 rgs @ 8:30 AM

Run 2 File name B* Date analyzed ↓
030800 C

48) After removing the last sample (after 1 week of stirring), transfer the sample back to the graduated cylinder. Record sample ID, volume and mass of material.

AW-101 EVAP B-2

3-8-00 @ 8:44 AM

Total 296.6 g Total 91.5 ml
Tare 140.2 g Solids 91.5 ml
Slurry _____ g Liquids 0 ml

switched to 12 hr REC mode
@ 9 AM since separation
appeared really slow

49) Focus a video camera on the solids-liquid interface of the sample and collect video for one week taking care not to disturb the sample. Report any observed gas retention and/or release behavior to the cognizant scientist. rgs

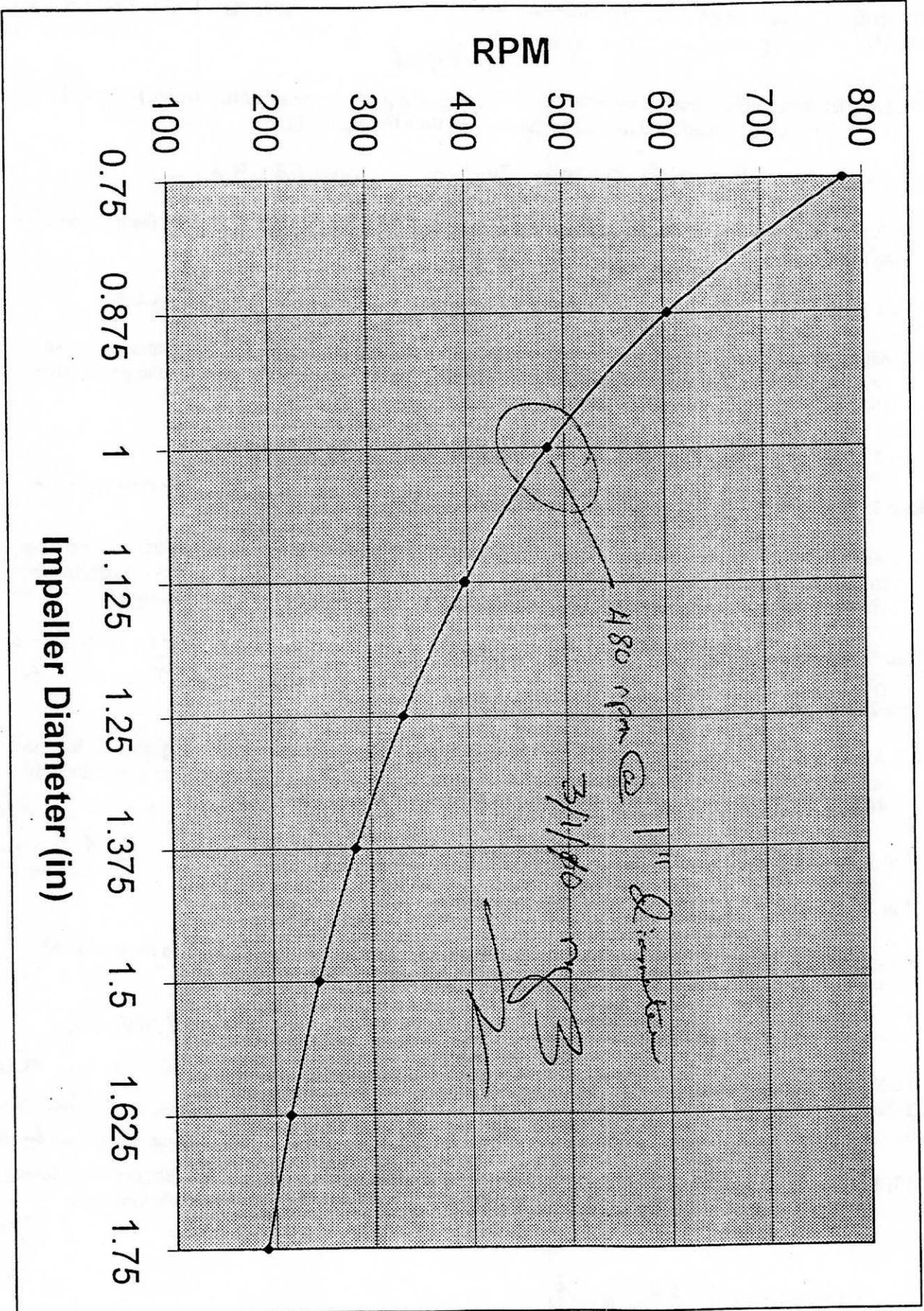
3/8/2000

* Not enough sample at this point.

