

DEVELOPMENT OF VISCOMETERS FOR KRAFT BLACK LIQUOR

SUMMARY REPORT-PHASE II AND IIA

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by

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Four of the six original on-line designs considered were thoroughly tested in this study. In particular, the following three designs were deemed as adequate viscometers for black-liquor applications and were recommended for further evaluation on industrial sites during the ensuing Phase III of the project: (i) a Brookfield coaxial rotational viscometer, (ii) a Micro Motion coriolis capillary viscometer, and (iii) a Nametre torsional vibratory viscometer. In addition, an experimental Magnetic Resonance on-line viscometer prototype developed by Southwest Research Institute and Quantum Magnetics was subjected to a preliminary evaluation with the objective of gathering data for instrument design purposes. Although it was determined that the latter instrument holds good potential as a black-liquor viscosity sensor, the instrument is still in its early development phases and is not yet ready for field deployment.

The final recommendations for pairing of sensors with pulping mill sites during Phase III of the project is as follows. First, the Brookfield instrument has been recommended for use in the Georgia Pacific Corporation mill in Palatka, Florida. The recommendation is based on the observation that this site processes liquors with viscosities varying from the low and to medium values, a range for which the Brookfield instrument is best suited. Second, the Micro Motion sensor is recommended for trials at the Canadian Forest Products site in Prince George, British Columbia. This site is currently firing liquor with high solids contents, a condition of suitability to the Micro Motion sensor, which is particularly accurate at the higher viscosities. Finally, the Nametre sensor is recommended for trials at the mill of the Chesapeake Paper Company in West Point Virginia where the solids content of the black liquor at the evaporators and at the ring header may experience significant variations due to solids precipitation effects. The Nametre instrument is adequate for use in this site because it features the widest range of viscosity applicability, and because multiple sensors can be used with a single electronics package.

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1 INTRODUCTION

This report documents the results of the evaluation of the on-line prototype viscometers for Kraft Black Liquors carried out at the Pilot Plant facilities of the University of Florida. The work reported in this document is sponsored by the US Department of Energy under DOE Cooperative Agreement DE-FC02-93CH10564 and satisfies the goals of Phase II and Phase IIA of the project.

1.1 PARTICIPATING ON-LINE PROTOTYPE VISCOMETERS

The original plan called for the evaluation of five prototype on-line viscometers along with laboratory bench versions. At a later stage in the project an additional experimental prototype under development at Southwest Research Institute was added. The on-line viscometers considered were the following:

- 1. Brookfield TT-100 coaxial rotational viscometer. Manufactured by Brookfield Engineering Laboratories, Inc., Stoughton, MA.
- 2. Micro Motion CMF025 coriolis capillary viscometer. Manufactured by Micro Motion Inc., Boulder CO.
- 3. Nametre 2010MX Viscoliner torsional vibratory viscometer. Manufactured by Nametre Company, Metchen, NJ.
- 4. Southwest Research Institute/Quantum Magnetics Magnetic Resonance Viscometer. Experimental prototype under development at Southwest Research Institute, San Antonio, TX.
- 5. Cambridge Applied Systems Sliding Element Viscometer. Manufactured by Cambridge Applied Systems Inc., Medford, MA.
- 6. Stevens Institute Capillary Viscometer. Manufactured by the Stevens Institute, Hoboken, NJ.

Of the instruments listed above, the first four manufacturers were able to deliver functional on-line prototypes that underwent extensive testing and evaluation. The Cambridge instrument (fifth in the list) was delivered only in its off-line (laboratory) form. Although it was not suitable for on-line utilization in an industrial environment, the laboratory Cambridge instrument nevertheless underwent a preliminary test of performance in the experimental pilot flow facility.

In addition to the Cambridge laboratory unit, two other manufacturers indicated in the list above were also able to deliver off-line (laboratory) versions of their on line instruments. More precisely, the laboratory units used in the study were the following:

- 1. Brookfield TT-PVS rotating cup viscometer for laboratory use. Manufactured by Brookfield Engineering Laboratories, Inc., Stoughton, MA.
- 2. Nametre 2010MX Viscoliner torsional vibratory viscometer for laboratory use. Manufactured Nametre Company, Metchen, NJ.
- 3. Cambridge Applied Systems Sliding-Element Viscometer for laboratory use. Manufactured by Cambridge Applied Systems Inc., Medford, MA.

1.2 EXPERIMENTAL OVERVIEW

The viscosity of black liquor is dependent not only on temperature but also on the type of liquor (wood species and cooking conditions) and the solids concentration. The experimental plan was to examine the performance of the on-line prototype viscometers under various process conditions to determine the range and suitability of each instrument in typical mill environments. Four different types of black liquor were utilized after concentrated to various levels of solids content:

- 1. Canadian Forest Products LTD., Prince George BC, Canada
- 2. Chesapeake Paper Products Company, West Point VA, USA
- 3. Georgia Pacific Corporation, Palatka FL, USA
- 4. Canadian Forest and Georgia Pacific Mixture

Each prototype on-line viscometer was evaluated in a pilot flow-loop facility running the different types of black liquors at different levels of solids content and at varying flow rates and temperatures. The accuracy of the of the viscosity measurements produced by the prototypes was determined by comparing the measurements to those obtained by laboratory measurements taken under well-controlled conditions. The laboratory instruments utilized as references included the three off-line versions of instruments acquired from the instrument manufacturers participating in the project as well as standard laboratory viscometers, such as a Haake Rotovisco RV12 rotational viscometer. The range, accuracy, and repeatability of the measured response were examined at various temperatures and flow rates under steady state conditions. The time response, sample acquisition, and preparation were additional performance variables that were also under consideration. Finally, the reliability and maintenance of the instruments were long term factors that were considered in the evaluation.

Extensive data were acquired during the evaluation phase using all four black liquor types at 13 different solids concentrations under various temperature and flow rate conditions. The experiments tested the accuracy and the reproducibility of the on-line instruments under steady state conditions, and also tested the speed of response of the instruments through transient-response experiments. The steady-state data were mostly reported in plots of the viscosity as a function of flow rate, and the transient-response tests data were plotted as viscosity as a function of time.

1.3 ORGANIZATION OF THE REPORT

Chapter 2 of the report discusses the experimental design of the testing phase. The experimental plan and methodology are discussed in detail along with the specifics of the laboratory characterizations.

Chapters 3-6 respectively discuss the experimental evaluations of the Brookfield, Micro Motion, Nametre, and Southwest Research Institute/Quantum Magnetics on-line instruments.

Chapter 7 discusses data gathered with the Laboratory Cambridge Viscometer. The instrument was installed in the pilot flow loop to carry out a conceptual evaluation of its capabilities with low-solids black liquor and to measure the instrument's response time. No information is given on the on-line prototype because no instrument was delivered for evaluation. Chapter 8 discusses the Stevens Institute Viscometer in concept only, since neither an on-line prototype or a laboratory bench version were delivered. No experimental data was gathered for this system.

Chapter 10 closes the report with a number of conclusions on the performance of the individual viscometer evaluations and offers recommendations for design modification and for the ensuing mill-site trials for Phase III.

2 EXPERIMENTAL DESIGN

This section describes the matrix of experiments that are carried out to evaluate the performance of the prototype viscometers in terms of (i) instrument variables such as accuracy and response time, and (ii) process variables such as flow rates and temperature. A list of relevant variables considered are shown in Table 2.1

(ii) Instrument variables
Accuracy and repeatability
Range of applicability
Response time
Sample acquisition and
conditioning
Reliability and maintenance

Table 2.1. Process and instrument variables of interest.

Of the process variables listed on the first column of Table 2.1, the temperature and the solids content of the liquor have a dominant effect on the viscosity of black liquor are (Fricke, 1987; Zaman, 1993; Zaman and Fricke, 1994<u>b</u>). Therefore, the experimental plan for evaluating the prototype viscometers includes subjecting the prototype viscometers to operation regimes at various temperatures and solid concentrations that are representative of pulping-mill conditions. Black liquors obtained from different sources—such as a liquor produced at the Palatka site of Georgia Pacific in Florida, or the liquor from Chesapeake Paper Company produced at the West Point site in Virginia—have different viscosity behavior because the sources use different wood species and different pulping recipes. The experimental program includes testing black liquors provided by three different sources of different geographical locations. It is now well known that black liquor behaves mostly as

a Newtonian fluid (Zaman and Fricke, 1991; Zaman and Fricke, 1994b), hence, the on-line viscometers should produce viscosity measurements that are independent of flow rate. Therefore, it is important to evaluate the viscometers under different flow rate regimes in order to determine if the design of the instruments permits them to effectively cope with the effects that a varying flow may have on the probes and other sensing elements. Finally the presence of contaminants and additives in the liquor may adversely impact the performance of an on-line viscometer. Contaminants such as fly ash augment the concentration of particles in the flow, and hence increase the likelihood of fouling in the sensors, as well as of causing malfunctions due to obstructions and interactions between the solid particles and the sensors.

The instrument variables shown in the second column of Table 2.1 include prominently the accuracy and the repeatability of the instrument. Accuracy refers to the agreement between the viscosity measurement produced by the on-line instrument and the viscosity measurement produced by a reliable instrument under laboratory conditions. On the other hand, repeatability (or precision) refers to a situation where the on-line instrument produces the same measurement after repeated readings under the same process conditions. Ideally one would want the viscometers to be both accurate and precise; however, often in processing operations it is sufficient to obtain a precise measurement because a repeatable measurement permits the deployment of effective process control strategies. Another instrument variable of interest is the range of applicability of the viscometers. For example, it may be possible that the principle of operation of a viscometer makes it reliable at low viscosities, but not reliable at high viscosities. Hence, the range of applicability is a critical variable of interest. Different viscometers acquire black-liquor samples from the flow stream using different techniques, such as passing the flow through a rotating cup, through a pair of bent capillary tubes, or through a sampling chamber. Furthermore, conditioning of the flow may also be done, such as adjustment of the temperature. The experimental evaluation study includes an assessment of the suitability of the different sampling methods used by the prototype viscometers considered. Finally, a primary concern with all prototype viscometers is their reliability and their maintenance requirements. Obviously, an instrument that operates without malfunctions and without need for maintenance for a long period of time is highly desirable.

2.1 Experimental Plan

The testing plan was designed to examine factors affecting performance of the prototype viscometers under various process conditions. Three different types of black liquor have been acquired for the evaluation phase from the following pulping-mill sources:

- Canadian Forest Products LTD., Prince George, Canada
- Chesapeake Paper Products Company, West Point VA, USA
- Georgia Pacific Corporation, Palatka FL, USA

A forth type of black liquor type was generated at the University of Florida by combining Georgia Pacific and Canadian Forest black liquors in an approximate 50%/50% mix. This mixture was made with the objective of obtaining a liquor with high solids content.

Five hundred gallons each of Canadian Forest and Chesapeake Paper black liquors were shipped to the pilot flow loop with a concentration of approximately 50% solids. A total of 700 gallons of Georgia Pacific black liquor was received at concentrations of approximately 34% and 42% solids. It was necessary to have on site such large samples of black liquor so that they could be concentrated to higher solids contents using the University of Florida evaporation system.

For each of the black liquor types, a range of solids concentrations was chosen to represent different locations where the viscometers might be placed within a pulp and paper mill. Once a black liquor type at a fixed solids concentration was loaded into the pilot flow loop, experiments were performed at various temperatures. Typically the temperature was

progressively increased in the main tank from run to run so that the loop was thermally equilibrated; however, since the main holding tank is at atmospheric pressure, the highest temperature was limited to 10-20°C below the boiling point of the black liquor. At least one run was performed using the heater in the sample loop so that temperatures above the atmospheric boiling point of the black liquor could be tested. Higher temperatures are more likely to represent the operating conditions of a pulp and paper mill.

At each temperature, several runs at different flow rates were made. These included one batch (or zero flow) run in which the viscometer line was isolated from the flowing stream. The flow rate range used depended upon the limits of the pilot flow loop. In general, the flow rates in a run were set in a sequence of steps. Typically, the first step was a run at 0.5 gpm. The second and subsequent steps consisted of an increase in the flow rate by 0.5 gpm, as shown in Table 2.2, until a maximum sustainable flow rate was attained. Finally, the zero-flow run was made.

Step	Flow rate (gpm \pm 0.25)
1	0.5
2	1.0
3	1.5
4	2.5
5	0.0

Table 2.2. Flow rate steps examined in experimental runs

Overall, five flow rate runs were performed within each temperature run. The upper limit of flow rate for these runs were dependent upon the output of the main progressive cavity pump at its maximum operational limit of 150 psig. In general, however, the main line pressure was kept at 70 psig so as not to stress the main pump to its maximum.

Two modes of operation were also examined: Steady state and transient. A majority of the experimental runs were at a thermal and flow steady state. Several thermal transients were examined to determine response times from the instruments. Thermal transient experiments were done by introducing step changes in the opening of the steam

control value of the heater to induce a viscosity step change resulting from a step change in temperature.

Once the experiments for a given black liquor type and solids concentrations were completed, the black liquor was off-loaded from the loop into transfer tanks. Then the Viscometer Line was cleaned first with low-pressure steam, and then with circulating hot water. The black liquor in the transfer tanks was taken to the evaporator and loaded into the evaporator feed tank. The black liquor was concentrated to the next solids concentration of interest using a wiped-film evaporator operating at a jacket pressure of 115 psig of saturated steam and process pressure of 0.5 atmosphere. Under these conditions, the average evaporation rate is approximately $15lbm_{water}/hr$. Once the evaporation was completed the black liquor was then loaded back into the pilot flow loop for further experiments.

2.2 Experimental Method

During the evaluation phase, we used a comprehensive experimental plan to examine various black liquor solids concentrations ranging from 34% to 73% with four different types of black liquor (Figure 2.3). Of the four black liquor types, the Georgia Pacific was the easiest to handle and work with because of its low level of air entrainment and ease of evaporation; therefore, more experiments were performed with this black liquor. Although the Canadian Forest and Chesapeake Paper liquors were not evaporated to high solids contents, two runs were made at low temperatures to simulate the highviscosity behavior that these liquors would have displayed at higher solids concentration.

A main problem experienced with the handling of the Canadian Forest and Chesapeake Paper black liquors was the formation of crystalline particles. Crystals formed in stagnant flow portions in the Main Holding Tank T1 (*cf.* P&ID diagram in Figure 9.1), and were eventually pumped throughout the loop where they collected and

created blockages in the lines. There is a strainer with a 40 mesh screen installed in the Viscometer Line to protect the viscometer from larger particles. The viscometers tested were not affected by particles smaller than 40 mesh.

		Annonananananananananananananananananana	36666666666666666666666666666666666666
Georgia Pacific	Canadian Forest	Chesapeake Paper	GP & Canadian Forest Mix
34.14%	47.96%	49.39%	67.71%
42.24%	57.78%	59.28%	71.38%
48.29%			73.40%
55.84%			
65.51%			
68.37%			
***************************************		Preventional procession and a second se	

Table 2.3. Black liquor types and solids concentration examined.

The Chesapeake Paper black liquor created more difficulties due to air entrainment, which made laboratory characterization, especially density correlations, extremely difficult to perform. The air entrainment in the loop is normally insignificant due to the high operating pressures and would not be a problem in actual mill operation. The effects of air entrainment can be determined by examining the density measurement as a function of flow rate with the Micro Motion instrument at a constant temperature. Black liquor can be assumed to be an incompressible fluid, therefore its density is independent of pressure. Since the flow rate in the Viscometer Line is a function of the Main Line pressure, the density of the black liquor is independent of flow rate. Significant aeration of the black liquor would give an artificially low density measurement with the Micro Motion device, and the density would increase as the flow rate increases; however, the density measurement of the Chesapeake Paper black liquor was unaffected by flow rate and remained constant at a constant temperature.

Once the Pilot Flow Loop was loaded for the current black liquor run, the Main Holding Tank and the flow loop were kept at a nominal temperature for several hours to ensure that the black liquor was thoroughly mixed and homogeneous. Flow in the loop was stopped briefly and a 1-liter sample was withdrawn for laboratory characterization. This procedure was followed to prevent flashing of the liquor when the laboratory sample was withdrawn, since the sample point was upstream of the heater, and the main tank was well below the boiling point at this point. Flashing is an undesirable effect because the loss of water and organics makes the sample appear more highly concentrated. If possible, the experimental runs were delayed until laboratory characterizations were completed so that an immediate check could be performed on the viscometers before testing.

In preparation for an experimental run, various instruments and sensors needed to be zeroed or calibrated.

- The flow loop was turned off momentarily and the Main Line Flow Meter FT1 (*cf.* PI&D diagram in Figure 9.1) and the Sample Line Flow Meter FT2 (*cf.* PI&D diagram in Figure 9.1) were zeroed at zero flow.
- Valves V4 and V16 were closed so the differential pressure cells (DP1 and DP2) across the Heater E1 and Cooler E2 could be zeroed.
- The Viscometer Line is bypassed by directing flow through the bypass using the Viscometer Line Isolation Valves (V7 and V8) so that the Brookfield and Micro Motion viscometers could be zeroed.

Once the preliminary equipment and instrumentation checks and calibrations were completed, the main line flow was set to approximately 30-40 gpm at a pressure of 70 psig and the flow was passed through the sample and viscometer lines to establish thermal equilibrium. In the control room, the National Instruments LabVIEW software and Micro Motion Prolink package were turned on concurrently with the DAQ computer and the data files were initialized. The values recorded by LabVIEW and Prolink were verified against the displayed values of indicators and controllers on the control panel. For the final step in preparing the data acquisition system, the Southwest Research Institute/Quantum Magnetics computer was then setup to acquire its own data. For the first flow rate run at the first temperature of interest, the Sample Line Controller FC1 (cf. P&ID diagram in Figure 9.1) was manually adjusted until the desired flow rate was attained. The Viscometer Line was allowed to achieve steady state, with the Cooling Water Controller TC2 (cf. P&ID diagram in Figure 9.1) set to manual at 99.9% open. Once at steady state, the Southwest Research Institute/Quantum Magnetics data acquisition system is turned on first, followed by Prolink and then LabVIEW. For each flow rate run, three different Brookfield rotational speeds were usually examined. The LabVIEW program paused while the Brookfield rotational speed were changed. In the meantime, the other systems were left running. LabVIEW was restarted after the rotational speed had been changed and the new calibration factors were entered into the LabVIEW program. Once all the data for the current flow rate had been acquired, all the DAQ systems were turned off, and flow through the Viscometer Line was increased to the next flow rate of interest. After steady state was achieved once again the data acquisition process was repeated.

The final flow rate run was always the zero-flow (or batch run) condition which was handled in two steps. In the first step, the same procedure was followed as in the other flow rate runs except that the Southwest Research Institute/Quantum Magnetics system was turned off. On the second step, the Southwest Research Institute/Quantum Magnetics instrument was run alone in the batch mode, since the Southwest Research Institute/Quantum Magnetics DAQ system can control the Viscometer Line Isolation Valves (V7 and V8). The Southwest Research Institute/Quantum Magnetics instrument in the automatic mode first opened the viscometer line for a predetermined time, and then closed the Viscometer Line and analyzed the black liquor sample. When the test was completed, the isolation valves were opened and a new sample was loaded into the line. This was repeated until the Southwest Research Institute/Quantum Magnetics DAQ system was turned off.

When the first temperature run was completed, the temperature in the main holding tank was increased using the Tank Temperature Controller TC1. After the flow loop has reached a new thermal equilibrium, the flow rate step runs were duplicated as described above. The procedure was repeated for the later temperature runs until the last run, in which the sample line heater was utilized to raise the temperature up past the boiling point of the black liquor sample. The flow rate runs were handled in the same manner, except that more care was needed to maintain a constant flow rate and temperature. The Process Heater Output Controller TC2, the Sample Flow Rate Controller FC1, the Main Line Pressure Controller PC1, and the Main Pump Speed Control were adjusted manually until the desired temperature and flow rate were achieved. At this point, The Sample Line Steam Tracing Controller PC2 is set to approximately to 25 psig to help offset heat losses in the sample line. Once steady state was attained, the flow rate run was initiated. This procedure was followed for the remaining flow rate runs.

For the transient heater runs, the flow through the viscometer line was fixed with the Process Heater Output Controller TC2 set manually to 0% open. Only the LabVIEW software was used for transient experiments, since LabVIEW preempts Prolink when multitasking with the DAQ computer. Although Prolink works well in the background of LabVIEW under steady state conditions, it does not respond quickly enough to rapidly changing variables. The LabVIEW software was turned on and then the Process Heater Output Controller TC2 was set to manually open the valve by small increments. The sample line flow rate could not be held to an absolutely constant value, but an approximate value could be maintained by adjusting the Motor Speed Controller. Once the viscometers had reached steady state, TC2 was setpped open to a new value. This procedure was repeated until the process output was saturated.

The set of data were collected as ANSI files from three locations; LabVIEW, Prolink and the Southwest Research Institute/Quantum Magnetics. The data were then transferred into Excel 5.0 files for analysis. Later, the data is divided for analysis of individual instrument and then analyzed.

2.3 Laboratory Characterizations

Well mixed representative black liquor samples from the pilot flow loop were characterized In the laboratory to develop viscosity, density, and heat capacity correlations. The laboratory characterization included measurements and correlations of:

- Viscosity
- Density
- Heat Capacity
- Solids Content
- Boiling Point Elevation

The viscosity for each black liquor sample was measured over the operating temperature range of the pilot flow loop using, at a minimum, three laboratory viscometers. The laboratory models (the Lab Cambridge, the Lab Nametre, and the Lab Brookfield) operated with the same principles and specifications of their on-line counterparts. The Haake Rotovisco RV12 Rotational Viscometer is a standard concentric cylindrical rheometer. In the laboratory, two types of units are employed: One is the open cup viscometer for use at low temperature for high viscosity samples, and the other is a pressure cell that was modified unit for use at high temperature and high pressure.

The viscosity data for each laboratory viscometer is gathered as a function of temperature and fitted to the form

$$\eta(cP) = \exp\left(a + \frac{b}{T(K)} + \frac{c}{T(K)^2}\right)$$
(2.1)

where a, b, and c are the correlated parameters (Zaman, 1993; Zaman and Fricke, 1994<u>a</u>). Afterwards, the smoothed correlations from at least three viscometers are compared with one another over the operational range to provide a separate and independent verification of the data, and to determine the upper and lower ranges of the laboratory reference. Generally, these correlations are within 5% of one another, which is the normally experienced agreement between instruments in the laboratory. Even though the black liquor samples were expected to be Newtonian at the solids content and viscosity ranges that were being tested, the viscosity was determined as a function of shear rate to provide verification.

Similar procedures are utilized in developing correlations of density and heat capacity as functions of temperature, as well as boiling point rise as a function of the solids content of each black liquor type. Density correlations of black liquors as a function of temperature are determined using the AccuPyc 1330 Pycnometer to measure the density of the sample at a fixed temperature and dilatometer to measure thermal expansion of the black liquor sample. The density correlation have the form of a second order polynomial

$$\rho(cP) = a + bT(^{\circ}C) + cT^{2}(^{\circ}C)$$
(2.2)

where a, b, and c are calibration constants (Fricke, 1987; Fricke, 1990).

2.4 Evaluation of On-Line Prototype Viscometers

In evaluating the experimental results from the on-line prototype viscometers, the viscosity measurements recorded on-line at the flow loop were compared with the laboratory reference viscosity measurements. The laboratory reference viscosities were calculated using the corresponding Viscometer Line temperature that was recorded on-line. Table 2.4 show the specific temperature sensors that were used to determine the

temperature of each on-line viscometer during the on-line experiments. The locations for each temperature sensor is given in Figure 9.1.

On-line Viscometer	Temperature Sensor
Brookfield	TT7
Micro Motion	TT7 & Internal
Nametre	TT4 & Internal
SWRI/OM	TT7
Lab Cambridge	Internal

Table 2.4. Temperature sensor used for laboratory reference for each instrument.

The Micro Motion, Nametre, and the laboratory version of the Cambridge possess internal temperature sensors, as shown in the Table 2.4. The Micro Motion internal temperature sensor is located on the outer casing and not in the flowing fluid. This reading is not accurate for the transient temperature run, but is suitable for steady sate analysis. Near the end of the instrument evaluations program the Nametre internal RTD failed and the external Viscometer Line RTD TT4 was used due to its proximity to the instrument.

It is important to remember that the laboratory reference for a liquor is a function of temperature only since the liquor is Newtonian at a constant solid concentration. In practically all the runs carried out, the viscosity versus flow rate plots show a downward trend of viscosity as the flow rate increases; however, this is not due to increases in shear stress but to increasing temperatures. As the flow rate increases, the net loss of heat in the viscometer line decreases and consequently the temperature in the viscometers increases. The approximate constant temperature attained in any given run can vary by as much as $\pm 5^{\circ}$ C. This was especially true for the high temperature runs in which the heater is employed, since maintaining a constant temperature with the fluctuations of black liquor flow rate and steam flow was very difficult.

For the transient temperature response runs, the flow rate is maintained at a low flow rate (~0.75 gpm) by manually adjusting the Sample Line Flow Rate Controller FC1

and the Motor Speed Controller. The Process Heater Controller TC2 is manually opened in small increments to introduce a temperature step change in the Viscometer Line utilizing the Heater E1. The flow rate is kept constant as best as possible though there are some shifts in the flow rate as the temperature increases. Once the temperature in the Viscometer Line reaches steady state, another step change is introduced by manually adjusting the Process Heater Controller Output. The Viscometer Line RTDs are assumed to instantaneously measure changes in the Viscometer Line during the transient temperature response runs. The time delays of the instruments are calculated from the moment the nearest RTD responses to the Viscometer Line temperature change.

3 BROOKFIELD COAXIAL ROTATIONAL VISCOMETER

The Brookfield Prototype Viscometer is an On-Line Coaxial Cylinder Rotational Viscometer which is designed to operate in a fully-flooded process stream under pressure or vacuum. The specific configuration tested is as follows:

- Sensor: TT100SD Rotational Viscometer
- Control Unit: 64-C0795 Rev A

The basic idea of the design is the use of a rotating-cup configuration. The process stream flows into a sample chamber where it contacts a measuring annulus comprised of a rotor and a stator. The laminar viscous drag of the process fluid generated by the constant rotation of the rotor is resisted by the stator (Brookfield, 1992; Brookfield, 1995, Zaman, 1993). This drag is transmitted as an angular deflection to the rotary transformer from a flexure element and torque tube. This signal is then processed into a 4-20 mA current which is proportional to the viscosity range. The control unit outputs the viscosity as a 4-20 mA linear signal which is proportional to the rotaries to the rotaries of the rotor.

