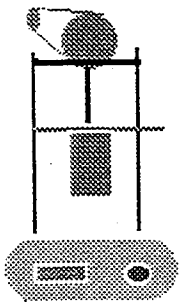
Capillary Rheometer Manual
for
KAYENESS Inc. Models
Galaxy III, IV and V



Kayeness Inc.

Version 2.02

SI Units

TABLE OF CONTENTS

I. Introduction

A. How to Use this Manual

This manual describes the setup procedure and basic operation of the *Kayeness* Galaxy class rheometers. With the accompanying KARS software manual (if software was purchased) it provides the complete description of resources at your command. It is not necessary to read this manual in its entirety; however, even experienced rheologists and technicians can benefit from the SAFETY tips and cleaning suggestions learned over many years of operating these machines in our in-house labs.

The Getting Started Chapter explains the details of setting up the instrument, important safety issues and walks you through two standard tests. Experienced users may wish to skip over the initial runs if familiar with entering programs into the rheometer.

B. Typographic Conventions

Typefaces are used in the following fashion:

Italics : Rheological items which have defining equations presented in the manual are shown in italics. If you come across an italicized item which is unclear you can be sure it has a mathematical definition previously defined in the manual.

Bold Italics : These are parameters which are set from the front panel on the rheometer (i.e. ***Melt Time, Delay Time*** etc.). These parameters are entered into the rheometer's control programs via the rheometer key pad.

BOLD ALL CAPITALS : This indicates an actual key found on the rheometer key pad. It is used in instructions like: Press the **RESET** button to stop the machine movement.

Underlined Items : Underlined items head paragraphs or sections which pertain to the particular item or model underlined. If you do not have or are not interested in the underlined item skip the section that follows it. Underlining is also used to emphasize safety issues.

This manual was produced using WORD® for windows. Figures from Kayeness's Advanced Rheology Software (KARS)© were imported directly from plot files made using ALT-F3 from the KARS© graphics screen. Figures can also be imported into WordPerfect® using the Graphics Retrieve command.

C. How to Contact Kayeness Inc.

Before calling Kayeness be sure you have gone through the "Answers to common questions" section of the manual. To help us handle your questions as quickly as possible, have the following items ready before you call:

- Machine name and model number
- Machine serial number (on back panel)
- Computer system make and model
- Current version of software (top left on start up screen)

Call Kayeness directly at (610) 286-7555 and ask for customer service, should you wish to comment or query in writing address it to:

Kayeness Inc. : Customer Service
115 Thousand Oaks Blvd., POB 709
Morgantown, PA 19543

You can also reach us through compuserve ID's 73537, 644 or 73642, 1405

II. Getting Started

A. Uncrating and Setup

1. Bench Requirements and Placement

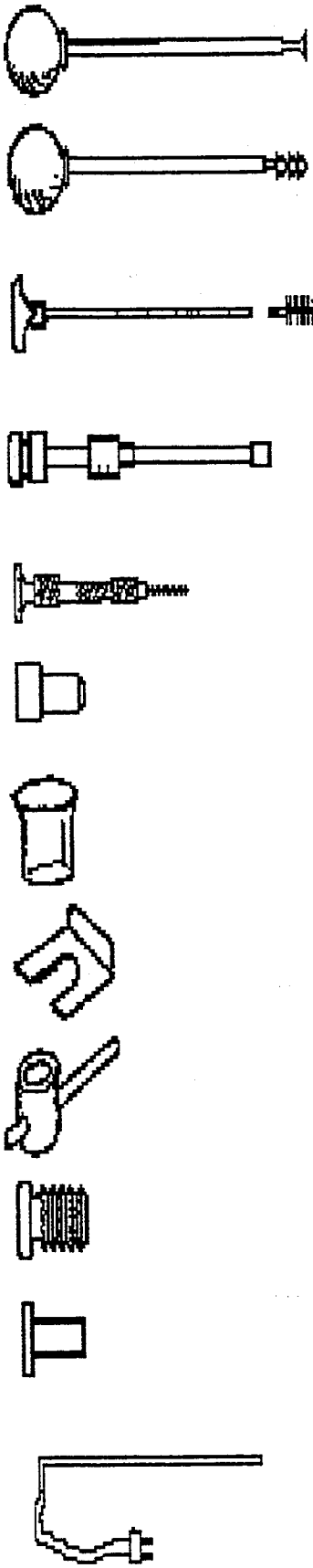
Typical laboratory benches are too high for efficient use of the rheometers. Cleaning can be difficult and requires awkward hand positions and forces which could lead to carpal tunnel syndrome or back discomfort. We strongly suggest a bench height of 29 inches (desk top height) for an average height operator. Place the front of the rheometer flush with the edge of the table. This will prevent the operator from having to bend forward excessively when cleaning the barrel and allow easier access to the back of the machine. As a minimum, the lab bench should easily be able to support the rheometer and operator (approximately 500 lbs.). The bench top should also be able to withstand hot dies and tools being dropped on them. Carpet protection is necessary near the rheometer since a hot die dropped on the carpet will quickly burn spots in them.

Adequate ventilation will also be required to remove potentially harmful fumes from samples being tested. Consult the Material Safety Data Sheets (MSDS) on the products to be tested and your material supplier to assess the magnitude of your ventilation needs. You may wish to consider these ventilation needs when positioning the instrument in the laboratory.

2. Unpacking the Rheometer

Most of the machine comes pre-assembled to your door, however certain parts are prone to breakage if they were placed in their normal operating position during shipping. These items will need to be installed before safe operation of the machine is possible. These items include:

The load cell, PRT temperature sensor, the load cell safety shield, the top support shield (Galaxy V & III only), Cable connection for PC (Computer), printer, plotter, AC Power connections.



Supplied Hand Tools

1. Charging tool for compacting pellets into barrel. Relief area to allow air to escape during packing. Part # 0051-36

2. Cleaning tool for barrel . Use with two patches for proper friction. Part # 0051-40

3. Orange handled cleaning rod with bronze scouring brush. Use after primary cleaning tool to get the barrel nicely polished. Handle Part# 0051-47, Bronze brushes 0051-48

4. Plunger for main rheometer barrel. Guide bushing should move freely up and down. Seat guide bushing in place prior to pressing RUN on rheometer. Complete assembly Part # 2052-300, Tip is removable, Tip Part # 2052-66; Sliding guide bushing Part # 0051-44

5. Clean out drill bit and drill bit vise for cleaning capillary dies. Bit Vise Part # 0051-38, specify die size.

6. Sample filling funnel. Fits into counter sink on top of barrel. Part # 0051-45

7. Pyrex beaker used for sample filling of pellets or powder. Part # GP0300.

8. Pull clip to automatically pull out plunger (use optional). When plunger is in barrel with no ram movement place pull clip into slots in load cap connected to the load cell. Press UP to remove the plunger. Part #2052-151

9. Die nut removal wrench. 7/8" T handle. Tighten only until snug. Part # 2052-156.

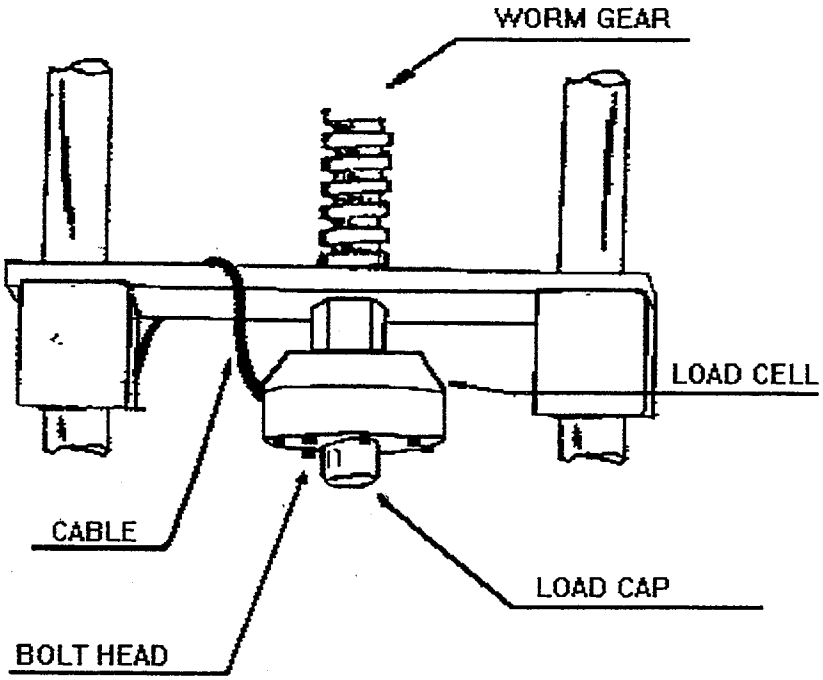
10. Die nut. The capillary die slides into this. It is then screwed into the base of the barrel and allowed to heat up before tightening. Part # 2052-73

11. Rheometer Die. Tungsten Carbide. Don't drop hot die into solvent! See table in appendix for ordering information.

12. PRT platinum resistance thermometer. Temperature sensor for measurement and control of temperature. Has red banana clip connector. Part # GP1752

The optional power cleaning tool is a reversible drill with a steel shaft topped with a knurled aluminum cap to hold patches. This is a battery powered unit that makes cleaning much easier. It comes with a rechargeable battery and charging unit and lasts about a week per charge in your average laboratory (works great with indexers too). Part # 8052-97K

3. Load cell assembly



Find the round load cell (blue) with connecting cable and be sure it is NOT labeled calibration cell. The actual deflection (which measures the force) occurs on the side of the cell which has the exposed bolt heads. The opposite end is considered the mechanical ground. Screw the plunger load cap (hollow-cup shaped with screw threads on one end) into the side of the load cell which has the exposed bolt heads (active end). The rheometer plunger will fit into the load cap when a test is underway. Depending on your machine connect the load cell to the cross-head of the machine as follows:

<u>Galaxy III & V</u>

Screw the open end onto the bolt in the center of the cross head located between the two rheometer posts and directly above the barrel hole. Be careful not to twist the cable excessively while screwing the load cell into place. A two handed snug tightening is all that is required. Loop the wire once around the cross members on the left side (when facing the rheometer keypad) then connect the lead to the only mating gold plated connector on the back of the rheometer.

Galaxy IV

Bolt the load cell to the bottom of the cross head member with the Allen head bolt provided. Connect the load cell wire to the plug located on the top left side of the machine just to the left of the left actuator post.

4. Connecting the Barrel Pressure Transducer Plug

(Skip this section if you do not have the barrel pressure transducer option).

The first trail tests should be run without the pressure transducer in place. Find the pressure transducer plug. It is a rod with 1/2" 20 thread shaped on the end like a pressure transducer. Put a small amount of anti-seize material onto the threads and insert it into the barrel pressure transducer hole located on the backside of the rheometer barrel. This is easiest to do by pulling the swing arm fully forward. Tighten the plug until only "snug" when the machine heats up it will tighten more.

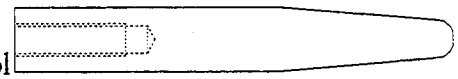
5. Installing an O-ring on the plunger tip

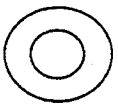
(Skip this section if you have not ordered an O-ring seal option).

You may also have purchased a grooved tip which allows for the use of O-ring on the piston tip.