~

Based on equation provided by

$$N^3 = 1.85 \times 10^7 \nu / D_i^5 \quad \text{PRB 3/20/99}$$



Prepared by Staff Member 3/1/00

3/1/00

Attachment to Test Plan PRB 3/20/99
29953-046 step 44

50) After the sample has remained undisturbed for 1 week, remove the standing liquid using a glass pipette.

51) Gently collect a subsample of the settled solids and immediately analyze for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C from 0 to approximately 1000 s⁻¹. Print out a copy of the rheograms and attach to this test plan.

loosely settled
slightly
Hled

Solids analyzed in HLRF using MS head with SVI sensor. 2 types of settled solids identified 1) loosely settled 2) tightly settled. Both types analyzed separately.

Run 1	File name	<u>031500C</u>	Date analyzed	<u>3/15/00</u>
	Dup	<u>031500E</u>		<u>3/15/00</u>
Run 2	File name	<u>031500F</u>	Date analyzed	<u>3/15/00</u>
		<u>031500G</u>		<u>3/15/00</u>
Standard File		<u>031500A</u>	95.5 cP Brookfield lot 111199 run on 3/15/00	
		<u>031500B</u>		

See 030800 D₁ } for 95.5 cP viscosity @ C Data
↓ E }
3/9/00 rgs

TAPE # 2 Labeled - AW-101 Settling Study
in 48 hr record mode

forgot to change out on Friday 8/10/2000

However Monday @ 8AM - Rec was still operating
and ~ 1/3 tape remained.

No sign of gas evolution was noticed over any time
I looked at the sample rgs 3/13/00

AW-101 B-2
Aqueous

AW-101 B-2
Solids

After 1 week
settling

Final wt 72.3 g

Final wt 207.9

Total Vol 91.5 ml

Solids 48.5 ml

Aqueous 43.0 ml

~ TARE

~ Tare

rgs 3/15/00

Attachment
 Test Plan 29953-046
 st-p 53
 PRB 3/20/00

From Dean Kurath

2/25/00 1:14 PM

PRB
 2/25/00

AN-107 Feed composition: not sulfate removal (Tc IX effluent)							
	Tc Effluent/vit feed average				Tc Effluent/vit feed average		
	ug/mL				ug/mL		
Al	2378			TOC	13,600		
Ba	--			TIC	7940		
Ca	163			Br	< 500		
Cd	26.9			Cl	< 500		
Co	2.1			F	3492		
Cr	44.3			NO2	28392		
Cu	11.7			NO3	112500		
Fe	8.5			oxalate	1375		
K	715			PO4	1400		
La	--			SO4	4008		
Mg	--			OH	0.8 M	estimated	
Mn	1.4						
Mo	16.4						
Na	111124						
Ni	214.5						
Pb	58.7						
Si	31.5						
Sn	--						
Ti	--						
U	--						
Zn	8.0						
	--						
B	17.4						
P	303						
Nd	--						
Sr	135						
W	76						
Y	--						
Zr	2.9						

Notes: 1) F content is probably due to organic interference.

Note: -- indicates below detection limit

$$\begin{aligned}
 [Na] &= 1.11 \times 10^5 \text{ ng/L} \cdot \frac{10^{-6} \text{ mol}}{\text{ng}} \cdot \frac{1 \text{ M.L}}{23 \text{ g}} \cdot \frac{1000 \text{ L}}{1} \\
 &= \underline{4.8} \text{ } 4.83 \text{ M}
 \end{aligned}$$

AN-107

52) Record the current sodium concentration of the pretreated AN-107 feed.

[Na] = 4.83 M (C1) Data Source Dean Kurath TCI report See previous page attached.

53) Record the [Na] targets provided by BNFL.

Target [Na] = 6 M (C2a) Ratio to 4.83 M 1.2422

Target [Na] = 8 M (C2b) 1.6563

Target [Na] = 10 M (C2c) 2.070

ryg
2/29/00

54) 200 ml of each evaporated slurry are required for this testing. Use the formula below to calculate the volume of pretreated slurry (V1) required to achieve 200 ml of evaporated slurry.

