Establishment of Harrop, High-Temperature Viscometer

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Summary

This report explains how the Harrop, High-Temperature Viscometer was installed, calibrated, and operated. The report includes assembly and alignment of the furnace, viscometer, and spindle, and explains the operation of the Brookfield Viscometer, the Harrop furnace, and the UDC furnace controller. Calibration data and the development of the spindle constant from NIST standard reference glasses is presented. A simple operational procedure is included.

Introduction

Viscosity is a liquid defining property which has great importance for the manufacture and control of glass materials. The viscosity is related to the force required to move one layer of a fluid past another, or to cause one solid surface to move past another when there is a layer of fluid between the two surfaces. The application of a shearing force causes the atoms or molecular groups in the liquid to undergo displacement with respect to each other that continues with time so long as the application of force continues.[1] The time rate of deformation or shear rate is linearly related to the shearing stress on the liquid through Newton's law of viscosity:

$$F/A = \mu dv/L \quad \text{Eq.1}$$

Where F is the force, A the area of the plates, dv the velocity difference between the plates, and L the separation between the plates. The symbol μ is the physical constant for a given material and conditions and is called viscosity. A simple schematic follows as Figure 1.

Figure 1. Schematic Representation of Newton's Law.

Using this simple representation, viscosity may also be defined by Equation 2.

$$\text{Viscosity} = \mu = \text{Shear Stress} / \text{Shear Rate} \quad \text{Eq. 2}$$

The fluidity of a liquid is defined as the reciprocal of the viscosity. A Newtonian fluid is independent of the rate of shear e.g., twice the force would move the liquid twice as fast. Fortunately, glass is considered to be a Newtonian liquid except perhaps at extreme shear rates, such as those found in high speed forming operations.

Viscosity is measured in Pa.s (pascal second), the accepted SI unit, although the older unit Poise is still more commonly used in discussions of glass. For conversion, 1 Pa.s is equal to 10 Poise. The term centipoise or cP is 1/100th of a Poise. This report will continue discussions with the use of Poise. Additional conversion units are listed below:

	<u>SI units</u>		<u>cgs units</u>
Viscosity	1 mPa.s	=	1 cP
Shear Stress	1 Newton/m ²	=	10 dyne/cm ²
Torque	1 Newton-m	=	107 dyne-cm.

Glass viscosity curves are often described mathematically by the Fulcher-Fogel-Tammann equation or more simply the Fulcher equation: [2]

$$\log(\mu) = -A + B \times 1000/(T - T_0) \quad \text{Eq. 3}$$

where A, B, and T₀ are empirical constants. The temperature (T) is the measured in degrees Celsius. This equation permits reasonable extrapolation of measurements to specific temperatures or viscosities. The determination of the three constants by manual methods is difficult, however, advanced software packages such as JMP®, Deltagraph®, etc. can solve the three constants through curve fitting. The constants tend to vary within the following limits:

	A	2 to 5
	B	5 to 8
and	T ₀	400 to 0.

Viscosity is one of the key physical property determinations for development of the vitrification process for nuclear or hazardous wastes. The viscosity - temperature relationship is a key parameter in the rate at which the glass melt homogenizes and refines. The glass viscosity establishes the melting temperature range and controls the rate of pouring through a given opening. In the glass industry, the viscosity also controls the ability to form the glass into the desired shape or form. The viscosity of glasses are often measured between 50 Poise to levels as high as 10 to the 14th power. Certain glass viscosity-temperature points have been commonly defined as shown in Table 1. The typical methods for the viscosity determinations are also noted.

Table 1. Viscosity Ranges and Methods of Determination

Log Viscosity (Poise)	Common Glass Term	Method of Determination
2.0	Melting Point	 Rotating Cylinder or
4.0	Working Point	Bubble Rise
7.65	Softening Point	Penetration
13.0	Annealing Point	 Fiber Elongation
14.5	Strain Point	Beam Bending

The rotating type viscometer, the most common determination method, was first introduced by Margules [3] and further adapted by successive investigators [4-10]. Two types of rotating viscometers have generally been employed to measure the high temperature viscosity of glass. In one case the crucible is rotated and the spindle deflection is measured. In the other case, the spindle is rotated and the viscosity determined from the percent torque and the rate of rotation [11]. This report will consider only the latter case.

Experimental

Set up and Alignment

The Harrop SP-4A High Temperature Glass Viscometer consists of the equipment listed in Table 2. A simple electrical schematic of the lay out is shown as Figure 2 and a detail of the equipment arrangement is presented in Figure 3.