3.1 Principles of Operation

The Brookfield Viscometer utilizes a direct measure of force for a fixed rate of shear. For Newtonian fluids the dynamic viscosity of the process fluid is related to the shear stress and shear rate as follows:

$$\eta = \frac{\tau}{\dot{\gamma}} \tag{3.1}$$

where

.

$$\tau = \frac{T}{2\pi R_i^2 L} \tag{3.2}$$

$$\dot{\gamma} = \frac{2\omega}{1 - \left(\frac{R_i}{R_o}\right)^2} \tag{3.3}$$

where

where

 τ = shear stress at the wall $\dot{\gamma}$ = shear rate T = torque R_i = radius of the rotor R_o = radius of the stator ω = velocity of the rotor η = dynamic viscosity

The Brookfield Viscometer is calibrated with a torsion bar which sets the calibration constant for the range of viscosity of interest and at a fixed rotational speed. The instrument tested had a dual-range capability for improved response in the low viscosity range. To determine viscosity with the current control unit, first the rotational speed would need to be calculated from a calibration correlation of rotational speed as a function of motor voltage. Then the viscosity range can then be calculated at that a fixed rotational speed (usually at 300 rpm) using the torsion calibration constant. The viscosity output at 300 rpm is calculated by (Brookfield, 1995)

$$\eta(@300rpm) = 1.1 \cdot \frac{100}{300} \cdot \frac{C}{x-4} \cdot 100$$

$$C = \text{torsion bar constant}$$

$$x = \text{Viscosity output in mA}$$
(3.4)

The 4-20 mA response from the control unit is linearly proportional to the calculated viscosity range, which is calculated by

$$0 \leftrightarrow \frac{300}{R} \cdot \eta \ (@ \ 300 \ \text{rpm}) \tag{3.5}$$

where R is the rotational speed measured in rpm.

3.2 Experimental Overview

Table 3.1 following lists solids compositions of the black liquor used with the experimental runs that were performed on the Brookfield Viscometer. In total, the instrument was tested on 13 different black liquor concentrations, ranging from a 42.20% Georgia Pacific liquor to a 73.40% liquor obtained by mixing a Georgia Pacific liquor with Canadian Forest liquor.

Georgia Pacific	Canadian Forest	Chesapeake	GP & Canadian Forest Mix
42.24% 48.29% 55.84% 58.38% 65.51% 68.38%	47.96% 57.73%	49.39% 59.58%	67.72% 71.38% 73.40%

Table 3.1. Black liquor types and solids content tested with the Brookfield Viscometer.

In addition to temperature and flow rate, the rotational speed of the viscometer was another variable investigated during the experimental trials for this instrument. For each flow rate, three different rotational speeds were normally run, although speeds were sometimes limited by the viscosity range of the instrument, especially at high viscosities. Due to the drift in the rotational speed voltage, very low rotational speeds may introduce large offsets; therefore, for a voltage range of 0-30 Vac, the minimum rotational speed examined was 50 rpm (or 5.4 Vac), with an 9:1 gear ratio. At low range, this viscometer has a viscosity range of 0-15.57 cP in the low-range setting, and 0-70.23 cP in the high-range setting. For the very high viscosity runs, the instrument was modified by changing the gear ratio to 18:1, which allowed lower rotational speeds (18 rpm) at a higher voltage setting.

The control unit has two outputs, namely a torque and a rotational speed signal. The torque signal is sent to the UF Data Acquisition System (DAQ). The rotational speed voltage is not sent to the UF DAQ System, but is monitored off-line by an attached multimeter. Because the Brookfield Control Unit available does not have any displays, to insure that the viscosity signal was being retransmitted properly, a multimeter was placed in series with the output line to measure the signal current. The viscosity is calculated using the relationship (Brookfield, 1995)

$$\eta = \frac{300}{R} \frac{\eta(@300rpm)}{16} (x-4)$$
(3.6)

With the present control unit, rotational speed transients (which are important for non-Newtonian fluid viscosity measurements) are difficult to coordinate with inputting the proper *rpm* value into the UF DAQ System and simultaneously adjusting the rotational speed on the control unit simultaneously.

3.3 Selected Experimental Results

The selected data presented in this section represent typical results for the Brookfield instrument. Extensive data are given in Section 3.5. The data presented is comprised of 4 steady-state runs of viscosity versus flow rate at different temperatures and solids compositions, and 2 transient response of the instrument to temperature changes.

Figure 3.1 shows a low viscosity run at 300 rpm with Georgia Pacific 58.38% solids black liquor at approximately 125°C. This is a steady-state run, and the results are presented on two plots. The upper plot shows the viscosity values measured by the instrument, and are indicated by the diamond-shaped markers. The continuous line joining the markers is included as a visual aid to track the data trend, and is obtained through arbitrary curve-fits. The upper plot also shows a laboratory reference band of viscosity values defined by two dash-dot lines. The band is constructed using viscosity correlations produced for the various laboratory instruments described in Section 2.4. In particular, the upper curve of the band corresponds to the highest viscosity prediction produced when

considering all the laboratory correlations at the temperature of the flow rate in question. Analogously, the lower curve of the band corresponds to the lowest viscosity prediction. Any measurements that fall inside the reference band are considered to be consistent with the laboratory measurements.

The lower plot in Figure 3.1 is a measure of the relative difference between the viscosity measurement (η) produced by the instrument and the viscosity (η_{ref}) predicted by a correlation produced using data acquired with a reliable laboratory instrument and valid for the particular solids-content of the black liquor used in the test. Hence, the relative difference is defined as follows:

Relative difference =
$$\frac{\eta - \eta_{ref}}{\eta_{ref}}$$
 (3.7)

We typically used a Haake Rotovisco RV12 Rotational Viscometer or a Cambridge Sliding Element Laboratory Viscometer as the reference laboratory instrument. Note that, for example, a relative difference of 0.08 indicates that the instrument viscosity differs from the reference measurement by 8%. We consider an instrument to be accurate if it realizes a relative difference of $\pm 10\%$. A relevant remark concerning this figure is that the temperature is not strictly constant during the run; in fact, as the flow rate varies, the temperature also varies, though it remains close to the approximate value reported in the figure title. For this particular figure the viscosity, flow-rate, and temperature information for 10 selected flow rates is as follows:

Flow Rate (gpm)	Temperature (°C)	Viscosity (cP)
0.88	118.45	13.99
0.80	118.17	14.18 m
0.81	118.08	14.26
0.79	118.08	14.25
0.74	119.56	14.75
1.06	118.82	14.78
1.05	117.89	14.76
0.98	117.62	15.02
0.00	108.46	20.2
0.00	108.36	20.4

Table 3.2. Flow-rate, temperature, and viscosity data for Figure 3.1.

Analysis of Figure 3.1 shows that the Brookfield Viscometer produces measurements with excellent accuracy because the measured response from the instrument lie within the boundary of the laboratory reference band. Note that the instrument tracks well with the changes in viscosity that occur due to the change of the temperature caused with the variation of flow rates. The lower plot was generated using the laboratory reference correlation

$$\log \eta_{ref}(cP) = 24.6362 - 20322.9/T(K) + 4594277/T(K)^2$$
(3.8)

The instrument tracked within the laboratory reference bounds with a relative difference of approximately 10% throughout the volumetric flow rate range investigated, the maximum flow rate for this run was 1.15 gpm.

Figure 3.2 is a mid-range viscosity run at 50 rpm with Georgia Pacific 65.51% solids black liquor at approximately 95°C. The instrument again tracked within the laboratory reference bounds with a $\pm 5\%$ relative difference from the laboratory results. The maximum flow rate for this run was increased to almost 2.5 gpm. The instrument started to demonstrate a slight downward trend in the viscosity measurement at the higher flow rates.

Figure 3.3 is a high-viscosity run at 50 rpm with Georgia Pacific 65.51% solids black liquor at approximately 90°C. The instrument tracked within $\pm 10\%$ of the laboratory

reference. At the upper end of the flow rate range, about 2.0 gpm, the instrument began to demonstrate a slight downward trend in the viscosity measurement. This is represented by the -10% relative difference at the high end of the flow rate.

Figure 3.4 is a high viscosity run at 100 rpm with Georgia Pacific 68.38% solids black liquor at approximately 95°C. In this case, the instrument tracked the laboratory reference bounds between 0 to -10% relative difference with a slight downward trend in viscosity as flow rate increases.

Figure 3.5 (A) plots viscosity as a function of time to examine the transient response of the Brookfield Viscometer with a GP and Canadian Forest Mix 67.46% black liquor. In this experiment the temperature of the flowing black liquor steam was changed in a step-fashion by manually opening the steam valve controlled by the Process Heater Output Controller (instrument TC2 in the P&ID diagram in Figure 9.1). The step changes were made in discrete increments, as shown in the legend of the figure, until the output from the Heater became saturated. The four markers (circle, square, triangle, and cross) on the figure denote the instants when the valve opening is changed. The dotted line represents the viscosity determined using a laboratory correlation of viscosity versus temperature, and the continuous line represents the measurement by the instrument. Further details on the execution of this may be found in Section 2.4. Initially, the viscosity of the black liquor was above the maximum torque for this instrument and a response was not given until the viscosity dropped to 400 cP at ~20 minutes. At the second step change (TC2 was opened 20%) there was a small delay in the temperature response from Viscometer Line RTD TT7 (cf. P&ID diagram in Figure 9.1). After the temperature step change is picked up at TT7, there was a short time delay from the measured response from the Brookfield instrument. By comparing the slopes from the viscosity transition between steady-states after the second step input, there appears to be negligible dynamics in the response or an insignificant time constant for this instrument. The transient response in the instrument is what is important in this plot, not the offset in the instrument to the absolute value, because the instrument could not be zeroed prior to the run.

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Figure 3.5 (B) plots viscosity as a function of time to examine the transient response of the Brookfield Viscometer with GP and Canadian Forest Mix 67.46% black liquor. Obtaining small transients while changing the rotational speed is an important feature if the Brookfield instrument is to be used for measuring the viscosity of non-Newtonian fluids. By this fashion, the effects of different shear rates on apparent viscosity can be examined in rapid succession without having to vary the flow rate. For the first step change, the base rotational speed was maintained at 50 rpm and then the rotation speed was shifted to 100 rpm and allowed to reach steady state. Once steady state was achieved, the rotational speed was shifted again. The time delays shown in Figure 3.5 B are not an accurate representation of the capabilities of this unit. The actual time delay is much shorter, and this is due to the cumbersome procedure followed to adjust the control unit. To change the rotational speed of the Brookfield with the current control unit requires that the operator first adjust the motor speed voltage to the proper value, then input the calibration factor into the UF DAQ System in the Pilot Plant. This accounts for the spikes in viscosity on the plot when the rotational speed is changed and for a large fraction of the delay.



Figure 3.1 Brookfield Viscometer at 300 rpm with Georgia Pacific 58.38% Solids Black Liquor at ~125°C.



Figure 3.2 Brookfield Viscometer at 50 rpm with Georgia Pacific 65.51% Solids Black Liquor at ~95°C.



Figure 3.3 Brookfield Viscometer at 50 rpm with Georgia Pacific 65.51% Solids Black Liquor at ~90°C.



Figure 3.4 Brookfield Viscometer at 100 rpm with Georgia Pacific 68.38% Solids Black Liquor at ~95°C.



Figure 3.5. Brookfield Viscometer transient temperature with GP and Canadian Forest Mix 67.46% Solids Black Liquor. (A) Step changes in temperature as a function of time. (B) Step changes in rotational speed.

3.4 Summary of Observations

The general trends observed with the Brookfield Viscometer experiments are summarized as follows:

- The instrument tracked within $\pm 10\%$ of the laboratory reference.
- There was no evidence of fouling during the trials.
- The instrument readings were unaffected by particles smaller than 40 mesh.
- The rotational-speed transition has a very small time delay; hence the instrument is adequate for use for non-Newtonian measurements.
- The instrument does not require laboratory calibration for operation.
- At higher flow rates, the instrument viscosity reading tended to be lower than the laboratory reference.
- The current control unit demonstrated an offset in both the torque and rotational speed outputs that need to be manually compensated for by the operator.

A decrease in viscosity when the flow rate is increased is expected. As the black liquor enters the measuring cell, the flow is diverted from the stator and torque tube by the spinning rotor. Under low flow conditions, the annulus has essentially static flow and the measurement is made at essentially drag flow conditions; however, as the flow rate increases, some flow develops across the stator which reduces the torque generated by the viscous drag of the spinning rotor. Due to the limitations of the viscometer line, it was not possible to evaluate the upper flow rate limits of the instrument. With flow rates less than 3 gpm, the flow effects are relatively minor and there are no problems anticipated with installing the instrument in a slip stream at a mill site.

With the current set up, the lower viscosity limit with the geometry and the 18:1 gear ratio used for this instrument is about 12-20 cP, and the upper limit, is approximately 600 cP. Increasing the bob length by nearly a factor of two will yield an instrument that
can measure viscosities of 3.5 cP with a $\pm 10\%$ accuracy, if an auto-zero is built into the control unit. With current mill recovery furnaces, it is unlikely that there will be situations in which viscosities above 400 cP will be exceeded.

During the trials there was no evidence of fouling or build up in the instrument. This conclusion tends to be supported by the fact that, on several occasions, the viscometer line was steam purged with 80 psig saturated steam with practically no change in the instruments performance. The measuring chamber in the viscometer was physically opened and inspected after long exposure to black liquor, and no significant build up was noticed.

Several electronic repairs were attempted to correct the offset drift in the torque and rotational speed signal with no success. The ground connections on the electronics unit, and the instrument were inspected regularly, and additional chassis grounds were also applied at the suggestion of Brookfield personnel; however, the offset drift remains. According to the Brookfield representatives, this problem has not been experienced with any other installation. The problem is not related to the data acquisition system used in the pilot plant because the problem persisted after isolating the control unit, and the system grounds were connected to several grounding rods and to the water pipes. The only possible non-Brookfield source of this offset drift may be from the line current, which is not isolated.

The original unit included a prototype single flush seal to reduce maintenance on the viscometer; however, the seal failed and the standard double-flush seal was installed. The double-flush seal requires a slight positive pressure on the side of the seal flush fluid. In the pilot flow loop, water under air pressure was used as the flush fluid. In mill installations, oil may be required as a seal fluid. The double-flush type of seal requires more maintenance than the single seal design.

The Brookfield Control Unit would benefit from a number of upgrades. With the current unit, the viscosity calculation is handled off-line by the UF DAQ System at a fixed

rotational speed. Not only would this situation not be practical in a mill environment, this control unit does not take advantage of the capability of this viscometer in handling non-Newtonian measurements. In order to improve the usefulness of the system, the output from the control unit should be based on viscosity, not torque, with the calculations handled on-line with digital displays of viscosity and rotational speed. In addition, an auto zeroing function for the viscosity and rotational speed signals would reduce errors caused by drifts and offsets.

The recommendations based on our experience with the Brookfield Viscometer are summarized as follows:

- The control unit needs to be upgraded.
- A temperature sensor should be added to the instrument for accurate measurement validation with a laboratory reference.
- The viscometer should be used only in a slip stream for flow rate control through the instrument and to protect the sensor from debris in the process line.

With proper sizing of the cylinder and the torque range, the instrument is capable of satisfactory operating over the expected ranges to be encountered in a mill.

3.5 Extensive Experimental Results

Included in this section are graphical representations of the results of the on-line viscometer trials performed with the Brookfield instrument. The figures describe the data gathered for all the black liquors listed in Table 3.1. Figures labeled as "d" represent the viscosity as measured by the instrument as a function of flow rate and the figures labeled as "b" show the relative difference of the measured response from the laboratory reference as a function of flow rate.

Figures 1-3 represent results for the Canadian Forest 47.96% Solids at 30°C for speeds of 100, 200, and 300 rpm. The relative difference is virtually constant with respect to flow rate, and is well within a difference of $\pm 10\%$ from the laboratory reference. Figures 4-12 present results in a similar fashion for the same liquor at various test temperatures up to 125°C. Note that for temperatures up to 75°C (viscosity of 9-11 cP), the instrument performed very well, with a relative difference within acceptable limits, and no apparent effects due to flow rate (up to 3 gpm), or rotor speed. The only problem observed was an occasional shift in the base signal output. Figures 10-12 are data for this liquor at the higher temperature of 125°C (viscosity of 3.5-2.5 cP), where the instrument was generally tracking the viscosity change; however, the error was too large and the problems with the zero offset were magnified. At 200 and 300 rpm (Figures 11 and 12), the instrument was tracking changes satisfactorily

Figures 13-21 represent similar results for Canadian Forest 57.73% Solids at temperatures between 50-125°C (viscosities of 20-280 cP), and at varying flow rates and rotor speeds. The instrument tracked viscosity acceptably; however, the accuracy was poor due to the ~1 Vac zero shifting offset. In addition, there was no obvious effect of flow rate on these results.

Figures 22-32 represent results for Chesapeake Paper 49.39% Solids at temperatures between 35-120°C (viscosities form 5-100 cP), and at varying flow rates and rotor speeds. This liquor was noticeably "dirtier" than the Canadian Forest or Georgia Pacific liquor. The performance at 35°C (Figures 22 and 23) is very good while performance at 50°C and 80°C (Figures 24-28) is acceptable; however, the effect in the zero shift is obvious at 300 rpm in the 80°C data (Figure 29). At 120°C (Figures 30-32), the viscosity is at the limit of the range for this configuration, there is questionable tracking of viscosity change, and the zero offset error is large, in general. There were no noticeable difficulties resulting from particulates in the liquor that passed a 40-mesh screen. The black liquor characteristics changed somewhat during these tests due to precipitation of solids or

association changes in the liquor. The liquor was recharacterized in the laboratory a number of times in order to account for any time-dependent variations.

Figures 33-41 represent results for Chesapeake Paper 59.29% Solids at temperatures from 65-120° (viscosities from 17 to 200 cP), and at varying flow rates and rotor speeds. Tracking of viscosity is good up to 90°C (Figures 33-35); however, at these temperatures there are problems with the zero shift. At the low viscosities (~120°C), performance was erratic at rotor speeds below 300 rpm (Figures 39 and 40) and the performance was marginal at 300 rpm (Figure 41), with the difference probably due primarily to the zero shift.

Figures 42-48 represent results for Georgia Pacific 42.20% Solids at temperatures from 35-85°C (viscosities from 4-20 cP) and at different flow rates and rotor speeds. Performance at 35°C was acceptable (Figure 45). While the tracking was acceptable at 60°C (Figures 42 and 43) and 85°C (Figure 46-48), there was a large offset. At 85°C, the viscosity measurement is at the lower range for this configuration.

Figures 49-56 represent results for Georgia Pacific 48.29% Solids at temperatures from 30-125°C (viscosities from 3-90 cP), and at different flow rates and rotor speeds. The results at 30-85°C (Figures 49-54) are generally acceptable for tracking and reproducibility, but the offset differences are large. During this series, the single-flush seal on the instrument failed and was replaced. At the same time, the instrument electronics were replaced and the sensor was recalibrated.

Figures 57-62 represent results for Georgia Pacific 55.84% Solids at temperatures from 55-95°C (viscosities from 35-200 cP), and at different flow rates and rotor speeds. At 55°C and 75°C, the instrument tracked well with marginally acceptable differences (Figures 57-59). There were erratic static readings at 75°C and 100 rpm (Figure 58) which were more than likely caused by the zero voltage drift. At 95°C (Figure 60), the performance was poor at 100 rpm. There were problems with the data acquisition system in the pilot plant during this time, which could explain the erratic readings.

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Figures 63-71 represent results for Georgia Pacific 58.38% Solids at temperatures from 60-125°C (viscosities from 10-230 cP) and at different flow rates and rotor speeds. Data taken up to 85°C were excellent (Figures 63-68) except for some drift at zero flow rate in Figure 65. There was very good tracking, accuracy was acceptable, and there was no noticeable effect of flow rate. This was also true for data at 125°C for rotor speeds above 100 rpm (Figures 69-71).

Figures 72-79 represent results for Georgia Pacific 65.51% Solids at temperatures from 95-125°C (viscosities from 30-310 cP), and at different flow rates and rotor speeds. At 85°C to 95°C (Figures 72-76), the instrument tracked well, with what perhaps may be a small effect of flow rate on the relative difference. At 125°C, there was a "glitch" during the 100 rpm and 200 rpm runs. This may be due to problems with the data acquisition system in the pilot plant which were experienced with some frequency during these runs.

Figures 80-89 represent results for Georgia Pacific 68.38% Solids at temperatures from 85-125°C (viscosities from 30-340 cP), and at different flow rates and rotor speeds. Data at the 85°C and 95°C (Figures 80-82) were restricted due to the limits on the torque. In general, the instrument tracked viscosity change with an offset, and comparison of results at 50 rpm (Figure 81) and 100 rpm (Figure 82) would indicate this is only a scaling problem for the torque. Tracking at 110°C (Figures 83-85) was perfect and relative differences were small at all rotor speeds and flow rates. Tracking was good at 125°C (Figures 86 and 87) with an offset. At 300 rpm (Figure 88), tracking and accuracy were good. This liquor contained particulates and showed evidence of solids build up in the strainers. The Viscometer Line was drained and steam purged, and then runs at 125°C were repeated at 200 rpm. Tracking and accuracy were not affected (Figure 89). In a number of cases, the instrument was steam cleaned in a similar fashion with no noticeable change in performance.

Figures 90-93 represent results for Georgia Pacific and Canadian Forest Mix 67.46% Solids at temperatures from 110-140°C (viscosities from 100-500 cP), and at

different flow rates and rotor speeds. The data gathered represent both transient and steady state temperature runs with this instrument. During these trials, the instrument was operating at the maximum torque limit at 110°C. At 110°C, the instrument had excellent tracking and accuracy while operating at a very low rotational speed (Figure 91). At 140°C, there was a good amount of scatter in the data, probably due to the difficulty in maintaining thermal stability in the Viscometer Line (Figure 92). However, the instrument tracked reasonably well considering the thermal variations present. The offset is due in large part to operating at a very low rotational speed where the zero shift would be most evident.

Figures 94-98 represent results for Georgia Pacific and Canadian Forest Mix 71.38% Solids at temperatures from 110-140°C (viscosities from 100-700 cP), and at different flow rates and rotor speeds. To operate at lower rotation speeds, the gear reducers were changed from 9:1 to 18:1. The data gathered represent both transient and steady state temperature runs with this instrument. Operating at these viscosities was very difficult with severe problems with the zero shift. At 110°C, the reading were erratic due to the zero shift error (Figure 94). At 130°C and a repeat run at 110°C, tracking was much better as the zero shift was somewhat brought under control (Figures 95 and 96).

Figures 99-100 represent results for Georgia Pacific and Canadian Forest Mix 743.40% Solids at temperatures from 110-140°C (viscosities from 150-1000 cP), and at different flow rates and rotor speeds. The data gathered represent both transient and steady state temperature runs with this instrument. At 135°C, the instrument exhibited reasonable tracking with a significant offset (Figure 99). The offset was due to in part to the zero shift, but also to difficulties in characterizing this sample in the laboratory due to the high viscosity of the sample.

Viscometer Test Run 960130B1 Figure No. 1a

Brookfield (100 rpm) with Canadian Forest 47.96% Solids at ~30°C



Viscometer Test Run 960130B1 Figure No. 1b

Brookfield (100 rpm) with Canadian Forest 47.96% Solids at ~30°C



Viscometer Test Run 960130B1 Figure No. 2a

Brookfield (200 rpm) with Canadian Forest 47.96% Solids at ~30°C



Viscometer Test Run 960130B1 Figure No. 2b

Brookfield (200 rpm) with Canadian Forest 47.96% Solids at ~30°C



Viscometer Test Run 960130B1 Figure No. 3a

Brookfield (300 rpm) with Canadian Forest 47.96% Solids at ~30°C



Viscometer Test Run 960130B1 Figure No. 3b

Brookfield (300 rpm) with Canadian Forest 47.96% Solids at ~30°C



Viscometer Test Run 960130B2 Figure No. 4a

Brookfield (100 rpm) with Canadian Forest 47.96% Solids at ~50°C



Viscometer Test Run 960130B2 Figure No. 4b

Brookfield (100 rpm) with Canadian Forest 47.96% Solids at ~50°C



Viscometer Test Run 960130B2 Figure No. 5a

Brookfield (200 rpm) with Canadian Forest 47.96% Solids at ~50°C



Viscometer Test Run 960130B2 Figure No. 5b

Brookfield (200 rpm) with Canadian Forest 47.96% Solids at ~50°C



Viscometer Test Run 960130B2 Figure No. 6a

Brookfield (300 rpm) with Canadian Forest 47.96% Solids at ~50°C



Viscometer Test Run 960130B2 Figure No. 6b

Brookfield (300 rpm) with Canadian Forest 47.96% Solids at ~50°C



Viscometer Test Run 960131B3 Figure No. 7a

Brookfield (100 rpm) with Canadian Forest 47.96% Solids at ~75°C



Viscometer Test Run 960131B3 Figure No. 7b

Brookfield (100 rpm) with Canadian Forest 47.96% Solids at ~75°C

;