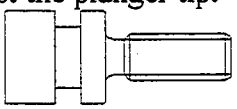
Tools & Parts

1. TFE O-Ring Insertion Tool
(Kayeness Part No. 2052-152)





2. TFE O-Ring (Kayeness part No. 2052-66R)
For testing low viscosity materials at temperatures below 300 °C a PTFE O-ring can minimize leakage past the plunger tip.



3. O-Ring Grooved Tip
(Kayeness Part No. 2052-66G)

Remove the O-Ring Grooved Tip from the piston rod assembly. If the tip is difficult to remove, try heating the tip up to melt/loosen any polymer that may have gathered around the tip edge. If the tip is still stuck, use a vise to grip the rod, but first carefully wrap the rod in a thick towel or other suitable material to protect the rod surface. With the rod in a vise, carefully wrap the tip with fine grit sandpaper (500 or higher, crocus cloth is preferred). Wrap the tip with a thick layer of sandpaper - enough so a set of pliers' teeth will not mar the tip surface. Then carefully use some pliers to nudge the tip loose.

With the tip free, screw the tip into the insertion tool, and then remove the old o-ring from the groove using a knife. Soften the new o-ring by heating

it (the top of the barrel works well), and insert the new ring over the tool and into the groove in the tip.
The new o-ring must now be sized to fit the barrel. To do this, apply a small amount of anti-seize to the tip threads and reassemble the tip onto the rod. With the barrel at its normal operating temperature and the die removed, try to install the tip and o-ring into the barrel. While twisting the rod clockwise (counterclockwise will loosen the tip), slowly push the tip into the barrel, removing the extra o-ring material and conforming the new o-ring to the barrel size. This operation will actually shear a small amount of the o-ring off. Once the tip and new o-ring can be inserted into the barrel, clean the barrel. The machine is now ready to be operated again.

The first one or two runs with a new o-ring will serve as a break-in period for the o-ring, and after this, the runs should be very consistent. A new o-ring will be required only if the plunger forces begin to drift lower or if the amount of material that is escaping past the tip and up the rod during a test (blow-by) exceeds 0.1g. Do not use this tip without an o-ring. If an o-ring is not used, the blow-by of material for most polymers will far exceed the 0.1g limit causing the plunger forces and shear stresses to be too low.

6. Connecting the PRT (temperature sensor)

Current machines require no installation of the PRT probe as was done with earlier version of the rheometers. The Galaxy III, IV and V machines come with the PRT already installed under the top of the barrel insulator plate.

7. Safety guards assembly

Remove the four screws found on the outer edges of the aluminum blocks found on the cross head . These screws will hold the small clear plastic shield in place over the load cell. Remove the protective paper covering from the shield and secure the latter in place with the recently removed screws. Be careful not to scratch the clear plastic with the screw driver.

The larger shield covers the pulley system on the Galaxy III and V models. Remove the four screws found on outer edges the upper (stationary) aluminum blocks. Remove the protective paper covering from the shield and secure the latter into place with the longest portion on top. Be careful not to scratch the clear plastic with the screw driver. WARNING: Be sure the longest portion of the shield is at the top otherwise the top shield may collide with the bottom shield. The shield should NOT come into contact with any part of the pulleys or bolt heads on the pulleys.

8. Power connection

First find the surge protector/power box. This is an extension cord of sorts with many sockets available for use. Plug it in and make sure the power is

OFF (the on switch should not be lit). There are a number of power cords supplied with the rheometer, the thickest (heaviest) of these is used for the rheometer. Connect the thickest cord to the back of the rheometer and into the isolated filter bank #3 in the power box. Power to the PC, and monitor should connect to the isolated filter bank #1. The printer and plotter can be connected to any remaining outlet.

9. Printer connection

Using the printer with KARS

When using the KARS software package the printer should be connected directly to the PC. Typically this is done with a 25 pin DB25 connector to the parallel port on the PC into the centronics connection on the printer. This cable is a standard IBM parallel port printer cable.

Connecting Printer Directly to the rheometer

If your PC is broken or you do not wish to use the PC, connect the printer directly to the rheometer by attaching the female DB25 connector directly to the DB25 male connector on the back of the rheometer. The rheometer has only one connector which mates with the printer cable. Connect the other end to the printer as usual. Be sure the printer is OFF when connecting this cable!

10. Computer connection

The data processing system (optional) consists of a PC and the KARS software package. The link between the PC COM1 port and the rheometer is made via a DB9 on the PC side to a round AMP connector on the rheometer side. Generally there is only one round 9 pin connector on the back of the rheometer to connect to; however, sometimes real time analog force output is also supplied which has the same type connector. Connect the PC COM1 line to the 9 pin round amp connector on the back left side of the rheometer which does not have a round zero adjust knob above it. This knob adjusts for a zero offset on the analog force signal (when the option is requested).

11. Plotter or Laser Micrometer Connection

If you have only one of these devices (Plotter or Laser micrometer) it can be connected directly to COM2 on the PC. If both devices are to be used; first, find the cable connecting the laser box (not the scanning unit) to the PC. The cable connects to the back of the Series 900 Laser scanner box to the 25 pin connector labeled SERIAL. The other end of the cable, a 9 pin (DB9) connector, goes into Channel A of the switch box. Second, connect the end of the molded cable (DB25, 25 pin) to the plotter serial connector and the 9 pin (DB9) side to Channel B of the switch Box. Finally connect the output from the switch box (middle bottom) to the DB9 COM2 port on the back of the PC. Leave the switch box set to Channel A (LASER) normally, this will allow KARS to initialize the laser when the KARS

program is started. Switch to Channel B only when plotted output is desired. Laser data collection will not be possible when the plotter is being used.

12. Software installation

If you purchased your PC from Kayeness the software should already be installed on your computer. If you have accidentally erased the program or have purchased your own computer with the rheometer system, insert the KARS floppy diskette in drive a: then type a:install. KARS will then install itself onto the hard drive C: creating the directory path c:\KARS if it does not exist. If the install routine finds an existing directory it will only delete the main KARS program replacing it with the current version on the floppy. It may add additional example files to those that exist. No data directories nor data files in the c:\KARS directory will be deleted. Should you wish to save the current version of KARS perform the following steps prior to installation or re-installation from floppy.

```
c:
cd\kars
ren kars.exe karsbu.exe
```

To run the old version of KARS simply type KARSBU to start the old KARS program.

Should you have any problems starting the program delete the initialization files **last.kay** and **defaults.pcs**. Kars will re-generate these files next time it runs.

13. Turning on the Rheometer

After following all the previous steps, be sure there is nothing to prevent the crosshead on the rheometer from moving up or down. With the power turned OFF pull out the red **EMERGENCY STOP** button on the front panel of the rheometer. Turn on the power to the main power box, then turn on the rheometer by pressing down the top half of the black switch to the right of the emergency stop button. Except in an emergency, never interfere with the initial movements of the rheometer (i.e. DO NOT press **RESET**) until the machine has reached ***Park Position*** and stops moving. The rheometer is resetting its Position based on the upper limit switch stop. If it is stopped during this process the machine position will be incorrect. Simply shut the machine off wait for 10 seconds then turn it on. Let the machine warm-up for a minimum of 20 minutes before performing any tests.

IF NOTHING HAPPENS

If nothing happens when you turn the machine on, check that the **EMERGENCY STOP** button is pulled out (be sure to press **RESET** before pulling out the emergency stop button) and all power connections

have been made. If still nothing happens proceed to the troubleshooting section.

14. Firmware Defaults

Firmware is the programming embedded into computer chips (EPROMS etc.) found inside the rheometer. The firmware governs most of the rheometers behavior and all of its communications to outside devices like the PC or a directly connected printer. Software (like KARS) runs on your PC and helps you save and analyze your data. When a rheometer system is built the firmware needs to know specific details about the machine in order to function properly. Press **SHIFT** then **9** on the front panel of the rheometer and the rheometer reveals the machine ID code. The following is a table of ID codes you can use to verify that code. A lightening strike or major brownout has been known to change this code.
If your configuration code differs from the one in the table you should first make sure you know the contents of all the programs in memory. In order to correctly enter the proper machine ID code the machines memory will need to be cleared ERASING all current programs (Test conditions!).

Clear all the program memory by pressing **SHIFT NO** the rheometer front display will show CLR ALL MEMORY? press **YES** to clear the memory then press **SHIFT 9** and enter in the appropriate code. Caution! All programs previously set up in the rheometer will need to be re-entered.

Machine Configuration Codes

Configuration Codes (SHIFT 9) Rheometer	Standard 4400 N Load Cell	High Force 8800 N Load Cell	Low Force
	1000 LB load cell	2000 LB load cell	
Galaxy III 9052	0034	--	0162 (500 LB)
Galaxy IV 0052	0098	0114	0226 (250 LB)
Galaxy IV 0052-72 (LS)	0099		0227
Galaxy V 8052			
A pulley (8:1)	0002	0018	
B pulley (4:1)	0001	0017	
C pulley (2:1)	0000	0016	
Metric Galaxy V	0005	0021 (9900 N)	
Metric Galaxy III	0036		

B. SAFETY

1. Use gloves its HOT!

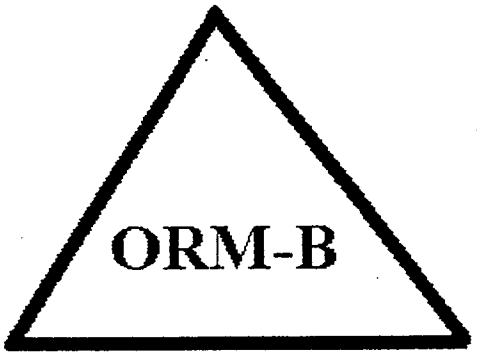
Gloves and a long sleeve shirt (or lab coat) are essentials needed to prevent burns. Dies, die nut holders and piston rods are extremely hot and are designed to transfer heat quickly to the sample being tested, unfortunately this means they will transfer heat very quickly to you as well. Even brief contact with a hot item can cause a burn. The barrel swing arm and connecting lower barrel cover can also get fairly hot, however at barrel temperatures lower than 350 °C these will not cause burns if touched for a brief period. Keep the swing arm free of items since they will fall off during opening or closing, plastic pens or other items may also melt! Consider where dies or the die nut holder may fall. If they are dropped on Nylon carpet or similar materials they can quickly form holes. Protective mats may be needed.

2. Electrical Hazard

Your Kayeness rheometer contains high voltage inside the stainless steel base. When this steel cover is opened a plastic cover is revealed. The plastic shield aids in protecting you from these voltages. DO NOT remove this cover unless you are instructed to do so by a KAYENESS representative or are experienced with high voltage devices. There are holes cut in the shield which allow access to the necessary items used in routine calibrations. Be sure the outlet used to power the rheometer is properly grounded.

3. Calibration Thermometers use Mercury

To calibrate the temperature on the rheometer a thermometer containing about 8 grams of mercury is used. Every lab with mercury thermometers or equipment containing mercury must be prepared for breakage. Note that mercury exposed to air "evaporates" at room temperature, producing an invisible, tasteless, odorless and dangerous vapor. Thermometers have been used for decades in laboratory equipment and when used properly provide an accurate and effective means of calibration. Keep the thermometer in a safe place were it will not be crushed or otherwise broken. When using the thermometer be careful not to drop or bend the glass. Place a hot thermometer onto cotton patches to cool . Never put a hot thermometer in contact with cold metal or cold solvent, the thermal shock can crack or shatter the glass. Mercury is extremely toxic and should be handled accordingly. A material safety data sheet (MSDS) for mercury (Hg) can be found in the appendix. Observe local, state and federal hazards waste disposal laws when disposing of any broken thermometers. You can find the names of mercury spill kit suppliers in the appendix under support vendors. If packaged in a sealed plastic container and labeled with the following symbol:



broken thermometers and their spilled mercury can be sent back to the manufacturer. UPS will accept these packages provided they are labeled and the material is in a secure container. See Princo support vendor for address information.