Table 2. Harrop High Temperature Glass Viscometer

Harrop platinum wound, water cooled, tube furnace (1600°C), Motorized Furnace Lift, Honeywell Universal Digital Controller (UDC3000), Brookfield DV-II+ Programable Viscometer, Viscometer Base and Stand, Platinum Crucible and Spindle Brookfield Oils and NIST Glass Standards.
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This equipment was designed jointly by Harrop Industries Inc. and the Brookfield Engineering Laboratories Inc. [11]. The Clemson University, Environmental Technologies Laboratory initially established the equipment settings [12]. The furnace was designed to use a limited amount of radioactive glass and measure the viscosity over a fairly wide range of viscosity and temperature.

The UDC controller requires the attachment of three lines from the viscometer base to the UDC controller. The heavy black power cable provides power to the furnace and the two smaller leads provide the thermocouple inputs to the controller [13]. All connections should be clearly labeled as shown in Figure 2. The UDC is supplied with 110 volt power from a common outlet as shown. The viscometer base has a plug to a wall outlet which provides power to the motorized furnace lift. The Brookfield Viscometer must be attached to the Viscometer Base and also requires 110 volt power. The Brookfield viscometer has a hand adjustment to raise and lower the instrument. The Harrop furnace has a motor driven lift and cooling water must be supplied to the furnace to prevent overheating of the outer surface.

Alignment of the viscometer base, the crucible holder/platinum crucible and the Brookfield viscometer is critical to this measurement. A level bubble is attached to the base and to the Brookfield to assist alignment [14]. An independent leveling devices such as a carpenters level is recommended. The crucible holder is an alumina tube which can be adjusted by loosening a bolt on the bottom of the viscometer base. The viscometer, hang down wire, and spindle should be centered in the alumina tube and then the platinum crucible. The spindle should be true. Note that the hook which attaches to the Brookfield has a left hand thread. Some of the Brookfield shafts do not appear to turn true and cause oscillation at high speeds. This could be confirmed by evaluating other Brookfield viscometers.

Initially during the set up, the platinum spindle should just touch the bottom of the platinum crucible. Locate the Brookfield viscometer travel stop at this position on the support bar. Mark the alumina tube, crucible holder at the base and then another mark at 0.200 inches or 0.50 cm above the first mark. Loosen the alumina tube with the set screw and lower the sample holder to the new mark. This will insure that the spindle is off the bottom of the crucible by 0.200 inch. When the furnace is at high temperature (1500°C) the spindle and crucible will increase in length due to thermal expansion by about 0.120 inch. This allows 0.080 inch between the spindle and the bottom of the crucible for clearance. The dimensions of the crucible and spindle are presented in Figure 4. The spindle has a maximum diameter of 0.344 inch and the crucible well has an inside diameter of 0.500 inch. The difference is only 0.156 inch or roughly about 1/16 th of an inch on either side of the spindle.

The furnace position has been marked on the right side of the stand and should be operated at the upper stop position "0" to perform the viscosity determination. See Figure 5 for the approximate furnace dimensions and the furnace positions. A check of the furnace gradient was made and is shown as Figure 6. This data shows that the flat portion of the gradient is in or near the raised stop position (0 position).

If alignments are not correct and the spindle shaft is not straight there will be poor operation of the system, particularly at high rotational speeds (>60 rpm). The HADV-II Brookfield viscometer has some axial play and creates a portion of the alignment problem. This may require correction at a later date. The Brookfield also has some side to side play on the Brookfield stand.

Set Up of Brookfield Programmable Viscometer

Turn on the power to the Brookfield Viscometer with the power switch on the back left hand side of the unit. Allow it to warm for about ten minutes. Per the directions from the viscometer, remove the hangdown wire and spindle and allow the instrument to autozero. A schematic of the Brookfield face is reproduced as Figure 7. Press the up arrow and the speed setting should appear while the motor is off (OFFRPM). The speed of the HADVII+ can be adjusted over two independent scales:

0, 0.5, 1, 2, 2.5, 4, 5, 10, 20, 50, and 100 RPM or

0, 0.3, 0.6, 1.5, 3.0, 6, 12, 30, and 60 RPM.

At speeds above 60 RPM there may be excessive oscillation of the spindle. Use the arrows to scroll through the speeds, use SET SPEED to select the speed, and use the MOTOR ON/OFF to turn on the motor. The SELECT SPINDLE and ENTER/AUTO RANGE are not used at this time. The % Torque will be used in these measurements. Some caution is required to insure the starting of the motor at a low speed so that the torque is not too high. The speed can be adjusted upward during measurement. This unit has additional capabilities which are presently unnecessary for this viscosity determination.