Viscometer Test Run 960131B3 Figure No. 8a

Brookfield (200 rpm) with Canadian Forest 47.96% Solids at ~75°C



Viscometer Test Run 960131B3 Figure No. 8b

Brookfield (200 rpm) with Canadian Forest 47.96% Solids at ~75°C



Viscometer Test Run 960131B3 Figure No. 9a

Brookfield (300 rpm) with Canadian Forest 47.96% Solids at ~75°C



Viscometer Test Run 960131B3 Figure No. 9b

Brookfield (300 rpm) with Canadian Forest 47.96% Solids at ~75°C



Viscometer Test Run 960131B4 Figure No. 10a

Brookfield (100 rpm) with Canadian Forest 47.96% Solids at ~125°C



Viscometer Test Run 960131B4 Figure No. 10b

Brookfield (100 rpm) with Canadian Forest 47.96% Solids at ~125°C



Viscometer Test Run 960131B4 Figure No. 11a

Brookfield (200 rpm) with Canadian Forest 47.96% Solids at ~125°C



Viscometer Test Run 960131B4 Figure No. 11b

Brookfield (200 rpm) with Canadian Forest 47.96% Solids at ~125°C



Viscometer Test Run 960131B4 Figure No. 12a

Brookfield (300 rpm) with Canadian Forest 47.96% Solids at ~125°C



Viscometer Test Run 960131B4 Figure No. 12b

Brookfield (300 rpm) with Canadian Forest 47.96% Solids at ~125°C



Viscometer Test Run 960409B1 Figure No. 13a

Brookfield (50 rpm) with Canadian Forest 57.73% Solids at ~50°C



Viscometer Test Run 960409B1 Figure No. 13b

Brookfield (50 rpm) with Canadian Forest 57.73% Solids at ~50°C



Viscometer Test Run 960409B2 Figure No. 14a

Brookfield (50 rpm) with Canadian Forest 57.73% Solids at ~55°C



Viscometer Test Run 960409B2 Figure No. 14b

Brookfield (50 rpm) with Canadian Forest 57.73% Solids at ~55°C



Viscometer Test Run 960410B3 Figure No. 15a

Brookfield (100 rpm) with Canadian Forest 57.73% Solids at ~80°C



Viscometer Test Run 960410B3 Figure No. 15b

Brookfield (100 rpm) with Canadian Forest 57.73% Solids at ~80°C


Viscometer Test Run 960409B3 Figure No. 16a

Brookfield (200 rpm) with Canadian Forest 57.73% Solids at ~80°C



Viscometer Test Run 960409B3 Figure No. 16b

Brookfield (200 rpm) with Canadian Forest 57.73% Solids at ~80°C



Viscometer Test Run 960410B3 Figure No. 17a

Brookfield (300 rpm) with Canadian Forest 57.73% Solids at ~80°C



Viscometer Test Run 960410B3 Figure No. 17b

Brookfield (300 rpm) with Canadian Forest 57.73% Solids at ~80°C



Viscometer Test Run 960410B4 Figure No. 18a

Brookfield (100 rpm) with Canadian Forest 57.73% Solids at ~125°C



Viscometer Test Run 960410B4 Figure No. 18b

Brookfield (100 rpm) with Canadian Forest 57.73% Solids at ~125°C



Viscometer Test Run 960410B4 Figure No. 19a

Brookfield (200 rpm) with Canadian Forest 57.73% Solids at ~125°C



Viscometer Test Run 960410B4 Figure No. 19b

Brookfield (200 rpm) with Canadian Forest 57.73% Solids at ~125°C



Viscometer Test Run 960410B4 Figure No. 20a

Brookfield (300 rpm) with Canadian Forest 57.73% Solids at ~125°C



Viscometer Test Run 960410B4 Figure No. 20b

Brookfield (300 rpm) with Canadian Forest 57.73% Solids at ~125°C



Viscometer Test Run 960412B5 Figure No. 21a

Brookfield (200 rpm) with Canadian Forest 57.73% Solids at ~80°C



Viscometer Test Run 960412B5 Figure No. 21b

Brookfield (200 rpm) with Canadian Forest 57.73% Solids at ~80°C



Viscometer Test Run 960207B1 Figure No. 22a

Brookfield (100 rpm) with Chesapeake Paper 49.39% Solids at ~35°C



Viscometer Test Run 960207B1 Figure No. 22b

Brookfield (100 rpm) with Chesapeake Paper 49.39% Solids at ~35°C



Viscometer Test Run 960207B1 Figure No. 23a

Brookfield (200 rpm) with Chesapeake Paper 49.39% Solids at ~35°C



Viscometer Test Run 960207B1 Figure No. 23b

Brookfield (200 rpm) with Chesapeake Paper 49.39% Solids at ~35°C



Viscometer Test Run 960207B2 Figure No. 24a

Brookfield (100 rpm) with Chesapeake Paper 49.39% Solids at ~50°C



Viscometer Test Run 960207B2 Figure No. 24b

Brookfield (100 rpm) with Chesapeake Paper 49.39% Solids at ~50°C



Viscometer Test Run 960207B2 Figure No. 25a

Brookfield (200 rpm) with Chesapeake Paper 49.39% Solids at ~50°C



Viscometer Test Run 960207B2 Figure No. 25b

Brookfield (200 rpm) with Chesapeake Paper 49.39% Solids at ~50°C



Viscometer Test Run 960207B2 Figure No. 26a

Brookfield (300 rpm) with Chesapeake Paper 49.39% Solids at ~50°C



Viscometer Test Run 960207B2 Figure No. 26b

Brookfield (300 rpm) with Chesapeake Paper 49.39% Solids at ~50°C



Viscometer Test Run 960208B3 Figure No. 27a

Brookfield (100 rpm) with Chesapeake Paper 49.39% Solids at ~80°C



Viscometer Test Run 960208B3 Figure No. 27b

Brookfield (100 rpm) with Chesapeake Paper 49.39% Solids at ~80°C



Viscometer Test Run 960208B3 Figure No. 28a

Brookfield (200 rpm) with Chesapeake Paper 49.39% Solids at ~80°C



Viscometer Test Run 960208B3 Figure No. 28b

Brookfield (200 rpm) with Chesapeake Paper 49.39% Solids at ~80°C



Viscometer Test Run 960208B3 Figure No. 29a

Brookfield (300 rpm) with Chesapeake Paper 49.39% Solids at ~80°C



Viscometer Test Run 960208B3 Figure No. 29b

Brookfield (300 rpm) with Chesapeake Paper 49.39% Solids at ~80°C



Viscometer Test Run 960208B4 Figure No. 30a

Brookfield (100 rpm) with Chesapeake Paper 49.39% Solids at ~120°C



Viscometer Test Run 960208B4 Figure No. 30b

Brookfield (100 rpm) with Chesapeake Paper 49.39% Solids at ~120°C



Viscometer Test Run 960208B4 Figure No. 31a

Brookfield (200 rpm) with Chesapeake Paper 49.39% Solids at ~120°C



Viscometer Test Run 960208B4 Figure No. 31b

Brookfield (200 rpm) with Chesapeake Paper 49.39% Solids at ${\sim}120^{\circ}\text{C}$



Viscometer Test Run 960208B4 Figure No. 32a

Brookfield (300 rpm) with Chesapeake Paper 49.39% Solids at ~120°C



Viscometer Test Run 960208B4 Figure No. 32b

Brookfield (300 rpm) with Chesapeake Paper 49.39% Solids at ~120°C



Viscometer Test Run 960217B1 Figure No. 33a

Brookfield (50 rpm) with Chesapeake Paper 59.28% Solids at ~65°C



Viscometer Test Run 960217B1 Figure No. 33b

Brookfield (50 rpm) with Chesapeake Paper 59.28% Solids at ~65°C


Viscometer Test Run 960217B2 Figure No. 34a

> Brookfield (100 rpm) with Chesapeake Paper 59.28% Solids at ~80°C



Viscometer Test Run 960217B2 Figure No. 34b

> Brookfield (100 rpm) with Chesapeake Paper 59.28% Solids at ~80°C



Viscometer Test Run 960217B2 Figure No. 35a

> Brookfield (200 rpm) with Chesapeake Paper 59.28% Solids at ~80°C



Viscometer Test Run 960217B2 Figure No. 35b

Brookfield (200 rpm) with Chesapeake Paper 59.28% Solids at ~80°C



Viscometer Test Run 960208B4 Figure No. 31b

Brookfield (200 rpm) with Chesapeake Paper 49.39% Solids at ~120°C



Viscometer Test Run 960219B3 Figure No. 36a

Brookfield (100 rpm) with Chesapeake Paper 59.28% Solids at ~90°C



Viscometer Test Run 960219B3 Figure No. 36b

> Brookfield (100 rpm) with Chesapeake Paper 59.28% Solids at ~90°C



Viscometer Test Run 960219B3 Figure No. 37a

Brookfield (200 rpm) with Chesapeake Paper 59.28% Solids at ~90°C



Viscometer Test Run 960219B3 Figure No. 37b

Brookfield (200 rpm) with Chesapeake Paper 59.28% Solids at ~90°C



Viscometer Test Run 960219B3 Figure No. 38a

Brookfield (300 rpm) with Chesapeake Paper 59.28% Solids at ~90°C



Viscometer Test Run 960219B3 Figure No. 38b

Brookfield (300 rpm) with Chesapeake Paper 59.28% Solids at ~90°C



Viscometer Test Run 960220B4 Figure No. 39a

Brookfield (100 rpm) with Chesapeake Paper 59.28% Solids at ~120°C



Viscometer Test Run 960220B4 Figure No. 39b

Brookfield (100 rpm) with Chesapeake Paper 59.28% Solids at ~120°C



Viscometer Test Run 960220B4 Figure No. 40a

Brookfield (200 rpm) with Chesapeake Paper 59.28% Solids at ~120°C



Viscometer Test Run 960220B4 Figure No. 40b

Brookfield (200 rpm) with Chesapeake Paper 59.28% Solids at ~120°C



Viscometer Test Run 960220B4 Figure No. 41a

Brookfield (300 rpm) with Chesapeake Paper 59.28% Solids at ~120°C



Viscometer Test Run 960220B4 Figure No. 41b

Brookfield (300 rpm) with Chesapeake Paper 59.28% Solids at ~120°C



Viscometer Test Run 950912B1 Figure No. 42a Brookfield (200 rpm) with Georgia Pacific 42.20% Solids at ~60°C



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Viscometer Test Run 950912B1 Figure No. 42b

Brookfield (200 rpm) with Georgia Pacific 42.20% Solids at ~60°C



Viscometer Test Run 950912B2 Figure No. 43a

Brookfield (200 rpm) with Georgia Pacific 42.20% Solids at ~60°C



Viscometer Test Run 950912B2 Figure No. 43b

Brookfield (200 rpm) with Georgia Pacific 42.20% Solids at ~60°C



Viscometer Test Run 950912B3 Figure No. 44a

Brookfield (100 rpm) with Georgia Pacific 42.20% Solids at ~60°C



Viscometer Test Run 950912B3 Figure No. 44b

Brookfield (100 rpm) with Georgia Pacific 42.20% Solids at ~60°C



Viscometer Test Run 951102B1 Figure No. 45a

Brookfield (100 rpm) with Georgia Pacific 42.20% Solids at ~35°C



Viscometer Test Run 951102B1 Figure No. 45b

Brookfield (100 rpm) with Georgia Pacific 42.20% Solids at ~35°C



Viscometer Test Run 951116B4 Figure No. 46a

Brookfield (100 rpm) with Georgia Pacific 42.20% Solids at ~85°C



Viscometer Test Run 951116B4 Figure No. 46b

Brookfield (100 rpm) with Georgia Pacific 42.20% Solids at ~85°C



Viscometer Test Run 951116B4 Figure No. 47a

Brookfield (200 rpm) with Georgia Pacific 42.20% Solids at ~85°C



Viscometer Test Run 951116B4 Figure No. 47b

Brookfield (200 rpm) with Georgia Pacific 42.20% Solids at ~85°C



Viscometer Test Run 951116B4 Figure No. 48a

Brookfield (300 rpm) with Georgia Pacific 42.20% Solids at ~85°C



Viscometer Test Run 951116B4 Figure No. 48b

Brookfield (300 rpm) with Georgia Pacific 42.20% Solids at ~85°C



Viscometer Test Run 960113B1 Figure No. 49a

Brookfield (100 rpm) with Georgia Pacific 48.29% Solids at ~30°C



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Viscometer Test Run 960113B1 Figure No. 49b

Brookfield (100 rpm) with Georgia Pacific 48.29% Solids at ~30°C



Viscometer Test Run 960113B1 Figure No. 50a

Brookfield (200 rpm) with Georgia Pacific 48.29% Solids at ~30°C



Viscometer Test Run 960113B1 Figure No. 50b

Brookfield (200 rpm) with Georgia Pacific 48.29% Solids at ~30°C



Viscometer Test Run 960113B2 Figure No. 51a

Brookfield (100 rpm) with Georgia Pacific 48.29% Solids at ~60°C


Viscometer Test Run 960113B2 Figure No. 51b

Brookfield (100 rpm) with Georgia Pacific 48.29% Solids at ~60°C



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Viscometer Test Run 960113B2 Figure No. 52a

Brookfield (200 rpm) with Georgia Pacific 48.29% Solids at ~60°C



Viscometer Test Run 960113B2 Figure No. 52b

Brookfield (200 rpm) with Georgia Pacific 48.29% Solids at ~60°C



Viscometer Test Run 960113B3 Figure No. 53a

Brookfield (100 rpm) with Georgia Pacific 48.29% Solids at ~85°C



Viscometer Test Run 960113B3 Figure No. 53b

Brookfield (100 rpm) with Georgia Pacific 48.29% Solids at ~85°C



Viscometer Test Run 960113B3 Figure No. 54a

Brookfield (200 rpm) with Georgia Pacific 48.29% Solids at ~85°C



Viscometer Test Run 960113B3 Figure No. 54b

Brookfield (200 rpm) with Georgia Pacific 48.29% Solids at ~85°C



Viscometer Test Run 960114B4 Figure No. 55a

Brookfield (100 rpm) with Georgia Pacific 48.29% Solids at ~125°C



Viscometer Test Run 960114B4 Figure No. 55b

Brookfield (100 rpm) with Georgia Pacific 48.29% Solids at ~125°C



Viscometer Test Run 960114B4 Figure No. 56a

Brookfield (200 rpm) with Georgia Pacific 48.29% Solids at ~125°C



Viscometer Test Run 960114B4 Figure No. 56b

Brookfield (200 rpm) with Georgia Pacific 48.29% Solids at ~125°C



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Viscometer Test Run 960503B1 Figure No. 57a

Brookfield (50 rpm) with Georgia Pacific 55.84% at ~55°C



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Viscometer Test Run 960503B1 Figure No. 57b

Brookfield (50 rpm) with Georgia Pacific 55.84% at ~55°C



Viscometer Test Run 960503B2 Figure No. 58a

Brookfield (100 rpm) with Georgia Pacific 55.84% at ~75°C



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Viscometer Test Run 960503B2 Figure No. 58b

Brookfield (100 rpm) with Georgia Pacific 55.84% at ~75°C



Viscometer Test Run 960503B2 Figure No. 59a

Brookfield (200 rpm) with Georgia Pacific 55.84% at ~75°C

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Viscometer Test Run 960503B2 Figure No. 59b

Brookfield (200 rpm) with Georgia Pacific 55.84% at ~75°C



Viscometer Test Run 960504B3 Figure No. 60a

Brookfield (100 rpm) with Georgia Pacific 55.84% at ~95°C



Viscometer Test Run 960504B3 Figure No. 60b

Brookfield (100 rpm) with Georgia Pacific 55.84% at ~95°C



Viscometer Test Run 960504B3 Figure No. 61a

Brookfield (200 rpm) with Georgia Pacific 55.84% at ~95°C



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Viscometer Test Run 960504B3 Figure No. 61b

Brookfield (200 rpm) with Georgia Pacific 55.84% at ~95°C



Viscometer Test Run 960504B3 Figure No. 62a

Brookfield (300 rpm) with Georgia Pacific 55.84% at ~95°C



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Viscometer Test Run 960504B3 Figure No. 62b

Brookfield (300 rpm) with Georgia Pacific 55.84% at ~95°C



Viscometer Test Run 960211B1 Figure No. 63a

Brookfield (50 rpm) with Georgia Pacific 58.38% Solids at ~60°C



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Viscometer Test Run 960211B1 Figure No. 63b

Brookfield (50 rpm) with Georgia Pacific 58.38% Solids at ~60°C



Viscometer Test Run 960213B2 Figure No. 64a

Brookfield (50 rpm) with Georgia Pacific 58.38% Solids at ~75°C



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Viscometer Test Run 960213B2 Figure No. 64b

Brookfield (50 rpm) with Georgia Pacific 58.38% Solids at ~75°C



Viscometer Test Run 960213B2 Figure No. 65a

Brookfield (100 rpm) with Georgia Pacific 58.38% Solids at ~75°C



Viscometer Test Run 960213B2 Figure No. 65b

Brookfield (100 rpm) with Georgia Pacific 58.38% Solids at ~75°C



Viscometer Test Run 960213B3 Figure No. 66a

Brookfield (100 rpm) with Georgia Pacific 58.38% Solids at ~85°C



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Viscometer Test Run 960213B3 Figure No. 66b

Brookfield (100 rpm) with Georgia Pacific 58.38% Solids at ~85°C



Viscometer Test Run 960213B3 Figure No. 67a

Brookfield (200 rpm) with Georgia Pacific 58.38% Solids at ~85°C



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Viscometer Test Run 960213B3 Figure No. 67b

Brookfield (200 rpm) with Georgia Pacific 58.38% Solids at ~85°C



Viscometer Test Run 960213B3 Figure No. 68a

Brookfield (300 rpm) with Georgia Pacific 58.38% Solids at ~85°C



Viscometer Test Run 960213B3 Figure No. 68b

Brookfield (300 rpm) with Georgia Pacific 58.38% Solids at ~85°C



Viscometer Test Run 960213B4 Figure No. 69a

Brookfield (100 rpm) with Georgia Pacific 58.38% Solids at ~125°C



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Viscometer Test Run 960213B4 Figure No. 69b

Brookfield (100 rpm) with Georgia Pacific 58.38% Solids at ~125°C



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Viscometer Test Run 960213B4 Figure No. 70a

Brookfield (200 rpm) with Georgia Pacific 58.38% Solids at ~125°C



Viscometer Test Run 960213B4 Figure No. 70b

Brookfield (200 rpm) with Georgia Pacific 58.38% Solids at ~125°C



Viscometer Test Run 960213B4 Figure No. 71a

Brookfield (300 rpm) with Georgia Pacific 58.38% Solids at ~125°C



Viscometer Test Run 960213B4 Figure No. 71b

Brookfield (300 rpm) with Georgia Pacific 58.38% Solids at ~125°C



Viscometer Test Run 960520B1 Figure No. 72a

Brookfield (50 rpm) with Georgia Pacific 65.51% Solids at ~85°C



Viscometer Test Run 960520B1 Figure No. 72b

Brookfield (50 rpm) with Georgia Pacific 65.51% Solids at ~85°C



Viscometer Test Run 960520B2 Figure No. 73a

Brookfield (50 rpm) with Georgia Pacific 65.51% Solids at ~90°C



Viscometer Test Run 960520B2 Figure No. 73b

Brookfield (50 rpm) with Georgia Pacific 65.51% Solids at ~90°C



Viscometer Test Run 960520B2 Figure No. 74a

Brookfield (100 rpm) with Georgia Pacific 65.51% Solids at ~90°C



Viscometer Test Run 960520B2 Figure No. 74b

Brookfield (100 rpm) with Georgia Pacific 65.51% Solids at ~90°C



Viscometer Test Run 960520B3 Figure No. 75a

Brookfield (50 rpm) with Georgia Pacific 65.51% Solids at ~95°C



Viscometer Test Run 960520B3 Figure No. 75b

Brookfield (50 rpm) with Georgia Pacific 65.51% Solids at ~95°C



Viscometer Test Run 960520B3 Figure No. 76a

Brookfield (100 rpm) with Georgia Pacific 65.51% Solids at ~95°C



Viscometer Test Run 960520B3 Figure No. 76b

Brookfield (100 rpm) with Georgia Pacific 65.51% Solids at ~95°C



Viscometer Test Run 960521B4 Figure No. 77a

Brookfield (100 rpm) with Georgia Pacific 65.51% Solids at ~125°C



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Viscometer Test Run 960521B4 Figure No. 77b Brookfield (100 rpm) with Georgia Pacific 65.51% Solids at ~125°C



Viscometer Test Run 960521B4 Figure No. 78a

Brookfield (200 rpm) with Georgia Pacific 65.51% Solids at ~125°C



Viscometer Test Run 960521B4 Figure No. 78b

Brookfield (200 rpm) with Georgia Pacific 65.51% Solids at ~125°C



Viscometer Test Run 960521B4 Figure No. 79a

Brookfield (300 rpm) with Georgia Pacific 65.51% Solids at ~125°C



Viscometer Test Run 960521B4 Figure No. 79b

Brookfield (300 rpm) with Georgia Pacific 65.51% Solids at ~125°C



Viscometer Test Run 960301B1 Figure No. 80a

Brookfield (50 rpm) with Georgia Pacific 68.38 % Solids at ~85°C



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Viscometer Test Run 960301B1 Figure No. 80b

Brookfield (50 rpm) with Georgia Pacific 68.38 % Solids at ~85°C



Viscometer Test Run 960302B2 Figure No. 81a

Brookfield (50 rpm) with Georgia Pacific 68.38 % Solids at ~95°C



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Viscometer Test Run 960302B2 Figure No. 81b

Brookfield (50 rpm) with Georgia Pacific 68.38 % Solids at ~95°C



Viscometer Test Run 960302B2 Figure No. 82a

Brookfield (100 rpm) with Georgia Pacific 68.38 % Solids at ~95°C



Viscometer Test Run 960302B2 Figure No. 82b

Brookfield (100 rpm) with Georgia Pacific 68.38 % Solids at ~95°C



Viscometer Test Run 960302B3 Figure No. 83a

Brookfield (100 rpm) with Georgia Pacific 68.38 % Solids at ~110°C



Viscometer Test Run 960302B3 Figure No. 83b

Brookfield (100 rpm) with Georgia Pacific 68.38 % Solids at ~110°C



Viscometer Test Run 960302B3 Figure No. 84a

Brookfield (200 rpm) with Georgia Pacific 68.38 % Solids at ~110°C



Viscometer Test Run 960302B3 Figure No. 84b

Brookfield (200 rpm) with Georgia Pacific 68.38 % Solids at ~110°C



Viscometer Test Run 960302B3 Figure No. 85a

Brookfield (300 rpm) with Georgia Pacific 68.38 % Solids at ~110°C



Viscometer Test Run 960302B3 Figure No. 85b

Brookfield (300 rpm) with Georgia Pacific 68.38 % Solids at ~110°C



Viscometer Test Run 960302B4 Figure No. 86a

Brookfield (100 rpm) with Georgia Pacific 68.38 % Solids at ~125°C



Viscometer Test Run 960302B4 Figure No. 86b

Brookfield (100 rpm) with Georgia Pacific 68.38 % Solids at ~125°C



Viscometer Test Run 960302B4 Figure No. 87a

Brookfield (200 rpm) with Georgia Pacific 68.38 % Solids at ~125°C



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Viscometer Test Run 960302B4 Figure No. 87b

Brookfield (200 rpm) with Georgia Pacific 68.38 % Solids at ~125°C



Viscometer Test Run 960302B4 Figure No. 88a

Brookfield (300 rpm) with Georgia Pacific 68.38 % Solids at ~125°C



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Viscometer Test Run 960302B4 Figure No. 88b

Brookfield (300 rpm) with Georgia Pacific 68.38 % Solids at ~125°C



Viscometer Test Run 960302B5 Figure No. 89a

> Brookfield (200 rpm) with Georgia Pacific 68.38 % Solids at ~125°C After Steam Purge



Viscometer Test Run 960302B5 Figure No. 89b

> Brookfield (200 rpm) with Georgia Pacific 68.38 % Solids at ~125°C After Steam Purge



Viscometer Test Run 960719B1 Figure No. 90

> Brookfield (50 rpm) Transient Temperature Run with GP & Canadian Forest Mix 67.46% Solids



Viscometer Test Run 960719B3 Figure No. 91a

Brookfield (50 rpm) with GP & Canadian Forest Mix 67.46% at ~110°C



Viscometer Test Run 960719B3 Figure No. 91b

Brookfield (50 rpm) with GP & Canadian Forest Mix 67.46% at ~110°C



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Viscometer Test Run 960720B4 Figure No. 92a

Brookfield (50 rpm) with GP & Canadian Forest Mix 67.46% at ~140°C



Viscometer Test Run 960720B4 Figure No. 92b

Brookfield (50 rpm) with GP & Canadian Forest Mix 67.46% at ~140°C



Viscometer Test Run 960720B5 Figure No. 93a



Brookfield (50 rpm) Transient Temperature Run with GP & Canadian Forest Mix 67.46% Solids

Viscometer Test Run 960720B5 Figure No. 93b

> Brookfield (50 rpm) Transient Temperature Run with GP & Canadian Forest Mix 67.46% Solids



Viscometer Test Run 960726B1 Figure No. 94a





Viscometer Test Run 960726B1 Figure No. 94b





Viscometer Test Run 960726B2 Figure No. 95a





Viscometer Test Run 960726B2 Figure No. 95b





Viscometer Test Run 960729B3 Figure No. 96a





Viscometer Test Run 960729B3 Figure No. 96b

> Brookfield (25 rpm) with GP & Canadian Forest Mix 71.38% Solids at ~110°C



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Viscometer Test Run 960730B4 Figure No. 97

> Brookfield (25 rpm) Transient Temperature Run with GP & Canadian Forest Mix 71.38% Solids at ~110°C



Viscometer Test Run 960730B5 Figure No. 98

> Brookfield (25 rpm) Transient Temperature Run with GP & Canadian Forest Mix 71.38% Solids at ~110°C



Viscometer Test Run 960801B1 Figure No. 99a





Viscometer Test Run 960801B1 Figure No. 99b

> Brookfield (50 rpm) with GP & Canadian Forest Mix 73.40% Solids at ~135°C



Viscometer Test Run 960801B2 Figure No. 100

> Brookfield (50 rpm) Transient Temperature Run with GP & Canadian Forest Mix 73.40% Solids



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4 MICRO MOTION CORIOLIS CAPILLARY VISCOMETER

The specific configuration of the Micro Motion Coriolis Capillary Viscometer tested is as follows.

•	Coriolis Mass Flow Sensor:	Micro Motion CMF025M
•	Differential Pressure Cell:	Rosemount 3051CD
•	Transmitter:	Micro Motion RFT9739
•	DAO Software:	Prolink version 2.2

4.1 Principles of Operation

The Micro Motion Viscometer is based on the Hagen-Poiseuille Equation for Newtonian fluids in laminar flow applied to fully developed flow conditions in a capillary (Bird, *et al.*, 1960; Micro Motion, 1994; Zaman, 1993)

$$\eta = \phi \frac{\Delta p \rho}{\dot{m}} \tag{4.1}$$

where	$\phi =$	proportionality constant
	$\Delta p =$	differential pressure
	\dot{q} =	volumetric flow rate
	$\dot{m} =$	mass flow rate

The Micro Motion coriolis mass flow sensor provides a direct measure of mass flow rate (\dot{m}) and, of the density (ρ), hence permitting the calculation of viscosity from Equation (4.1). Since the density of the fluid in the sensor is measured, a volumetric flow rate (\dot{q}) can be calculated as $\dot{q} = \dot{m}/\rho$. An electromagnetic drive coil located at the center of the tube bend vibrates the flow tubes at its natural frequency. On the upward cycle of the tube

vibration, the fluid entering the sensor counters the upward motion by applying a downward force on the tube. With the flow tubes momentum as the fluid travels around the turn in the tube, the fluid resists the decreasing upward motion by applying an upward force as the fluid exits the sensor. These simultaneous opposing forces cause the tubes to bend slightly. During the downward cycle of the flow tube vibration, the opposite occurs which establishes a twisting motion. This phenomena is called the Coriolis Effect (Micro Motion, 1994).

Due to Newton's Second Law of Motion, F=ma, the amount of twist is directly proportional to the mass flow rate through the sensor. The amount of twist caused by the flow is measured as the time difference in phase between two electromagnetic velocity sensors on either side of the flow tube. With no flow through the sensor, the two velocity pickups are in phase. With flow, the velocity signals are no longer in phase and a time difference is detected. A flow calibration factor is determined by using the mass flow meter with a reference fluid, and along with the time difference, the mass flow rate is ascertained (Micro Motion, 1994).

The density of a fluid flowing through the flow tubes of the sensor is inversely proportional to the square of the natural frequency vibration. The sensor mass and the mass of the fluid present in the flow tubes can be related to a mass-and-spring assembly in which the mass, once set in motion, will vibrate at its resonant frequency according to the equation (Micro Motion, 1994)

$$f = \frac{1}{2\pi} \sqrt{\frac{K}{m_{tot}}}$$
(4.2)

where

f = resonant frequency K = calibration constant m_{tot} = mass of the fully flooded sensor

Using the fact that $m_{tot}=m_{tube}-m_{fluid}$, where the mass of the fluid in the flow tubes is m_{fluid} and the mass of the sensor is m_{tube} , then the density can then be calculated by

$$\phi = \phi_1 f^{-2} - \phi_2 \tag{4.3}$$

 $\phi_1 =$ calibration constant $\phi_2 =$ physical constant of the sensor

The density calibration constant ϕ_1 is determined using two fluids of known density (air and water) as a reference. The mass and volume of the sensor is fixed for any given device, and the tube frequency is established by the instrument.

From the measurements of pressure drop, density, and mass flow rate, the Hagen-Poiseuille Equation (4.1) can be used to calculate viscosity, as long as the flow is laminar. The viscosity proportionality constant ϕ is set by calibrating the sensor at a point of known viscosity for a Newtonian fluids. A Non-Newtonian fluid uses two-points: One in the upper part and one in the lower part of the operating viscosity range. It is important to keep in mind that the Hagen-Poiseuille Equation is developed for a straight capillary tube and for fully developed flow, two conditions that do not hold for the Micro Motion Mass Flow Sensor. At relatively high flow rates, secondary flow characteristics, as well as entry and exit effects can produce pressure drops higher than that predicted by the Hagen-Poiseuille equation, hence adversely affecting the reading of the Micro Motion sensor.