4. Pinch Points

When the machine is in operation the crosshead (which holds the load cell and load cap) moves downward creating an area where anything lying on the swing arm could potentially get crushed. When the **RUN** button is pressed the FULL FORCE of the machine can be brought to bear. When the down button **DN** is pressed on the rheometer a 2 to 3 LB safety load limit on the plunger will stop the movement of the cross head / plunger. In addition all front panel keys on the rheometer become stop keys when **DN** is active. Any item between the cross head and the lower swing arm which does not effect the load reading is not "seen" by the rheometer and will be crushed! The **DN** button should be used for routinely moving the cross-head down to do force calibrations or to check motor operation. The operator should be sure the area between the cross head and the rheometer swing arm is clear prior to any machine movement. The large red **EMERGENCY STOP** button will stop any machine movement and allow the pulley system to be rotated by hand (if needed). The **EMERGENCY STOP** button is similar to a clutch on a car. When engaged it disconnects the motor from the driving mechanism when released it re-engages any current commands. Press RESET before pulling the emergency stop button out when the machine is on. This is like putting the car in neutral before letting the clutch out.

An optional front guard with power interlocks can be provided. This shield is tied to a mechanism which prevents operation of the machine until the shield is in place. The shield inhibits access to the area between the cross head and the swing arm.

5. Don't Remove PRT

Don't remove the platinum resistance thermometer (PRT) **UNLESS**, the set point temperature (Melt Temperature) has been set to zero (0.0). If you wish to check the temperature in the barrel use the separate hole drilled just for the thermometer which can be found by removing the small

Allen head screw just in front of the PRT well. The PRT is a 2-3 mm thin rectangular section of platinum wire grid which is used to measure the temperature. It is housed in a 4.75 mm (3/16") steel tube about 125 mm (5 inches) long with a 90 degree bend on one end. On Galaxy V machine this tube is just to the left of the top of the rheometer barrel under a protective metal shield.

When the PRT is removed with the set point temperature higher than room temperature the machine senses cold and pumps more heat to the heaters. This can ruin the heat treatment on the cylinder and supporting insulators if the temperature gets too high. **Always set the temperature to 0.0 before removing the PRT!** If you are not calibrating turn the machine off when removing the PRT.

The Galaxy Series rheometer have the PRT probe under the top insulator plate. The top insulator plate must be removed to remove the PRT.

6. Fumes from Materials

Plan for the unexpected when it comes to materials giving off hazardous vapors. Many polymers (PVC etc.) are well known for giving off hazardous fumes at elevated temperatures. An exhaust system which removes fumes from both the die exit and near the top of the barrel is strongly recommended. Consideration should also be given to additives which may degrade or decompose at elevated test temperatures.

7. Before Testing Safety Check

- 120V power outlet properly grounded? (220 V Europe/Asia)
- Rheometer on sturdy level bench?
- Exhaust hood or snorkel operational?
- Operator safety glasses?
- Operator high temp gloves?
- Arm protection long sleeves or lab coat
- Protective oil wiped out of barrel?
- PRT (temperature sensor) in barrel?
- Safety shields installed?

8. Purging the machine

The rheometer barrel leaves the factory coated with oil to protect it from rust. This oil must be removed before accurate rheological data can be obtained. Put two cotton patches half overlapped directly over the top of the barrel of the still cold machine. Use rheometer cleaning tool #2 pictured on page 8 to run the patches up and down the full length of the barrel. The die and die holder nut should be removed. Repeat the process

with fresh patches until the patches come out clean.

Turn on the machine . If you hear a continuous beeping sound, turn the machine off and be sure you have connected the PRT temperature sensor and the load cell correctly as described under Getting Started. If the machine's front panel lights up but there is no movement on the cross-head, shut the machine off, pull out the **EMERGENCY STOP** button, then turn the machine back on. If the machine's cross head moves up then down and stops it is operating properly and you may continue. If the machine is not working correctly proceed to the trouble shooting section.

Press **EDIT** on the front panel of the rheometer
Press **YES** until the word TEMP. = XXX will appear.
Press 2 3 0 then **YES;** the top display should show 230 for a second or two then the current temperature will be displayed. You should see it rising slowly to 230° C.

At 230° C the machine is sufficiently hot that we can purge it with Polyethylene (PE) or Polypropylene (PP). A purge is simply a charge of material run through the machine without collecting data. The purge run helps eliminate any remnants from previous tests. It also coats and fills micro cavities in the metals which can be a cause of variability if the first charge of material is treated like the following charges.

Putting the Die in the Barrel

The machine is shipped with a die (Tool #11, Page 8) in the die nut and the die nut is screwed into the bottom of the barrel. The die holder nut threads come coated with a thin layer of high temperature anti-seize to keep the die holder nut from seizing in the barrel. Drop the die into the die nut then screw the nut into the bottom of the barrel. Use the die wrench to just snug the die nut into place **DO NOT OVER TIGHTEN.** As the die heats it will tighten. See Page 8

Loading the Barrel

Turn the round ball handle on the right side of the swing arm counter clockwise and pull the swing arm open until it is in a comfortable position for you to load the sample. Don't be afraid of opening it too far; you can't. Use the 10 mL beaker to scoop a heaping beaker of pellets from the sample you wish to run. Get the filling funnel and place it directly over the top of the barrel. The lip on the base of the funnel will fit onto the top of the barrel insulator. Holding the funnel in your left hand gently pour about 2/3 of the pellets from the beaker into the funnel (you may need to tap the beaker against the funnel lightly). After putting in only 2/3 of the beaker content use the packing tool to firmly press the material down into the barrel. Push down until you feel little movement of the packing tool. It is not necessary to use large amounts of force in packing.

Note: Try not to leave the packing tool in the barrel for too long.

If the packing tool heats up material will melt around it and make packing difficult.

Add the remaining material and pack it down as before. A good rule of thumb for knowing whether you have enough material in the barrel is that the end of the packing tool relief area (where the diameter gets big again) should be about level with the top of the barrel. Adding too much material is a common mistake for beginners and will make it difficult to get the plunger and guide bushing into place.

Place the plunger into the barrel giving it a twist clockwise (looking down from above the plunger) be sure the guide bushing slides down into the top part of the barrel. This guide bushing forces the plunger top to go directly into the load cap attached to the load cell. Check to be sure the plunger is not moving (some materials can force the plunger upwards, especially if overfilling or degassing occurs) then close the swing arm. Tighten the ball capped screw to fix the swing arm in position. Let the material heat up for about 3 minutes or longer. Time is not critical here; we merely want to give the material enough time to melt so we don't over force the machine trying to push it out. After about 3 minutes press **SHIFT** then **DN** (down key). The front panel of the rheometer will display ***Purge?*** press **YES** to empty the contents of the barrel through the die. The rheometer will drive down until it sees 2 to 3 lbs of load. This occurs when coming into contact with the plunger. The rheometer will then increase the plunger speed until either maximum speed is achieved or half the ***Terminal Force*** is felt by the load cell. The plunger is always driven to the bottom limit switch setting which is about 3 mm (1/8 ") above where physical contact between the plunger and die would be made.

9. Cleaning Up

Loosen the ball screw handle on the swing arm and pull the swing arm and barrel forward for cleaning. Put two patches directly over the barrel about 1/2 way overlapped. Using the cleaning tool push the patch down into the barrel. Run the patch up and down a half dozen times or so, then repeat the process. The second set of patches should come out fairly clean, if not repeat the process until they come out clean. When done put the plunger back into the barrel. This allows the plunger to heat up before the next test.

With materials that are thermally stable (less than 5% viscosity change over 1/2 hour) we recommend only cleaning the barrel between runs of the same material. For materials that degrade we recommend cleaning both the barrel and the die completely. You can clean the die by first running a drill bit up into the die while it is hot and in the rheometer. This will make it much easier to get the cleaning drill bit in when the die is removed and the material starts to solidify. Remove the die by extending the arm on the die nut removal wrench to its full length. Attach the socket completely over

the die nut. A quick hit on the end of the socket arm is all it takes to remove even an over tightened die. Slide the arm back to its center position on the wrench and spin the wrench while pressing up with one finger below the socket. Be careful as the die is very hot when it comes out. If you don't use gloves you will get burned eventually. Using a cotton rag wipe the top of the die clean. Be sure to get the material out of the conical top section, if your die has such a tapered entrance. Run the supplied drill bit into the die. Remove material that collects in the grooves and repeat until the drill passes easily through the die. On long dies it may be necessary to clean from both ends to completely clean the entire length of the die. While the die is out look down the barrel bore to be sure it is clean. If it is not run a couple of patches up and down it before putting the die back into position. When the die is out of the barrel it cools down quickly. The longer it is out the longer the wait for the temperature to stabilize. Minimizing the time the die stays out of the machine will increase the number of tests you can run. When the temperature on the front display is within two degrees of the setpoint you can begin your next test. Loading material will cause a small temperature change even if temperature setpoint was achieved. The melt time (360 seconds) will allow ample time to get the temperature to setpoint before the first data point is collected. Always leave the machine clean. If it is going to sit at room temperature for an extended period of time you may wish to coat the barrel with a light machine oil to prevent rusting. The oil will need to be purged from the machine before accurate data can be obtained.

For reference, the difference between a die and an orifice is that an orifice is a very short length die.

10. Your First Test: A Time Sweep Step-by Step

The first test you should perform on the machine is a standard thermal degradation run or a time sweep. It is called a thermal degradation run because it looks to see how the viscosity of the material changes with time, typically the longer the time, the more the material degrades. The run is very simple, we will ask the machine to move at only one speed 5 mm/min. (ca. 0.2"/min.) and take measurements at positions in the barrel that are 5 mm apart (1 minute per measurement). Additionally we will ask that the machine pause (stop plunger movement) for two minutes after each data point is taken.

Press **EDIT** on the front panel of the rheometer to begin setting the rheometer program. Continue by pressing the keys under the PRESS THESE KEYS column in the table below. Don't worry too much about what it means right now, we will cover more details as we go along.