Revised TARGET V₁

$V1a = (C2a/C1) \times 200 \text{ ml}$

90 ~~100~~ ml $V1a = \frac{6 \text{ M}}{4.83 \text{ M}} \times \frac{90}{100} = 111.8 \text{ ml}$ *checked PRB 2/29/00*

180 ml $V1b = \frac{8}{4.83} \times 180 = 298.1 \text{ ml}$

90 ~~100~~ ml $V1c = \frac{10}{4.83} \times \frac{90}{100} = 186.3 \text{ ml}$

Total = 629.3 ml 596.2 ml reqs 2/29/00

Had to decrease volume sample was 2635 ml

55) Weight three 500 ml glass graduated cylinders labeled AN-107 EVAP A, AN-107 EVAP B, and AN-107 EVAP C (Fisher cat #08-566-11F or equivalent).

AN-107 EVAP A

AN-107 EVAP B

AN-107 EVAP C

Tare 234.0 g

Tare 239.8 g

Tare 230.8 g

With Rubber Stoppers in

56) Measure the distance between the highest and lowest graduation on the graduated cylinders using a place ruler.

High 250 ml

Low 10 ml

Distance 23.3 cm

AN-107 EVAP A

AN-107 EVAP B

AN-107 EVAP C

Distance _____ cm

Distance _____ cm

Distance _____ cm

all cylinders are the same PRB 3/30/00

57) Place approximately V1a ml of pretreated AN-107 feed into graduated cylinder AN-107 EVAP A.
Record the mass and volume, and then return this material to the primary AN-107 feed container.

AN-107 EVAP A

Total 1039.2 g
Tare 421.2 g
Slurry 618.0 g
Volume 500.0 ml

diff 0.45%

58) Calculate the Density of the AN-107 feed.

Density = 1.236 g/ml

Expected Density = 1.2244 g/ml

See e-mail from B. RAPKO referring
Dean Kurth's report. rg > 2/29/00

59) Using this density, calculate the mass of material required for samples A, B, and C.

M1a = V1a x Density
TARGET Vol 111.8 ml
90 mL M1a = 124.2 x 1.236 = 156.5 g
180 mL M1b = 298.1 x 1.236 = 368.5 g
90 mL M1c = 207.0 x 1.236 = 255.9 g
~ 737g

60) Weigh three 250 ml glass jars.

AN-107 EVAP 1 (250 mL Beaker) Tare 106.01 g
AN-107 EVAP 2 (400 mL Beaker) Tare 121.91 g
AN-107 EVAP 3 (250 mL Beaker) Tare 105.02 g

61) Transfer the required mass calculated above (M1a, M1b, and M1c) into each of the 250 ml jars (M1a in AN-107 EVAP 1, M1b in AN-107 EVAP 2, and M1c in AN-107 EVAP 3).

AN-107 EVAP 1	AN-107 EVAP 2	AN-107 EVAP 3
Total <u>244.21</u> g	Total <u>490.52</u> g	Total <u>335.38</u> g
Tare <u>106.01</u> g	Tare <u>121.91</u> g	Tare <u>105.02</u> g
Slurry <u>138.20</u> g	Slurry <u>368.61</u> g	Slurry <u>230.36</u> g

62) Calculate the target mass for each sample assuming all mass loss is the result of water evaporation.

Target_a = M1a - (V1a - Target Vol) x Density + Beaker Tare
 Target_a = 138.2 - (111.8 mL - 90 mL) x 1.236 + 106.01 = 222.41 g
 Target_b = 368.5 - (298.1 - 180) x 1.236 + 121.91 = 372.31 g
 Target_c = 230.3 - (186.3 - 90) x 1.236 + 105.02 = 239.02 g

2/29/00 rg B

Swoboda, Robert G

From: Bredt, Paul R
Sent: Tuesday, February 29, 2000 7:47 AM
Subject: Swoboda, Robert G
FW: composition

Attachment
to Test Plan
29953-046
Step 58

PRB
4/4/00

Thanks,

Paul

XX
Paul R Bredt, Ph.D. Pacific Northwest National Laboratory
Senior Research Scientist II Battelle Blvd., PO Box 999
Radiochemical Processing Group Richland, WA 99352
(509) 376-3777 paul.bredt@pnl.gov
XX

-----Original Message-----

From: Rapko, Brian M
Sent: Thursday, February 24, 2000 10:56 AM
To: Bredt, Paul R; Swoboda, Robert G
Subject: FW: composition

From this spreadsheet Dean sent me the recorded density of the feed is 1.2244 g/mL. - Brian

From: Kurath, Dean E
Sent: Tuesday, January 11, 2000 11:19 AM
To: Rapko, Brian M
Subject: composition

Brian, See the attached spreadsheet. Use the composition given for the Tc effluent/SO4 feed. Since we removed no Tc during Tc IX the composition should be ok.