4.2 Experimental Overview

where

Table 4.1 lists the solids composition of black liquors used in the experimental runs that were performed with the Micro Motion Viscometer. In total, the instrument was tested on 14 different black liquor concentrations, ranging from a 34.14% Georgia Pacific liquor to a 73.40% liquor obtained by mixing a Georgia Pacific liquor with Canadian Forest liquor.

In the configuration in the pilot flow loop, the Rosemount 3051CD differential pressure cell chambers are mounted on both ends of the Micro Motion CMF025 coriolis mass flow sensor, with the 4-20 mA analog differential pressure signal going to the Micro Motion transmitter. From the Micro Motion transmitter, an RS-232 HART signal is sent to

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the Micro Motion Prolink Interface, while two user-selectable analog 4-20 mA signals are also sent to the UF Data Acquisition (DAQ) System in the pilot plant. Viscosity and differential pressure data were selected to be transmitted to the DAQ system, while viscosity, density, temperature, differential pressure, mass flow rate, and volumetric flow rate were digitally transmitted to Prolink.

Georgia Pacific	Canadian Forest	Chesapeake	GP & Canadian Forest Mix
34.14%	47.96%	49.39%	67.72%
42.24%	57.73%	59.58%	71.38%
48.29%			73.40%
55.84%			
58.38%			
65.51%			
68.38%			

Table 4.1. Black liquor types and solids content tested with the Micro Motion Viscometer.

In steady-state trials, Prolink and the UF DAQ System were run concurrently with the data time stamped for off-line analysis. For transient trials, only the UF DAQ System was active, since Prolink is preempted by the UF DAQ Software and cannot update the sample data quickly enough to record the transitions.

The Micro Motion Viscometer must be zeroed, and a one-point viscosity calibration needs to be performed on the sensor through Prolink. If the Rosemount DP cell and the Micro Motion transmitter were digitally linked, then the DP cell could also be zeroed using Prolink. For the analog signals, the DP cell needs to be manually zeroed. To zero the Micro Motion sensor and the DP cell, the Viscometer Line is fully flooded and then the isolation valves are activated to develop a no-flow condition in the Viscometer Line. The instruments are then zeroed. To provide a one-point viscosity calibration, the black liquor needs to be at its most viscous level, and must be flowing through the Viscometer Line at a velocity so as to create the highest differential pressure measurable while still in the laminar regime. Once these conditions are met, the laboratory reference viscosity at the temperature of the sensor is entered into Prolink. This sets the viscosity calibration constant. For a Newtonian fluid, this constant should not need to be changed once it is set.

4.3 Selected Experimental Results

The selected data presented in this section represent typical results for the Micro Motion instrument. The data presented is comprised of 4 steady-state runs of viscosity versus flow rate at different temperatures and solids compositions, and 1 transient response of the instrument to temperature changes.

Figure 4.1 shows a high viscosity run with Georgia Pacific 68.39% solids black liquor at approximately 90°C. This is a steady-state run, and the results are presented on two plots. The upper plot shows the viscosity values measured by the instrument, and are indicated by the diamond-shaped markers. The continuous line joining the markers is included as a visual aid to track the data trend, and is obtained through arbitrary curve-fits. The upper plot also shows a laboratory reference band of viscosity values defined by two dash-dot lines. The band is constructed using viscosity correlations produced for the various laboratory instruments described in Section 2.4. In particular, the upper curve of the band corresponds to the highest viscosity prediction produced when considering all the laboratory correlations at the temperature of the flow rate in question. Analogously, the lower curve of the band corresponds to the lowest viscosity prediction. Any measurements that fall inside the reference band are considered to be consistent with the laboratory measurements.

The lower plot in Figure 4.1 is a measure of the relative difference between the viscosity measurement (η) produced by the instrument and the viscosity (η_{ref}) predicted by a correlation produced using data acquired with a reliable laboratory instrument and valid for the particular solids-content of the black liquor used in the test. Hence, the relative difference is defined as follows:

Relative difference =
$$\frac{\eta - \eta_{ref}}{\eta_{ref}}$$
 (4.3)

We typically used a Haake Rotovisco RV12 Rotational Viscometer or a Cambridge Sliding Element Laboratory Viscometer as the reference laboratory instrument. Note that, for example, a relative difference of 0.08 indicates that the instrument viscosity differs from the reference measurement by 8%. We consider an instrument to be accurate if it realizes a relative difference of $\pm 10\%$. A relevant remark concerning this figure is that the temperature is not strictly constant during the run; in fact, as the flow rate varies, the temperature also varies, though it remains close to the approximate value reported in the figure title. For this particular figure the viscosity, flow-rate, and temperature information for 10 selected flow rates is as follows:

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Flow Rate (gpm)	Temperature (°C)	Viscosity (cP)
0.544	84.8	286.62
0.524	84.7	288.94
0.474	84.4	294.85
0.911	84.4	296.12
0.937	84.7	289.28
1.208	85.1	286.67
1.225	85.3	284.75
1.250	85.5	281.95
1.243	85.4	282.78
1.250	85.5	281.95

Table 4.2. Flow-rate, temperature, and viscosity data for Figure 4.1.

Analysis of Figure 4.1 shows that the Micro Motion Viscometer produces measurements with generally good accuracy because the measured response from the instrument lie near the outer boundary of the laboratory reference band. Note that the instrument tracks well with the changes in viscosity that occur due to the change of the temperature caused with the variation of flow rates. The lower plot was generated using the laboratory reference correlation

$$\log \eta_{ref}(cP) = 18.2073771 - 17633.4415/T(K) + 4709530.7768/T(K)^2$$
(4.4)

The plot shows that throughout this run the instrument experiences a relative-difference offset of approximately -5%. In this case, the range of the viscometer is limited by the differential pressure cell.

Figure 4.2 shows another high viscosity run with Georgia Pacific 65.51% solids black liquor approximately at 80°C. Again, the viscometer tracks just below the lower laboratory reference boundary with a relative difference offset of -10%. Higher flow rates were not possible due to limitations of the differential pressure cell selected for this application. The results from this run and from the previous run are typical high viscosity trials for this instrument. In both cases, there is a minor offset with excellent tracking.

Figure 4.3 shows a mid-ranged viscosity run with Georgia Pacific 65.51% solids black liquor at approximately 95°C. The viscosity of the black liquor starts out about 140 cP and slowly decreases to about 120 cP as flow rate increases. At 0.5 gpm, the Micro Motion Viscometer is within the laboratory reference band; however, as the flow rate increases, so does the Micro Motion viscosity reading. This run demonstrates the transition to a linear rising response in the viscosity measurement.

Figure 4.4 shows a low viscosity run with Georgia Pacific 68.39% solids black liquor approximately at 125°C. This run demonstrates a more dramatic increase in the Micro Motion viscosity measurement as the flow rate increases. In this run, the Micro Motion viscosity measurement at 0.75 gpm is slightly above the laboratory reference boundary at 55 cP, and the viscosity measurement steadily increases as the flow rate increases, even though the laboratory reference viscosity is relatively constant.

Figure 4.5 plots viscosity as a function of time to examine the transient response of the Micro Motion Viscometer with GP and Canadian Forest Mix 67.46% black liquor. In this experiment the temperature of the flowing black liquor steam was changed in a step-fashion by manually opening the steam valve controlled by the Process Heater Output

Controller (instrument TC2 in the P&ID diagram in Figure 9.1). The step changes were made in discrete increments, as shown in the legend of the figure, until the output from the Heater became saturated. The four markers (circle, square, triangle, and cross) on the figure denote the instants when the valve opening is changed. The dotted line represents the viscosity determined using a laboratory correlation of viscosity versus temperature, and the continuous line represents the measurement by the instrument. Further details on the execution of this may be found in Section 2.2. Note that in Figure 4.5 at time zero, the Viscometer Line is at a thermal and flow rate steady-state. At approximately 2 minutes into the run, the first step input was introduced by manually opening the Process Heater Output Controller TC2 to 10%. Since there was no immediate temperature response from the Heater Steam Output Indicator TI8, the second temperature step input (TC2 is set to 15%) open) was applied 3 minutes into the run. There is a ~3 minute time-delay before the Viscometer Line RTD TT7 (cf. P&ID diagram in Figure 9.1) responses to the temperature step input. The time-delay of the Micro Motion Viscometer from the Viscometer Line RTD TT7 is less than 30 seconds. During this temperature step input, there is a drop of viscosity from 580 cP to 250 cP. For the third step input (TC2 is set to 20% open), the time-delay of the Micro Motion Viscometer is actually slightly shorter than the delay for the Viscometer Line RTD TT7 for a viscosity decrease of 250 cP to 100 cP. In order to maintain accurate viscosity measurements from the instrument, a low flow rate was maintained through the Viscometer Line during this run. When examining this plot, the focus of attention should be placed on the transient response of the instrument and not the difference from the laboratory reference because the sensor is not able to achieve thermal stability which can lead to erratic measured responses.

The linear increase of the Micro Motion viscosity as a function of flow rate with low-viscosity fluids was commonly encountered through out the tests. Apparently, the secondary flow effects within the Micro Motion flow tubes is creating the erroneous viscosity measurement. At flow rates lower than 0.5 gpm, the instrument used measures the viscosity correctly even at low viscosities (≤ 80 cP); however, on the test runs with low solids, the instrument may have been erroneously calibrated when the secondary flows were affecting the viscosity measurements, instead of the normal procedure where it is normally calibrated in the regime of high viscosity and high pressure drop. We have verified that in these cases the viscosity measurement is still linear, but the Y-intercept (viscosity) is lower than the laboratory reference boundary, thus creating an offset.



Figure 4.1 Micro Motion Viscometer with Georgia Pacific 68.39% Solids Black Liquor at ~90°C.



Figure 4.2 Micro Motion Viscometer with Georgia Pacific 65.51% Solids Black Liquor at ~80°C.



Figure 4.3 Micro Motion Viscometer with Georgia Pacific 65.51% Solids Black Liquor at ~95°C.


Figure 4.4 Micro Motion Viscometer with Georgia Pacific 68.39% Solids Black Liquor at ~125°C.



Figure 4.5 Micro Motion Viscometer transient temperature run with GP and Canadian Forest Mix 67.46% Solids Black Liquor.

4.4 Summary of Observations

The general trends observed with the Micro Motion Viscometer are summarized as follows:

- The Micro Motion Viscometer tracked within ±10% of the laboratory reference at high viscosities (> 150 cP).
- There was no evidence of fouling during the trials.
- Transient response time is very short.
- There are no moving parts in the process stream.
- In addition to viscosity data the instrument provides information on mass flow rate, volumetric flow rate, temperature, differential pressure, and density (within ±3% of laboratory reference values).

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- Secondary flows affect the viscosity measurement for low viscosity and high flow rate systems.
- This instrument requires a one-point calibration with a laboratory reference.

The setup in the pilot plant is comprised of includes the CMF025 coriolis mass flow meter with 0.25" flow tubes, and the 3051CD differential pressure cell which has a range of 0-36 psig. The viscosity measurement and the flow rate range are limited by the pressure drop across the instrument. By maintaining very low flow rate (0.35-0.5 gpm), the instrument can be used for measuring viscosities up to 600 cP. With current mill recovery furnaces, it is unlikely that there will be situations in which viscosities above 400 cP will be exceeded.

Under conditions of high flow rate and low viscosities, a combination of coriolis and entrance and exiting effects create secondary flows which develop an additional pressure term. The additional pressure introduces an errors in the reading, causing a linearly rising viscosity measurement with increasing flow rate. The response to the secondary flows appears to be systematic; hence, a compensation correlation may be developed. Micro Motion has proposed using to use a power-law equation in an attempt to provide a correction to the secondary flows

$$\eta = k \frac{\Delta p}{\dot{q}^n} \tag{4.5}$$

where *n* is the correction term for the secondary flows. From Figures 4.6 and 4.7, the Micro Motion, the corrected Micro Motion, and the corrected UF responses are compared with the laboratory reference. The UF correction parameters were determined with a non-linear model fit of the power law equation. The calibration constant k in the UF model is in units of psig/gpm; whereas, the Micro Motion calibration constant is in units of psig*g/mL*min/lbm.

In Figures 4.6 and 4.7, the corrected Micro Motion and UF viscosity measurements are within $\pm 20\%$ of the laboratory reference using; however, this *n* value is not necessarily a constant for different black liquor types. In addition, at very low flow rates (< 0.5 gpm), the power law model can cause a very large viscosity reading, as shown in Figure 4.6, although this may have been caused by errors in transmitting very small pressure drop measurements to the Micro Motion transmitter over an analog line. Figure 4.7 shows far less scatter at low flow rates. The UF and Micro Motion viscosity corrections are within $\pm 10\%$ of the laboratory reference. Additional analysis of the data gathered at the pilot plant may yield a universal model for each coriolis mass flow sensor.

Table 4	4.2.	Micro M	lotion visc	osity con	npensation
parameters.					
	*****	Figure 4.6		Figure 4.7	
UI	7	k=6.98	n=1.31	k=8.9	n=1.14
Micro M	lotion	k=100	n=1.3	k=150	n=1.3

The Micro Motion Viscometer is more than capable of operating satisfactorily by maintaining the flow rate below the critical point where the effects of the secondary flow become significant. The effects of the secondary flows can be minimized by examining the viscosity ranges of the application and limiting the flow through the mass flow sensor. The upper flow rate limits with black liquor viscosities between 200-300 cP were not reached in the pilot flow loop due to limitations in the differential pressure cell. Experimental trials with Chesapeake Paper 59.28% solids black liquor at approximately 65°C demonstrated that the viscosity measurement remained constant at 200 cP for flow rates up to 1.8 gpm before the DP cell reached its limit. The critical flow rate can be increased by using a larger diameter mass flow sensor.

The recommendations based on our experience with the Micro Motion Viscometer is summarized as follows:

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- Use in slip stream only with necessary controls to maintain a flow rate below the critical flow rate.
 - Use digital communication between the differential pressure cell and the Micro Motion transmitter to increase the accuracy of the viscosity measurement, especially at low pressure drops.
 - Develop a universal viscosity correction model to compensate for the secondary flow effects.

With proper sizing of the mass flow sensor and of the differential pressure cell, the instrument is capable of satisfactorily operating over the expected viscosity ranges to be encountered in a mill.



Figure 4.6 Micro Motion Viscometer viscosity compensation with Georgia Pacific 65.51% Solids.



Figure 4.7 Micro Motion Viscometer viscosity compensation with Chesapeake Paper 59.28% Solids.

4.5 Extensive Experimental Results

Included in this section are graphical representations of the results of the on-line viscometer trials performed with the Micro Motion instrument. The figures describes the data gathered for all the black liquors listed in Table 4.1. Figures labeled as "a" represent the viscosity as measured by the instrument as a function of flow rate and the figures labeled as "b" show the relative difference of the measured response from the laboratory reference as a function of flow rate. In addition, the density from the Micro Motion Viscometer was also examined. Figures labeled as "c" represent the density as measured by the instrument as a function of flow rate and the figures labeled as "d" show the relative difference of the measured response the density as measured by the instrument as a function of flow rate and the figures labeled as "d" show the relative difference of the measured response the density as measured by the instrument as a function of flow rate and the figures labeled as "d" show the relative difference of the measured response from the laboratory reference as a function of flow rate and the figures labeled as "d" show the relative difference of the measured response from the laboratory reference as a function of flow rate and the figures labeled as "d" show the relative difference of the measured response from the laboratory reference as a function of flow rate.

Figures 500 represent results for the Canadian Forest 47.96% Solids at 30-120°C (viscosities of 5-60 cP), and at varying flow rates. At 30°C, the data represents what has been typical for lower viscosities. The instrument gives very reproducible results; however, the viscosity measured on-line does not agree with the viscosity determined in the laboratory. The difference was a linear function of flow rate. The relative difference, in this case, was linear with respect to flow rate.

Figures 501-503 represent results for the same liquor at 50-120°C as a function of flow rate. In every case, the viscosity measured with the Micro-Motion instrument on-line was linear with respect to flow rate and results were very reproducible. Other on-line viscometers in place during the same tests followed the predicted viscosities, with offset errors in some cases, but never with the high errors that we observed with this instrument. On the other hand, reproducibility was probably the best with the Micro Motion Viscometer. The trials were performed in laminar flow at all conditions, it was not believed that the problem was with the sensitivity of the DP cell, because the results were very reproducible. Figures 504-507 represent similar results for Canadian Forest 57.73% Solids at temperatures from 50 to 125°C (viscosities of 20-290 cP), and at varying flow rates. The results at 50°C and 55°C for the liquor are excellent (Figures 504 and 505). The viscosity was tracked exactly and accurately with essentially zero relative difference. Figure 506 represent data for the same liquor at 80°C where the predicted viscosity ranges from about 60 to 75 cp. The data show the same type of linear increase in measured viscosity as was observed for the same liquor at 47.96% solids. Data taken at 125°C shows similar behavior (Figures 507).

Figures 508-511 represent results for Chesapeake Paper 49.39% Solids at temperatures from 35 to 120°C (viscosities from 5 to 110 cP), and at different flow rates. This liquor was noticeably "dirtier" than the Canadian Forest liquor due to association and particulate formation in the liquor. Performance at 35°C shows good reproducibility and a linear change in measured viscosity with respect to flow rate (Figure 508). This occurs for all tests run at temperatures up to 120°C for this liquor (Figures 509-511). It should be noted that this liquor was quite "dirty" at this concentration, but there was no evidence of fouling of the instrument.

Figures 512-515 represent results for Chesapeake Paper 59.28% Solids at temperatures from 65 to 120°C (viscosities from 15 to 220 cP), and at varying flow rates. Results at 65°C are very good with the predicted viscosity in this case is about 200 cP (Figure 512). At 80°C and 95°C, the data may be showing the critical flow rate at which the secondary flows become significant after which the viscosity measured increases linearly with increasing flow rate (Figures 513 and 514). At 120°C, the same overall general trends were observed (Figure 515). One important fact: The instrument gives very reproducible results, even for a "dirty" liquor.

Figures 516-519 represent results for Georgia Pacific 42.20% Solids at temperatures from 35 to 120°C (viscosities of 2-20 cP), and at different flow rates. Most of these conditions are out of range for this instrument. However, results seem

reproducible, and the same general response characteristics were observed as before with low-viscosity liquors. Data represented were taken while the DP cell may have been failing. After this, the DP cell was replaced.

Figures 520-523 represent results for Georgia Pacific 48.29% Solids at temperatures from 35 to 125°C (viscosities of 5-80 cP), and at different flow rates. The same general response characteristics were observed as before with low-viscosity liquors. Again there was a linear response in measured viscosity as flow rate was increased which appears highly reproducible.

Figures 523-525 represent results for Georgia Pacific 55.84% Solids at temperatures from 55 to 85°C (viscosities from 30 to 190 cP), and at different flow rates. At 55°C, the instrument gave excellent results when the average viscosity was about 180 cP with a slight linear increase in the viscosity measurement (Figure 523). Results at 75°C and 85°C were similar to earlier results at lower viscosities; that is, the measured viscosity was a linear function of flow rate (Figures 524 and 525). There is one noticeable difference in this and subsequent data. The line for measured viscosity always started from within the predicted viscosity band at 0.5 gpm and then increased; this line did not cross the predicted viscosity band at different flow rates as the viscosity decreased. The instrument was calibrated using the same procedure listed earlier, except that the viscosity of the black liquor was high enough so that secondary flows were not very significant and an accurate calibration was performed.

Figures 526-529 represent results for Georgia-Pacific 58.38% Solids at temperatures from 60 to 125°C (viscosities from 10 to 200 cP), and at different flow rates. Data taken at 60°C were very good (Figure 526). There was very good tracking, accuracy was acceptable, and there was no noticeable effect of flow rate. Data at higher temperatures for conditions with predicted viscosities below about 80 cP showed the same type of response of measured viscosity with flow rate as observed before for lower viscosities (Figures 527-529).

Figures 530-533 represent results for Georgia-Pacific 65.51% Solids at temperatures from 80 to 125°C (viscosities from 30 to 280 cP), and at different flow rates. At 80°C, the instrument tracked well with a relative difference that was slightly greater than -10%, and reproducibility was excellent (Figure 530). At 85°C and 95°C, the measured viscosity began to show some variation with flow rate, but data were reproducible (Figures 531 and 532). At 125°C, however, where the predicted viscosity is in the range of 30-40 cP, the measured viscosity increases with increasing flow rate (Figure 533). This increase was linear over the region for which the predicted viscosity band was constant.

Figures 534-537 represent results for Georgia-Pacific 68.38% Solids at temperatures from 90 to 125C (viscosities from 35 to 320 cP), and at different flow rates. Data at 90°C tracked the viscosity very well with little relative difference where the predicted viscosity was high (Figure 534). Results at 100°C were reasonably good with all but one set of data points falling within the predicted viscosity band and a slight linear increase in the measure viscosity as the flow rate increases (Figure 534). At 115°C and 125°C, however, where the predicted viscosity ranges from 35-80 cP, the same type of linear increase was observed in measured viscosity with increased flow rate that was previously observed in this viscosity range (Figures 536 and 537).

Figures 538-542 represent results for Georgia Pacific and Canadian Forest Mix 67.46% Solids at temperatures from 110 to 140°C (viscosities from 100 to 500 cP), and at different flow rates. The data gathered represent both transient and steady state temperature runs with this instrument. At 100°C and 105°C, the instrument had excellent tracking and good accuracy while operating at a very low flow rates (Figures 539 and 540). At 140°C, there was a good amount of scatter in the data that was probably due to the difficulty in maintaining thermal stability in the Viscometer Line, and the same type of linear increase was observed in measured viscosity with increased flow rate that was previously observed in this viscosity range (Figure 541). Figure 538 plots viscosity as a function of time to examine the performance of the instrument during transient temperature runs. There are no

references to the actual time the temperature step-input was introduced by the Heater because this data was gathered with Prolink and an accurate time stamp was not possible. During the transient response experiments, the correlation of one laboratory viscometer was used as the lab reference. In this case, the Micro Motion instrument had a faster response to the temperature step-input than the Viscometer Line RTD. The change of viscosity was from 560 cP to 150 cP. Figure 542 shows the transient temperature experiment with the data gathered by the UF DAQ system in the pilot plant. During the experiment, temperature step-inputs were introduced by manually opening the Process Heater Output Controller (TC2) in discrete increments until the output becomes saturated. The Micro Motion instrument had a faster response to the temperature step-input than the Viscometer Line RTD.

Figures 543-546 represent results for Georgia Pacific and Canadian Forest Mix 71.38% Solids at temperatures from 100 to 140°C (viscosities of 100-700 cP), and at different flow rates. The data gathered represent both transient and steady state temperature runs with this instrument. At 100°C, the instrument demonstrated excellent tracking and accuracy even at a very low flow rate (Figure 543). At 125°C, the linear increase with increasing flow rate was again observed (Figure 544). During the transient temperature experiments, the instrument had a very rapid response to the temperature step inputs (Figures 545 and 546).

Figures 547 and 548 represent results for Georgia Pacific and Canadian Forest Mix 73.40% Solids at temperatures from 100 to 140°C (viscosities of 150-1000 cP) and at different flow rates. The data gathered represent both transient and steady state temperature runs with this instrument. At 135°C, the instrument had excellent tracking with a significant offset from the laboratory reference (Figure 547). The offset may be explained by the difficulty in characterizing this liquor due to the high viscosity. There is a good possibility that the reference viscosity was low, since the behavior of the Micro Motion Viscometer was consistent for a higher viscosity fluid. During the transient temperature

experiments, the instrument had a very rapid response to the temperature step inputs (Figures 548).