Set Up of Honeywell UDC Controller

Turn on the power to the UDC by pushing on the "Power On" switch on the face of the controller. See Figure 8. The "Stand By" switch, white light, should be pushed so that power is available to the instrumentation but the furnace relay contact is open. The "Power On" switch cannot operate unless it is in the "Stand By" mode. The Honeywell controller can now be adjusted.

To check the tuning of this controller press the "Setup" button to see SETUP TUNING. Next press the "function" button to see 10.0 PROP BD. Keep pressing the "function" button to check tuning parameters. See Attachment 1 for the record of the original settings for all functions.

To establish the heating schedule press the "Setup" button twice to see SP RAMP. Prepare a heating program as shown in Figure 9. This will reduce mistakes in the heating program. Press the function button to see ENABLE -- SP PROGRAM. Press function button again to start the programming segment with START SEG --1. Continue pressing function button and follow directions on Figure 9 to establish a heating program. The heating program can contain up to 12 segments. The up and down arrows are used to adjust ramp rates, soak set points, and soak times. The start up of the furnace will be covered under the Procedures section.

Calibration Standards

Silicone Fluids

Before viscosity can be determined with the non-standard platinum spindle and crucible it is necessary to calibrate the unit with standard materials. There are standard Brookfield fluids and NIST glass standards available for this purpose. Both were used in this set up and the results will be reported here [15]. The use of these standard materials permits the calculation of a constant which can later be used to convert the Brookfield % Torque data to viscosity. The constant may be calculated from the following formula:

$$K = [(Standard\ viscosity) \times RPM] / \% Torque \quad Eq. 4$$

The Brookfield fluids are easy to use but the calibration does not include the effects of high temperature. These fluids are recommended for initial set-up but the NIST glasses are recommended for actual calibration of the viscometer. The Brookfield fluids are identified and characterized in Table 3. There are two types of general purpose fluid. One is standardized at 25°C and the other, thermal fluid, which can be used over the temperature range of 25.0 to 149°C. In this calibration the high temperature HT30000 was employed and K was found to be between 240 and 247. These fluids are recommended for initial set u, but the NIST glass calibration is required for the high temperature calibration.

Table 3. Brookfield Viscosity Oil Standards

Brookfield Part #	Nominal Viscosity- cP	Specific Gravity	Temp °C
<u>General Purpose Fluids</u>			
5 cps	5	0.92	25.0
10 cps	10	0.94	"
50 cps	50	0.96	"
100 cps	100	0.96	"
500 cps	500	0.97	"
1000 cps	1,000	0.97	"
5000 cps	5,000	0.97	"
12500 cps	12,500	"	"
30000 cps	30,000	"	"
60000 cps	60,000	"	"
100000 cps	100,000	"	"

Table 3. Continued

Brookfield Part #	Nominal Viscosity cP	Specific Gravity	Temp °C
<u>High Temperature Fluids</u>			
HT 30000	30,000	N.A.	25.0
	9,000	"	93.3
	4,500	"	149.0
HT 60000	60,000	"	25.0
	18,000	"	93.3
	9,000	"	149.0
HT 100000	100,000	"	25.0
	30,000	"	93.3
	15,000	"	149.0

NIST Standard Glasses

Standard glasses for viscosity may be obtained from the National Institute of Standards and Technology, Gaithersburg, MD. Two glasses were obtained for this purpose: a borosilicate No. 717a and a soda-lime glass No. 710a. The viscosity-temperature relationships and densities are presented in Tables 4 and 5. The use of these glasses is recommended to develop the viscosity spindle constant. This method should replace the need for separate thermocouple or viscometer calibration as the data should be traceable to NIST as long as the viscometer set up, thermocouple, or furnace conditions are not changed.