As for the amount. The remaining feed is less than 1033 g (840 mL).



eed.effluent.composi
on.AN-1...

Dean Kurath
Staff Engineer
MSIN: P7-28
Chemical Separations and Slurry Processing Group
Pacific Northwest National Laboratory
Richland WA, 99352
email: dean.kurath@pnl.gov
phone: (509)376-6752
fax: (509)376-7127

Vacuum
Started Evap. 2/29/00 @ ~2pm

PR Bredt
09/24/99

Test Plan: BNFL-TP-29953-046
Page 32 of 54

Start wt 244.21 490.52 335.38

63) Place glass jars labeled AN-107 EVAP 1, AN-107 EVAP 2, and AN-107 EVAP 3 in a vacuum oven at 50°C (±5°C) and adjust the vacuum to an indicated pressure of 40-80 torr (this need not be done with a calibrated vacuum gauge).

64) Monitor the mass of each sample on a regular basis to assess the rate of evaporation.

TARGET Gross Wt. 222.41 372.31 239.02
AN-107 EVAP 1 (A) AN-107 EVAP 2 (B) AN-107 EVAP 3 (C)

Date	Mass	Date	Mass	Date	Mass
3-1-00 @ 8am	236.0		439.5		311.4
3-1-00 @ 3pm	231.2		419.3		301.4
3-2-00 @ 8am	220.1		402		255.6
3-3-00 @ 8:30	220.1		365.3		244.1
3/1/2000	222.6	3-2-00	372.7		
				3/9-00	238.9

Adjusted
with
water

Done

Done

65) When the mass of each of the slurries reaches the targets calculated above, remove them from the vacuum and cap the sample. Record the mass.

AN-107 EVAP 1		AN-107 EVAP 2		AN-107 EVAP 3	
Total	222.6 g	Total	372.7 g	Total	238.9 g
Tare	106.01 g	Tare	121.9 g	Tare	105.0 g
Slurry	116.6 g	Slurry	250.8 g	Slurry	133.9 g

[Signature] 3/8/00

66) Transfer the material in AN-107 EVAP 1 to graduated cylinder AN-107 EVAP A, AN-107 EVAP 2 to graduated cylinder AN-107 EVAP B, and AN-107 EVAP 3 into graduated cylinder AN-107 EVAP C. Record the mass and volume of material in each of the graduated cylinders.

AN-107 EVAP A	AN-107 EVAP B - 1	AN-107 EVAP C	AN107 Evap B-2
Total <u>350.1</u> g	Total <u>362.3</u> g	Total <u>363.7</u> g	<u>365.4</u> g
Tare <u>234.0</u> g	Tare <u>239.8</u> g	Tare <u>230.8</u> g	<u>239.7</u> g
Slurry <u>116.1</u> g	Slurry <u>122.5</u> g	Slurry <u>132.9</u> g	<u>125.7</u> g
Volume <u>90.5</u> ml	Volume <u>90.0</u> ml	Volume <u>92.5</u> ml	<u>91.5</u> ml
$\rho = 1.283 \text{ g/ml}$	$\rho = 1.361 \text{ g/ml}$	$\rho = 1.937 \text{ g/ml}$	$\rho = 1.374 \text{ g/ml}$

67) Prewrite 3 teflon coated magnetic stir bars.

A
B
C
see page 8

_____ g
_____ g
_____ g

68) Pre weigh 3 10 ml volumetric flasks.

A
B
C
PRB 3/20/00

_____ g
_____ g
_____ g

69) Place one stir bar in each of the volumetric flasks and fill to the mark with DI water. Record the mass of the volumetric flasks. Calculate the volume of the stir bars by the displacement of water.

A		B		C	
Total	_____ g	Total	_____ g	Total	_____ g
Flask	(_____)g	Flask	(_____)g	Flask	(_____)g
Bar	(_____)g	Bar	(_____)g	Bar	(_____)g
Water	_____ g	Water	_____ g	Water	_____ g

NOT done (No visible solids)

70) Place stir bar A in AN-107 EVAP A, stir bar B in AN-107 EVAP B, and stir bar C in AN-107 EVAP C.

71) Mobilize the material in each of the graduated cylinders using a magnetic stir plate. After ~2 minutes, turn off the stir plate and record the time and date for each of the three samples.