Viscometer Test Run 960130M1 Figure No. 500a

Micro Motion with Canadian Forest 47.96% Solids at 30°C



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Viscometer Test Run 960130M1 Figure No. 500b

Micro Motion with Canadian Forest 47.96% Solids at 30°C



Viscometer Test Run 960130M1 Figure No. 500c

Micro Motion Density with Canadian Forest 47.96% Solids at 30°C



Viscometer Test Run 960130M1 Figure No. 500d

Micro Motion Density with Canadian Forest 47.96% Solids at 30°C



Viscometer Test Run 960130M2 Figure No. 501a

Micro Motion with Canadian Forest 47.96% Solids at 50°C



Viscometer Test Run 960130M2 Figure No. 501b

Micro Motion with Canadian Forest 47.96% Solids at 50°C



Viscometer Test Run 960130M2 Figure No. 501c

Micro Motion Density with Canadian Forest 47.96% Solids at 50°C



Viscometer Test Run 960130M2 Figure No. 501d

Micro Motion Density with Canadian Forest 47.96% Solids at 50°C



Viscometer Test Run 960131M3 Figure No. 502a

Micro Motion with Canadian Forest 47.96% Solids at 80°C



Viscometer Test Run 960131M3 Figure No. 502b

Micro Motion with Canadian Forest 47.96% Solids at 80°C



Viscometer Test Run 960131M3 Figure No. 502c

Micro Motion Density with Canadian Forest 47.96% Solids at 80°C



Viscometer Test Run 960131M3 Figure No. 502d

Micro Motion Density with Canadian Forest 47.96% Solids at 80°C



Viscometer Test Run 960131M4 Figure No. 503a

Micro Motion with Canadian Forest 47.96% Solids at 120°C



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Viscometer Test Run 960131M4 Figure No. 503b

Micro Motion with Canadian Forest 47.96% Solids at 120°C





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Viscometer Test Run 960131M4 Figure No. 503d

Micro Motion Density with Canadian Forest 47.96% Solids at 120°C



Viscometer Test Run 960409M1 Figure No. 504a

Micro Motion with Canadian Forest 57.73% Solids at ~50°C



Viscometer Test Run 960409M1 Figure No. 504b

Micro Motion with Canadian Forest 57.73% Solids at ~50°C



Viscometer Test Run 960409M1 Figure No. 504c

Micro Motion Density with Canadian Forest 57.73% Solids at ~50°C



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Viscometer Test Run 960409M1 Figure No. 504d

Micro Motion Density with Canadian Forest 57.73% Solids at ~50°C



Viscometer Test Run 960409M2 Figure No. 505a

Micro Motion with Canadian Forest 57.73% Solids at ~55°C



Viscometer Test Run 960409M2 Figure No. 505b

Micro Motion with Canadian Forest 57.73% Solids at ~55°C



Viscometer Test Run 960409M2 Figure No. 505c

Micro Motion Density with Canadian Forest 57.73% Solids at ~55°C


Viscometer Test Run 960409M2 Figure No. 505d

Micro Motion Density with Canadian Forest 57.73% Solids at ~55°C



Viscometer Test Run 960410M3 Figure No. 506a

Micro Motion with Canadian Forest 57.73% Solids at ~80°C



Viscometer Test Run 960410M3 Figure No. 506b

Micro Motion with Canadian Forest 57.73% Solids at ~80°C



Viscometer Test Run 960410M3 Figure No. 506c

Micro Motion Density with Canadian Forest 57.73% Solids at ~80°C



Viscometer Test Run 960410M3 Figure No. 506d

Micro Motion Density with Canadian Forest 57.73% Solids at ~80°C



Viscometer Test Run 960410M4 Figure No. 507a

Micro Motion with Canadian Forest 57.73% Solids at ~125°C



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Viscometer Test Run 960410M4 Figure No. 507b

Micro Motion with Canadian Forest 57.73% Solids at ~125°C



Viscometer Test Run 960410M4 Figure No. 507d

Micro Motion Density with Canadian Forest 57.73% Solids at ~125°C



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Viscometer Test Run 960207M1 Figure No. 508a

Micro Motion with Chesapeake Paper 49.39% Solids at ~35°C



Viscometer Test Run 960207M1 Figure No. 508b

Micro Motion with Chesapeake Paper 49.39% Solids at ~35°C



Viscometer Test Run 960410M4 Figure No. 507c

Micro Motion Density with Canadian Forest 57.73% Solids at ~125°C



Viscometer Test Run 960207M1 Figure No. 508c

Micro Motion Density with Chesapeake Paper 49.39% Solids at ~35°C



7.67

Viscometer Test Run 960207M1 Figure No. 508d

Micro Motion Density with Chesapeake Paper 49.39% Solids at ~35°C



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Viscometer Test Run 960207M2 Figure No. 509a

Micro Motion with Chesapeake Paper 49.39% Solids at ~50°C



Viscometer Test Run 960207M2 Figure No. 509b

Micro Motion with Chesapeake Paper 49.39% Solids at ~50°C



Viscometer Test Run 960207M2 Figure No. 509c

Micro Motion Density with Chesapeake Paper 49.39% Solids at ~50°C



Viscometer Test Run 960207M2 Figure No. 509d

Micro Motion Density with Chesapeake Paper 49.39% Solids at ~50°C



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Viscometer Test Run 960208M3 Figure No. 510a

Micro Motion with Chesapeake Paper 49.39% Solids at ~75°C



Viscometer Test Run 960208M3 Figure No. 510b

Micro Motion with Chesapeake Paper 49.39% Solids at ~75°C



Viscometer Test Run 960208M3 Figure No. 510c

Micro Motion Density with Chesapeake Paper 49.39% Solids at ~75°C



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Viscometer Test Run 960208M3 Figure No. 510d

Micro Motion Density with Chesapeake Paper 49.39% Solids at ~75°C



Viscometer Test Run 960208M4 Figure No. 511a

Micro Motion with Chesapeake Paper 49.39% Solids at ~120°C



Viscometer Test Run 960208M4 Figure No. 511b

Micro Motion with Chesapeake Paper 49.39% Solids at ~120°C



Viscometer Test Run 960208M4 Figure No. 511c

Micro Motion Density with Chesapeake Paper 49.39% Solids at ~120°C



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Viscometer Test Run 960208M4 Figure No. 511d

Micro Motion Density with Chesapeake Paper 49.39% Solids at ~120°C



Viscometer Test Run 960217M1 Figure No. 512a

Micro Motion with Chesapeake Paper 59.28% Solids at ~65°C



Viscometer Test Run 960217M1 Figure No. 512b

Micro Motion with Chesapeake Paper 59.28% Solids at ~65°C



Viscometer Test Run 960217M1 Figure No. 512c

Micro Motion Density with Chesapeake Paper 59.28% Solids at ~65°C



Viscometer Test Run 960217M1 Figure No. 512d

Micro Motion Density with Chesapeake Paper 59.28% Solids at ~65°C



Viscometer Test Run 960217M2 Figure No. 513a

Micro Motion with Chesapeake Paper 59.28% Solids at ~80°C



Viscometer Test Run 960217M2 Figure No. 513b

Micro Motion with Chesapeake Paper 59.28% Solids at ~80°C



Viscometer Test Run 960217M2 Figure No. 513c

Micro Motion Density with Chesapeake Paper 59.28% Solids at ~80°C



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Viscometer Test Run 960217M2 Figure No. 513d

Micro Motion Density with Chesapeake Paper 59.28% Solids at ~80°C



Viscometer Test Run 960219M3 Figure No. 514a

Micro Motion with Chesapeake Paper 59.28% Solids at ~95°C



Viscometer Test Run 960219M3 Figure No. 514b

Micro Motion with Chesapeake Paper 59.28% Solids at ~95°C



Viscometer Test Run 960219M3 Figure No. 514c

Micro Motion Density with Chesapeake Paper 59.28% Solids at ~95°C


Viscometer Test Run 960219M3 Figure No. 514d

Micro Motion Density with Chesapeake Paper 59.28% Solids at ~95°C



Viscometer Test Run 960219M4 Figure No. 515a

Micro Motion with Chesapeake Paper 59.28% Solids at ~120°C



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Viscometer Test Run 960219M4 Figure No. 515b

Micro Motion with Chesapeake Paper 59.28% Solids at ~120°C



Viscometer Test Run 960219M4 Figure No. 515c

Micro Motion Density with Chesapeake Paper 59.28% Solids at ~120°C



Viscometer Test Run 960219M4 Figure No. 515d

Micro Motion Density with Chesapeake Paper 59.28% Solids at ~120°C



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Viscometer Test Run 950903M1 Figure No. 516a

Micro Motion with Georgia Pacific 42.20% Solids at ~35°C



Viscometer Test Run 950903M1 Figure No. 516b

Micro Motion with Georgia Pacific 42.20% Solids at ~35°C



Viscometer Test Run 950903M1 Figure No. 516c

Micro Motion Density with Georgia Pacific 42.20% Solids at ~35°C



Viscometer Test Run 950903M1 Figure No. 516d

Micro Motion Density with Georgia Pacific 42.20% Solids at ~35°C



Viscometer Test Run 950905M2 Figure No. 517a

Micro Motion with Georgia Pacific 42.20% Solids at ~55°C



Viscometer Test Run 950905M2 Figure No. 517b

Micro Motion with Georgia Pacific 42.20% Solids at ~55°C



Viscometer Test Run 950905M2 Figure No. 517c

Micro Motion Density with Georgia Pacific 42.20% Solids at ~55°C



Viscometer Test Run 950905M2 Figure No. 517d

Micro Motion Density with Georgia Pacific 42.20% Solids at ~55°C



Viscometer Test Run 950906M3 Figure No. 518a

Micro Motion with Georgia Pacific 42.20% Solids at ~85°C



Viscometer Test Run 950906M3 Figure No. 518b

Micro Motion with Georgia Pacific 42.20% Solids at ~85°C



Viscometer Test Run 950906M3 Figure No. 518c

Micro Motion Density with Georgia Pacific 42.20% Solids at ~85°C



Viscometer Test Run 950906M3 Figure No. 518d

Micro Motion Density with Georgia Pacific 42.20% Solids at ~85°C



Viscometer Test Run 950907M4 Figure No. 519a

Micro Motion with Georgia Pacific 42.20% Solids at ~120°C



Viscometer Test Run 950907M4 Figure No. 519b

Micro Motion with Georgia Pacific 42.20% Solids at ~120°C



Viscometer Test Run 950907M4 Figure No. 519c

Micro Motion Density with Georgia Pacific 42.20% Solids at ~120°C



Viscometer Test Run 950907M4 Figure No. 519d

Micro Motion Density with Georgia Pacific 42.20% Solids at ~120°C



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Viscometer Test Run 960126M1 Figure No. 520a

Micro Motion with Georgia Pacific 48.29% Solids at ~30°C



Viscometer Test Run 960126M1 Figure No. 520b

Micro Motion with Georgia Pacific 48.29% Solids at ~30°C



Viscometer Test Run 960126M1 Figure No. 520c

Micro Motion with Georgia Pacific 48.29% Solids at ~30°C



Viscometer Test Run 960126M1 Figure No. 520d

Micro Motion with Georgia Pacific 48.29% Solids at ~30°C



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Viscometer Test Run 960126M2 Figure No. 521a

Micro Motion with Georgia Pacific 48.29% Solids at ~60°C



Viscometer Test Run 960126M2 Figure No. 521b

Micro Motion with Georgia Pacific 48.29% Solids at ~60°C



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Viscometer Test Run 960126M2 Figure No. 521c

Micro Motion with Georgia Pacific 48.29% Solids at ~60°C



Viscometer Test Run 960126M2 Figure No. 521d

Micro Motion with Georgia Pacific 48.29% Solids at ~60°C



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Viscometer Test Run 960126M3 Figure No. 522a

Micro Motion with Georgia Pacific 48.29% Solids at ~85°C



Viscometer Test Run 960126M3 Figure No. 522b

Micro Motion with Georgia Pacific 48.29% Solids at ~85°C

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Viscometer Test Run 960126M3 Figure No. 522c

Micro Motion with Georgia Pacific 48.29% Solids at ~85°C



Viscometer Test Run 960126M3 Figure No. 522d

Micro Motion Density with Georgia Pacific 48.29% Solids at ~85°C



Viscometer Test Run 960126M4 Figure No. 522a

Micro Motion with Georgia Pacific 48.29% Solids at ~125°C



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Viscometer Test Run 960126M4 Figure No. 522b

Micro Motion with Georgia Pacific 48.29% Solids at ~125°C



Viscometer Test Run 960126M4 Figure No. 522c

Micro Motion with Georgia Pacific 48.29% Solids at ~125°C


Viscometer Test Run 960126M4 Figure No. 522d

Micro Motion Density with Georgia Pacific 48.29% Solids at ~125°C



Viscometer Test Run 960503M1 Figure No. 523a

Micro Motion with Georgia Pacific 55.84% Solids at ~55°C



Viscometer Test Run 960503M1 Figure No. 523b

Micro Motion with Georgia Pacific 55.84% Solids at ~55°C



Viscometer Test Run 960503M1 Figure No. 523c

Micro Motion Density with Georgia Pacific 55.84% Solids at ~55°C



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Viscometer Test Run 960503M1 Figure No. 523d

Micro Motion Density with Georgia Pacific 55.84% Solids at ~55°C



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Viscometer Test Run 960503M2 Figure No. 524a

Micro Motion with Georgia Pacific 55.84% Solids at ~75°C



Viscometer Test Run 960503M2 Figure No. 524b

Micro Motion with Georgia Pacific 55.84% Solids at ~75°C



Viscometer Test Run 960503M2 Figure No. 524c

Micro Motion Density with Georgia Pacific 55.84% Solids at ~75°C



Viscometer Test Run 960503M2 Figure No. 524d

Micro Motion Density with Georgia Pacific 55.84% Solids at ~75°C



Viscometer Test Run 960504M3 Figure No. 525a

Micro Motion with Georgia Pacific 55.84% Solids at ~85°C



Viscometer Test Run 960504M3 Figure No. 525b

Micro Motion with Georgia Pacific 55.84% Solids at ~85°C



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Viscometer Test Run 960504M3 Figure No. 525c

Micro Motion Density with Georgia Pacific 55.84% Solids at ~85°C



Viscometer Test Run 960504M3 Figure No. 525d

Micro Motion with Georgia Pacific 55.84% Solids at ~85°C



Viscometer Test Run 960211M1 Figure No. 526a

Micro Motion with Georgia Pacific 58.38% Solids at ~60°C



Viscometer Test Run 960211M1 Figure No. 526b

Micro Motion with Georgia Pacific 58.38% Solids at ~60°C



Viscometer Test Run 960211M1 Figure No. 526c

Micro Motion Density with Georgia Pacific 58.38% Solids at ~60°C



Viscometer Test Run 960211M1 Figure No. 526d

Micro Motion Density with Georgia Pacific 58.38% Solids at ~60°C



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Viscometer Test Run 960213M2 Figure No. 527a

Micro Motion with Georgia Pacific 58.38% Solids at ~75°C



Viscometer Test Run 960213M2 Figure No. 527b

Micro Motion with Georgia Pacific 58.38% Solids at ~75°C



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Viscometer Test Run 960213M2 Figure No. 527c

Micro Motion Density with Georgia Pacific 58.38% Solids at ~75°C



Viscometer Test Run 960213M2 Figure No. 527d

Micro Motion Density with Georgia Pacific 58.38% Solids at ~75°C



Viscometer Test Run 960213M3 Figure No. 528a

Micro Motion with Georgia Pacific 58.38% Solids at ~90°C



Viscometer Test Run 960213M3 Figure No. 528b

Micro Motion with Georgia Pacific 58.38% Solids at ~90°C



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Viscometer Test Run 960213M3 Figure No. 528c

Micro Motion Density with Georgia Pacific 58.38% Solids at ~90°C



Viscometer Test Run 960213M3 Figure No. 528d

Micro Motion Density with Georgia Pacific 58.38% Solids at ~90°C



Viscometer Test Run 960213M4 Figure No. 529a

Micro Motion with Georgia Pacific 58.38% Solids at ~120°C



Viscometer Test Run 960213M4 Figure No. 529b

Micro Motion with Georgia Pacific 58.38% Solids at ~120°C



Viscometer Test Run 960213M4 Figure No. 529c

Micro Motion Density with Georgia Pacific 58.38% Solids at ~120°C



Viscometer Test Run 960213M4 Figure No. 529d

Micro Motion Density with Georgia Pacific 58.38% Solids at ~120°C



Viscometer Test Run 960520M1 Figure No. 530a

Micro Motion with Georgia Pacific 65.51% Solids at ~80°C



Viscometer Test Run 960520M1 Figure No. 530b

Micro Motion with Georgia Pacific 65.51% Solids at ~80°C



Viscometer Test Run 960520M1 Figure No. 530c

Micro Motion Density with Georgia Pacific 65.51% Solids at ~80°C



Viscometer Test Run 960520M1 Figure No. 530d

Micro Motion Density with Georgia Pacific 65.51% Solids at ~80°C



Viscometer Test Run 960520M2 Figure No. 531a

Micro Motion with Georgia Pacific 65.51% Solids at ~85°C



Viscometer Test Run 960520M2 Figure No. 531b

Micro Motion with Georgia Pacific 65.51% Solids at ~85°C



Viscometer Test Run 960520M2 Figure No. 531c

Micro Motion Density with Georgia Pacific 65.51% Solids at ~85°C


Viscometer Test Run 960520M2 Figure No. 531d

Micro Motion Density with Georgia Pacific 65.51% Solids at ~85°C



Viscometer Test Run 960520M3 Figure No. 532a

Micro Motion with Georgia Pacific 65.51% Solids at ~95°C



Viscometer Test Run 960520M3 Figure No. 532b

Micro Motion with Georgia Pacific 65.51% Solids at ~95°C



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Viscometer Test Run 960520M3 Figure No. 532c

Micro Motion Density with Georgia Pacific 65.51% Solids at ~95°C



Viscometer Test Run 960520M3 Figure No. 532d

Micro Motion Density with Georgia Pacific 65.51% Solids at ~95°C

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Viscometer Test Run 960521M4 Figure No. 533a

Micro Motion with Georgia Pacific 65.51% Solids at ~125°C



Viscometer Test Run 960521M4 Figure No. 533b

Micro Motion with Georgia Pacific 65.51% Solids at ~125°C



Viscometer Test Run 960521M4 Figure No. 533c

Micro Motion Density with Georgia Pacific 65.51% Solids at ~125°C



Viscometer Test Run 960521M4 Figure No. 533d

Micro Motion Density with Georgia Pacific 65.51% Solids at ~125°C



Viscometer Test Run 960301M1 Figure No. 534a

Micro Motion with Georgia Pacific 68.39% Solids at ~90°C



Viscometer Test Run 960301M1 Figure No. 534b

Micro Motion with Georgia Pacific 68.39% Solids at ~90°C



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Viscometer Test Run 960301M1 Figure No. 534c

Micro Motion Density with Georgia Pacific 68.39% Solids at ~90°C



Viscometer Test Run 960301M1 Figure No. 534d

Micro Motion Density with Georgia Pacific 68.39% Solids at ~90°C



Viscometer Test Run 960302M2 Figure No. 535a

Micro Motion with Georgia Pacific 68.39% Solids at ~100°C



Viscometer Test Run 960302M2 Figure No. 535b

Micro Motion with Georgia Pacific 68.39% Solids at ~100°C



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Viscometer Test Run 960302M2 Figure No. 535c

Micro Motion Density with Georgia Pacific 68.39% Solids at ~100°C



Viscometer Test Run 960302M2 Figure No. 535d

Micro Motion Density with Georgia Pacific 68.39% Solids at ~100°C



Viscometer Test Run 960302M3 Figure No. 536c

Micro Motion Density with Georgia Pacific 68.39% Solids at ~115°C



Viscometer Test Run 960302M3 Figure No. 536a

Micro Motion with Georgia Pacific 68.39% Solids at ~115°C



Viscometer Test Run 960302M3 Figure No. 536b

Micro Motion with Georgia Pacific 68.39% Solids at ~115°C



Viscometer Test Run 960302M3 Figure No. 536d

1

Micro Motion Density with Georgia Pacific 68.39% Solids at ~115°C



Viscometer Test Run 960302M4 Figure No. 537a

Micro Motion with Georgia Pacific 68.39% Solids at ~125°C



Viscometer Test Run 960302M4 Figure No. 537b

Micro Motion with Georgia Pacific 68.39% Solids at ~125°C



Viscometer Test Run 960302M4 Figure No. 537c

Micro Motion Density with Georgia Pacific 68.39% Solids at ~125°C



Viscometer Test Run 960302M4 Figure No. 537d

Micro Motion Density with Georgia Pacific 68.39% Solids at ~125°C



Viscometer Test Run 960719M1 Figure No. 538a

> Micro Motion Transient Temperature Run with GP & Canadian Forest Mix 67.46% Solids



Viscometer Test Run 960719M1 Figure No. 538b

Micro Motion Density Transient Temperature Run with GP & Canadian Forest Mix 67.46% Solids



Viscometer Test Run 960719M2 Figure No. 539a

Micro Motion with GP & Canadian Forest Mix 67.46% Solids ~100°C



Viscometer Test Run 960719M2 Figure No. 539b

Micro Motion with GP & Canadian Forest Mix 67.46% Solids ~100°C



Viscometer Test Run 960719M2 Figure No. 539c

> Micro Motion Density with GP & Canadian Forest Mix 67.46% Solids ~100°C



Viscometer Test Run 960719M2 Figure No. 539d





Viscometer Test Run 960719M3 Figure No. 540a

Micro Motion with GP & Canadian Forest Mix 67.46% Solids ~105°C



Viscometer Test Run 960719M3 Figure No. 540b

Micro Motion with GP & Canadian Forest Mix 67.46% Solids ~105°C



Viscometer Test Run 960719M3 Figure No. 540c

> Micro Motion Density with GP & Canadian Forest Mix 67.46% Solids ~105°C



Viscometer Test Run 960719M3 Figure No. 540d





Viscometer Test Run 960720M4 Figure No. 541a

Micro Motion with GP & Canadian Forest Mix 67.46% Solids at ~140°C



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Viscometer Test Run 960720M4 Figure No. 541b

Micro Motion with GP & Canadian Forest Mix 67.46% Solids at ~140°C



Viscometer Test Run 960720M4 Figure No. 541c

> Micro Motion Density with GP & Canadian Forest Mix 67.46% Solids at ~140°C



Viscometer Test Run 960720M4 Figure No. 541d

> Micro Motion Density with GP & Canadian Forest Mix 67.46% Solids at ~140°C



Viscometer Test Run 960720L5 Figure No. 542



Viscometer Test Run 960726M1 Figure No. 543a

Micro Motion with GP & Canadian Forest Mix 71.38% Solids ~100°C



Viscometer Test Run 960726M1 Figure No. 543b

Micro Motion with GP & Canadian Forest Mix 71.38% Solids ~100°C



Viscometer Test Run 960726M1 Figure No. 543c

> Micro Motion Density with GP & Canadian Forest Mix 71.38% Solids ~100°C



Viscometer Test Run 960726M1 Figure No. 543d





Viscometer Test Run 960726M2 Figure No. 544a

Micro Motion with GP & Canadian Forest Mix 71.38% Solids ~125°C



Viscometer Test Run 960726M2 Figure No. 544b

Micro Motion with GP & Canadian Forest Mix 71.38% Solids ~125°C



Viscometer Test Run 960726M2 Figure No. 544c





Viscometer Test Run 960726M2 Figure No. 544d

> Micro Motion Density with GP & Canadian Forest Mix 71.38% Solids ~125°C



Viscometer Test Run 960726L4 Figure No. 545

Micro Motion Transient Temperature Run with GP & Canadian Forest Mix 71.38% Solids



Viscometer Test Run 960726L5 Figure No. 546

Micro Motion Transient Temperature Run with GP & Canadian Forest Mix 71.38% Solids



Viscometer Test Run 960801M1 Figure No. 547

Micro Motion with GP & Canadian Forest Mix 73.40% Solids ~135°C



Viscometer Test Run 960801L2 Figure No. 548

Micro Motion Transient Temperature Run with GP & Canadian Forest Mix 73.40% Solids 900.00 Micro Motion (cP) 800.00-Lab Reference (cP) 700.00--0 TC2 12% Open 0 (a) 500.00-500.00-500.00-300.00-TC2 20% Open TC2 100% Open Δ 300.00-200.00-100.00-0.00-45 50 15 20 25 30 35 40 5 0 10 Time (min)

5 NAMETRE TORSIONAL VIBRATORY VISCOMETER

The specific configuration of the Nametre Viscoliner Torisional Vibratory Viscometer tested is as follows.

- Sensor: Cylindrical Torsional Vibratory Viscometer
- Instrument: 1810MX Viscoliner

The Nametre Viscoliner is an On-Line Torsional Oscillatory Viscometer in which the shear wave created by the mechanical oscillation of the sensor arm is related to the viscous loss of the material and provides a measure of the viscosity of the fluid. The fluid enters the measuring chamber and fully envelopes the sensor arm which is vibrating at a constant amplitude and frequency. The power required to maintain this reference oscillation is related to the product of viscosity and density (Nametre, 1973; Nametre, 1991; Nametre, 1995).

5.1 Principles of Operation

The viscometer is comprised of three parts: The sensor, transducer, and control unit. The sensor or torsional member is a hollow shank with an enlarged hollow cylindrical end that provides a relatively large surface area for creating the shear wave, although the mass of the end of the sensor must be small enough for the rapid thermal stabilization that is necessary for accurate measurements. A magnetic coil causes the sensor to oscillate and the amplitude of oscillation against the damping effects of the sample fluid is sensed by the transducer and is converted into a DC voltage. This signal is then compared to a reference signal of the sensor oscillating in a vacuum or a low viscosity fluid, such as air, and the error signal generated is used to control the gain on the magnetic coil to maintain a set oscillatory amplitude and frequency (Nametre, 1973; Nametre, 1991).

$$\eta \rho = k(i - i_0)^2 \tag{5.1}$$

where

$$k =$$
 proportionality constant
 $i =$ current through the driver coil
 $i_0 =$ reference current through the driver coil

During the installation of the viscometer, zero calibration in air is performed at the process operating temperature or, in the case of the pilot flow loop where temperature is one of the variables, $\eta \rho$ as a function of temperature in air is determined. Although the air zero correction as a function of temperature may be significant at low viscosity, it is insignificant at viscosities above 30 cP. At lower viscosities the affect of temperature on the zero (reading in air) can be corrected for by using the correlation Equation 5.2.

$$Error(\eta \rho) = 3 * 10^{-5} T(^{\circ}C)^{2} - 0.0058T(^{\circ}C) + 0.3442$$
(5.2)

The control unit receives the output from the transducer and displays a fluid temperature reading, as there is an RTD located at the end of the sensor, and $\eta\rho$ reading. This control unit is capable of handling up to four sensors and a 4-20 mA densitometer input to determine an absolute viscosity.

With additional sensors, this viscometer can be used with non-Newtonian fluids, where the viscosity varies with shear rate, by setting the various transducers to different frequencies. Any set viscometer relates to shear rate by

$$\dot{\gamma} = 2\pi f \tag{5.3}$$

where f is the frequency of mechanical oscillation. Though it maybe possible to have a Nametre Viscometer with variable frequencies with one transducer, the 1810MX does not have this option and it would be important to examine the residence time between frequency changes as long transients would make process control difficult.

5.2 Experimental Overview

Table 5.1 lists the solids composition of black liquors used in the experimental runs that were performed with the Nametre Viscometer. In total, the instrument was tested on 14 different black liquor concentrations, ranging from a 34.14% Georgia Pacific liquor to a 73.40% liquor obtained by mixing a Georgia Pacific liquor with Canadian Forest liquor.

Georgia Pacific	Canadian Forest	Chesapeake	GP & Canadian Forest Mix
34.14%	47.96%	49.39%	67.72%
42.24%	57.73%	59.58%	71.38%
48.29%			73.40%
55.84%			
58.38%			
65.51%			
68.38%			

Table 5.1. Black liquor types and solids content tested with the Nametre Viscometer.

For installation in the pilot flow loop, a containment vessel was manufactured for the Nametre sensor. The black liquor enters the bottom of the containment vessel through a 1" flex hose, and then against an deflection plate that diverts the flow, envelops the Nametre sensor; afterwards, and exits through a side port at the top of the containment vessel. The deflection plate was designed to prevent any debris in the flow from striking and damaging the sensor. The sensor is isolated from high frequency vibrations from the pilot flow loop through the use of flex hoses on the input and output ports on the containment vessels. Since this instrument operates on the principles of a vibrating rod viscometer, high frequency vibrations may interfere with the vibrations of the probe. There has not been any evidence of any high frequency interference from the day-to-day operation of the pilot flow loop.

The control unit has two output signals, the product of $\eta \rho$ and temperature, which are transmitted as 4-20 mA signals into the UF DAQ (University of Florida Data

Acquisition) System. For most of the experimental trials, the internal RTD with the Nametre Viscometer was used as the reference temperature and this closely matched the upstream Viscometer Line RTD TT4 (*cf.* P&ID diagram in Figure 9.1) reading. The internal RTD experienced some malfunctions in the later runs; therefore, the upstream Viscometer Line TT4 was used as the reference temperature for later runs. The malfunction of the internal RTD has no affect on the viscosity measurement of the Nametre Viscometer.

The laboratory reference density, as a function of the viscometer line temperature, is used to calculate the absolute viscosity determined by the Nametre Viscometer off-line. The control unit require a minimal interaction with the operator during the experimental trials, except for setting the initial auto zero and possibly reranging the retransmission outputs. Overall, the instrument is very self reliant operationally.

5.3 Selected Experimental Results

The selected data presented in this section represent typical results for the Nametre instrument. The data presented is comprised of 5 steady-state runs of viscosity versus flow rate at different temperatures and solids compositions, and 1 transient response of the instrument to temperature changes.

The Figure 5.1 shows a low viscosity run with Canadian Forest 47.96% solids black liquor at approximately 50°C. This is a steady-state run, and the results are presented on two plots. The upper plot shows the viscosity values measured by the instrument, and are indicated by the diamond-shaped markers. The continuous line joining the markers is included as a visual aid to track the data trend, and is obtained through arbitrary curve-fits. The upper plot also shows a laboratory reference band of viscosity values defined by two dash-dot lines. The band is constructed using viscosity correlations produced for the various laboratory instruments described in Section 2.4. In particular, the upper curve of the band corresponds to the highest viscosity prediction produced when considering all the laboratory correlations at the temperature of the flow rate in question. Analogously, the lower curve of the band corresponds to the lowest viscosity prediction. Any measurements that fall inside the reference band are considered to be consistent with the laboratory measurements.