Setting a Time Sweep rheometer *PROGRAM*

Note press **EDIT** on the front panel of the rheometer to start the process

Machine Responses	PRESS THESE KEYS	Comments
Melt Time = XXX.X	**"NO" 2 4 0 "YES"**	XXX.X is any given number, in seconds
MATL ID= ????????	**"NO" A S K "YES"**	NO clears, ++ makes "B" appear. YES accepts value.
TEMP. = XXX.X	**"NO" 2 3 0 "YES"**	NO clears, 230 C entered. YES accepts value.
ORIF.DIA..=X.XXX	**"NO" 1 0 0 0 "YES"**	Assumes capillary die is 1mm in diameter.
ORIF. LEN.=XX.XX	**"NO" 2000 "YES"**	Assumes capillary die is 20 mm in length
SAMPL LEN.=XXX.XX	**"NO" "YES"**	Set sample rate to zero for rate run
RATE #1= XXX.XX	**"NO" 00500 "YES"**	Set Ram Rate to 5mm/min
RATE #2= XXX.XX	**"NO" 00500 "YES"**	Set Ram Rate to 5mm/min
RATE #3= XXX.XX	**"NO" 00500 "YES"**	Set Ram Rate to 5mm/min
RATE #4= XXX.XX	**"NO" 00500 "YES"**	Set Ram Rate to 5mm/min
RATE #5= XXX.XX	**"NO" 00500 "YES"**	Set Ram Rate to 5mm/min
RATE #6= XXX.XX	**"NO" 00500 "YES"**	Set Ram Rate to 5mm/min
RATE #7 = XXX.XX	**"NO" 00500 "YES"**	Set ram rate to 5mm/min
RATE #8 = XXX.XX	**"NO" 00500 "YES"**	Set ram rate to 5mm/min
RATE #9 = XXX.XX	**"NO" "YES"**	Zero rate means no more speeds
MELT FORCE=XXXX	**NO 1000 "YES"**	1000N Pre-Start Force to Pack
TERM FORCE=XXXX	**"NO" 3300 "YES"**	3300N Safety Overload
START POS.=XXX.XX	**"NO" 16000 "YES"**	Begin test at 160mm from top
POS. #1 = XXX.XX	**"NO" 17000 "YES"**	Force acquired at 170mm position
POS. #2 = XXX.XX	**NO" 17500 "YES"**	Force acquired at 175mm position
POS. #3 = XXX.XX	**"NO" 18000 "YES"**	Force acquired at 180mm position
POS. #4 = XXX.XX	**NO" 18500 "YES"**	Force acquired at 185mm position
POS. #5 =XXX.XX	**"NO" 19000 "YES"**	Force acquired at 190mm position
POS. #6 = XXX.XX	**"NO" 19500 "YES"**	Force acquired at 195mm position
POS #7 = XXX.XX	**"NO" 20000 "YES"**	Force acquired at 200mm position
POS. #8 = XXX.XX	**"NO" 20500 "YES"**	Force acquired at 205mm position
PARK POS. = XXX.XX	**"NO" 05500 "YES"**	Crosshead Park at 55mm position
TEST DELAY=XXX.XX	**"NO" 1200 "YES"**	Pause for 120 sec between points
RUN# START	**"NO" "YES"**	Clears RUN# to zero
PROGRAM ENTERED		

If at any time you wish to start again at the beginning of the program entry press **RESET** then **EDIT.** If a program parameter is already set to the correct value simply press **YES** to accept it, there is no need to type it in again. Pressing the **NO** key clears the current numeric value to zero. Pressing **NO** on non-numeric values causes them to toggle through the various items allowed. Once a program is entered the machine will remember it, even if the power is turned off for an extended period.

Get the Die Diameter Right!

If your die diameter is not 1mm but 1.25mm make sure the line is:

(NOTE: older machines may call for you to enter the **radius** of the die not the diameter)

ORIF. DIA.=1.25
not ***ORIF. DIA.***=1.0

similarly if the die length is 25 mm

ORIF. LEN. *=25.0*
not ***ORIF. LEN.*** =20.0

We have been conservative on the terminal force setting. This setting may be adjusted higher based on your load cell and system configuration. See you machine specifications/order for details.

Now you have told the machine the details it needs to know about running a test. This can be run over and over again without re-entering these machine control settings. We'll get into exactly what these settings mean later. Now let's enter a Sample ID and the operator initials.

Entering alphabetic characters on the rheometer

You'll notice that there are no letters on the key pad. Alphabetic characters are entered by press the +, - and **SKIP** buttons as a response to a question asking for alphabetic input like *Sample ID*, *Material ID* or *Operator ID*. To get the letter "A" to appear press the + (plus key). Pressing the + key again causes "B" to appear, press again "C" and so forth. Pressing - (minus key) causes the previous letter in the alphabet to appear. The **SKIP** key jumps 6 letter at a time through the alphabet. After the last character is selected press **YES** again to accept the entire word. To enter a Sample ID of "TEST" and an operator ID of "JFR" type the following. When the character you wish appears press **YES**.

Setting the SAMPLE ID name

Note Press **ID** key to start input of Sample ID

MACHINE RESPONSES	**PRESS THESE KEYS**	*COMMENTS*
SAMPLE ID = *????????*	**SKIP SKIP SKIP + +**	? is any letter
T	**YES**	YES accepts the letter T
E	**SKIP - YES**	E appears before YES pressed
S	**SKIP SKIP SKIP + YES**	
T	**SKIP SKIP SKIP + + YES YES**	Second YES accepts word TEST
OPER. = ???		Use + and - and SKIP to get your first Initial
?	**YES**	Press YES to accept letter
?	**YES**	2nd initial Use + and - andSKIP
?	**YES YES**	3rd initial, Final extra YES to accept all letters

Note that the - (minus key) wraps back around the alphabet thus pressing - four times gets you to "W" fast. If you are using a PC with KARS software you can set the ***Sample ID*** to the letters ASK when the data is transferred to the PC it will then ASK you for the ***Sample ID***. It is much easier to type the Sample ID at the computer keyboard. The ***Sample ID*** will be the PC's filename for the data.

OPTIONS PROGRAM

One last thing to check before we actually run the sample, is the Options. Press **SHIFT** then **PRNT** on the front panel of the rheometer. The machine will briefly display **OPTION PROGRAM**. After viewing the option proceed to the next item by pressing **YES** . You should see in order:

- MANUAL MODE off?
- PRINTER off?
- PC Attached: YES?
- Auto F Zero on?
- LASER Mike: off?
- MACHINE ID.=0004

If the display does not match the listing above press the NO button to change it to its other possible settings or in the case of MACHINE ID simply type in the 4 digit value you wish. When the display matches the above press YES to continue to the next item in the option. Turn on your PC and start the KARS program. Check to see that the connections are READY as shown on top left of your computer monitor. Leave the KARS program running on the PC, your rheometer will be talking to it during the run. The Rheometer should always be turned on first then your PC. This allows the PC to communicate to the Rheometer and set up the communications lines properly.

Direct connection to printer (No PC)

If you are not using a PC and have your rheometer connected directly to a printer,

Set **PRINTER ON** and **PC attached OFF .**

Performing the test:

Now that the machine is programmed to run the test all that is needed is to load material and start the rheometer. We suggest starting with an easy material like a polyethylene (PE, LLDPE or HDPE) or polypropylene (PP). Polyethylene and Polypropylene materials **do not** need to be dried to remove moisture. In other materials trapped moisture can have dramatic effects in lowering the viscosity of the material (e.g. PET, Nylon, PBT, PEEK and others).

Load the barrel as described on Page 18 under the purging section. Do not press **SHIFT DN** to purge the machine of its contents as before, instead press **RUN** to start your test. If KARS isn't already running on your PC now is a good time to start it. If the rheometer is stopped and showing F0 on its front panel then your computer or printer connection is not made correctly or the KARS program has not been started.

The test will run automatically from here on, when it finishes it will purge the remaining material and the cross head will move back up to the park position and data will be transferred to the PC or directly to the printer.

If you are running KARS on the PC a green box appears asking for any comments you may have. It then asks for a second comment. Press enter if you wish to leave the second comment blank. The just acquired data will be analyzed and appear on the front screen. Press F5 to show a plot on the computer screen and admire your work. Press ESCape to return to the front screen.

11. Your second Test : A RATE SWEEP

The second test you should perform is a standard shear rate run or rate sweep. It is called a rate sweep run because it looks to see how the viscosity of the material changes with the rate it flows through the die. For most polymers the quicker the flow rate the lower the viscosity. The run is very simple, we will ask the machine to move through 7 unique speeds, it will make measurements at positions in the barrel specified (more about how these are obtained later). We want the machine to perform the test as quickly as possible so we will ask that no time delay be added between each measurement point. Press **EDIT** on the front panel of the rheometer to begin setting the rheometer program, continue by doing the following:

Setting a Rate Sweep rheometer *PROGRAM*

Note press **EDIT** on the front panel of the rheometer to start the process

Machine Responses	PRESS THESE KEYS	Comments
Melt Time = XXX.X	"NO" 3 6 0 " YES"	XXX X is any given number, in seconds
MATL ID= ????????	"NO" ASK YES	NO clears, ++ makes "B' appear, YES accepts value
TEMP.= XXX.X	"NO" 2 3 0 "YES"	NO clears, 230 C entered, YES accepts value
ORIF. RAD.=X.XXX	"NO" 1000 "YES"	assumes capillary die is 1mm in diameter
ORIF. LEN.=XX.XX	"NO" 2000 "YES"	assumes capillary die is 20 mm in length
SAMPL LEN=X.XXXX	"NO" "YES"	Set sample rate to zero for rate run
RATE #1= XXX.XX	"NO" 3000 "YES"	Set Ram Rate to 300 mm/min
RATE #2= XXX.XX	"NO" 1000 "YES"	Set Ram Rate to 100 mm/min
RATE #3= XXX.XX	"NO" 03000 "YES"	Set Ram Rate to 30 mm/min
RATE #4= XXX.XX	"NO" 01000 "YES"	Set Ram Rate to 1 mm/min
RATE #5= XXX.XX	"NO" 00300 "YES"	Set Ram Rate to 3 mm/min
RATE #6= XXX.XX	"NO" 00100 "YES"	Set Ram Rate to 1mm/min
RATE #7= XXX.XX	"NO" 00040 "YES"	Set ram rate to 0.4 mm/min
Rate #8 = XXX.XX	"NO" 01000 "YES"	Set ram rate to 10 mm/min (again)
Rate #9 = XXX.XX	"NO" "YES"	Zero rate means no more speeds
MELT FORCE=XXXX	"NO" 1000 "YES"	1000N Pre-Start Force to Pack
TERM FORCE=XXXX	"NO" 3300 "YES"	3300N Safety Overload
START POS.=XXX.XX	"NO" 14000 "YES"	Begin test at 140 mm from top
POS. #1 = XXX.XX	"NO" 17500 "YES"	Force acquired at 175mm position
POS. #2 = XXX.XX	"NO" 19000 "YES"	Force acquired at 190 mm position
POS. #3 = XXX.XX	"NO" 19800 "YES"	Force acquired at 198 mm position
POS. #4 = XXX.XX	"NO" 20400 "YES"	Force acquired at 204 mm position
POS. #5 = XXX.XX	"NO" 20800 "YES"	Force acquired at 208 mm position
POS. #6 = XXX.XX	"NO" 21000 "YES"	Force acquired at 210 mm position
POS. #7 = XXX.XX	"NO" 21100 "YES"	Force acquired at 211 mm position
POS. #8 = XXX.XX	"NO" 21700 "YES"	Force acquired at 217 mm position
PARK POS.=XXX.XX	"NO" 05500 "YES"	Crosshead Park at 5 mm position
TEST DELAY=XXX.XX	"NO" "YES"	No Pause between points
RUN# START	"NO" "YES"	Clears RUN# to zero
PROGRAM ENTERED		

Verify the options program (see Page 23 for more details) to be sure ***Manual Mode is OFF.*** Load the barrel as described previously on Page 18. Once the plunger is in place, the swing arm fully closed, the plunger guide bushing is in place press; ***RUN***. The test will automatically acquire the data, purge the barrel of remaining material, move back to park position and send the data to the printer or the PC.