Table 4. Standard Reference Material 717a - Borosilicate Glass

Glass No. NIST	Temp (°C)	Visc. Poise	Log Visc.	Est. Density (gm/cc)
<u>Borosilicate</u>				
717a	1555	100	2.00	2.29 @ 25°C
	1466	178	2.25	
	1388	316	2.50	
	1318	562	2.75	
	1256	1,000	3.00	
	1201	1,778	3.25	
	1151	3,162	3.50	
	1106	5,623	3.75	
	1065	10,000	4.00	
	1028	17,782	4.25	
	993	31,622	4.50	
	961	56,234	4.75	
	932	100,000	5.00	
	905	177,828	5.25	
	880	316,228	5.50	

Fulcher Equation, $\text{Log Visc. (Poise)} = -1.5602 + 4852.2 / (T(^{\circ}\text{C}) - 192.462)$

Table 5. Standard Reference Material 710a Soda-Lime-Silica

Glass No. NIST	Temp. °C	Viscosity Poise	Log 10 Vis.	Density gram/cc (Est. *)
Soda Lime Silica				
710 a	1464	100	2.00	2.55 @ 25°C
	1432	126	2.10	
	1387	178	2.25	
	1319	316	2.50	
	1259	562	2.75	
	1205	1,000	3.00	
	1157	1,778	3.25	
	1113	3,162	3.50	
	1037	10,000	4.00	
	973	31,622	4.50	
	918	100,000	5.00	

Fulcher Equation, Log Viscosity (Poise) = $-1.729 + 4560/(T(^{\circ}\text{C}) - 240.8)$

* Density of 1826 soda lime NIST standard.

Calibration Results

The viscometer should be calibrated with one or both of the NIST glass standards as required. The initial calibration of the top cooled Harrop Furnace with the 717a glass is presented as Table 6. The plotted data is shown as Figure 11. The actual data taken during these runs is presented as Attachments 2 and 3. The two constant determinations were averaged and the best straight line fit was obtained. The equation for the spindle constant is presented at the bottom of Table 6.

Table 6 Calibration with NIST 717a Borosilicate Glass

Crucible Temp. °C	Viscos. (Poise)	Spindle RPM	Avg. % Torque	Calc. K Value
8/19/98				
				Glass Wt. 5.988 gr
1513	130.1	30	26.88	145.2
1406	274.3	30	51.12	161.0
1299	668.1	12	48.9	163.9
1193	1947	6	69.6	167.8
1088	7211	1.5	62.7	172.5
8/27/98				
				Glass Wt. 5.966 gr
1512	130.9	30	21.7	181.0
1405	276.4	30	41.8	198.4
1298	674.2	12	39.6	204.3
1192	1969	6	56.17	210.3
1087	7312	1.5	50.4	217.6

Average Spindle Constant $K = 272.04 - 0.069137 \times \text{Temp}^{\circ}\text{C}$.

Viscosity Determination Procedures - Glass

- 1) The Harrop/Brookfield viscometer should be setup and all units connected per schematics presented in Figures 2 to 4. See the previous section on the set up and alignment of the unit. The furnace, sample holder, and Brookfield viscometer should be leveled and aligned. The carpenter's level should be used to check all surfaces. The hang down wire and spindle should be attached to the viscometer to check the alignment with the center of the ceramic tube and the crucible. The hang down rod and spindle should be straight and true when rotating below 60 rpm. When the spindle is lowered into the crucible it should come to rest 0.200 inch from the bottom of the crucible. Raise the Brookfield Viscometer and spindle 2 to 3 inches above the stop.
- 2) Setup the Brookfield viscometer and become familiar with its operation. See Figure 5 and previous section covering the viscometer. Set the viscometer at a relatively slow speed.
- 3) Setup the Honeywell UDC controller checking the setup and general configuration. See Figure 6, Attachment 1 and the previous section. Establish the heat up and hold schedule similar to that shown in Figure 7.
- 4) Determine the density of the sample glass at room temperature.[16] Weigh out 2.6 cc of the glass (Density x 2.60 = grams of glass) and place into the platinum crucible. Place the platinum crucible on the alumina sample holder. Carefully raise the furnace with the lift. Some assistance by hand is required to raise the furnace.
- 5) Insure that the cooling water is flowing to the furnace. A slight vibration will be felt when the Tygon® tube is compressed between the fingers. On the UDC controller, push the Manual/Auto button to Auto. An "A" should appear at the top of the Honeywell control. Push the Run/Hold button to Run and an "R" should appear on the left side of the Honeywell control. Check the "Lower Display" for the set point. It may be necessary to adjust the Set Point with the Select Set Point switch to ensure the amp loading is not too high on start up (approx. 70 to 80°C). Push the Std By/ Run button to Run - a blue light should appear. Something similar to the following should be seen on the Honeywell screen:

	<u>Honeywell Screen</u>			<u>Digital Read Out</u>
	C		A	Temperature Indicator
(run)	R	105		98 (crucible)
(set pt.)	SP	108		
- 6) Check ammeter - initially should have a low value (< 5 amp). If not adjust Set Point.
- 7) To Stop program advancement simply push Run/Hold to Hold. To stop heating the furnace push StdBy/Run to Stand By (White Light). To completely reset the program shut controller off and start at Step 3 again.
- 8) When the furnace reaches the high temperature hold and the glass has melted and homogenized, lower the Brookfield Instrument and spindle to the stop.