AN-107 EVAP A Time Not needed - no solids Date _____

AN-107 EVAP B Time _____ Date _____

AN-107 EVAP C Time _____ Date _____

72) After turning off the stirrer, record the ambient temperature volume of settled solids and liquids after 5 minutes, then every 10 minutes for the first hour, and then every half hour until the end of the work day. As well, record these volumes every 4 hours during the second and third day.

AN-107 EVAP A without glass formers at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

No Solids

3/9/99
pp
rgz

AN-107 EVAP B without glass formers at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

No Solids
3/7/00 rjb

*Did not Add stir bar
Not. Very many solids*

AN-107 EVAP C without glass formers at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min	8/9/00	7:30	92.5	~1ml	
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour	8/9/00	8:30	92.5	<1ml	
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour	8/9/00	4 pm	92.5	<1ml	
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour	8/13/00	8 AM	92.5	<1ml	

*Estimated (no increments
from 0 - 10ml on
250ml Grad.
ryB*

ryB

8/13/00

See Below for Start Vol & wts

PR Bredt
09/24/99

Test Plan: BNFL-TP-29953-046
Page 37 of 54

After
Removed Sub-Aliquots for Viscosity

73) Place the graduated cylinders in an oven at 50°C for 1 day. Remove the samples and record the mass and volume.

in Oven Start 3/21/00 → 3/23/00 @ 8am

AN-107 EVAP A		AN-107 EVAP B - 1		AN-107 EVAP C	
Total	336.1 g	Total	350.3 g	Total	347.9 g
Tare	234.0 g	Tare	239.8 g	Tare	230.8 g
Slurry	102.1 g	Slurry	110.5 g	Slurry	117.1 g
Volume	81.0 ml	Volume	83 ml	Volume	85 ml
$\rho =$	1.26 g/ml	$\rho =$	1.33 g/ml	$\rho =$	1.38 g/ml

← deg of flocculant ppt observed

74) Mobilize the material in each of the graduated cylinders using a magnetic stir plate. After ~2 minutes, turn off the stir plate and record the time and date for each of the three samples.

	Date	Time
AN-107 EVAP A	3/23/00	9 AM
AN-107 EVAP B	↓	↓
AN-107 EVAP C	↓	↓

Prior to mobilizing this sample had visible flocculant ppt. - much more than @ 25°C.

75) Return the graduated cylinders to the oven at 50°C.

No solids in A or B samples
3/21/00
ryg

As per Pauli request I did this part of the test.
3/21

Before oven measurements

	A	B-1	C	B-2
Total	337.4	350.5	348.0	356.4
Tare	234.0	239.8	230.8	239.7
Slurry	103.4	101.7	117.2	116.7
Volume	81.0	82.0	84.0	86.5

Reserved for stir studies

1KB
4/4/00

76) After turning off the stirrer, record the volume of settled solids and liquids after 5 minutes, then every 10 minutes for the first hour, and then every half hour until the end of the work day. As well, record these volumes every 4 hours during the second and third day.

AN-107 EVAP A without glass formers at 50°C

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min	3/23/00	9 AM	81.0	0	81
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

No

Solids in EVAP A

3/21/00 (9)

EVAP B-1

AN-107 EVAP B without glass formers at 50°C

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min	3/23/00	9 AM	83	—	83
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

No

Solids

EVAP B sample
3/2/00 YRS

~~AN-107 EVAP C without glass formers at 50°C~~

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min	3/23/00	9:15	85.0	-	85
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

solids settled
too fast at 25°C
RKB
3/20/00

77) If not performed in the last 30 days, analyze one standard between 10 and 100 cP for shear stress as a function of shear rate at 25°C from 0 to approximately 1000 s⁻¹. Print out a copy of the rheogram and attach to this test plan.

Viscometer Bohlin CS Location 325/506 Geometry C-25 Short
Viscosity 95.5 cP Lot 111195 Manufacturer Brookfield
File name 031600A Date analyzed 3/6/00
030800D
↓ E) Also these verify within 30 days

78) Remove the graduated cylinders from the oven and allow to cool overnight.

79) While stirring the samples on a magnetic stir plate remove subsamples from each of the graduated cylinders and analyze for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C and 50°C from 0 to approximately 1000 s⁻¹. Print out a copy of the rheograms and attach to this test plan.