What's happening automatically during the run

When the **RUN** button is pressed a number of things happen. First the rheometer starts an internal timer which keeps track of the total test time and the ***Melt Time.*** The rheometer then moves the cross head down until it senses 2-3 lbs of force. This occurs when the load cap connects to the top of the plunger. A count down time then appears on the rheometers' front display which also shows the ***Melt Time*** remaining. On the left side and the current plunger location is displayed (e.g. L=111 MT=305). The rheometer will then gradually increase the plunger speed until one of two things happens, a) either it achieves maximum speed, or b) it sees a force on the load cell equal to the ***Melt Force***. The ***Melt Time*** counter on the front display will not be updated until ***Melt Force*** is achieved or the plunger has reached the ***Start Position***. While the rheometer is trying to apply the ***Melt Force*** to the material it also watches to see if it has reached

the ***Start Position***, if it has it stops moving downward and waits for the ***Melt Time*** to expire. The first test rate begins once the timer is equal to the ***Melt Time*** and the plunger is at ***Start Position***. If the ***Melt Force*** is set too low the plunger will not reach the ***Start Position*** within the available ***Melt Time***. When this occurs the rheometers front display will switch from a count down timer showing ***Melt Time*** (L=130.12 MT=12.3) to a run time counter (L=130.12 RT=380.1). The run timer shows how long it has been, in seconds, since the **RUN** button was pressed. The actual testing of the sample will begin only after the ***Melt Time*** has expired and the plunger is at or past the ***Start Position***. When the ***Melt Force*** is high the plunger may go a few millimeters past the start position; this is normal.

12. Cleaning Up a really Big Mess

Big messes tend to occur when the die nut is not screwed in completely. Material runs down the die nut holder threads and coats the threads with a sticky polymeric mass. This can be very difficult to remove. We tried several methods including solvents but found a tap and die set for the die holder nut works best. The tap is an English 3/4" 10 with a mating die. It is imperative the tap be ground flat at the top ("Bottom Tap") so that the tapered end does not go into the main bore and destroy the finish. A tap ground flat at the top will hit the bottom of the barrel when it is screwed up from below. Low torque (force) turning of the tap is usually all that is necessary to remove material tenaciously stuck in the threads. Similarly, the die holder nut is screwed into a thread cutting die. This will clear the threads of material. The inside of the die nut can also be clogged with material. For short dies running Nylon this is common. A 12.7 mm (1/2) diameter rat tail (tapered) round file, run up and down the inside of the die holder nut does an excellent job. Placing the hot die nut onto a rat tail and then brushing the outer threads with a stiff wire brush is an excellent cleaning method.

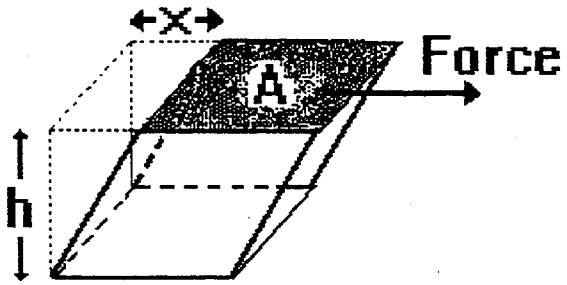
Oven cleaner (Easy-Off®) sprayed onto a cold plunger and left overnight will do an excellent job of cleaning degraded material off the shaft, the outside of the die and on the die holder nut. Be careful not to breath in oven cleaner vapors.

If running at high temperatures (>300 ^{o}C) you may wish to coat the outside of the die with anti-seize material prior to placing it in the die holder nut. This will make it much easier to clean should any material get into that area and cross link. Be careful not to get anti-seize material onto the top of the die or in the barrel as this will drastically effect your results. Some customers have used a brass wire flywheel to clean piston tips between runs at high temperatures. Should you also chose this route, be sure to check the plunger tip diameters often for excessive wear. (Tips less than 9.515 mm would be out of ASTM specifications for K&S machines). Material leakage past the tip should also be less than 0.1 g.

III. Introduction to Rheology

A. Geometries of Deformation

Simple Shear Deformation



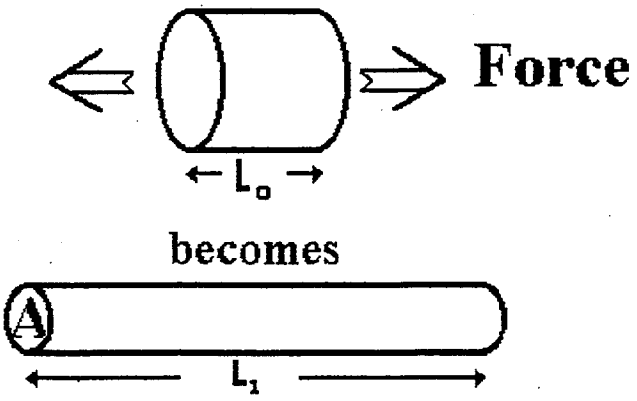
Shear Stress $= \tau =$ Force / Area $= F / A$

Shear Strain $= \gamma =$ Displacement / Gap $= x / h$

Strain Rate $= \dot{\gamma} =$ Displacement Rate / Gap $= \dot{x} / h$

In the simple shear geometry the gap and area remain constant throughout the deformation process.

Extensional Deformation



Extensional Stress $= \sigma =$ Force / Area $= F / A$

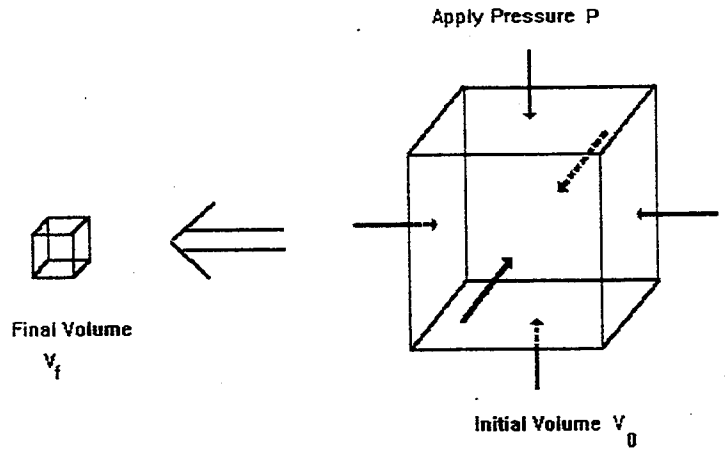
Extensional Strain $= \varepsilon =$ Change in Displacement/ Length

$$= \int_{l_0}^{l_1} \frac{dl}{l} = \ln\left(\frac{l_1}{l_0}\right)$$

Extensional Rate of Strain $= \dot{\varepsilon} =$ Change in strain with time $= \dot{x} / l$

Other definitions are used and depend upon the reference frame chosen for the observation. The above definitions are the common.

Volumetric Deformation (BULK)



Stress is the applied pressure, P.
Strain is the change in volume divided by the original volume.

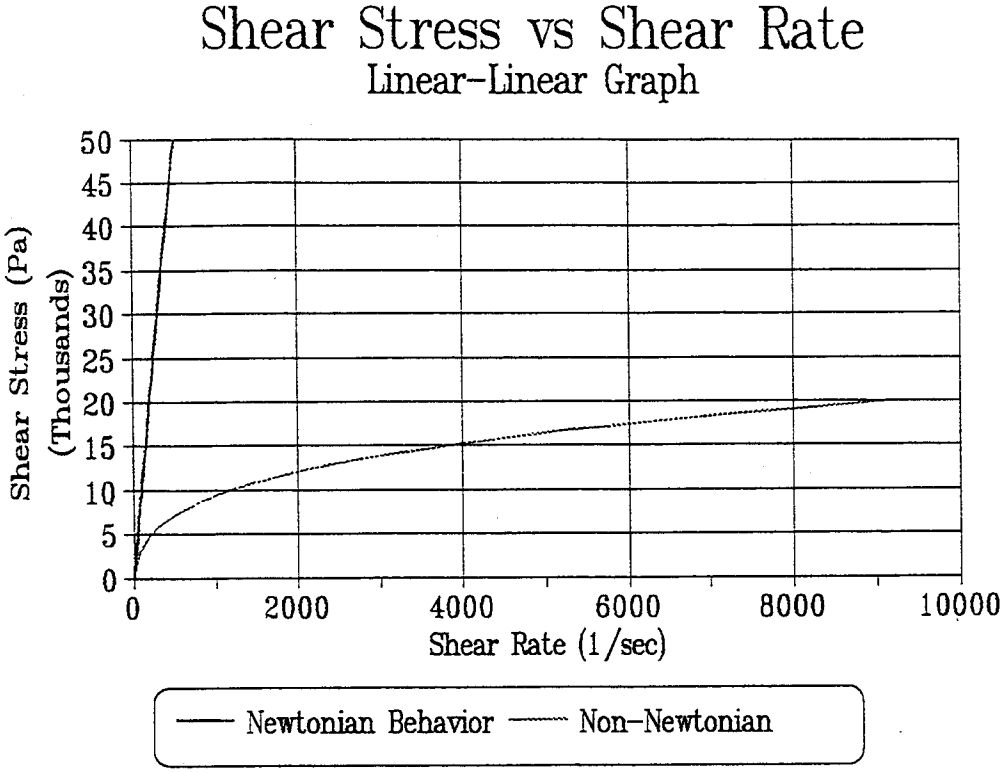
Most real flows are a combination of these three fundamental deformation geometries shown above. Flow in the rheometer contains all three types of deformation. When the molten material is compressed in the barrel it under goes a volumetric compression which increases its density slightly. Material flowing from the barrel into the entrance of the die experiences an extensional flow. Imagine stretching the material in the barrel until it is thin enough to fit through the die and you understand the extension aspect of the flow. Finally in the capillary die the material immediately adjacent to the walls is not moving (more on this assumption later) yet the material in the very center of the capillary is coming out quickly. The die wall is like the bottom plate of the simple shear geometry and the center of the stream is similar to the top plate. Capillary rheometers are designed primarily to measure shear viscosity of a material. With some additional measurements, extensional viscosity (see Page 57) can be estimated and limited volumetric data (PVT) can be collected in the molten range of the material using a solid die, special sealing plunger and pressure transducer.