- 9) The Brookfield should be set for a slow speed and gradually increased to obtain a % Torque reading of at least 10% or more. The initial high temperature hold is important to allow bubbles to escape and the glass to equilibrate. A time of at least one half hour to three quarters hour should be allowed before measurements are taken.
- 10) Three to four measurements should be obtained at five minute intervals while the furnace is holding. The following data should be recorded: Set Point, Furnace Temperature, Sample Temperature, RPM, % Torque and Time. See Figure 10 for a typical data sheet. Stop the Brookfield instrument until the furnace temperature has reached the next hold.
- 11) Wait till furnace has decreased in temperature to the next lower hold and has equilibrated for about one quarter hour. Reduce speed on the viscometer with the set speed adjustment prior to initial start up. Obtain three to four measurements at this temperature at about 5 minute intervals. If more time is needed to obtain additional measurements, press the Hold button which stops the program advance.
- 12) Repeat step 10 and 11 until all measurements are taken. This usually requires 5 to 6 hold periods. It is better to obtain 5 or 6 precise measurements than many measurements which were not given sufficient time to equilibrate.
- 13) Slowly raise the Brookfield and spindle 1 inch above stop point and allow glass to drain off the spindle. Raise the spindle another 1 inch to clear the crucible. Allow the furnace to cool to room temperature in this position. Turn off the furnace power and when the furnace has cooled down turn off the water cooling.
- 14) Remove the crucible and place upside down in an alumina crucible and place in a furnace. Heat the crucible to about 1000°C and allow it to drain. The crucible will require cleaning in HF for several days to clean. Special training must be obtained for work with HF.
- 15) The viscosity is calculated with the following formula.
$$\text{Viscosity (Poise)} = K \times \% \text{ Torque/RPM}$$
- 16) The calibration of the viscometer using the NIST glass should be carried out after any major disturbance of the viscometer set up or the furnace etc. The unit should be calibrated after every 20 to 30 measurements.

References

1. A. K. Varshneya, "Fundamentals of Inorganic Glasses," Chapter 9, p183-210, Academic Press, San Diego, CA, 1994.
2. G.S. Fulcher, "Analysis of Recent Measurements of the Viscosity of Glasses," J. Amer. Ceram. Soc., 8: [6] 339-55, (1925)
3. M. Margules, "Über die Bestimmung des Reibungs und Gleitungs Coefficienten aus ebenen Bewegungen einer Flüssigkeit," Sitzber.Akad.Wiss.Wien, 83, Part II, 588 (1881).
4. S. English, J.Soc. Glass Tech. Trans., 7, 25 (1923) and 8, 205 (September, 1924).
5. E.W. Washburn, G.R. Shelton, and E.E. Libman, Univ. of Illinois Eng. Exp. Station, Bull. 140, 74 (April, 1924).
6. H.R. Lillie, "Viscosity Measurements in Glass," J. Amer. Ceram. Soc., 12 [8] 516-529, (1929).
7. H.A. Robinson & C.A. Peterson, "Viscosity of Recent Container Glass," J. Amer. Ceram. Soc., 27, [5], 129-138, (1944).
8. R.L. Tiede, "Improved Apparatus for Rapid Measurement of viscosity of Glass at High Temperatures," J.Amer.Ceram.Soc. 42 [11] 537-541, (1959).
9. A. Napolitano & E.G. Hawkins, "Viscosity of a Standard Soda-Lime-Silica Glass," J Research of NBS, 68A [5], 439-448, (1961)
10. ASTM C 965-81, "Measuring Viscosity of Glass Above the Softening Point," 1010-1016, Part 17, Annual book of ASTM Standards.
11. A. Blum, "Instruction Manual for Harrop Model SP-4A, High Temperature Viscometer," Serial No. 10137, May 1997, Harrop Industries, Inc.
12. T. Pruett, "Final Report on Shakedown of Glass Viscometer to Support the Pu Program at SRTC," Task Order 228, SCUREF, Clemson Environmental Technologies Laboratory, Clemson, SC, April 15, 1998.
13. "UDC 3000, Universal Digital Controller Product Manual," No. 51-52-25-07E, October, 1995, Fort Washington, PA.
14. "Brookfield DV-II+ Programable Viscometer Operating Instructions," Manual No. M/97-164, Brookfield Engineering Laboratory, Inc. Stoughton, MA.
15. Notebook No. WSRC-NB-89-30, (E-57157) 74-89, R.F. Schumcher
16. GTOP 3-038 "Density of Glass by Buoyancy," 5/31/96.