The lower plot in Figure 5.1 is a measure of the relative difference between the viscosity measurement (η) produced by the instrument and the viscosity (η_{ref}) predicted by a correlation produced using data acquired with a reliable laboratory instrument and valid for the particular solids-content of the black liquor used in the test. Hence, the relative difference is defined as follows:

Relative difference =
$$\frac{\eta - \eta_{ref}}{\eta_{ref}}$$
 (5.4)

We typically used a Haake Rotovisco RV12 Rotational Viscometer or a Cambridge Sliding Element Laboratory Viscometer as the reference laboratory instrument. Note that, for example, a relative difference of 0.08 indicates that the instrument viscosity differs from the reference measurement by 8%. We consider an instrument to be accurate if it realizes a relative difference of $\pm 10\%$. A relevant remark concerning this figure is that the temperature is not strictly constant during the run; in fact, as the flow rate varies, the temperature also varies, though it remains close to the approximate value reported in the figure title. For this particular figure the viscosity, flow-rate, and temperature information for 10 selected flow rates are listed in Figure 5.2.

Flow Rate (gpm)	Temperature (°C)	Viscosity (cP)
0.65	46.99	23.07
0.65	46.99	23.19
1.25	46.99	23.43
1.24	47.08	23.44
1.15	47.35	23.07
1.14	47.26	23.07
1.95	47.72	22.47
1.95	47.72	22.47
0.00	47.45	23.20
0.00	47.45	23.20

Table 5.2. Flow-rate, temperature, and viscosity data for Figure 5.1.

Analysis of Figure 5.1 shows that the Nametre Viscometer produces measurements with excellent accuracy because the measured response from the instrument lie within the boundary of the laboratory reference band. Note that the instrument tracks well with the changes in viscosity that occur due to the change of the temperature caused with the variation of flow rates. The lower plot was generated using the laboratory reference correlation

$$\eta_{ref}(cP) = \frac{\exp(5.4020648 - 5537.289/T(K) + 1551100.6/T(K)^2)}{(1 - error)}$$
(5.5)

where
$$error = 4.240718 - 0.02276T(K) + 0.0000311T(K)^2$$
 (5.6)

The Nametre Viscometer measures a product of $\eta \rho$. The laboratory density correlation as a function of temperature was used to determine an absolute viscosity

$$\rho(g/mL) = 1.3082518 - 0.00057992949T(^{\circ}C) - 1.0614077 \cdot 10^{(-6)}T(^{\circ}C)$$
(5.7)

With a viscosity of about 22-25 cP, the instrument tracked well within the laboratory reference boundary with a relative difference of less than +5% and was unaffected by the sample flow rate.

Figure 5.2 is another low viscosity run with the Canadian Forest 47.96% solids at approximately 30°C. With the viscosity about 54-62 cP, the Nametre Viscometer tracked on the upper boundary of the laboratory reference viscosity with a relative difference of +5%. Again, the instrument was unaffected by flow rate.

In Figure 5.3, the viscosity range of the sample flow is similar to the previous figure, except this is with Canadian Forest 57.73% solids black liquor at approximately 80°C. The Nametre Viscometer measurements were above the laboratory reference boundary with a relative difference greater than +60%. Build up along the sensor was suspected in causing the large offset in the measured viscosity. Due to the limitations of the Pilot Flow Loop, the flow rate in the Viscometer Line could not be increased above 3.5 gpm.

The conditions for the last figure were repeated after the viscometer line was steam purged. To steam clean the Nametre Viscometer, a 100 psig steam line is connected to the sample point and the Valve V3 (*cf.* P&ID diagram in Figure 9.1) is opened with the main pump turned off and the Valve V20 (*cf.* P&ID diagram in Figure 9.1) was closed. The steam purges the black liquor from the viscometer line and back into the main holding tank. The steam is left on until the Nametre $\eta \rho$ measurement is close to or below zero. Afterwards, the main pump is started and the flow is passed through the Viscometer Line. The Viscometer Line is allowed to thermally equilibrate quickly and the experimental run is started. After the steam purge, Figure 5.4 shows that the Nametre viscosity measurement is at the lower laboratory reference boundary with a relative difference of -20%.

Figure 5.5 is a high viscosity run with Georgia Pacific 68.39% solids black liquor at approximately 85°C. In this experimental run, the viscosity of the black liquor is about 280-310 cP and the instrument is at the lower laboratory reference boundary with a relative difference of -5%. In this case, the sample flow rate is restricted to about 1.8 gpm because of the limitations of the pilot flow loop.

Figure 5.6 plots viscosity as a function of time to examine the transient response of the Nametre Viscometer with a GP and Canadian Forest mixed black liquor at 71.38% solids. At time zero, the Viscometer Line is at a thermal and flow rate steady-state. In this experiment the temperature of the flowing black liquor steam was changed in a step-fashion by manually opening the steam valve controlled by the Process Heater Output Controller (instrument TC2 in the P&ID diagram in Figure 9.1). The step changes were made in discrete increments, as shown in the legend of the figure, until the output from the Heater became saturated. The four markers (circle, square, triangle, and cross) on the figure denote the instants when the valve opening is changed. The dotted line represents the viscosity determined using a laboratory correlation of viscosity versus temperature, and the continuous line represents the measurement by the instrument. Further details on the execution of this may be found in Section 2.1. Note that in Figure 4.5 at time zero, the Viscometer Line is at a thermal and flow rate steady-state. For the first step change (TC2 is set to 15% open), there is a 2 minute time delay before the Viscometer Line RTD TT4 (cf. P&ID diagram in Figure 9.1) responses to the step input. The Nametre Viscometer has a delay of approximately 2 minutes from the step response in viscosity within the Viscometer Line. For the second step input (TC2 is set to 20% open), the time-delay of the Nametre Viscometer is approximately 3 minutes from the Viscometer Line RTD TT4 temperature response. From examining the transients of the Nametre Viscometer, the time constant for this instrument appears to be the longest of those tested, but is still reasonably short for even a major change in a process variable (the temperature).

With the current Nametre Viscometer setup in the pilot flow loop, fouling builds up on the ledge where the hollow shank joins the cylindrical probe. The location of the fouling has been verified physically by disassembling the containment vessel on several occasions. The fouling appears to be a problem with black liquors above about 50% solids. The first two figures demonstrate that the instrument is capable of operating in low viscosity ranges; however, at equal viscosities at higher solids, the fouling caused

erroneous viscosity measurements. This trend continued throughout the high solids and low viscosity trials. Figure 5.5 is typical of a high solids and high viscosity run where the amount of fouling was insignificant when compared with the viscosity of the sample and did not affect the viscosity measurement. At low viscosities, the amount of fouling is significant, and this gives a high error in viscosity as the additional mass on the probe presents a false measure of fluid resistance. Fouling accumulated rapidly on the probe after steam cleaning the Nametre Viscometer and there was only a window of an about hour to complete a run before fouling again affected the viscosity measurement.

Steam purging the viscometer line is adequate to clean the sensor for the most part; however, the performance was not completely restored in every case. The source of some of the error and erratic measurements after the steam purging may be traced to the fact that fouling occurs very rapidly and that since the experimental runs needed to be performed soon after the steam purge, that the instrument may not have had adequate time to thermally equilibrate.

One must keep in mind that the design of the containment vessel is for the pilot flow loop only and that the flow pattern enveloping the probe is restricted by the location of the inlet port, the defection plate, and the flow rate of the black liquor. Through minor modifications of the installation of the Nametre sensor and increasing the sample flow rate, the stagnant flow areas above the cylindrical probe could be eliminated. The flow pattern could reduce or remove any fouling that might build up on the sensor. In addition, streamlining the sensor geometry will further reduce the stagnant flow areas around the probe and prevent or greatly reduce fouling; moreover, this modification could increase the operational range by allowing the instrument to operate under low flow conditions.



Figure 5.1 Nametre Viscometer measured response with Canadian Forest 47.96% Solids Black Liquor at ~50°C.



3.0



Figure 5.2 Nametre Viscometer measured response with Canadian Forest 47.96% Solids Black Liquor at ~30°C.

70.0

60.0

50.0

Viscosity (cP) 30.0

20.0

10.0

0.0



Figure 5.3 Nametre Viscometer measured response with Canadian Forest 57.73% Solids Black Liquor at ~80°C.



Figure 5.4 Nametre Viscometer measured response with Canadian Forest 57.73% Solids Black Liquor at ~80°C after the Viscometer Line has been steam purged.



Figure 5.5 Nametre Viscometer measured response with Georgia Pacific 68.39% Solids Black Liquor at ~85°C.



Figure 5.6 Nametre Viscometer tranisent temperature run with GP and Canadian Forest Mix 71.38% Solids Black Liquor.

5.4 Summary of Observations

The general trends observed with the Nametre Viscometer are summarized as follows:

- The Nametre Viscometer tracked within ±10% of the laboratory reference at high viscosity (> 175 cP) or at low viscosities and low solids (< 50% solids).
- The instrument was unaffected by particles smaller than 40 mesh.
- The instrument was affected by second phases such as air entrainment.
- The instrument was unaffected by the flow rates achieved in the Viscometer Line; regulating the flow in a slip stream should not be a concern.
- The instrument provides a direct measure of force.

- With the current installation in the pilot flow loop, the instrument was subject to fouling at high solids.
- The instrument measures a product of ηρ and a density input is required to determine absolute viscosity.

The Nametre Viscoliner is capable of measurement for a wide range of viscosity from 10 cP up to 1000 cP. In the pilot flow loop, the instrument was used to measure viscosities up to 600 cP. Once the unit has been auto zeroed in the installation phase, the instrument did not require recalibration throughout the experimental trials. For use in an environment where there is a wide operating temperature range, the instrument requires a temperature zero correction, especially when utilized for low viscosity processes. At high viscosities, the temperature zero correction is insignificant.

The problem with fouling on the Nametre sensor can more than likely be eliminated by proper installation of the probe so as to reduce any stagnant flow areas and by increasing the sample line flow rate. The instrument reading was unaffected by the flow rates generated in the pilot flow loop and increasing the flow rate in a slip stream may reduce the fouling on the sensor. A further improvement would include streamlining the probe so as to reduce the stagnant flow area for fouling build up. Fouling always occurred at the same location, which is on the small ledge where the cylindrical probe end joins the hollow sensor arm. The Teflon coating on the sensor did not show any signs of damage or wear, and there was no evidence of fouling on the sides or bottom of probe.

The control unit is capable of handling up to four sensors and an on-line densitometer. The instrument could be useful for non-Newtonian fluids, if multiple sensor operating at the same temperature, but different oscillatory frequencies, were utilized. With the addition of a densitometer, an absolute viscosity can be determined on-line. Since density is not as strong a function of temperature as is viscosity, an absolute viscosity

measurement may not be necessary for all applications. In those cases, a densitometer may not be necessary or the density could be estimated from correlations.

The recommendations based on experience with the Nametre Viscometer are summarized as follows:

- Use in a slip stream for the protection of the probe and also to provide easy access to the sensor for inspection and cleaning.
- Properly install to reduce stagnant flow areas around the probe.
- Streamline the probe so as to reduce the area for fouling to build up.
- With the removal of the potential of fouling on the sensor, the instrument is capable of satisfactorily operating over the expected viscosity ranges to be encountered in a mill.

5.5 Extensive Experimental Results

Included in this section are the graphical representations of the results of the on-line viscometer trials performed with the Nametre instrument. The figures describes the data gathered for all the black liquors listed in Table 5.1. Figures labeled as "a" represent the viscosity as measured by the instrument as a function of flow rate and the figures labeled as "b" show the relative difference of the measured response from the laboratory reference as a function of flow rate. Laboratory data on density determined from measurements of density near ambient temperatures and thermal expansion data for the liquor were used to determine viscosity from the instrument readings.

Figures 600-604 represent results for Canadian Forest 47.96% Solids at 30 to 120°C (viscosities from 3 to 60 cP), at varying flow rates. The instrument demonstrated excellent tracking and accuracy, and flow rates up to 3 gpm through this viscometer did not affect the measurement.

Figures 605-609 represent similar results for Canadian Forest 57.73% Solids at temperatures from 50 to 125°C (viscosities from 20 to 240 cP), and at different flow rates. At 50°C and 55°C, the instrument data track viscosity well, shows no effect of flow rate, and show good precision (Figure 605 and 607). The data at 80°C show a large relative error and were more erratic (Figure 607). The on-line value of viscosity is always well above the value of viscosity determined in the laboratory, and the result is even worse at 125°C (Figure 608). Note the erratic behavior that is reflected very clearly in the relative difference plot. The Viscometer Line was drained and steam purged and then data again taken at 80°C again. Comparing the results given in Figures 607 with those given in Figures 609, it can be seen that the viscosity readings track the viscosity much better, readings are much less erratic, and the relative difference is nearly within acceptable limits.

Both Canadian Forest and Chesapeake liquors had precipitated solids present in the 48-58% range. Several times, there was build up of solids on the active element with some of the liquors tested. When the viscometer chamber was isolated and drained and the viscometer opened for inspection, the solids deposits were always in the stagnant flow area behind the cylinder; there was never any evidence of deposit on the sides of the cylinder nor on the upstream end. Build up seems to result in erratic readings from the viscometer, as can be seen from the relative difference graphs. Steam purging was successful in removing the deposits and in returning the operation of the viscometer to normal. The RTD in the instrument failed and was not replaced. This was not serious, since we had determined that the RTD reading in the fluid stream and the RTD reading for the instrument were nearly identical.

Figures 610-613 represent results for Chesapeake Paper 49.39% Solids at temperatures from 35 to 120°C (viscosities from 5 to 100 cP), and at different flow rates. This liquor was noticeably "dirtier" than the Canadian Forest liquor. Performance at 35°C and 50°C was very good (Figures 610 and 611). Performance at 80°C began to show the effects of deposits (Figure 612). Note that the readings were more erratic and that the

measured viscosity was much higher than the laboratory standard. The results for this liquor at 120°C are worse (Figure 613). Part of the problem may have been problems with the data acquisition system in the pilot plant. However, the high readings and erratic behavior are characteristic of results obtained when there is a build up of solids at the back of the active element. The viscometer was opened and inspected after this data was taken, and the active element was clean, except for the area at the back of the cylinder. Nearly all of this data was taken with a 40 mesh screen in place upstream of the viscometer. While the build up on the sensor is a problem, the build up can be detected from erratic reading behavior and the active element probably can be steam cleaned and certainly can be cleaned by steaming followed by a hot water wash.

Figures 614-616 represent results for Chesapeake Paper 59.28% Solids at temperatures from 65 to 90°C (viscosities from 40 to 250 cP), and at different flow rates. Tracking of viscosity is good up to 90°C (Figures 614 and 615), but the data at 90°C again shows evidence of buildup on the active element by the more erratic readings (Figure 616). Also, as the temperature was increased, the relative differences increased. Data were not taken at temperatures above 90°C, because of the evidence of build up of deposits on the back of the element.

Figures 617-622 represent results for Georgia-Pacific 42.20% Solids at temperatures from 35 to 120C (viscosities from 2 to 20 cP), and at different flow rates. The viscometer tracked viscosity change very well for data taken at temperatures from 35-100°C; however, the relative difference ranged from 12 to 90% (Figures 617-621). Currently, there is no explanation for this. Data at 120°C shows some evidence of erratic readings that could be the result of some deposit, and the viscometer does not track the viscosity very well, but the viscosity was very low (Figure 622).

Figures 623-626 represent results for Georgia Pacific 48.29% Solids at temperatures from 30 to 125°C (viscosities from 3.5 to 72 cP), and at different flow rates. The results at 30-85°C are generally acceptable for tracking and reproducibility with

marginal to good accuracy (Figures623-625). At 125°C, the instrument tracked viscosity well, but reproducibility was not as good for this test and the relative difference was more than 10%; however, the viscosity is low (Figure 626).

Figures 627-629 represent results for Georgia Pacific 55.84% Solids at temperatures from 60 to 90°C (viscosities from 25 to 190 cP), and at different flow rates. At 60°C and 75°C, the instrument tracked well with acceptable or marginally acceptable relative differences (Figures 627 and 628). At 90°C, the readings were erratic with a large relative error (Figure 629). This may have been due to solids build up on the active element; in addition, there were some problems with the UF DAQ system in the pilot plant during these runs.

Figures 630-633 represent results for Georgia-Pacific 58.38% Solids at temperatures from 60 to 125°C (viscosities from 10 to 210 cP), and at different flow rates. Data taken at 60°C were excellent (Figure 630). There was very good tracking, accuracy was acceptable, and there was no noticeable effect of flow rate. Data taken at 70°C were also good, tracking the viscosity with good reproducibility (Figure 631). At 85C, the viscosity tracking is good, but the relative difference was very large (Figure 632). Data at 125°C shows the effect of solids buildup on the active element; viscosity is not tracked, readings are erratic, and the relative difference is very large (Figure 633).

Figures 634-638 represent results for Georgia-Pacific 65.51% Solids at temperatures from 85 to 125°C (viscosities from 20 to 260 cP), and at different flow rates. At 85-95°C, the instrument tracked well with no effect of flow rate(Figures 634-636). The relative difference was small at 85°C, but large and positive at 90°C and 95°C. At 125°C, the error was very large (Figure 637). In this latter case, the instrument gave erratic and high readings even after steam purging (Figure 638). The fouling occurs very quickly once the sensor is immersed in the liquor which may explain the previous results.

Figures 639-643 represent results for Georgia-Pacific 68.38% Solids at temperatures from 85 to 125°C (viscosities from 40 to 330 cP) and at different flow rates.

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Data at 85°C and 95°C were very good, tracking viscosity very well with no effect of flow rate, good reproducibility, and small relative error (Figures 639 and 640). At 115°C, the instrument tracked viscosity well, but the relative error was too high (Figure 641). At 125°C, the data began to be erratic and the viscosity was not tracked as well as at lower temperatures (Figure 642). Also, the relative difference was large and positive. The viscometer was purged with steam extensively, washed out with hot water, and data again taken at 125°C. The results are shown in Figures 643. Viscosity tracking has been improved, the reproducibility was restored, and the relative difference has been reduced markedly.

Figures 644-648 represent results for Georgia Pacific and Canadian Forest Mix 67.46% Solids at temperatures from 110 to 140°C (viscosities from 100 to 500 cP) and at different flow rates. The data gathered represent both transient and steady state temperature runs with this instrument. At 100°C and 105°C, the instrument had good tracking, although the relative difference was high at 30% above the laboratory reference (Figures 645 and 646). At 140°C, there was a good amount of scatter in the data that was probably due to the difficulty in maintaining thermal stability in the Viscometer Line, especially at low flow rates (Figure 647). The instrument displayed a large positive relative difference. Figure 644 pots viscosity as a function of time to examine the performance of the instrument duing transient temperature runs. During the transient response experiments, the correlation of one laboratory viscometer was used as the lab reference. The Heater (E1) was used to introduce the temperature step input into the Viscometer Line by manually opening the Process Heater Output Controller (TC2) in discrete increments. At the first step input (TC2 was opened 15%), the instrument experienced a time-delay of ~2 minutes from the Viscometer Line RTD (TT4) temperature response. There was an overall decrease in viscosity from 650 to 250 cP. For second step input (TC2 was opened 20%), there was a time-delay of ~2 minutes from the Viscometer Line RTD with a decrease in viscosity from 200 to 100 cP.

Figures 649-653 represent results for Georgia Pacific and Canadian Forest Mix 71.38% Solids at temperatures from 105 to 130°C (viscosities from 100 to 700 cP), and at different flow rates. At 105°C and 110°C, the instrument demonstrated good tracking and accuracy (Figure 649 and 651). At 130°C, the data tracked well with a 20% relative difference from the laboratory reference (Figure 650).

Figures 654 and 655 represent results for Georgia Pacific and Canadian Forest Mix 73.40% Solids at temperatures from 100 to 140°C (viscosities from 150 to 1000 cP) and at different flow rates. The data gathered represents both transient and steady state temperature runs with this instrument. At 140°C, the instrument tracked well; however, there is a very large relative difference from the laboratory reference which is probably due to build up on the sensor probe (Figure 654).

ON-LINE VISCOMETER DEVELOPMENT FOR KRAFT BLACK LIQUOR Viscometer Test Run 960130N1

Figure No. 600a

Nametre with Canadian Forest 47.96% Solids at ~30°C



Viscometer Test Run 960130N1 Figure No. 600b

Nametre with Canadian Forest 47.96% Solids at ~30°C



Viscometer Test Run 960130N2 Figure No. 601a

Nametre with Canadian Forest 47.96% Solids at ~50°C



Viscometer Test Run 960130N2 Figure No. 601b

Nametre with Canadian Forest 47.96% Solids at ~50°C



Viscometer Test Run 960130N3 Figure No. 602a

Nametre with Canadian Forest 47.96% Solids at ~75°C



Viscometer Test Run 960130N3 Figure No. 602b

Nametre with Canadian Forest 47.96% Solids at ~75°C



Viscometer Test Run 960131N4 Figure No. 603a

Nametre with Canadian Forest 47.96% Solids at ~120°C



Viscometer Test Run 960131N4 Figure No. 604b

Nametre with Canadian Forest 47.96% Solids at ~120°C



Viscometer Test Run 960409N1 Figure No. 605a

Nametre with Canadian Forest 57.73% Solids at ~50°C



Viscometer Test Run 960409N1 Figure No. 605b

Nametre with Canadian Forest 57.73% Solids at ~50°C



Viscometer Test Run 960409N2 Figure No. 606a

Nametre with Canadian Forest 57.73% Solids at ~55°C



Viscometer Test Run 960409N2 Figure No. 606b

Nametre with Canadian Forest 57.73% Solids at ~55°C



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Viscometer Test Run 960410N3 Figure No. 607a

Nametre with Canadian Forest 57.73% Solids at ~80°C



Viscometer Test Run 960410N3 Figure No. 607b

Nametre with Canadian Forest 57.73% Solids at ~80°C



Viscometer Test Run 960410N4 Figure No. 608a

Nametre with Canadian Forest 57.73% Solids at ~125°C



Viscometer Test Run 960410N4 Figure No. 608b

Nametre with Canadian Forest 57.73% Solids at ~125°C



Viscometer Test Run 960410N5 Figure No. 609a

> Nametre with Canadian Forest 57.73% Solids at ~80°C After Steam Purge



Viscometer Test Run 960410N5 Figure No. 609b

> Nametre with Canadian Forest 57.73% Solids at ~80°C After Steam Purge



Viscometer Test Run 960207N1 Figure No. 610a

Nametre with Chesapeake Paper 49.39% Solids at ~35°C



Viscometer Test Run 960207N1 Figure No. 610b

Nametre with Chesapeake Paper 49.39% Solids at ~35°C



Viscometer Test Run 960207N2 Figure No. 611a

Nametre with Chesapeake Paper 49.39% Solids at ~50°C



Viscometer Test Run 960207N2 Figure No. 611b

Nametre with Chesapeake Paper 49.39% Solids at ~50°C



Viscometer Test Run 960208N3 Figure No. 612a

Nametre with Chesapeake Paper 49.39% Solids at ~80°C



Viscometer Test Run 960208N3 Figure No. 612b

Nametre with Chesapeake Paper 49.39% Solids at ~80°C



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Viscometer Test Run 960208N4 Figure No. 613a

Nametre with Chesapeake Paper 49.39% Solids at ~120°C



Viscometer Test Run 960208N4 Figure No. 613b

Nametre with Chesapeake Paper 49.39% Solids at ~120°C



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Viscometer Test Run 960217N1 Figure No. 614a

Nametre with Chesapeake Paper 59.28% Solids at ~65°C



Viscometer Test Run 960217N1 Figure No. 614b

Nametre with Chesapeake Paper 59.28% Solids at ~65°C



Viscometer Test Run 960217N2 Figure No. 615a

Nametre with Chesapeake Paper 59.28% Solids at ~80°C



Viscometer Test Run 960217N2 Figure No. 615b

Nametre with Chesapeake Paper 59.28% Solids at ~80°C



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Viscometer Test Run 960219N3 Figure No. 616a

Nametre with Chesapeake Paper 59.28% Solids at ~90°C



Viscometer Test Run 960219N3 Figure No. 616b

Nametre with Chesapeake Paper 59.28% Solids at ~90°C



Viscometer Test Run 950903N1 Figure No. 617a

Nametre with Georgia Pacific 42.20% Solids at ~35°C



Figure No. 617b Viscometer Test Run 950903N1

Nametre with Georgia Pacific 42.20% Solids at ~35°C


Figure No. 618a Viscometer Test Run 950905N2

Nametre with Georgia Pacific 42.20% Solids at ~55°C



Figure No. 618b Viscometer Test Run 950905N2

Nametre with Georgia Pacific 42.20% Solids at ~55°C



Figure No. 619a Viscometer Test Run 950906N3

Nametre with Georgia Pacific 42.20% Solids at ~90°C



Figure No. 619b Viscometer Test Run 950906N3

Nametre with Georgia Pacific 42.20% Solids at ~90°C



Figure No. 620a Viscometer Test Run 950907N4

Nametre with Georgia Pacific 42.20% Solids at ~100°C



Figure No. 620b Viscometer Test Run 950907N4

Nametre with Georgia Pacific 42.20% Solids at ~100°C



Figure No. 621a Viscometer Test Run 951110N5

Nametre with Georgia Pacific 42.20% Solids at ~60°C



Figure No. 621b Viscometer Test Run 951110N5

Nametre with Georgia Pacific 42.20% Solids at ~60°C



Figure No. 622a Viscometer Test Run 951215N5

Nametre with Georgia Pacific 42.20% Solids at ~120°C



Figure No. 622b Viscometer Test Run 951215N5

Nametre with Georgia Pacific 42.20% Solids at ~120°C



Viscometer Test Run 960113N1 Figure No. 623a

Nametre with Georgia Pacific 48.29% Solids at ~30°C



Viscometer Test Run 960113N1 Figure No. 623b

Nametre with Georgia Pacific 48.29% Solids at ~30°C



Viscometer Test Run 960113N2 Figure No. 624a

Nametre with Georgia Pacific 48.29% Solids at ~60°C



Viscometer Test Run 960113N2 Figure No. 624b

Nametre with Georgia Pacific 48.29% Solids at ~60°C



Viscometer Test Run 960113N3 Figure No. 625a

Nametre with Georgia Pacific 48.29% Solids at ~85°C



Viscometer Test Run 960113N3 Figure No. 625b

Nametre with Georgia Pacific 48.29% Solids at ~85°C



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Viscometer Test Run 960114N4 Figure No. 626a

Nametre with Georgia Pacific 48.29% Solids at ~125°C



Viscometer Test Run 960114N4 Figure No. 626b

Nametre with Georgia Pacific 48.29% Solids at ~125°C



Viscometer Test Run 960503N1 Figure No. 627a

Nametre with Georgia Pacific 55.84% Solids at ~60°C



Viscometer Test Run 960503N1 Figure No. 627b

Nametre with Georgia Pacific 55.84% Solids at ~60°C



Viscometer Test Run 960503N2 Figure No. 628a

Nametre with Georgia Pacific 55.84% Solids at ~75°C



Viscometer Test Run 960503N2 Figure No. 628b

Nametre with Georgia Pacific 55.84% Solids at ~75°C



Viscometer Test Run 960504N3 Figure No. 629a

Nametre with Georgia Pacific 55.84% Solids at ~90°C



Viscometer Test Run 960504N3 Figure No. 629b

Nametre with Georgia Pacific 55.84% Solids at ~90°C



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Viscometer Test Run 960211N1 Figure No. 630a