B. Newton's Law

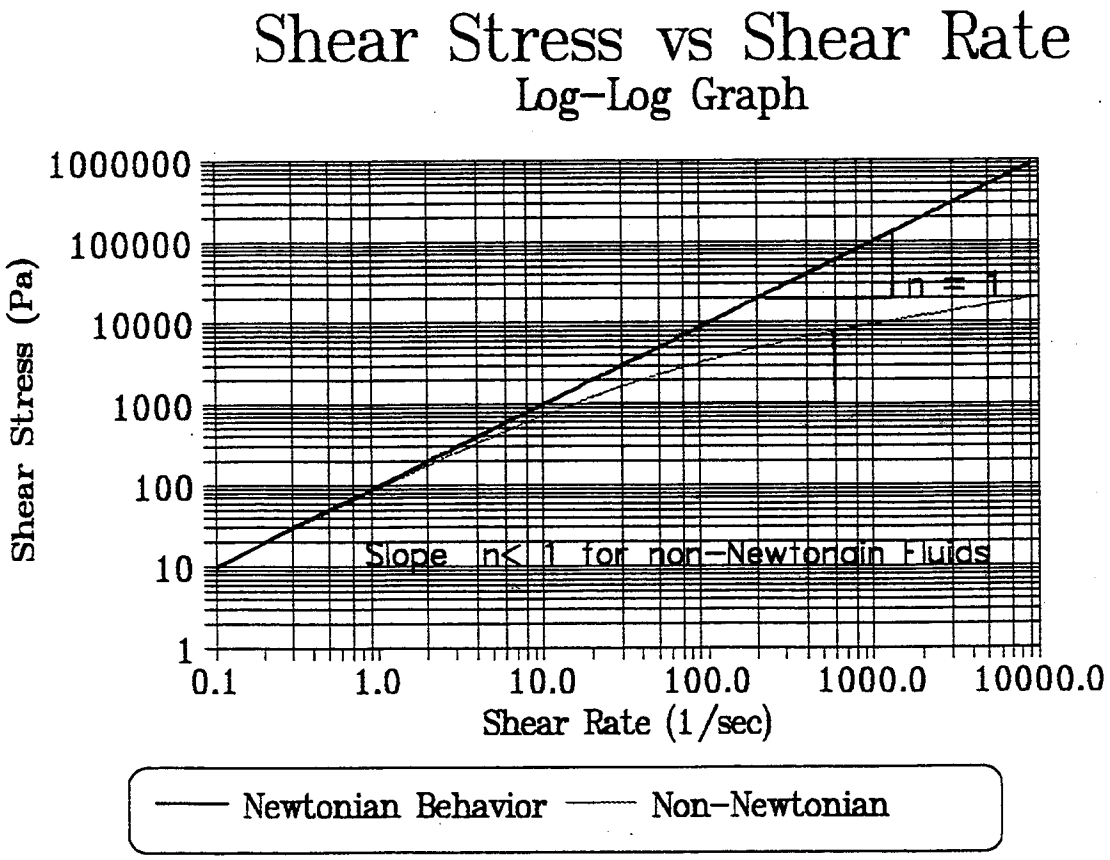
$$\tau = \mu \dot{\gamma}$$

Shear Stress	Newtonian Viscosity	Shear Rate

Newton's law states that the shear stress on a material is directly proportional to the shear rate to which it is subjected. For the simple shear deformation described previously this means the force necessary to move the top plate will increase proportionally with the increase in speed of the top plate. Graphically this means a plot of linear (as opposed to Log) shear stress vs. linear shear rate will yield a straight line as illustrated in the sharply increasing straight line in the next Figure. The slope at any given point on the curve is the viscosity of the material for that shear rate.

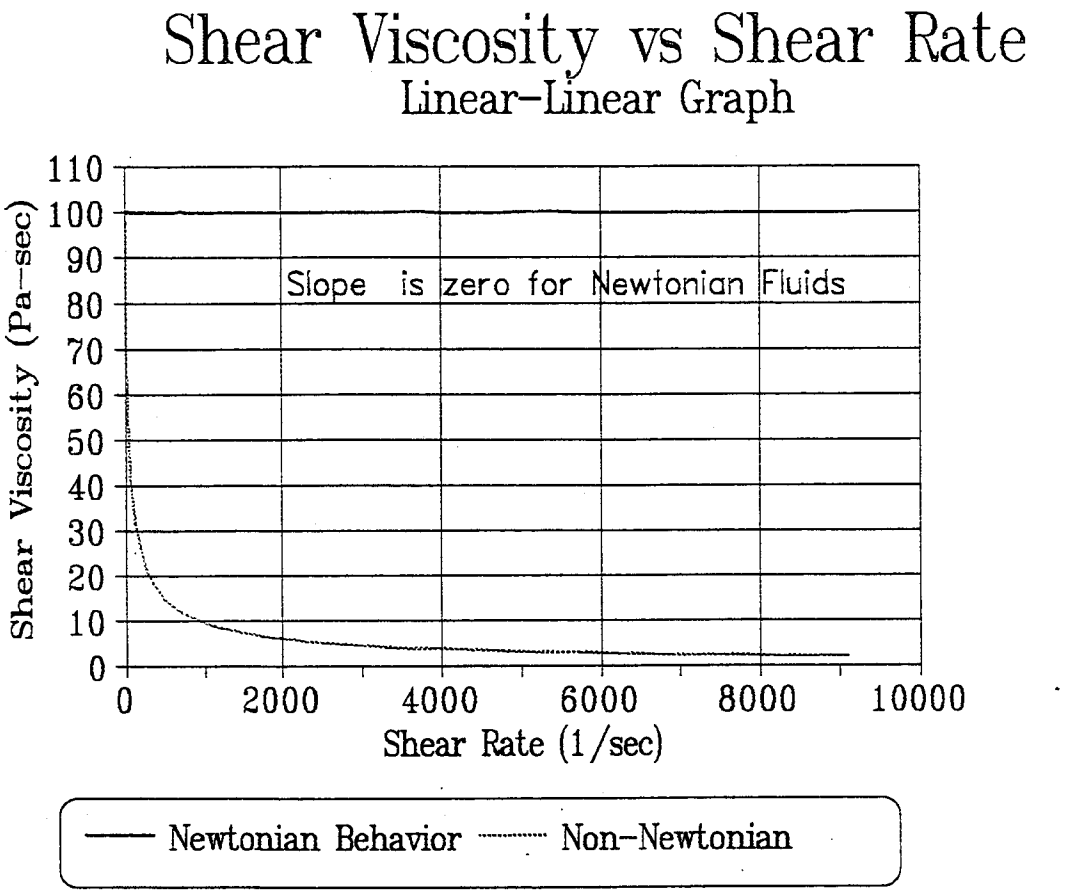


Because the magnitude of the change in viscosity is so large it is often more convenient to make a log-log plot.



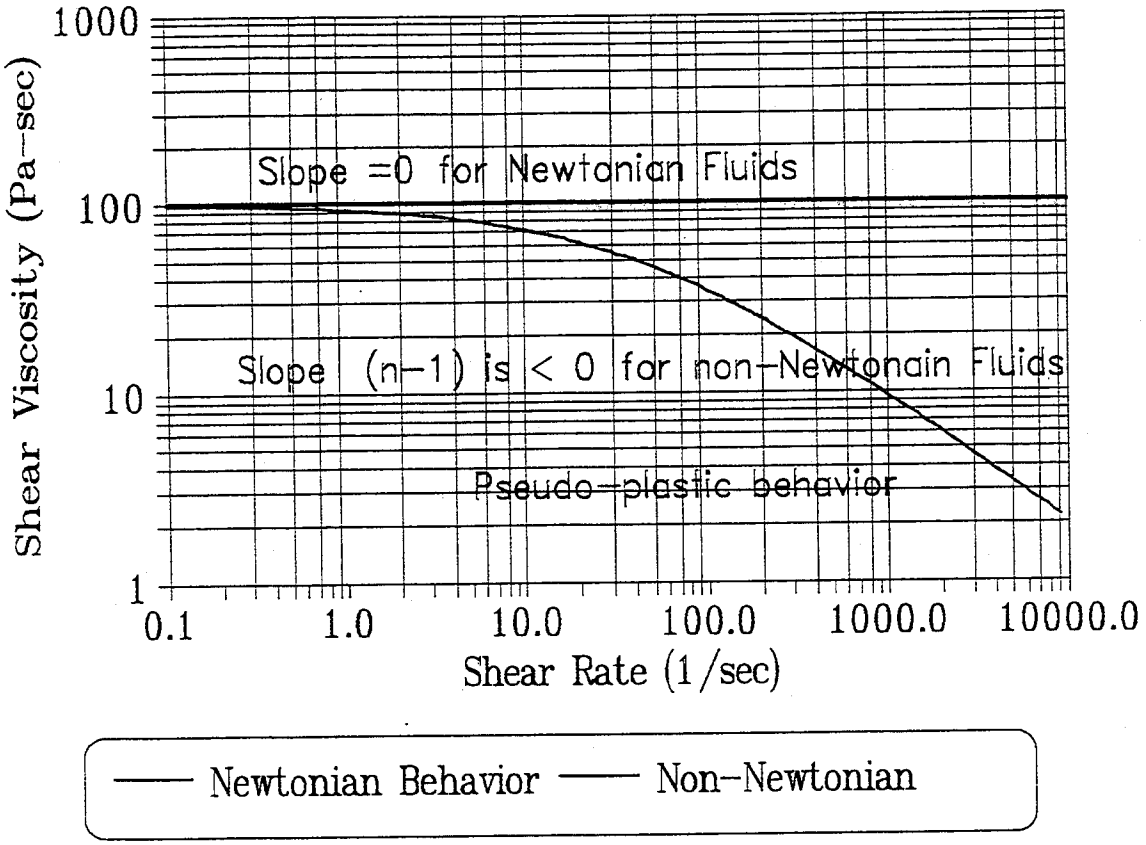
It is easy to tell the Newtonian region for materials on a log-log plot of shear stress vs. shear rate, the slope is equal to 1.0 in the Newtonian region. The slope drops to less than 1.0 for most polymers as the shear rate increases.

It is even easier to see if the material is behaving like a Newtonian material when looking at a viscosity vs. shear rate plot. Viscosity is constant (slope=0.0) for both log-log and linear-linear plots of viscosity vs. shear rate as follows:



For the exact same data the precipitous viscosity drop can be displayed more clearly in the Log-Log metric as shown below:

Shear Viscosity vs Shear Rate
Log-Log Graph



Here's a list of some common material and their viscosities at typical use temperatures. Note that some materials usually considered solid, such as glass, actually have a very low flow rate even at high stress levels, and thus an extremely high viscosity.

Viscosities of some Common Materials

Material	Viscosity (Pa-sec)	Consistency
Air	10^{-5}	gaseous
Water	10^{-3}	fluid
Olive Oil	10^{-1}	liquid
Glycerin	10^{0}	liquid
Pancake Syrup	10^{2}	thick liquid
Caramel	10^{2}	slightly thicker
Polymer Melts	10^{2}-10^{6}	toffee-like
Pitch	10^{9}	stiff
Glass	10^{21}	rigid

C. Effect of Shear Rate on Viscosity

Pseudoplastic

Most polymers are pseudoplastic which means their viscosity drops with increasing shear rate. For a given machine and die the viscosity is directly proportional to the force on plunger divided by speed, that is to say it's a force to speed ratio. Shear rate for a fixed die and machine depends only on the speed of the plunger. If for a Newtonian material you double the speed of the plunger, you double the force on the plunger. For pseudoplastic materials, doubling the plunger speed does not double the force; the force is less. The force needed to push the plunger has increased but not as much as we would have expected based upon Newtonian behavior. See the graphs under Newton's Law for more insight into this behavior.

When polymers flow they tend to orient the long molecules which make up the material. Once this alignment is made the flow process is easier causing the decrease in viscosity. Given enough time the chains will come back to their original configuration (provided the material is stable for that period of time) and the effect of shear rate thinning can be replicated.

Dilatant

Materials which increase in viscosity with shear rate are known as dilatant. Some filled systems exhibit this type of behavior, it is unusual for non-filled polymeric melts.

D. Effect of Time on Viscosity

Thixotropic

If a material is thixotropic this means it's viscosity decreases with time. Given enough time polymeric materials tend to orient themselves making flow easier. This effect is reproducible, in that, given enough time to recover the material will give the same time dependence when retested. When a material degrades chemically to a lower molecular weight this is a non-reversible effect.