2. Also -
- 1 gram
So 1 more time → Dup 2

@ 25°C Start * over stressed @ 7125⁻
File name 031600B Date analyzed 03-16-00 @ 10 am

File name 031600C Date analyzed ↓ Also over stressed
031600D - The best one (is)

File name 031600E Date analyzed 03-16-00 @ 10:45

AN-107 EVAP B Duplicate File name 031600F Date analyzed ↓ failed on return

AN-107 EVAP G File name 031600G Date analyzed 03-16-00 @ 11:15

AN-107 EVAP H Duplicate File name 031600H Date analyzed ↓ 11:35

80) If possible, return this material to the respective graduated cylinders.

@ 50°C

AN-107 Evap A File name 031600M Date 3-16-00 @

↓ A Dup

031600N

AN-107 EVAP B

↓ B Dup

031600K ← stress exceeded on down swing @ 3605⁻

3-16-00 @ 12:15 pm

031600L ← 2. & the same rgs @ 3605⁻

↓

AN-107 Evap C **

↓ C Dup

031600I

3-16-00 @ 11:50

031600J

↓ ↓

will rerun as per Print request See next page 41a

* - Sample dried while boosting temp to 50°C, therefore I changed out all sample in cup with fresh sample, I had enough 3/16/00 rgs

ENGINEERING WORKSHEET

Prepared By: RGJ Date: 3/21/00 Project: DNFL - AN107 Rheology
 Subject: _____

AN-107 EVAP B Rerun Viscosity @ 25°C & 50°C

25°C

File Name

Start Date/Time 3/21/00 @ 10 AM

Viscosity	File Name	Notes
~10 cP	03-2100A	over stressed on return @ ~200 s ⁻¹
~13	032100B	OK Run
~18	C	Just added a little more sample Stress exceeded on return @ ~350 s ⁻¹ ↑ Above Dumped sample that shot out back Over stressed again
~22	D	
~15 cP	E	changed out most of sample & cleaned wheel & edge of cup This run was good

@ 50°C

032100F

OK

~8 cP

~9.8

~9-10

G	=	over stressed on return @ ~200 s ⁻¹
H	-	used last of sample over stress

NOTE: Sample dries too quickly especially @ 50°C

RGJ 3/21/00

82) Cap the graduated cylinders and record the mass and volume.

AN-107 EVAP A		AN-107 EVAP B		AN-107 EVAP C	
Total	_____g	Total	_____g	Total	_____g
Tare	_____g	Tare	_____g	Tare	_____g
Slurry	_____g	Slurry	_____g	Slurry	_____g
Volume	_____ml	Volume	_____ml	Volume	_____ml

83) Mobilize the material in each of the graduated cylinders using a magnetic stir plate. After ~2 minutes, turn off the stir plate and record the time and date for each of the three samples.

AN-107 EVAP A	Time _____	Date _____
AN-107 EVAP B	Time _____	Date _____
AN-107 EVAP C	Time _____	Date _____

84) After turning off the stirrer, record the ambient temperature, volume of settled solids and liquids after 5 minutes, then every 10 minutes for the first hour, and then every half hour until the end of the work day. As well, record these volumes every 4 hours during the second and third day.

AN-107 EVAP A with glass formers at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

AN-107 EVAP B with glass formers at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

AN-107 EVAP C with glass formers at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

85) Place the graduated cylinders in an oven at 50°C for 1 day. Remove the samples and record the mass and volume.

AN-107 EVAP A		AN-107 EVAP B		AN-107 EVAP C	
Total	_____g	Total	_____g	Total	_____g
Tare	_____g	Tare	_____g	Tare	_____g
Slurry	_____g	Slurry	_____g	Slurry	_____g
Volume	_____ml	Volume	_____ml	Volume	_____ml

86) Mobilize the material in each of the graduated cylinders using a magnetic stir plate. After ~2 minutes, turn off the stir plate and record the time and date for each of the three samples.

AN-107 EVAP A	Time _____	Date _____
AN-107 EVAP B	Time _____	Date _____
AN-107 EVAP C	Time _____	Date _____

87) Return the graduated cylinders to the oven at 50°C.

88) After turning off the stirrer, record the volume of settled solids and liquids after 5 minutes, then every 10 minutes for the first hour, and then every half hour until the end of the work day. As well, record these volumes every 4 hours during the second and third day.

AN-107 EVAP A with glass formers at 50°C

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

AN-107 EVAP B with glass formers at 50°C

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

AN-107 EVAP C with glass formers at 50°C

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

89) If not performed in the last 30 days, analyze one standard between 10 and 100 cP for shear stress as a function of shear rate at 25°C from 0 to approximately 1000 s⁻¹. Print out a copy of the rheogram and attach to this test plan.