Figure 2. Simple Schematic Harrop Viscometer

Figure 3. Harrop High Temperature Viscometer

Figure 4. Platinum Crucible and Spindle

Figure 5. Approximate Dimensions of Viscosity Set Up

Figure 6. Furnace Gradient Top Cooled Harrop Visc. RFS - 8-14-98

READ OUT

cP 0.00 --- C

Off RPM % 0.0

LVDV-II 673.7 dyne-cm

RVDV-II 7187

→ HADV-II 14,374

HBDV-II 57,496

Options Menue

Motor On/Off
Escape exits options

Motor
on/off
Escape

Options
Tab

Enter
Auto
Range

Execute current option
Presents maximum torque

Causes unit to run at
selected speed

Set
Speed

↑

Select
Spindle

Spindle selection

Selects data/parameter
display cP,SS,SR, %
Torque

Select
Display

↓

Print

Print when printer attached

Figure 7. Brookfield Digital Viscometer

Figure 8. Honeywell UDC Controller

Prompt	Function	Segment	Value	Prompt	Function	Segment	Value
STRT SEG	Seg No.		1	SEG7 RAMP	Ramp Time	7	.15m
END SEG	Seg No.		12	SEG8 SP	Soak SP	8	1200
RECYCL	No. Recyc.		0	SEG8 TIME	Soak Time	8	.30m
SOAK DEV	Deviation Val		0	SEG9 RAMP	Ramp Time	9	.15m
SEG1 RAMP	Ramp Time	1	3.0	SEG10 SP	Soak SP	10	1100
SEG2 SP	Soak SP	2	1500	SEG10 TIME	Soak Time	10	.30m
SEG2 TIME	Soak Time	2	1.0	SEG11RAMP	Ramp Time	11	.3.00
SEG3 RAMP	Ramp Time	3	.15m	SEG12 SP	Soak SP	12	35
SEG4 SP	Soak SP	4	1400	SEG12 TIME	Soak Time	12	0
SEG4 TIME	Soak Time	4	.30m	STATE	Con.@ End		HOLD
SEG5 RAMP	Ramp Time	5	.15m	PROG END	Con. Status		LAST SP
SEG6 SP	Soak SP	6	1300	RAMP UNIT	Engr. Unit		Time
SEG6 TIME	Soak Time	6	30m		for Ramp		

SET UP to SET UP TUNING
 FUNCTION to parameters
 SET UP to SP RAMP
 FUNCTION to SP Prog. Enable
 FUNCTION to above....

Figure 9. Ramp/Soak Profile and Input to Program

Figure 10. Viscosity Test Report

VISCOSITY TEST REPORT				By: _____ Date: _____		
Sample ID No. _____				Density: _____		
Heat Treatment _____				Weight Glass _____		
Weight Cruc. _____		Weight Glass _____				
Time/ Time Remaining	Set Pt Temp. °C	Furn. Temp. °C	Cruc. Temp. °C	% Torque	RPM	Viscosity P

Glass Weight = $2.6 \times \text{Glass Density}$
 Spindle Constant, $K = (\text{Visc} \times \text{RPM}) / \% \text{Torque}$
 Viscosity = $K \times \% \text{Torque} / \text{RPM}$

Comments on Determination

Signed: _____ Date: _____

Figure 11. Graph of Measured Spindle Constants at Temperature - °C.

Configuration Record Sheet

P.21

Keep a record

Enter the value or selection for each prompt on this sheet so you will have a record of how your controller was configured.