Rheopectic

Rheopectic materials are those that generally increase in viscosity with time. Cross linking or further polymerization can cause this increase in viscosity to occur.

E. Effect of Temperature on Viscosity

In general polymeric materials tend to decrease in viscosity with an increase in temperature provided the material is chemically stable. If not chemically stable the material can increase in molecular weight causing a rise in viscosity (crosslinking increases viscosity dramatically) or it can break down via chain scission causing a larger than expected drop in viscosity with temperature.

A common method to represent the viscosity vs. temperature dependence involves the Arrhenius expression where a log of the viscosity (often the zero shear viscosity value) vs. 1/Temperature is fitted to a straight line. Sometimes a log(viscosity) vs. Temperature curve is also used. Both give similar goodness of curve fits. KARS uses the Arrhenius dependence in its curve fitting models.

The WLF dependence is also used and is found to be particularly useful at temperatures closer to the glass transition temperature of the material.

Suggest test temperatures for various materials are displayed in a table in the ASTM D3835 test method.

F. Effect of Fillers on Viscosity

Generally, inorganic fillers such as carbon black, $CaCO_3$,TiO_2 etc. increase the viscosity of a material based on their volume fraction. They also tend to reduce extrudate swell (rocks don't swell).

G. Effect of Moisture on Viscosity

Many materials adsorb (surface) and absorb (internal) moisture while at room temperature this often has little effect on the material. However at elevated temperature even small amounts of moisture can have dramatic effects on the viscosity of the material, especially on condensation

polymers. It is known that the fiber strength of PET is a strong function of the moisture in the material before the spinning process.

H. Capillary Rheological Equations and Corrections
1. Basic Capillary Rheometer Equations[1]

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

Viscosity is Shear Stress over Shear Rate

Where η is viscosity, τ the shear stress and $\dot{\gamma}$ the shear rate.

Apparent Shear Stress :

$$\tau = \frac{F}{A} = \frac{D_c \Delta P}{4L_c} = \frac{F_p r_c}{2\pi R_b^2 L_c}$$

Where τ is the shear stress, **F** is force acting on surface area **A**. $\mathbf{F_p}$ is the force on the plunger and r_c , D_c is the inner radius and diameter of the capillary respectively, R_b is the inner radius of the barrel and $\mathbf{L_c}$ is length of the capillary and ΔP is the pressure drop along the capillary.

Apparent Shear Rate:

$$\dot{\gamma} = \frac{32Q}{\pi D_c^3} = \frac{4R_b^2 S}{r_c^3}$$

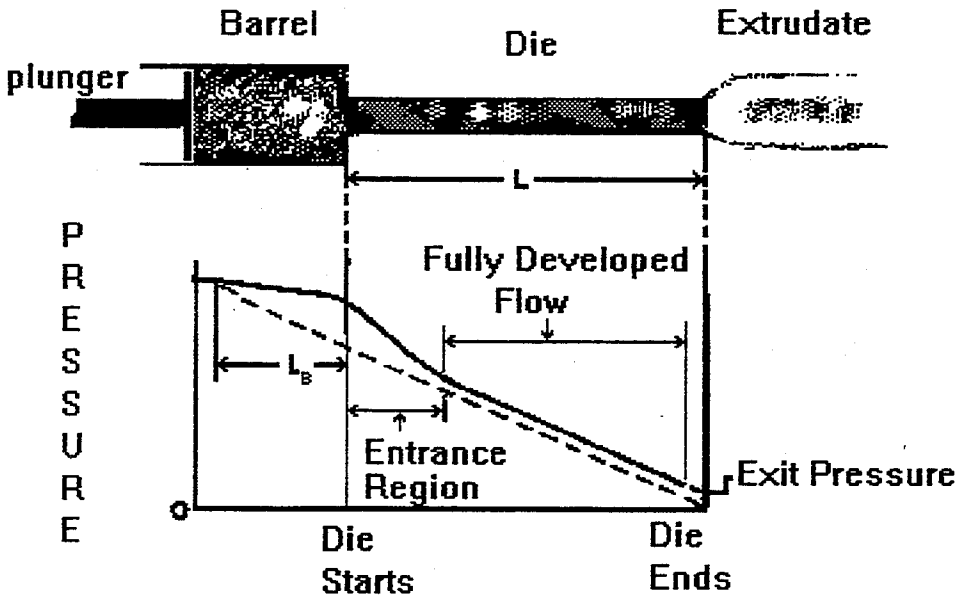
[1]J.M. Dealy, "Rheometer for Molten Plastics: A guide to Testing and Property Measuremnt", Van Nostrand Reinhold (SPE), NY, 1982

Where $\dot{\gamma}$ is the shear rate, $\mathbf{R_b}$ is the inner radius of the barrel, S is the speed of the plunger and r_c is the inner radius, D_c the diameter of the capillary and Q is the volumetric flow rate.

$$\eta = \frac{F r_c^4}{8 \pi R_b^4 L_c S}$$

Viscosity reduces to the above equation. Physically it is proportional to the force on the plunger divided by the speed of the plunger. That constant is strongly dependent on barrel and die radius.

2. Pressure Profile



From the above diagram of the pressure vs. position it should be clear that the pressure falls as the die exit is approached. Even within the barrel there is, at least, some small pressure drop known as the barrel pressure drop or barrel drag effect. Close to the die entrance and just inside the die the pressure drop is fairly dramatic as we force the material to undergo a primarily extensional deformation to enter the die. Once past the entrance

region the pressure falls in a linear fashion from the shearing action within the die. Note that the exit pressure is typically non-zero. The barrel pressure drop, the entrance pressure drop, frictional effects and the exit pressure all get measured in addition to the pressure drop in the die. In the previous equations we assumed we were just measuring the pressure drop in the die. If we use the data as collected, that is, the Apparent values we over-estimate the viscosity because of the extra pressures in the system. By using several dies of the same diameter but differing lengths and measuring the plunger force for a given shear rate at the same point in the barrel for each die, the extraneous pressures can be removed and the true viscosity calculated as described below under the section Corrected Shear Stress.

3. Why use a Pressure Transducer and When?

Advantages of a Pressure Transducer
a) Frictional effects of plunger tip are removed.
b) Barrel Pressure drop from plunger tip to the pressure sensor is removed.
c) Some precision improvements can be gained.

Disadvantages of a pressure transducer
a) Reservoir needs flow into it for pressure sensors to stabilize.
b) Increased thermal losses from pressure probe.
c) Pressure reservoir needs cleaning for materials which degrade.
d) Device itself is inherently not as accurate as a load cell
e) Smaller dynamic range.
f) Mercury used in transducers, care must be taken in handling and removal of device.
g) More tedious to clean.
h) Must be removed when hot to prevent damage of device. Polymer may adhere to the diaphragm and cause it to fail if removed cold.

4. Common Assumptions

- Fully developed, steady-state laminar flow
- No radial or circumferential velocity components
- No body forces (inertial effects etc.)
- Viscosity independent of pressure
- Isothermal conditions

5. Weissenberg-Rabinowitsch Correction

The Weissenberg-Rabinowitsch correction accounts for the fact that most polymeric materials when flowing through a capillary behave like a Newtonian fluid (shear stress is directly proportional to shear rate) only at very low shear rates. For a constant output flow rate, the velocity of the melt stream in the center of the die is at a maximum. The velocity then falls to zero at the capillary wall. The change in this velocity profile as we

move from the center of the die to the wall is the shear rate for that position in the die. Polymeric materials at high flow rates show a much larger change in velocity near the wall of the die when compared to Newtonian materials. At higher shear rates then, the calculated apparent shear rate (equation shown above) is an underestimate of the true shear rate and the viscosity is over estimated.

In summary the Weissenberg Rabinowitsch shear rate correction accounts for the fact that the true shear rate is often larger than the apparent shear rate for non-Newtonian materials. The true shear rate can be calculated using the following equation:

$$\dot{\gamma} = \frac{(3n+1)}{4n}\dot{\gamma}_a$$

where n is the tangent slope of the log true shear stress vs. log apparent shear rate curve at the apparent shear rate being corrected. $\dot{\gamma}$ is the true shear rate and $\dot{\gamma}_a$ is the apparent shear rate described previously.

Note that the shift in shear rate can be substantial causing a trace of ln (viscosity) vs. ln (shear rate) to move down and far to the right on the plot. The total viscosity change (viscosity drop at a fixed shear rate) however very rarely exceeds 15 % and occurs only in the non-newtonian range of the material.

6. Corrected Shear Stress

The correction method according to Bagley is used to calculate true stress. To obtain the true shear stress perform the following procedure:

Using a minimum of two dies (though preferably three or more) having the same entrance angle and same diameter (D) yet of differing capillary lengths (L) collect steady-state flow data on shear rate and test pressure (or plunger force). At least, one L/D ratio should be less than 10 and at least one should be greater than 16. Prepare a plot of Pressure (or Plunger Force) versus the Length to Diameter (L/D) ratio of the dies used. For points at constant apparent shear rate draw the best straight line through the data and determine the intercept with the Pressure axis (Pc) or Force axis (Fc). Obtain true shear stress using the following equation:

$$\tau = \frac{(P - P_c)D}{4L} = \frac{(F - F_c)D}{4L\,A_B}$$

where τ is the true shear stress, P is melt pressure, Pc is the intercept obtained for a given shear rate from the above described plot (see Figure 1). D is the die diameter, L is the die length. For plunger force measuring devices F is the force on the plunger, Fc is the intercept force on the

Bagley plot described above and A_B is the cross sectional area of the barrel. Devices which measure plunger force must acquire data for a given shear rate (a given line on the graph) at the same position in the barrel for the various dies used. In this way barrel pressure drop effects will be removed along with the other stationary pressures in the system when the Bagley correction is performed.

Note 1: Pressure transducers mounted just above the die in the barrel still require the Bagley correction. If the position of the pressure probe is a substantial distance from the capillary entrance then pressure drop from the pressure probe to the top of the die can be significant.

Note 2 : When using very long dies there may be a non-linear changes in the pressure vs. L/D plots due to the effects of pressure on viscosity or viscous heating. In such cases use only the data from shorter capillaries which do not exhibit the effect.

KARS assesses the validity of the assumption of having straight lines in the Bagley plot. Users are warned that the Bagley correction is not valid under circumstances where the straight line conditions are not met.

7. Extrudate Swell

Extrudate swell is a measure of the increase in diameter of the extrudate over that of the extrusion die. Most materials decrease in swell with residence time in the die. Thus at higher shear rates (short time in the die) the extrudate swell is generally larger than at lower rates. Swell also tends to decrease with temperature as most materials become less elastic as they are heated

PVC

PVC is odd compared to most materials. The degree of particle fusion and the fact that many crystallites exist at typical test conditions account for much of this odd behavior. PVC can be thought of more like a lightly crosslinked rubber than a true homogeneous melt. Because of this it has behavior which can be directly the opposite of most true polymer melts. It can show increases in extrudate swell with temperature and viscosity value which are very dependent on how (Temperature & shear history) the pellets were formed. Though PVC itself is not very moisture sensitive some of the additives used can be. You may wish to see if drying the sample affect your results.

IV. Details of Test Programs and Option Program

A. The start of any rheometer test.

There are four important control variables which govern the start of a test: ***Park Position, Melt Force, Melt Time*** and ***Start Position***.