Viscometer _____ Location _____ Geometry _____

Viscosity _____ cP Lot _____ Manufacturer _____

File name _____ Date analyzed _____

90) Remove the graduated cylinders from the oven and allow to cool overnight.

91) While stirring the samples on a magnetic stir plate remove subsamples from each of the graduated cylinders and analyze for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C and 50°C from 0 to approximately 1000 s⁻¹. Print out a copy of the rheograms and attach to this test plan.

AN-107 EVAP A File name _____ Date analyzed _____

AN-107 EVAP A Duplicate File name _____ Date analyzed _____

AN-107 EVAP B File name _____ Date analyzed _____

AN-107 EVAP B Duplicate File name _____ Date analyzed _____

AN-107 EVAP B File name _____ Date analyzed _____

AN-107 EVAP B Duplicate File name _____ Date analyzed _____

92) If possible, return this material to the respective graduated cylinders.

93) Assemble a mixing vessel using the following parts or equivalent. Attach impeller to a stirring motor capable of maintaining a constant rotational rate from 100-1400 rpm.

Part	Vendor	Catalog number
500 ml O-ring Sealed Kettle, 3.75 inch OD, 5 3/8 inch flange	Labglass	LG-8071-100
Clamp	Labglass	LG-7316-106
O-ring	Labglass	LG-1022-476
Kettle top with three 24/40 necks	Labglass	LG-8072-100
2 3/8 diameter 4 blade impeller	Fisher Scientific	14-505-20G

94) Transfer the sample specified by BNFL to the mixing vessel (do not transfer the stir bar). Record which sample was transferred as well as the day and time transferred below.

Sample transferred _____ Date _____ Time _____

95) Turn on the stirrer and adjust the rotational speed to that specified by BNFL. Record the time, date and speed below.

Speed _____ rpm Date _____ Time _____

96) After 1 hour of stirring, remove a sample through the sampling port and immediately analyze for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C from 0 to approximately 1000 s⁻¹. Print out a copy of the rheograms and attach to this test plan.

Run 1 File name _____ Date analyzed _____

Run 2 File name _____ Date analyzed _____

97) After 1 day of stirring, remove a sample through the sampling port and immediately analyze for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C from 0 to approximately 1000 s⁻¹. Print out a copy of the rheograms and attach to this test plan.

Run 1 File name _____ Date analyzed _____

Run 2 File name _____ Date analyzed _____

98) After 1 week of stirring, remove a sample through the sampling port and immediately analyze for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C from 0 to approximately 1000 s⁻¹. Print out a copy of the rheograms and attach to this test plan.

Run 1 File name _____ Date analyzed _____

Run 2 File name _____ Date analyzed _____

99) After removing the last sample (after 1 week of stirring), transfer the sample back to the graduated cylinder. Record sample ID, volume and mass of material.

AN-107 EVAP __

Total	_____ g	Total	_____ ml
Tare	_____ g	Solids	_____ ml
Slurry	_____ g	Liquids	_____ ml

100) Focus a video camera on the solids-liquid interface of the sample and collect video for one week taking care not to disturb the sample. Report any observed gas retention and/or release behavior to the cognizant scientist.

PR Bredt
09/24/99

Test Plan: BNFL-TP-29953-046
Page 54 of 54

- 101) After the sample has remained undisturbed for 1 week, remove the standing liquid using a glass pipette.
- 102) Gently collect a subsample of the settled solids and immediately analyze for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C from 0 to approximately 1000 s⁻¹. Print out a copy of the rheograms and attach to this test plan.

Run 1 File name _____ Date analyzed _____

Run 2 File name _____ Date analyzed _____

902 Battelle Boulevard
P.O. Box 999
Richland, Washington 99352
Telephone (509) 376-1982
Email eugene.morrey@pnl.gov
Fax (509) 376-7127

January 26, 2000

Dr. Stuart Arm
3000 George Washington Way
Mailstop: BN-FL
Richland, WA 99352

29953-109

Dear Dr. Arm:

**TRANSMITTAL OF TEST PLAN BNFL-TP-046 REV. 0 ADDENDUM, "LAW MELTER
FEED RHEOLOGICAL AND PHYSICAL PROPERTIES MEASUREMENTS
(ATTACHMENT 1),"**

References: 1. Test Plan from Battelle to BNFL, Inc. "LAW Melter Feed Rheological and Physical
Properties Measurements," BNFL-TP-29953-046, Rev. 0.