Nametre with Georgia Pacific 58.38% Solids at ~60°C



Viscometer Test Run 960211N1 Figure No. 630b

Nametre with Georgia Pacific 58.38% Solids at ~60°C



Viscometer Test Run 960213N2 Figure No. 631a

Nametre with Georgia Pacific 58.38% Solids at ~70°C



Viscometer Test Run 960213N2 Figure No. 631b

Nametre with Georgia Pacific 58.38% Solids at ~70°C

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Viscometer Test Run 960213N3 Figure No. 632a

Nametre with Georgia Pacific 58.38% Solids at ~85°C



Viscometer Test Run 960213N3 Figure No. 632b

Nametre with Georgia Pacific 58.38% Solids at ~85°C



Viscometer Test Run 960213N4 Figure No. 633a

Nametre with Georgia Pacific 58.38% Solids at ~125°C



Viscometer Test Run 960213N4 Figure No. 633b

Nametre with Georgia Pacific 58.38% Solids at ~125°C



Viscometer Test Run 960520N1 Figure No. 634a

Nametre with Georgia Pacific 65.51% Solids at ~85°C



Viscometer Test Run 960520N1 Figure No. 634b

Nametre with Georgia Pacific 65.51% Solids at ~85°C



Viscometer Test Run 960520N2 Figure No. 635a

Nametre with Georgia Pacific 65.51% Solids at ~90°C



Viscometer Test Run 960520N2 Figure No. 635b

Nametre with Georgia Pacific 65.51% Solids at ~90°C


Viscometer Test Run 960520N3 Figure No. 636a

Nametre with Georgia Pacific 65.51% Solids at ~95°C



Viscometer Test Run 960520N3 Figure No. 636b

Nametre with Georgia Pacific 65.51% Solids at ~95°C



Viscometer Test Run 960521N4 Figure No. 637a

Nametre with Georgia Pacific 65.51% Solids at ~125°C



Viscometer Test Run 960521N4 Figure No. 637b

Nametre with Georgia Pacific 65.51% Solids at ~125°C



Viscometer Test Run 960522N5 Figure No. 638a

> Nametre with Georgia Pacific 65.51% Solids at ~100°C After Steam Purge







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Viscometer Test Run 960301N1 Figure No. 639a

Nametre with Georgia Pacific 68.39% Solids at ~85°C



Viscometer Test Run 960301N1 Figure No. 639b

Nametre with Georgia Pacific 68.39% Solids at ~85°C



Viscometer Test Run 960302N2 Figure No. 640a

Nametre with Georgia Pacific 68.39% Solids at ~95°C



Viscometer Test Run 960302N2 Figure No. 640b

Nametre with Georgia Pacific 68.39% Solids at ~95°C



Viscometer Test Run 960302N3 Figure No. 641a

Nametre with Georgia Pacific 68.39% Solids at ~115°C



Viscometer Test Run 960302N3 Figure No. 641b

Nametre with Georgia Pacific 68.39% Solids at ~115°C



Viscometer Test Run 960302N4 Figure No. 642a

Nametre with Georgia Pacific 68.39% Solids at ~125°C



Viscometer Test Run 960302N4 Figure No. 642b

Nametre with Georgia Pacific 68.39% Solids at ~125°C



Viscometer Test Run 960302N5 Figure No. 643a

> Nametre with Georgia Pacific 68.39% Solids at ~125°C After Steam Purge



Viscometer Test Run 960302N5 Figure No. 643b

> Nametre with Georgia Pacific 68.39% Solids at ~125°C After Steam Purge



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Viscometer Test Run 960719N1 Figure No. 644

> Nametre Transient Temperature Run with GP & Canadian Forest Mix 67.46% Solids



Viscometer Test Run 960719N2 Figure No. 645a

Nametre with GP & Canadian Forest Mix 67.46% Solids at ~105°C



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Viscometer Test Run 960719N2 Figure No. 645b

Nametre with GP & Canadian Forest Mix 67.46% Solids at ~105°C



Viscometer Test Run 960719N3 Figure No. 646a

Nametre with GP & Canadian Forest Mix 67.46% Solids at ~110°C



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Viscometer Test Run 960719N3 Figure No. 646b

Nametre with GP & Canadian Forest Mix 67.46% Solids at ~110°C



Viscometer Test Run 960720N5 Figure No. 648

> Nametre Transient Temperature Run with GP & Canadian Forest Mix 67.46% Solids



Viscometer Test Run 960720N4 Figure No. 647a

Nametre with GP & Canadian Forest Mix 67.46% Solids at ~140°C



Viscometer Test Run 960720N4 Figure No. 647b

Nametre with GP & Canadian Forest Mix 67.46% Solids at ~140°C



Viscometer Test Run 960726N1 Figure No. 649a

Nametre With GP & Canadian Forest Mix 71.38% Solids at ~105°C



Viscometer Test Run 960726N1 Figure No. 649b

Nametre With GP & Canadian Forest Mix 71.38% Solids at ~105°C



Viscometer Test Run 960726N2 Figure No. 650a

Nametre With GP & Canadian Forest Mix 71.38% Solids at ~130°C



Viscometer Test Run 960726N2 Figure No. 650b

Nametre With GP & Canadian Forest Mix 71.38% Solids at ~130°C



Viscometer Test Run 960726N3 Figure No. 651a

Nametre With GP & Canadian Forest Mix 71.38% Solids at ~110°C



Viscometer Test Run 960726N3 Figure No. 651b

Nametre With GP & Canadian Forest Mix 71.38% Solids at ~110°C



Viscometer Test Run 960730N4 Figure No. 652

> Nametre Transient Temperature Run With GP & Canadian Forest Mix 71.38% Solids



Viscometer Test Run 960730N5 Figure No. 653

> Nametre Transient Temperature Run With GP & Canadian Forest Mix 71.38% Solids

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Viscometer Test Run 960801N1 Figure No. 654a

Nametre with GP & Canadian Forest Mix 73.40% Solids at ~140°C



Viscometer Test Run 960801N1 Figure No. 654b

Nametre with GP & Canadian Forest Mix 73.40% Solids at ~140°C



Viscometer Test Run 960801N1 Figure No. 655

> Nametre Transient Temperature Run with GP & Canadian Forest Mix 73.40% Solids



6 SOUTHWEST RESEARCH INSTITUTE-QUANTUM MAGNETICS MAGNETIC RESONANCE VISCOMETER

Magnetic resonance (MR) is a non-radioactive, non-invasive, non-destructive measuring technique based on the interactions of matter with a static and radio-frequency (RF) pulsed magnetic fields. The fluid is passed through the sensor probe that contains a static magnetic field, and a pulsed RF field is applied, after which the RF signals re-emitted from the fluid are analyzed to determine viscosity from laboratory correlations (Barrall, 1996<u>a</u>; Barrall, 1996<u>b</u>; De Los Santos, 1996). The specific configuration tested is as follows:

- Sensor: Magnetic Resonance Probe
- Control Cabinet: Controller and RF Power Amplifier

This instrument is being developed jointly by Southwest Research Institute of San Antonio, TX, and Quantum Magnetics of San Diego, CA. The instrument is currently an experimental unit, and is not yet fully developed for black-liquor viscosity applications. The objective of the experiments described in this section is to provide initial black liquor viscosity data to support the development of the MR Viscometer.

6.1 Principles of Operation

The nuclei in the sample fluid are arranged in random orientation until the sample comes into contact with the static magnetic field of the MR probe, where the nuclear spins align with the field direction and develop a magnetic moment. Since the alignment with the magnetic field is not instantaneous, the alignment time is characterized by the spin-lattice relaxation time (T_1) , which is one parameter that may be used in correlating viscosity. The
parameter T_1 is also useful for determining the time the sample needs to be exposed to the magnetic field before a measurement is acquired.

Once the sample has been magnetically aligned by the permanent magnetic field, the MR probe fires a pulse of RF energy that causes the magnetic moment of the aligned sample nuclei to tip away from the direction of the constant magnetic field (B_0) . The frequency of the RF pulse depends upon the strength of the constant magnetic field and the nuclei of the sample (Barrall, 1996a; Barrall, 1996b; De Los Santos, 1996)

$$\omega_0 = \gamma B_0 \tag{6.1}$$

where

$$\omega_0 = \text{frequency of RF pulse (Larmor}
 Frequency)

 $\gamma = \text{magnetogyric ratio}$$$

The constant γ has a different value for each nuclide, and in effect, determines the MR frequency for a given applied magnetic field. The MR Viscometer employs ¹H MR, with the magnetogyric ratio of $\gamma = 2.675 \times 10^8 \frac{rad}{s \cdot T}$. At the proper Larmor Frequency, the magnetic moment is shifted 90° from its equilibrium state. After the RF pulse, the nuclei realign with B_0 at its equilibrium state, and, as this occurs, the nuclei re-emit the RF energy that was absorbed. This energy is detected by the sensor coil in the MR probe. The magnitude of the MR signal depends upon the magnitude of the magnetization at the time of the pulse. The equilibrium magnetization, M_0 , in an applied field, B_0 , is given by (Barrall, 1996a)

$$M_0 = \frac{N\gamma^2 h I (I+1) B_0}{12\pi^2 kT}$$
(6.2)

where

I = 1/2 for protons N = total number of nuclei h = Planck's constant k = Boltzmann's constant T = absolute temperature To provide for maximum detection sensitivity in a single-pulse MR experiment, the RF pulse must by of sufficient energy to cause the nuclei to be tipped 90° from the applied static magnetic field.

Of primary importance in the evaluation of any MR application is the signal-to-noise ratio (SNR). The SNR of an MR system is a function of a large number of variables and is highly configuration-dependent. For the purposes of this study, Southwest Research Institute and Quantum Magnetics took on the responsibility for adjusting the key operational variables. The reader may contact them for further information. For a given configuration, the SNR of an MR system can be represented by (Barrall, 1996<u>a</u>)

$$SNR \propto \frac{\rho V_s^{\frac{1}{2}} \omega_0^{\frac{7}{4}}}{\delta_f^{\frac{1}{2}}}$$
 (6.3)

Where ρ is the density of resonant nuclei in the sensitive region of the system; V_s is the volume of the sensitive region of the instrument, which is assumed to be filled with the material under study; and δ_f is the bandwidth of he MR receiver. Due to the strong dependence of the SNR on ω_0 , it is desirable to operate at as high a field as is practical since higher field entails increased cost, while observing a nuclide with a relatively large magnetogyric ratio. Since γ for hydrogen nuclei is among the largest of the magnetogyric ratios and black liquor contains a high density of hydrogen nuclei, observation of the hydrogen nucleus is the natural choice for MR measurements for this fluid.

The magnetization created by the initial pulse of the MR experiment described above decays with respect to two different time constants, T_1 and T_2 . Constant T_1 (spin-lattice relaxation time) is the characteristic time for the nuclear spin system to return to equilibrium with its surroundings after a disturbance. The characteristic time for the nuclear spin system to return to internal equilibrium after a disturbance is characterized by the spin-spin relaxation time (T_2). The magnitude of T_2 is dependent on the magnetic field generated by the presence of the neighboring nuclei. In a solid with large internal magnetic fields, the T_2 value is very small (10-100 μ Sec). In a very low viscosity fluid, such as a gas, the T_2 value is large (Sec) where there is practically no internal magnetic field. The T_2 parameter can be useful in determining information about the molecular state of the sample in question (Barrall, 1996a; De Los Santos, 1996).

Southwest Research Institute and Quantum Magnetics first considered the relationship between the two relaxation times and the viscosity of the fluid. Both of these relaxation times are affected by intermolecular and by intramolecular motion. This is due to the nature of the spin-spin and spin-lattice couplings which drive the relaxation of the magnetization. Given certain assumptions, an analytical relationship between T_1 , T_2 , and the molecular reorientation time, τ_e , may be determined. Since the molecular reorientation time is proportional to the ratio of the zero shear viscosity to the absolute temperature, there should be a dependence of the MR relaxation parameters to viscosity (Barrall, 1996a; De Los Santos, 1996).

The analysis sequence utilized in the pilot flow loop was designed to gather as much information as possible, since a particular correlation has not yet been developed. In particular, this plan required long residence time (2-4 minutes) between measurements. On the other hand, for a practical application of the fully developed MR Viscometer, the measurement time should be in the milliseconds range. Since this viscometer is an experimental prototype, the objectives for our testing of this device are different from those of the on-line viscometers discussed in previous sections. Data acquired by the MR Viscometer was sent to Southwest Research Institute and to Quantum Magnetics to support the development of a correlation for black liquor viscosity utilizing MR principles, and to determine the effects of flow rate, liquor type and contaminants.

6.2 Experimental Overview

Table 6-1 lists the experimental runs that were performed on the Magnetic Resonance Viscometer.

Georgia Pacific	Canadian Forest	Chesapeake	GP & Canadian Forest Mix
55.84% 65.51%	57.73%		67.72% 71.38% 73.40%

Table 6.1. Black liquor types and solids content tested with the SWRI-Quantum Magnetics MR Viscometer.

The instrument was installed late in the evaluation phase and only a few experimental trials were performed. The main goals with this instrument were:

- To determine the MR parameter that best correlates with black liquor viscosity and solids content.
- To examine the effects of flow rate on the MR measurement.
- To determine the effects of various black liquor types on the viscosity correlations.

The control unit of the MR viscometer includes its own DAQ (Data Acquisition) system where all the MR parameters are collected and stored on disk. In turn, the sample temperature and the constant T_1 are retransmitted to the UF DAQ System. Time synchronizing of the two sets of data was very difficult, since the only flow-loop variable measured by the MR Viscometer was the sample temperature. Since the instrument has a 2-4 minute lag time between measurements, the flow rate was averaged from the number of flow rate samples collected by the UF DAQ System in the Pilot Plant divided by the number of samples taken by the MR instrument. It was found that the temperature measurement taken by the MR Viscometer is not very accurate; hence the Viscometer Line

RTD TT7 (*cf.* PI&D diagram Figure 9.1) was used instead. A temperature correlation between the MR sample temperature and TT7 was developed and this correction was then used to correct the sample temperature data from the MR instrument.

For measurements with no flow, the UF DAQ System was turned off and the MR Viscometer was set to automatic control. The MR control unit is capable of sending signals to control the relay isolation valves V7 and V8 (*cf.* PI&D diagram Figure 9.1), and of gathering information automatically. Before the MR instrument was allowed to take control of the viscometer line, the operating conditions of the viscometer line were fixed and the isolation control valves were set for remote-control operation. The delay time in the MR Viscometer DAQ program, which permitted flow through the viscometer line to provide a fresh sample for analysis, was set and then the program was activated. Flow entered the viscometer for the set delay time. Once this time had expired, the isolation valves closed for a zero-flow run. Once the analysis was completed, the delay time was reset, and flow through the viscometer line began again.

6.3 Selected Experimental Results

The data that was gathered during the experimental trials were first analyzed at UF where the files from the MR Viscometer were synchronized with the UF DAQ System. The data files were then sent to Southwest Research Institute and Quantum Mechanics for further analysis and development of MR-viscosity correlations. Southwest Research Institute and Quantum Magnetics investigated correlations between the viscosity of the black liquor sample and all of the measured MR parameters (relaxation time and amplitudes). They found that the constant T_1 and the long component of the constant T_2 are well correlated with the black liquor viscosity, but that the relative amplitudes of the relaxation times and the short component of the constant T_2 had poor correlations with

viscosity. Viscosity as a function of relaxation time was fitted by using nonlinear regression to the model (Barrall, 1996<u>a</u>)

$$\eta_H = c_0 + \frac{c_1}{T_m} + \frac{c_2}{T_m^2} + \frac{c_3}{T_m^3}$$
(6.4)

where T_m refers to either T_1^{sr} (saturation recover), T_1^{osir} (one-shot inversion recovery), or to T_2 ; with c_i represents the correlation coefficients, and η_H is the viscosity of the black liquor determined in the laboratory using a Haake Rotational Viscometer. Equation 6.4 does not represent a true physical relationship between viscosity and MR relaxation times; the model was simply chosen to provide a empirical relationship between these variables.

The coefficients of the model (6.4) were found by linear regression for the three possible choices of T_m . The resulting correlations are plotted in Figure 6.1, for three black liquor types, namely, a Canadian Forest liquor with 57.73% solids (shown as CF 57.73% in the figure), a Georgia Pacific liquor with 55.84% solids (GP 55.84%), and a mixture of Canadian Forest and Georgia Pacific liquors with a 73.40% solids content (GP & CF 73.40%). The T_1^{osir} constant appears to correlate best with viscosity, followed by T_1^{sr} , and then by T_2 . The relative difference with respect to the Haake-measured viscosities for the models plotted on Figures 6.1 A, B, and C, are respectively shown in Figure 7.2 A, B, and C. As a measure of overall error in the predicted viscosities, the average root mean square deviation from the laboratory reference viscosities of the model were calculated. The results are reported in Table 6.2 (Barrall, 1996<u>a</u>).

Due to the design of the sensor, the flow of black-liquor may have a considerable effects on the relaxation times and viscosity. Once the sample has become magnetically aligned, the MR probe fires a pulse of RF energy which causes the magnetic moment to tip away from the direction of the constant magnetic field B_0 . As the sample realigns itself with B_0 , the RF energy that was absorbed is re-emitted and detected. However, in a flowing sample stream, the sample may have had time to fully realign with B_0 before the

sample leaves the measuring chamber. This would cause an erroneous shorter relaxation time as long as the relaxation decay is longer than the necessary residence time. The constant T_1^{osir} is less affected by increasing flow rate, primarily because T_1^{osir} is consistently shorter than the other relaxation times.

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***************************************	Co	onstant T _m in Model (6	.4)
Black Liquor	T_1^{sr}	T_1^{osir}	<i>T</i> ₂
CF 57.73%	2.1%	1.1%	4.6%
GP 55.84%	5.3%	1.7%	12.8%
GP 65.51%	4.7%	2.2%	8.8%
GP&CF 67.73%	4.9%	2.3%	13.0%
GP&CF 71.38%	9.5%	2.5%	18.3%
GP&CF 73.40%	10.0%	1.8%	11.4%
Combined	59.4%	40.0%	52.4%

Table 6.2. Average root mean square deviation of the predicted viscosity from the actual viscosity.



Figure 6-1 SWRI/QM Viscometer relaxation times with selected black liquors for each of the three MR experiments: (A) T_1^{sr} , (B) T_1^{osir} , and (C) T_2 .



Figure 6.2 SWRI/QM Viscometer with Canadian Forest 57.73% Solids Black Liquor for each of the three MR experiments: (A) T_1^{sr} , (B) T_1^{osir} , and (C) T_2 .

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6.4 Summary of Observations

The MR instrument is an experimental prototype; hence it was not evaluate for it competence for practical applications. The intention of the study was to provide initial black liquor viscosity data to support further development. The MR Viscometer is a new and unique method of indirectly measuring viscosity and possibly solids content. Preliminary work has demonstrated that correlations between molecular properties and viscosity is possible. Further work needs to be done to determine if it is possible to develop a model that is independent of black liquor type, and if different MR properties may yield improved correlations for viscosity and solids content.

The long residence time between measurements can be reduced to milliseconds if only a single molecular parameter is to be measured and the effects of flow rate can be reduced or eliminated through redesigning the sensor to account for the measured relaxation decay. The advantages of this technique are very significant, namely, the MR viscometer is non-invasive with no moving parts in the process stream, non-destructive to the sample, and non-radioactive. This instrument may potentially prove to be a valuable tool in measuring on-line process viscosity.

7 CAMBRIDGE APPLIED SYSTEMS SLIDING ELEMENT VISCOMETER

The Cambridge Applied Systems Sliding Element Viscometer operates on principles similar to a falling-element viscometer, except that an external driving force is used to drive the element instead of gravity. The resulting velocity profile of he process fluid between the piston and the chamber wall is a combination of Poiseuille and Couette Flow, i.e., there is a drag flow in the direction of the element travel and there is a pressure driven flow in the opposite direction of the element travel (Cambridge, 1994; Gibbs, 1994). The viscosity of the process fluid is determined through a correlation determined using standard oils. The Cambridge Applied Systems Viscometer is a batch instrument, that is, the viscosity measurement is taken from a sample of the process fluid that is isolated from the process stream. The specific configuration tested is the following laboratory model.

Sensor: Sliding Element Viscometer

Cambridge Applied Systems did not deliver an on-line prototype for evaluation in the Pilot Plant. As an alternative, we carried out an evaluation of the laboratory. The major difference between the design of the on-line prototype and the laboratory model is that the former features an automated sampling and purging mechanism. In the on-line prototype design, an automatic actuating isolation valves opens to allow the fluid into the measuring chamber. The valves then closes to isolate the measuring chamber, and the element begins to oscillate. After the oscillation reaches steady-state, a viscosity measurement is recorded, the isolation valves open along with a steam purge valve, and the low pressure steam forces the sample back into the process stream while cleaning the measuring chamber. After this, the process is repeated for the next measurement. The on-line design is based on sound principles; however, we were not able to evaluate the performance of the automatic sample/purge mechanism because the instrument was not available for delivery from the manufacturer.

7.2 Principles of Operation

The sliding element viscometer contains two magnetic coils to provide an external electromagnetic driving force for the element. At any given moment, one coil is used to drive the element in one direction while the other coil is used to monitor the motion of the element. Once the element has moved a predetermined distance, the process is reversed, forcing the element in the other direction. Since the motion of the element is in two directions, any other external forces, such as gravity, are effectively eliminated during a complete two-way cycle. The time required for the element to complete a two-way cycle is directly proportional to the viscosity of an Newtonian fluid (Cambridge, 1994; Gibbs, 1994)

$$\eta = -\frac{tF}{L_c} \left(\frac{\left(r_o^2 - r_i^2 + \left(r_o^2 + r_i^2\right) \ln\left(\frac{r_i}{r_o}\right)\right)}{4\pi L_e \left(r_o^2 - r_i^2\right)} \right)$$
(7.1)

where

t = time of cycle F = force applied to the element $L_e = \text{length of the element}$ $L_c = \text{length of the chamber}$ $r_i = \text{radius of the element}$ $r_o = \text{radius of the chamber}$

To maintain isothermal conditions, the sensor is insulated and an external controlled electrical heater is installed around the measuring chamber. Controlled heating is necessary for this type of batch instrument because the residence time for a measurement can be several minutes long and the instrument is not heated by the natural convection of the process fluid. This instrument is limited to Newtonian fluids and can be adversely affected by the presence of particles in the sample.

7.3 Experimental Overview

Table 7.1 shows the types of black liquors used in the experimental runs that were performed on the Laboratory Cambridge Viscometer installed in the pilot flow loop

Table 7.1. Black liquor types and solids content tested with the Laboratory Cambridge Viscometer.

Georgia Pacific	Canadian Forest	Chesapeake	GP & Canadian Forest Mix
34.14%	-	-	-
42.20%	-	-	-
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The Laboratory Cambridge Viscometer was slightly modified and installed in the viscometer line to make a few preliminary evaluation runs with low-viscosity Georgia Pacific black liquor. Since the laboratory model was not equipped with retransmission lines for viscosity and temperature signals, all data were gathered by hand and timed with a stop watch. The variables of interest during these two runs were the residence time until steady state, the effects of process line vibration, and the accuracy of the viscosity measurement.

For these runs, the instrument was loaded with black liquor by opening the inlet and outlet valves of the viscometer, allowing a sample of black liquor to flow through the device, and then isolating the sample by closing the two valves. The Viscometer Line was brought to the desired temperature, and the external heater on the Laboratory Cambridge Viscometer was set to the same temperature. Once the instrument was at a thermal steady state with a temperature that closely matched the Viscometer Line temperature, the initial sample was then evacuated from the measuring chamber and a fresh sample was loaded. When the sample was isolated, the start time was recorded. The viscosity and temperature reading displayed on the control unit were recorded at regular intervals until after a steady state value of temperature and viscosity were reached. This procedure was repeated for all of the following runs.

7.4 Selected Experimental Results

The data presented for the Laboratory Cambridge Viscometer is independent of flow rate because this is a batch instrument. The results are shown as the natural log of viscosity plotted verses reciprocal absolute temperature in Figures 7.1 and 7.2. The diamond markers show the viscosity measured by the instrument connected to the sample in it the pilot plant. Samples taken from the pilot plant were taken to the laboratory and the viscosity was determined by glass capillary method (triangle markers) and were also measured in the laboratory using the Cambridge unit again (circle markers). The figures show that the viscosity readings from the Cambridge instrument tracked the laboratory measurements. The Cambridge readings shown required a residence time of 3-5 minutes, hence, the tracking capability was affected by a short delay. The instrument may have been capable of use at higher solids; however, further testing was not possible because using the laboratory instrument in the pilot flow loop was extremely man-power intensive due to the lack of automated controls and retransmission capabilities.

7.5 Summary of Observations

In the laboratory, the Cambridge Viscometer was proven to be an invaluable tool in measuring laboratory reference viscosities. The instrument is very easy to operate; essentially, the operator loads the instrument, turns on the power, and, after 30 minutes, records the temperature and viscosity. If the instrument is at thermal stability with the flowing liquor, the readings can be taken with a residence time of only 3-5 minutes. This

instrument has been shown to be a most reliable and reproducible laboratory viscometer for Newtonian fluids.



Figure 7.1 Laboratory Cambridge Viscometer installed in the Pilot Flow Loop with Georgia Pacific 34.14% Solids Black Liquor.

When conducting trials with the laboratory instrument, there was little question whether the device would properly measure low-medium solids black liquor. In fact, the Cambridge laboratory unit was used extensively in our development of high-solids viscosity correlations. On the other hand, the performance of the instrument on-line is still undetermined. In particular, it is not yet known what is the minimum residence time for the device, what are the effects of fouling, and what kinds of interference would be produced from vibrations in the line. Additionally, an prototype on-line instrument would need automated loading and purging of the measuring chamber, which would require further evaluation for reliability and performance. With the laboratory device installed in the viscometer line, only the residence time and sensitivity to vibrations could be tested. The instrument demonstrated no adverse effects from line vibration and required an average sample residence time of 3-5 minutes for the limited runs that were performed.



Figure 7.2 Laboratory Cambridge Viscometer installed in the Pilot Flow Loop with Georgia Pacific 42.20% Solids Black Liquor.

8 STEVENS INSTITUTE CAPILLARY VISCOMETER

The Stevens Institute Capillary Viscometer is an instrument designed utilizing a new principle of Δp measurement. Neither the on-line prototype or the laboratory model of this device were delivered. According to the design, the device was to be mounted on the process line and the sample was to be drawn into the measuring chamber by the upward stroke of a piston. On the downward stroke of the piston, the sample was to be forced back into the main stream, and the viscosity could be determined through the measurement of the Δp across a section of the tube and velocity of the piston (Bird, *et al.*, 1960).

8.1 Principle of Operation

This device would provide a direct measure of Δp , piston velocity, and temperature. The viscosity would be determined from measured values of Δp and shear rate as follows (Bird, *et al.*, 1960)

$$\eta = \frac{\tau}{\dot{\gamma}} = \frac{R^2 \Delta p}{8L\nu} \tag{8.1}$$

where

L = length of measuring chamber R = radius of measuring chamber $\nu =$ velocity of the piston

This viscometer, like the Cambridge Applied Systems, takes discrete (as opposed to continuous) measurements of viscosity. The residence time for this device should be very small and the problem of achieving thermal equilibrium with the process line should be easier to handle than with the Cambridge Applied Systems instrument. In addition, this viscometer should be able to measure the viscosity of non-Newtonian fluids by varying the velocity of the piston which in effect varies the shear rate.