Group Prompt	Function Prompt	Value or Selection	Factory Setting	Group Prompt	Function Prompt	Value or Selection	Factory Setting
TUNING	PROP BD	<u>10</u>	1.0	CONTROL	PID SETS	<u>1 ONLY</u>	1 ONLY
	or GAIN		1.0		SW VALUE		0.00
	RATE MIN	<u>0</u>	0.00		SP SOURC	<u>1 LOCAL</u>	1 LOCAL
	RSET MIN		1.0		RSP SRC		NONE
	or RSET RPM	<u>0.10</u>	1.0		RATIO		1.0
	or MAN RSET		0.0		BIAS		0
	PROP BD2		1.0		SP TRACK	<u>NONE</u>	NONE
	or GAIN2		1.0		POWER UP	<u>MANUAL</u>	MANUAL
	RATE2MIN		0.00		SP HILIM	<u>1610</u>	1000
	RSET2MIN		1.0		SP LOLIM	<u>0.0</u>	1000
	or RSET2RPM		1.0		ACTION	<u>REVERSE</u>	REVERSE
	CYC2SEC		20.0		OUT RATE	<u>DISABL</u>	DISABL
	CYC2SEC		20.0		PCTM UP		0
	SECURITY	<u>0</u>			PCTM DN		0
	LOCKOUT	<u>NONE</u>	CALIB		OUT HILIM	<u>100</u>	100.0
	AUTO MAN	<u>ENABLE</u>	ENAB		OUT LOLIM	<u>0</u>	0
	SP SEL	<u>ENABLE</u>	ENAB		DROPOFF	<u>0</u>	0.0
	RUN HOLD	<u>ENABLE</u>	ENAB		DEADBAND		2.0
SP RAMP	SP RAMP		DISABL	OPTIONS	AUX OUT		DISABL
	TIME MIN		3		4mA VAL		0
	FINAL SP				20mA VAL		0
	SP RATE		DISABL		DIG IN 1		NONE
	EU/HR UP				DIG IN 2		NONE
	EU/HR DN			Com	ComSTATE		DISABL
ADAPTIVE	SP PROG	<u>ENABLE</u>	DISABL		Com ADDR		0
	ADAPTIVE	<u>DISABL</u>	DISABL		SHEDTIME		0
	SP CHANG		10		PARITY		ODD
ALGORITHM	KPG		1.0		BAUD		300
	CONT ALG	<u>PID A</u>	PID A		DUPLEX		
	INPUT 2		DISABL		LOOPBACK		DISABL
	OUT ALG	<u>CURRENT</u>			SHEDMODE		LAST
	4-20RNG		50PGT	ALARMS	SHED SP		TO LSP
INPUT 1	RLY TYPE				UNITS		PERCENT
	DECIMAL	<u>XXXX</u>	XXXX		A1S1 VAL	<u>1610</u>	90
	UNITS	<u>DEGC</u>	NONE		A1S2 VAL		90
	IN1 TYPE	<u>RTE</u>	0-10mV		A2S1 VAL		90
	XMITTER		LINEAR		A2S2 VAL		90
	IN1 HI	<u>1704</u>	1000		A1S1TYPE	<u>PV</u>	NONE
	IN1 LO	<u>-17.78</u>	0		A1S2TYPE	<u>NONE</u>	NONE
	BIAS IN1	<u>0.0</u>	0		A2S1TYPE	<u>NONE</u>	NONE
	FILTER 1	<u>1</u>	1		A2S2TYPE	<u>NONE</u>	NONE
	BURNOUT	<u>UP</u>	NONE		A1S1 HL	<u>HI</u>	HI
INPUT 2	EMISSIV		0		A1S1 EV		HI
	PWR FREQ	<u>60 HZ</u>	60HZ		A1S2 HL		HI
	XMITTER 2		LINEAR		A1S2 EV		HI
	IN2 HI		1000		A2S1 HL		HI
	IN2 LO		0		A2S1 EV		HI
	FILTER 2		1		A2S2 HL		HI
					A2S2 EV		HI
					AL HYST	<u>0.1</u>	0.1