Enclosed is one fully signed copy of test plan addendum, "LAW Melter Feed Rheological
and Physical Properties Measurements (Attachment 1)," for your files. The electronic copy
of the addendum will be transmitted to you by Chrissy Charron.

If you have any questions, please call me at 376-1982.

Sincerely,



Eugene V. Morrey
Project Manager

EVM:c²

Enclosure

Cc: Nigel Lockwood (BNFL)
Paul Bredt (Battelle),
BNFL Project File/LB

PNNL Test Plan Addendum

Document No.: BNFL-TP-29953-46
Rev. No.: 0
Document Control: Only the original signed copy is controlled

Title: LAW Melter Feed Rheological and Physical Properties Measurements

Work Location: Radiochemical Processing Laboratory

Page 1 of 2

Author: Paul Bredt

Effective Date: Upon Final Approval
Supersedes Date: New

Use Category Identification: Reference

Identified Hazards:

- Radiological
- Hazardous Materials
- Physical Hazards
- Hazardous Environment
- Other:

Required Reviewers:

- Technical Reviewer
- Project Manager
- Building Manager
- RPL Manager
- Radiological Control
- SFO Manager
- ES&H
- Quality Engineer

Are One-Time Modifications Allowed to this Procedure? Yes No

NOTE: If Yes, then modifications are not anticipated to impact safety. For documentation requirements of a modification see SBMS or the controlling Project QA Plan as appropriate.

On-The Job Training Required? Yes or No

FOR REVISIONS:

Is retraining to this procedure required? Yes No

Does the OJT package associated with this procedure require revision to reflect procedure changes?
 Yes No N/A

Approval

Signature

Date

Author

Paul Bredt

12/16/99

Technical Reviewer

Henry D. Smith

12/23/99

Project Manager

JE Purath for

12/29/99

BNFL

ST Am
Stuart Am

1/21/00

LAW Melter Feed Rheological and Physical Properties Measurements

Instructions

Under Test Plan BNFL-29953-046, "LAW Melter Feed Rheological and Physical Properties Measurements", samples of the AW-101 melter feed were evaporated to 6, 8, and 10 M with respect to sodium. Duratek has provided BNFL with specifications for glass former addition to the initial feed (4.59 M). This addendum to the test plan defines specifications for glass formers additions to the evaporated AW-101 melter feed.

Table 1 provides the specifications provided by Duratek as well as the adjusted levels required for addition to the evaporated samples under step 30 of Test Plan 29953-46. Since the Duratek specifications were provided for a 4.59M feed, these were multiplied by 1.31, 1.74, and 2.18 to arrive at the targets for the 6, 8, and 10 M feeds respectively.

Table 1. Glass formers to be Added to AW-101 Melter Feed.

Additive	Vendor	Grade	Target (g) per liter of feed			
			4.59 M	6 M	8 M	10 M
Kyanite (Al ₂ SiO ₅)	Kyanite Mining Corp.	Raw Kyanite, 325 Mesh	41.74	54.56	72.75	90.94
Orthoboric Acid (H ₃ BO ₃)	US Borax Inc.	Technical Granular	127.75	166.99	222.65	278.32
Wollastonite (CaSiO ₃)	NYCO Minerals	Powder untreated, NYAD 325 Mesh	32.44	42.41	56.54	70.68
Red Iron Oxide (Fe ₂ O ₃)	The Prince Manufacturing Co.	Red Iron Oxide, 325 mesh, (5001)	37.35	48.82	65.10	81.37
Olivine (Mg ₂ SiO ₄ with some Fe ₂ SiO ₄)	UNIMIN Corp.	325 Mesh (#180)	22.10	28.89	38.52	48.14
Ground Silica Sand (SiO ₂)	US Silia Co.	Sil-co-Sil 75, 200 Mesh	262.84	343.58	458.10	572.63
Rutile (TiO ₂)	Chemalloy Co.	Premium Grade, Airfloated	14.32	18.72	24.96	31.20
Zinc Oxide (ZnO)	Zinc Corp. of America	KADOX-920	21.21	27.73	36.97	46.22
Zircon Sand (ZrSiO ₄)	American Minerals Inc.	Flour 325 Mesh	31.94	41.75	55.67	69.58
Sugar	C&H	Granular Sugar	51.34	67.12	89.49	111.86

* $\frac{6m}{4.59m} = 1.307$

$\frac{8m}{4.59} = 1.743$

$\frac{10m}{4.59} = 2.179$

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