8.2 Summary of Observations

The Stevens Institute Viscometer was not delivered for the evaluation trials. By utilizing a unique method in determining the Δp , theoretically the instrument should have been capable of measuring very small pressure drops for fully developed flow; however, this technology remains unproven for black liquor applications. Due to difficulties in making this technology reliable, the instrument could not be delivered in time for the evaluation trials. We anticipated that the Stevens Institute Viscometer would have particularly useful in high viscosity applications so that a sufficient pressure drop would be generated across the measuring chamber. The unanswered questions for this instrument include: What is the residence time for a measurement, what is the sampling rate or time between measurements, what are the possible effects of fouling in the instrument, and what is the reliability of the Δp measurement. This instrument design has the potential of being a valuable rheological device industrial applications. A comprehensive evaluation program should be carried out to assess the suitability of the instrument for practical applications.

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9 FINAL DESIGN OF THE PILOT FLOW LOOP

The design of the pilot flow loop has been an evolutionary process where modifications and improvements were incorporated into the design through experience of real-time operations. Specifics of the initial design and equipment will not be presented here as they may be referenced in the Master's degree thesis entitled "The Design of a Flow Loop for the Evaluation of On-Line Viscometers for Black Liquor Streams in Kraft Pulping Mills" by Richard A. Gibbs (1994). An overview of the pilot flow loop design will be presented in this section and specifics of modifications made to the flow loop will be discussed as a complement to Gibbs work.

9.1 Design Overview

The primary objective of the pilot flow loop is to simulate a main industrial process stream. It includes a bypass stream that provides a flexible means to test and evaluate prototype on-line viscometers and other instruments in a simulated mill environment. The pilot flow loop is not limited to use for black liquor trials or on-line viscometers, but it is a flexible platform that can be utilized for various other fluids and instruments, for the analysis of heat transfer properties through the Kenics Heat Exchangers, for the development and applications of process control strategies, and for the on-line fluid property measurements.

To simulate the main process stream, the black liquor is pumped continuously from a main holding tank by a progressive cavity pump through the main loop and is returned to the main holding tank. A bypass is located at one point in the Main Line, permitting a portion of the main process stream to be diverted to the Sample Line for analysis, and then returned to the Main Line to close the loop. For clarification, the instruments being tested are *on-line* devices which operate in a bypass, or slip, stream from the main process stream. This provides the necessary flow rate control and isolation for maintenance of the instrumentation without affecting the process. *In-line* devices are instruments that operate inside or on the main process. In-line instrument installations are in general less expensive that on-line configurations because the cost of installing a bypass line is avoided. Furthermore, for some particulate-rich steams, the bypass operations changes some characteristics of the flow by affection, for example, the mean particle size that is found in the bypass steam. On the other hand, on-line instruments have the advantage that the main process flow does not have to be interrupted when there is a need to maintenance the instrument. The pilot plant is currently configured for on-line testing of the devices.

The discussion pilot flow loop is divided into several major sections:

- Main Line
- Sample Line
 - Heat Exchanger Section
 - Viscometer Line
- Data Acquisition

The major sections are shown on Figure 9.1 along with key instrumentation details.

9.2 Main Line

The Main Line consists of a Main Holding Tank, a Moyno Progressive Cavity Pump, 2" 304 L stainless steel flanged pipe line, and associated control instrumentation. The Main Line is constructed to withstand a maximum pressure of 300 psig and to accommodate highly corrosive materials. The main line is steam-traced with 3/8" copper tubing used to preheat the piping before operation and to help maintain thermal equilibrium within the loop. Flanged pipe was utilized throughout the Main Line for durability, ease of maintenance and flexibility for modifications of the flow loop. The Moyno Progressive Cavity Pump is designed to handle highly viscous materials, up to 1,000,000 cP, and to provide a uniform, continuous, non-pulsating flow. In addition, this type of pump causes very little line vibration during operation, and offers very accurate and repeatable flow rates for which the pressure head is nearly independent of pump speed.

9.2.1 Main Pump

The Main Moyno Progressing Cavity Pump (shown as P1 in Figure 9.1) is designed for discharge pressures up to 150 psig with a maximum flow rate of approximately 55 gpm. During the evaluation trials, we maintained the flow rate between 30-40 gpm. The pressure was set to approximately 70 psig for low to medium viscosity trials, and to 120 psig for high viscosity trials. The rotor and stator are the two main components of a Moyno Progressive Cavity Pump. The rotor is a single external helix with a round cross section that fits inside the stator, which is a double internal helix molded of an elastomer and is permanently bonded within an alloy steel tube. As the rotor turns within the stator, cavities are formed which progress from the suction to the discharge end of the pump, conveying the pumped material.

The original stator was constructed of Viton, which is a high-temperature and chemically resistant polymer; however, Viton is not resistant to black liquor and becomes hard and brittle after long term exposure. Latter we chose EDPM (Ethylene Propylene Dimer Material) as a replacement for Viton and proved to be more resistant (though not completely resistant) to black liquor chemical attack. Operationally, using the EDPM stator limits the maximum temperature in the main loop to 175°C, but this did not affect these evaluation trials, since this temperature is well above the boiling point of black liquor. However, this temperature limit does affect maintenance and prevents cleaning the flow

loop with high pressure steam. The first EDPM stator failed after 4 months of hightemperature (~100°C) continuous operation. A second stator has been in continuous high temperature operation for the 4 months before requiring replacement.

9.2.2 Main Holding Tank

The black liquor is stored in the Main Holding Tank (shown at T1 on Figure 9.1) while the pilot flow loop is in operation. Since the tank is at atmospheric pressure, the holding temperature is limited to a temperature below the boiling point of the black liquor in the tank. Typically, the temperature in the Main Holding Tank is kept as low as possible while still maintaining fluidity and manageability of the black liquor to prevent thermal degradation prior to and during the experimental runs. An agitator M1 is utilized to maintain thermal and physical homogeneity of the stored black liquor sample.

The Tank Temperature TT1 (*cf.* P&ID diagram in Figure 9.1) and Tank Level LT1 (*cf.* P&ID diagram in Figure 9.1) are the two process variables that are monitored in the Main Holding Tank. The Tank Temperature thermocouple was replaced with an industrial covered RTD, which provides a more accurate temperature measurement and gives additional protection for the sensor. The tank temperature is controlled by either selecting the Tank Temperature Control Valve V43 or V44, where the first valve uses a small trim, and the latter, a larger trim. In normal operation, only Control Valve V44 is used, since Control Valve V43 is undersized and does not compensate for the heat loss in the flow loop. The Tank Level sensor measures the liquid head in the main tank and is calibrated to indicate the tank level in inches. Tank Level reading is a measurement of liquid head; therefore, the level is a function of the fluid density and LT1 must be calibrated for each black liquor sample for optimal accuracy.

9.2.3 Main Line Stream

Flow rate and line pressure are the two process variables measured in the Main Line. The flow rate is controlled by adjusting the Main Line Control Valve V5. The pressure is adjusted in the Main Line so as to maintain a constant flow rate through the Sample Line (which is a function of main line pressure and not the flow rate through the Main Line). The Main Line Flow Meter FT1 is a magnetic flow sensor that is capable of measuring flow rates up to a maximum of 60 gpm. A 16 perf strainer was installed just upstream of the Main Line Control Valve to prevent blockages in the seat of the control valve, and to protect the flow loop from the possibility of over-pressure in the line due to plugging of the Main Line Control Valve V5.

9.3 Sample Line

The purpose of the Sample Line is to simulate a bypass line from an industrial process main line, and to provide a controlled environment for evaluating the prototype online viscometers. At the Sample Point, a portion of the process stream from the Main Line enters the Sample Line passes through the Heater E1 flows through either the Viscometer Line or the bypass, and then flows through the Cooler E2, after which the liquor is returned to the Main Line at the Return Point to complete the loop. The Sample Line Control Valve V15 regulates the flow through the Sample Line and flow is controlled from the Sample Line Flow Meter FT2.

9.3.1 Heat Exchanger Section

The Heat Exchanger Section of the Sample Line is constructed of 304L stainless steel pipe with 300# NPT fittings with two 3-way pneumatic valves to form the bypass from the Viscometer Line. The Heater Exchanger Section includes two Chemineer Kenics Mixer High Efficiency Heat Exchangers that provide the heating and cooling of the black

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liquor through the Sample Line. These heat exchangers offer high efficiency and low pressure drop, while providing thermal homogeneity of the black liquor sample in a laminar flow regime. The primary purpose for the Heater was for the high temperature viscosity trials, where it was necessary to heat the black liquor above its atmospheric-pressure boiling point to simulate temperatures that would be experienced in typical mill operations. The Cooler was used to cool the black liquor down to the Main Holding Tank temperature to prevent a thermal build up in the flow loop; however, the Cooler was undersized for this purpose. When the Heater was in operation, the temperature in the Main Holding Tank needed to be closely monitored to prevent the thermal build up from causing the black liquor in the main tank to boil over.

By monitoring the process conditions of the heat exchangers, black liquor heat transfer properties and heat exchanger efficiency can be determined. The 2-wire RTDs on both the Heater and Cooler were replaced with 3-wire RTDs for improved accuracy in the temperature measurements, and an additional 3-wire RTD was added to the Heater shell outlet to measure the steam output temperature. In addition, two Rosemount 3051CD Differential Pressure Cells were mounted across the Heater and Cooler to measure the pressure drop across the heat exchangers at varying flow rates and viscosities.

9.3.2 Viscometer Line

The Viscometer Line provided the controlled environment for the evaluation of the prototype on-line viscometers. The Viscometer Line can be isolated from the Heat Exchanger Section by actuating the Viscometer Isolation Valves (V7 and V8), which are two 3-way pneumatic relay controlled valves that work in tandem. These isolation valves are controlled by the Sample Line Flow Meter FI2, either locally at the flow indicator, or remotely from the DAQ computer. The Viscometer Isolation Valves were used to isolate the viscometers from the Sample Line for zero-flow trials, and to allow for maintenance

and modifications to the viscometers or Viscometer Line without disturbing the flow through the Sample Line.

The Viscometer Line is constructed using 1" x 0.035" 304 stainless steel tubing with Swagelok compression fittings to provide the greatest flexibility for future modifications. The process tubing and each viscometer is steam-traced with 3/8" copper tubing used for preheating the Viscometer Line and to maintain thermal uniformity during the evaluation trials.

To protect the on-line instruments, the black liquor was passed through a 40-mesh strainer and then through a 200-mesh filter which was also being evaluated for black liquor applications. The Strainer/Filter Section could also be bypassed, either for maintenance or as an additional variable for the evaluation trials. The filter was not able to handle black liquor above 55% solids and was eventually eliminated. In addition to plugging at higher solids, the filter utilized Viton seals and gaskets that failed after long term exposure to black liquor. Teflon gaskets and seals were purchased as replacements, but were not installed. The 40-mesh strainer appears to able to provide adequate protection for the on-line viscometers and no plugging problems were experienced during the evaluation trials.

After the black liquor stream exits the Strainer/Filter Section, it passes through a static mixer that ensures physical and thermal homogeneity before the viscometers. The on-line viscometers and other instruments were installed in the following order: Nametre, Micro Motion, Brookfield, Electron Machine Refractometer (additional support equipment), and finally the Southwest Research Institute/Quantum Magnetics MR Viscometer. After passing through the instruments, the black liquor stream flowed through Valve V8 and entered the Cooler before rejoining the Main Line at the Return Point. Additional valves (V6, V12, V14, and the Nametre Steam Injection Valve) provide means for cleaning the viscometers with water, steam, or hot caustic, with forward or back flushing.

9.4 Data Acquisition System

The University of Florida Data Acquisition (UF DAQ) System is a comprehensive system that played a key role in the evaluation trials. Essentially, this system gathers the signals from all of the viscometers and process instruments, and records their values in raw form for off-line analysis. The data gathered is utilized for evaluating the on-line instruments, which is the primary goal of the project. Moreover, the data could possibly be used for determining black liquor heat transfer properties, the development of solids and density correlations, transport properties analysis, and refractive index studies.

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9.4.1 Instrumentation Overview

The instrumentation of the flow loop served a dual purpose: It provided centralized control of the equipment and process conditions of the flow loop, and provided a reliable and comprehensive data acquisition system. The major sensors, control valves, controllers and indicators were specified by Mr. R. Gibbs in his Masters Thesis (1994) and will not be given in detail here. The only changes that involve the flow loop instrumentation were with the trim sizing for the steam control valves (which were undersized and hence replaced with larger B-sized trims), and all the 2-wire RTDs (which were replaced by 3-wire RTDs for increased accuracy in the temperature measurement). All of the sensor signals from the flow loop are transmitted to the Control Room where the measurements are displayed on their respective indicators and controllers, and these readings are retransmitted as 4-20 mA signals to the UF DAQ system. From the control room, all data and control variables are manipulated from centrally located instrumentation.

The UF DAQ System consists of a Pentium PC computer, National Instruments DAQ equipment and LabVIEW software. The retransmitted signals from the controllers, indicators, and viscometer control units are converted from 4-20 mA to 1-5 Vdc signals by 250 Ω biasing resistors, and the voltage signals are then sent to the National Instruments

DAQ input cards. The National Instruments LabVIEW software was chosen for data collection due to the user-friendly graphical programming interface, its flexibility, and its seamless interface with National Instruments hardware. The National Instrument SCXI configuration offers flexibility, increased modular expandability (in addition to the expansion slots in the computer bus), and an open system for custom modules and instrumentation.

9.4.2 National Instrument Hardware

The SCXI configuration includes the SCXI-1000 4-Slot Chassis, SCXI-1300 Terminal Block, SCXI-1100 32-Channel Multiplexer Amplifier and Signal Conditioner, SCXI-1161 8-Channel Power Relay Module, AT-MIO-16E-10 Enhanced Multifunction I/O Board, and the AT-AO-10 Analog Output Board. The SCXI-1000 houses the modules SCXI-1100 and SCXI-1161, while the AT-MIO-16E-10 and AT-AO-10 take up two of the four expansions slots in the computer. The analog 1-5 Vdc signals are picked up by the SCXI-1300 Terminal Block and are conditioned by the SCXI-1100. The input signals are mulitplexed and sent to the AT-MIO-16E-10 card where the signals are processed by LabVIEW. The UF DAQ system has the planned capability of outputting ten 4-20 mA analog signals from the AT-AO-10 that can be sent to the control valves for direct computer control of the flow loop or back to the controllers as remote setpoints. At this time, the AT-AO-10 has not been fully tested; while the remote setpoints have been wired, additional switches and wiring will be necessary for the computer to take direct control of the flow loop. The SCXI-1161 is capable of actuating 8 relays that are controlled by LabVIEW. Two of there relays are currently wired to control the Viscometer Isolation Valves (V7 and V8) from the Sample Line Flow Indicator (FI2); however, this capability is not currently in use and has not been fully tested.

9.4.3 LabVIEW Software

The LabVIEW software has proven to be a very capable and flexible program. The DAQ system was broken down into a series of interlinking modules or VI's (virtual instruments) which make troubleshooting, updating, and expanding the DAQ system easier due to the modular programming design. The input signals are first processed by the MYSCXI.VI which is linked to the MASTER.VI along with each sensor block (temperature, pressure, flow meter, and tank level) and viscometer VI. Within each sensor block and viscometer VI, the signals from MYSCXI.VI are converted into the actual measured readings and are then recorded into data sheets within MASTER.VI. New VI's can be relatively easily incorporated into the MASTER.VI by duplicated existing VI's, incorporating the proper calibration factors and ranges, and designating appropriate channel numbers.

The UF DAQ System has been designed with expandability and flexibility as the key factors. During the evaluation trials, only a small part of the full capability of the system has been utilized. Many of the control functions of the pilot flow loop can be automated with the output features of the DAQ system along with full computer process control. Due to the time constraints, direct computer process control and flow loop automation were not implemented.





10 CONCLUSIONS AND RECOMMENDATIONS

10.1 Conclusions

Each of the on-line prototype viscometers that were evaluated are capable of satisfactorily operating over the expected viscosity ranges to be encountered in a mill with proper installation and considerations for flow rate control. Since these instruments are on-line devices, slip stream installation has been arranged at the participating mills. Installation of the viscometers in a slip steam will provide for the necessary physical protection, flow rate control, and isolation for maintenance that is required for proper operation.

10.1.1 Brookfield Coaxial Rotational Viscometer

The Brookfield Coaxial Rotational Viscometer tracked within $\pm 10\%$ of the laboratory reference throughout most of the experimental runs. Even though there was some concern of fouling with this design, there was no evidence of fouling or interference from particles smaller than 40 mesh through out these trials. Since this instrument is a direct measure of force, laboratory calibrations are not necessary for a viscosity measurement for Newtonian fluids, but laboratory verification would ensure accuracy. The instrument displayed a tendency to output a lower viscosity reading than the laboratory reference at higher flow rates (> 2.5 gpm) with the current configuration. Maintaining a relatively low flow rate should not be difficult when operating in a slip stream. The control unit demonstrated an offset in both the Viscosity Zero and Rotational Speed Voltage which

was manually compensated for by the operator. The electronics package needs to be upgraded with an auto-zero for the offsets and on-line viscosity calculation.

The Brookfield instrument has a number of significant advantages. First, this instrument has demonstrated excellent accuracy, precision, and tracking throughout the viscosity ranges encountered during the evaluation phase. Second, low flow rate conditions does not affect the accuracy of this instrument. Third, there were no significant fouling effects observed. Fourth, there were no effects of particles smaller that 40-mesh. Fifth, this instrument can be useful for non-Newtonian fluid measurements without changes in flow rate because of the short rotational speed transient. Finally, this viscometer does not require a laboratory reference for calibration.

The Brookfield instrument has a number of significant disadvantages. First, there is no temperature sensor in this instrument which would be helpful in validating the viscosity measurement from laboratory references. Second, this instrument is limited to low flow rates for accurate measurements. Third, the unit uses a double-flush seal which requires additional maintenance. Fourth, the instrument is limited to viscosities above 10 cP with the present configuration. Finally, the current electronics package is unsatisfactory for installation in an industrial process.

10.1.2 Micro Motion Coriolis Capillary Viscometer

The Micro Motion Viscometer was able to track within $\pm 10\%$ of the laboratory reference at high viscosities (> 150 cP) or at lower viscosities with low flow rates (< 0.5 gpm). This instrument has no moving parts in the process stream, and there was no evidence of any fouling or affect of particles smaller than 40 mesh on measurements during the experimental runs. The Micro Motion Viscometer provides a direct measure of mass flow rate, density, and pressure drop, along with measurements of volumetric flow rate and sensor temperature. The instrument has demonstrated a linearly rising response of

viscosity to increasing flow rate for low viscosity conditions. Under conditions of low viscosity and high flow rates, secondary flow effects cause a linearly increasing differential pressure in addition to the capillary pressure drop in the flow tubes, once the critical flow rate has been exceeded. The critical flow rate appears to be a function of the geometry of the flow tubes and viscosity. Correlations are being devised to compensate for this effect; however, if the mass flow meter is properly sized and if the slip stream flow rate is maintained at a level below the critical flow rate, the Micro Motion Viscometer is capable of accurate and repeatable viscosity measurements even at low viscosities (< 150 cP).

The Micro Motion instrument has a number of significant advantages. First, this instrument has demonstrated good accuracy at high viscosities (> 150 cP). Second, the instrument demonstrated excellent reproducibility at all viscosity ranges tested. Third, there were no significant fouling effects observed. Fourth, there were no effects of particles smaller that 40-mesh. Fifth, it may be possible to use this instrument for non-Newtonian fluid measurements at high viscosities by varying the flow rate through the instrument. Sixth, the viscosity measurement for a Newtonian fluid is nearly independent of flow at high viscosities. Seventh, this instrument provides excellent measurements of density and mass flow rate which are independent of flow rate. Finally, this viscometer has an excellent response time (fastest of all the instruments evaluated).

The Micro Motion instrument has a number of significant disadvantages. First, this instrument is not accurate at low viscosities in the current configuration tested. Second, this instrument is limited laminar flow for viscosity measurements. Third, the viscometer requires at least a one-point calibration from a laboratory reference. Finally, the flow rate must be controlled for accurate viscosity measurements.

10.1.3 Nametre Viscoliner Viscometer

The Nametre Viscometer tracked within $\pm 10\%$ of the laboratory reference at high viscosity (> 175 cP) and at low viscosities with low solids (< 50% solids). The instrument was unaffected by particles smaller than 40 mesh, by flow rates that were achieved in the Viscometer Line, and by second phases (such as air) in the process fluid. The Nametre Viscometer provides a direct measure of force and does not require laboratory calibration; however, the instrument outputs a product of $\eta \rho$ which would require a density input for an absolute viscosity measurement. With the current installation in the Pilot Flow Loop, the instrument is subject to fouling at high solids black liquor. The build up was verified physically by disassembling the viscometer housing and inspecting the probe. In most cases, steam purging the Viscometer Line was sufficient to clean the sensor probe and restore the performance of the instrument. With proper installation in the slip stream and stream lining the sensor probe, the effects of fouling can be minimized if not eliminated.

The Nametre instrument has a number of significant advantages. First, this instrument has demonstrated good accuracy, precision, and tracking when the sensor probe is free of fouling. Second, there were no effects of particles smaller that 40-mesh. Third, the viscosity measurement is independent of flow rate. Fourth, the instrument is unaffected by second phases (such as air entrainment) in the process fluid. Fifth, this instrument has the widest operational range of the viscometers evaluated. Finally, this viscometer has an excellent electronics package ready for on-line deployment; in addition, the control unit is capable of handling multiple sensors.

The Nametre instrument has a number of significant disadvantages. First, the sensor probe is subject to fouling with the current configuration tested. Second, this instrument measures dynamic viscosity ($\eta \rho$) on-line and would require an on-line density measurement for measurements of absolute viscosity (η). Third, the unit requires a zero

correction to compensate for temperature effects. Finally, background vibrations must be considered for optimal installation.

10.1.4 SWRI/Ouantum Magnetics MR Viscometer

This instrument is an experimental prototype and should not be considered to have been evaluated in the same way as the production on-line prototypes. The MR Viscometer is a new and unique method of indirectly measuring viscosity and possibly solids content. Preliminary work has demonstrated that correlations between molecular properties and viscosity is possible. Further work needs to be done to see if it is possible to develop a model that is independent of black liquor type and if different MR properties may yield improved correlations for viscosity and solids content.

10.1.5 Cambridge Applied Systems Sliding Element Viscometer

An on-line prototype viscometer was not delivered for evaluation, but a laboratory bench version employing the same principles of viscosity measurement was examined in the flow loop. The Laboratory Cambridge Viscometer proved to be a reliable and valuable laboratory instrument during these trials, but its use is limited to use with Newtonian fluids. When conducting trials with the laboratory instrument, there was little question whether the device would properly measure low-medium solids black liquor; however, what was not known was the minimum residence time for the device, effects of fouling, and interference from line vibrations. Additionally, a prototype on-line instrument would need automated loading and purging of the measuring chamber which would require further evaluation. With the laboratory device installed in the viscometer line, only the residence time and sensitivity to vibrations could be tested. The instrument demonstrated no adverse affects from line vibration and required an average residence time of the sample being tested of 3-5 minutes. Other questions could only be answered with an on-line prototype.

10.1.6 Stevens Institute Capillary Viscometer

The Stevens Institute Viscometer was not delivered for the evaluation trials. By utilizing a unique method in measuring the Δp , the instrument is theoretically capable of measuring very small pressure drops for fully developed flow; however, this technology is unproven. Due to the difficulties in making this technology reliable, the instrument could not be delivered in time for the evaluation trials. The Stevens Institute Viscometer would have been of most use in high viscosity applications where a large Δp could be generated across the measuring chamber, since, at low viscosities, the Δp may have been below the detection limits for this instrument. This instrument may have been the only viscometer in the evaluation (other than possibly the Nametre) capable of being installed *in-line* due to the design and principles of operation. This instrument has the potential of becoming a valuable rheological device for industry; however, there are still questions that must be answered.

10.2 Recommendations

Each of the on-line prototype viscometers evaluated are capable of satisfactorily operating in the mill; however, each instrument has strengths and weaknesses, and each mill has different requirements and expectations. The recommended pairing of instruments to mills took into consideration the needs of the mills, the black liquor type, and the strengths of the instruments. Several locations in the mill have been identified as possible sites of installation for these viscometers with the hope of meeting the expectations of the participating mills. Locations of the viscometers in the ring header is best suited for eventual closed loop control of the black liquor firing in the recovery furnaces; the final solids concentration needed for viscosity control can be set by locating the viscometers at the concentrator exit and at the exit of the multi-effect evaporator chain. This would permit solids concentration that is needed to maintain a constant firing viscosity to be predicted. In

the short term, monitoring the viscosity over time should prove helpful in identifying trends in black liquor viscosity and difficulties with the firing in the recovery furnaces. In this way, the viscometers can immediately provide useful information to the mills while proving their worth. The following discusses the recommendations for the on-line prototype viscometers.

10.2.1 Brookfield Coaxial Rotational Viscometer

The Brookfield Viscometer has been recommended to Georgia Pacific Corporation, Palatka, FL. The final firing black liquor viscosity for Georgia Pacific is typically low to mid-ranged, which is best suited for the Brookfield Viscometer. With the configuration tested, this instrument was able to handle fluids with viscosities from 10 to 200 cP. The following lists the recommended modifications to the instruments.

- The control unit needs to be upgraded.
- Addition of a temperature sensor near the measuring chamber.
- Use of a 12-20 mesh screen on the slip stream.

10.2.2 Micro Motion Coriolis Capillary Viscometer

The Micro Motion Viscometer has been recommended to the Canadian Forest Paper Products, Prince George, BC. The Canadian Forest Mill is going to be firing the highest solids content black liquor of the participating mills, and this will be best suited for the Micro Motion Viscometer which operates best under high viscosity conditions. Since this instrument does not have any moving parts in the process stream, maintenance and cleaning of the slip stream will be easier to perform, especially when dealing with a high solids content black liquor. In addition, the Canadian Forest Mill is also interested in the mass
flow rate into the recovery furnace. The recommendations based on experience with the Micro Motion Viscometer is summarized as follows:

- For use in slip stream only with necessary controls to maintain a sample stream flow rate below the critical flow rate.
- Digital communication between the differential pressure cell and the Micro Motion transmitter to increase the accuracy of viscosity measurement, especially at low pressure drops.
- Development of a broad range viscosity correction model to compensate the secondary flow effects at lower viscosities.

10.2.3 Nametre Viscoliner Viscometer

The Nametre Viscoliner Viscometer has been recommended to the Chesapeake Paper Company, West Point, VA. The Nametre Viscometer is capable of handing the widest range of viscosity measurements and multiple sensors can be used with one electronics package, which meets the needs of Chesapeake Paper Mill. Chesapeake Paper has expressed interest in monitoring the viscosity in the ring header as well as in the multieffect evaporator system. With one electronics package, the Nametre will be able to monitor each location but using the same type of sensor, even though, the viscosity of the black liquor is very different at these locations. The recommendations based upon experience with the Nametre Viscometer are summarized as follows:

- Use in a slip stream for the protection of the probe and also to provide easy access to the sensor for inspection and cleaning.
- Properly install to reduce stagnant flow areas around the probe, especially at the downstream end of the probe.

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• Steamline the probe so as to reduce the downstream area of the probe to prevent or minimize fouling due to solids deposition.