CALIBRATION WITH NIST 717a					Attachment 2	
Date:	8/19/98	FFS			Glass Wt:	5.988 gr
Time	Minute	Set Point	Furnace	Sample	FFM	Torque
	Remaining	Temp.-°C	Temp.-°C	Temp.-°C	Setting	Reading
1140	13H	1500	1499	1511	30	48.8
1145	13H	"	1500	1513	"	39.0
1150	"	"	"	"	"	42.0
1155	12	"	"	"	"	28.4
1200	11H	"	"	"	60	EE
1205	11H	"	"	"	30	30.8
1210	11H	"	"	"	"	27.0
1215	7	"	"	"	"	27.0
1220	5H	"	"	"	"	26.6
1225	1H	"	"	"	"	26.6
					Average	26.88
1300	16	1400	1399	1406	30	51.2
1305	10	"	"	"	"	51.1
1310	5	"	"	"	"	51.1
1315	2	"	1400	"	"	51.1
					Average	51.12
1340	20	1300	1296	1297	12	50.0
1345	15	"	1298	1299	"	49.2
1350	10	"	1299	1300	"	48.9
1355	5	"	"	"	"	48.8
1400	2	"	"	"	"	48.8
					Average	48.92
1430	15	1200	1198	1192	6	70.3
1435	10	"	1199	1193	"	69.8
1440	5	"	"	"	"	69.6
1445	5H	"	"	"	"	69.6
1450	0	"	"	"	"	69.4
					Average	69.60
1515	20	1100	1096	1085	1.5	64.8
1520	15	"	1098	1087	"	63.4
1525	10	"	"	1088	"	62.4
1530	5	"	1099	1088	"	62.3
					Average	62.70
SPINDLE CONSTANT CALCULATION						
	Log Viscosity					
	Fulcher Eq.	Reading	Avg. Reading	Spindle Const.		
Temp.-°C	Poise	FFM	% Torque	K		
1513	130.08	30	26.88	145.2		
1406	274.3	30	51.12	161.0		
1299	668.1	12	48.92	163.9		
1193	1947	6	69.6	167.8		
1088	7211	1.5	62.7	172.5		
$K = [Visc(P) \times RPM] / \% \text{ Torque}$						
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CALIBRATION WITH NIST 717a					Attachment 3	
Date:	8/27/98	FFS			Glass Wt:	5.966 gr
Time	Minute	Set Point	Furnace	Sample	FFM	Torque
	Remaining	Temp.-°C	Temp.-°C	Temp.-°C	Setting	Reading
1050	31	1500	1501	1512		
1055	27	"	1500	1511	30	38.3
1100	23	"	"	1512	"	25.8
1105	18H	"	"	"	"	22.2
1110	"	"	"	"	"	21.7
1115	"	"	"	"	"	21.9
1120	13	"	"	"	"	21.7
1125	8	"	"	"	"	21.6
1130	2	"	"	"	"	21.6
					Average	21.70
1150	24	1400	1395	1400	30	43.3
1155	20	"	1397	1402	"	42.4
1200	16	"	1398	1405	"	42.0
1205	12	"	1399	"	"	41.7
1210	7	"	"	"	"	41.8
1215	3	"	"	1406	"	41.7
					Average	41.80
1240	23	1300	1295	1293	12	41.8
1245	18	"	1298	1297	"	40.2
1250	13	"	1298	"	"	39.8
1255	9	"	1299	"	"	39.6
1300	5	"	"	"	"	39.5
1304	1	"	"	"	"	39.5
					Average	39.60
1325	23	1200	1195	1186	6	60.2
1330	18	"	1197	1190	"	57.5
1335	13	"	1198	1191	"	56.5
1340	8	"	1199	1192	"	56.1
1345	3	"	"	"	"	55.9
					Average	56.17
1410	21	1100	1095	1081	1.5	53.9
1415	18	"	1097	1085	"	52.2
1420	13	"	1098	1086	"	50.8
1425	8	"	1099	1087	"	50.4
1430	3	"	"	"	"	50.0
					Average	50.40
SPINDLE CONSTANT CALCULATION						
	Log Viscosity					
	Fulcher Eq.	Reading	Avg. Reading	Spindle Const.		
Temp.-°C	Poise	FFM	% Torque	K		
1512	130.9	30	21.7	181.0		
1405	276.4	30	41.8	198.4		
1298	674.2	12	39.6	204.3		
1192	1969	6	56.17	210.3		
1087	7312	1.5	50.4	217.6		
		$K = [\text{Visc}(P) \times \text{RPM}] / \% \text{ Torque}$				
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**Westinghouse Savannah River Co.
Savannah River Technology Center**

**WSRC-RP-98-00737
Revision 0**

Distribution:

**E.W. Holtzscheiter, 773-A
C.R. Goetzman, 773-A
N.E. Bibler, 773-A
L.F. Landon, 704-1T
C.T. Randall, 704-T
D.A. Crowley, 773-43A
J.R. Harbour, 773-43A
D.F. Bickford, 773-43A
C.M. Jantzen, 773-A
D.K. Peeler, 773-43A
A.D. Cozzi, 773-43A
T.L. Fellingner, 773-A
I.A. Reamer, 773-A
J.M. Pareizs, 773-A
R.J. Workman, 773-A
STI, 703-43A, (4)**

RECORDS ADMINISTRATION

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