The terms plunger positions and plunger locations refer to the same information and are used interchangeably. The plunger position is the distance in millimeters (inches U.S.) from the upper limit position or the HOME position. All positions displayed and requested are therefore the distance away from the HOME position. When the machine is first turned on the HOME position is found and set to zero by the rheometers internal program called firmware. Once the HOME position is established the machine then moves to the PARK position for sample loading. Since the HOME position is used as a reference for all other positions it is important not to interrupt the machine when it first starts up and finds the HOME position.

Park Position: The ***Park Position*** is the position from which a test begins and the position to which the rheometer returns after completing a run or sample purge. It is normally set at 50 mm on a Galaxy V.

Melt Force: The ***Melt Force*** is the force the rheometer tries to apply on the sample to get the plunger to start position. Note: If too little sample is placed in the barrel this force will never be achieved. Large diameter dies and low material viscosities may also prevent melt force from being reached. A typical setting is 650 N.

Melt Time: The ***Melt Time*** is the minimum amount of time the material will sit in the barrel before the first rate begins. If the ***Melt Force*** is insufficient to get the material to the ***Start Position*** then the set ***Melt Time*** will be exceeded. The front panel of the rheometer shows a count down time for the ***Melt Time,*** it show MT XXX where XXX is the number of seconds remaining in the ***Melt Time.*** If the rheometer is not at the ***Start Position*** when ***Melt Time is zero, then*** the front display changes to showing the Run Time or the amount of time passed (including the ***Melt Time***) in seconds since the **RUN** button was pressed (e.g. RT=412).

Start Position: The ***Start Position*** is the position where the first programmed rate begins. In controlled stress runs it is where the first programmed load begins.

Other Rheometer program parameters found under **EDIT**:
These parameters are values required to run a test and can be entered by

pressing the **EDIT** key on the rheometers front panel.

Orif. Len: This is the orifice length or die length. It is the length of the capillary region of the die. If you know the L/D of the die multiply by the diameter to get the length.

Orif. Diameter: This is the orifice diameter or die diameter. It is assumed the die is uniform in diameter along it's length. For accurate viscosity measurements the die diameter is the most critical dimension.(English is radius, refer to appendix).

Sample Length: This is the distance over which the speed of the plunger is measured during load control tests. Set it to 0.0 if you wish to do runs which control plunger speed. It should be set to values other than zero when mimicking a MCR machine or when constant load (stress) runs are required. A typical sample length is about 1/3 of the distance between measurement positions.

During a run ***Sample Length*** works like this: You've set up a run with all positions 1.25 mm apart starting from a 115 mm start position. The load is constant at each point and set at 900N. The ***Sample Length*** should be about 5mm. After pressing RUN the test begins like any other test (described in the previous section). When melt time expires, the system then moves at what ever speed is necessary to keep 900 on the load cell (or pressure transducer). When position 127.5 mm is reached an internal timer is started. When position 132.5 mm is reached the time is stopped and the second point in the test starts (after any delay time pause).

Pos. #1: Positions are the actual locations in the barrel where the data is collected. Typically this means that when this position is reached the reading is taken. The 0.0 position is when the rheometer hits the upper limit switch when it is first turned on. Positions will only be entered for ram rates other than zero. When in ***Manual Mode*** only the ***Start Position*** is used; all other positions are defined when the **END** key is pressed during the run. Any positions currently there will be overwritten. When the **END** key is pressed with ***Manual Mode*** ON, the position value at that instant is stored in a position value associated with that rate. If ***Manual Mode*** is turned off subsequent automatic runs will be collected at the exact same positions as the previously manual test.

Galaxy V

Start your tests at position values no lower 115 mm (130 mm is typical) and go no higher (plunger further in barrel) than 210 mm. The plunger would actually hit the die at about 231 mm if the limit switch failed. The top of the access hole for the pressure transducer is at about 210 mm.

Galaxy III and IV

Start tests at positions no lower than 84 mm(95 mm is typical) and go no higher than 7.0 inches into the barrel. Contact with the die would occur at about 7.8 inches on these machines if the limit switch failed. Getting very close to the die can also cause abnormally high force readings as the flow pattern changes.

Terminal Force: Terminal force is a software protection force for the machine. If the ***Terminal Force*** is observed by the rheometer's on board computer it will abort the test. The plunger movement will stop; the machine will try to purge the barrel of its contents. If it can not the crosshead will return to start position. The terminal force also controls the maximum force allowed during the purging of material from the barrel. The speed of purging will be automatically adjusted so that the maximum amount of force applied is half the terminal force. (See Page 87 if you are having trouble purging the machine) Normally this value is set just below your load cell capacity (e.g. 1000 LB cell set to 990 LB).

Test Delay: The ***Test Delay*** is a stop in plunger movement between each test point for a specified number of seconds. The total delay between two data points will be the delay time plus the actual plunger movement time. For example, if all rates are 8 mm/min and all positions are 8 mm apart then the measurement time is 1.0 minutes plus an additional ***Test Delay*** of 180 seconds would make the total time between points 4.0 minutes. For thermal degradation runs increase or decrease the ***Test Delay*** and or the number of points in the test to achieve the total time needed to quantify the degradation.

S.I., Pascal
ENGLISH

Metric, Poise: You will see one of these three items appear indicating the unit system that will be used on the printer if the printer is connected directly to the rheometer and not to the PC. If you are using KARS it does not matter how this is set as KARS will convert to what ever units you request in the KARS options screen [F9]). "Metric/SI" Rheometers will not ask this question.

RUN # START: This is the run number the rheometer includes with the data. Normally when a new ***Sample ID*** is entered this value should be cleared back to zero by pressing **NO** on the rheometer keyboard. This value is automatically incremented every time the **RUN** button is pressed. The run number is part of the extension name for file saved with KARS (e.g. SampleID.K01 or SampleID.D01). The automatic incrementing of the RUN # allows the file name to be unique even when the ***Sample ID*** is not changed.

B. Selecting a PROGRAM

Eight different programs #00 to #07 (Running conditions) can be stored inside the rheometer. The lower right part of the red LED displays the current Program # when a test is not running. The **EDIT** allows modification of the current program. To change programs press the **PRGM** key on the rheometer's front panel. To enter the program number, remember that two digits are required. Program 0 becomes active when the keys **PRGM 0 0 YES** are pressed. Similarly to switch to program conditions found in program #07 press: **PRGM 07 YES .** You may wish to use a certain program number for control sample tests like #07 and typically use #00 for making up new programs.

C. Options Program

Press the SHIFT key and then the PRNT key on the Rheometer touch pad to enter the options program. After pressing these keys the words "OPTION PROGRAM" will appear on the LED display then disappear. The first option that can be modified is manual mode.

You will see one of the following appear:

Manual Mode ON?

Manual Mode OFF?

(If the words Manual Mode do not appear then an older version of the firmware is installed).

If you are happy with the current setting press the YES key and the next option item will be displayed. If you wish to change the item press NO. The mode you wish will appear, then press YES to continue onto the next item. The options appear in the following order:

Manual Mode: Manual mode allows the operator to select when the data for a given point is taken. **When manual mode is selected ON, the force value is acquired when the END key is pressed on the front panel** (rather than when a given location is reached). Only the start position matters when MANUAL MODE is on, all other positions will be ignored. If the END key is never pressed no data will be collected!! Manual Mode will put the current positions (sometimes called locations) from each manually selected position into the current program memory. If manual mode is then turned OFF the last manual mode positions chosen will be used to collect the next automatic run. The positions chosen can be seen by going into EDIT and viewing the current program positions or by looking at the print out of the last run. Running a manual mode test on the highest viscosity material in a group first, then switching back to automatic mode (Manual Mode OFF) for the remaining runs assures steady-state conditions will be met for the series. It is also best to err on the side of

allowing too much time when using the manual mode.

Printer: This should be set to ON only if the printer is attached directly to the rheometer. If the printer is attached to the PC, printer should be set OFF.

PC Attached: If you wish to have the data transferred to the PC after a run is completed, set PC attached to YES. If PC function is set to YES and a PC is not attached, the rheometer will wait for a PC to be attached before a run begins. If the machine stops and shows F0 it is likely that it is waiting for PC communications before it begins the test.

Auto F Zero: The Auto Force Zero should be used when careful measurements are needed and the operator is aware of how the auto-zeroing mechanism works. When auto zero is set ON the rheometer will subtract the current load reading found when the RUN button is pressed from all subsequent data collected. The force value, which is treated as true zero can be displayed when auto F zero is ON by pressing **RESET**, **TEST**, then pressing **NO** until the word OFFSET is seen. Press **YES** and the machine will display Offset F #.## where #.## is the force in lbs being subtracted to give the true force reading. **The load cell cap must not be engaged on the plunger when RUN is pressed! Do not press Down then RUN, use the RUN button only!** If the Auto Force Zero is to be used all the time, it is recommended that the force zero value be set to 10.0N when the load cell cap is free from load. This force will later be subtracted from all data so it is corrected, however if the force value is at zero or below zero incorrect force data may be obtained (Force values will be very large!). When a run is aborted and force zero mode is on it is **imperative** that before the run is re-started that the cross head is moved up so that no force is on the load cell prior to pressing the **RUN** button. This is easily accomplished by pressing **RESET** to abort a run, adjust any program variables as needed, then press **UP** until the plunger is free from the loading cap on the load cell, then press **RUN**.
System with pressure transducers are zeroed and RCal'd when the **DELETE** key is pressed instead of the **RUN** key..

LASER Mike: If the LASER micrometer is attached and you wish to collect real time extrudate swelling measurement this option should be set to ON. This options signals the PC to talk to the LASER mike and acquire data at the same time the load data is acquired. An audible beep will be heard from the PC every time a laser value is captured. If a beep is not heard when data is being collected on the rheometer it is a signal the laser communications is not set up correctly or is not activated.

Machine ID: This value is just a tracking number based on the work order request when the machine was built. It is imperative this value be unique if one or more rheometers are connected to the same PC.

D. Generic Run Check List

Immediately prior to pressing RUN
Plunger in machine?
Guide Bushing in place?
Swing arm fully closed?
Area between crosshead and swing arm clear?
Die Nut tight? Pressure transducer tight?
PC (KARS) or printer ready?

Prior to loading Sample
Is material properly prepared (dried, mixed, check for contaminates)
Machine on 20 minutes?
Correct Die in machine?
Sufficient anti-seize coating on die nut
(especially at high temperatures >300 °C).
Correct program being run?
Hand tools in position (packing funnel and tool, cleaning drill bit etc.)
Is ***Manual Mode*** on or off as needed?

Long term items (in order of importance)
Are die diameters within spec. (passed G0-No Go gage, ASTM, ISO, DIN?)
Force calibration and zero OK?
Temperature calibration OK?
Die length OK?
Piston Tip Diameter within spec.?
Threads OK on die holder nut? (too little anti-seize causes excessive wear)
Barrel Diameter OK?

E. Selecting Load or Pressure Transducer

If the pressure transducer option has been purchased the active measurement device can be selected by

To Select a Transducer

When the rheometer LED display reads "ready>"
The rheometer front panel +/- keys perform the following:
+ - Selects Load Cell
- - Selects Pressure

Display will show either LC or PT on screen

E.G. Press -
Display reads "READY> PT #01"
The pressure transducer is now active. Pressing + will make the load cell the active measurement device.

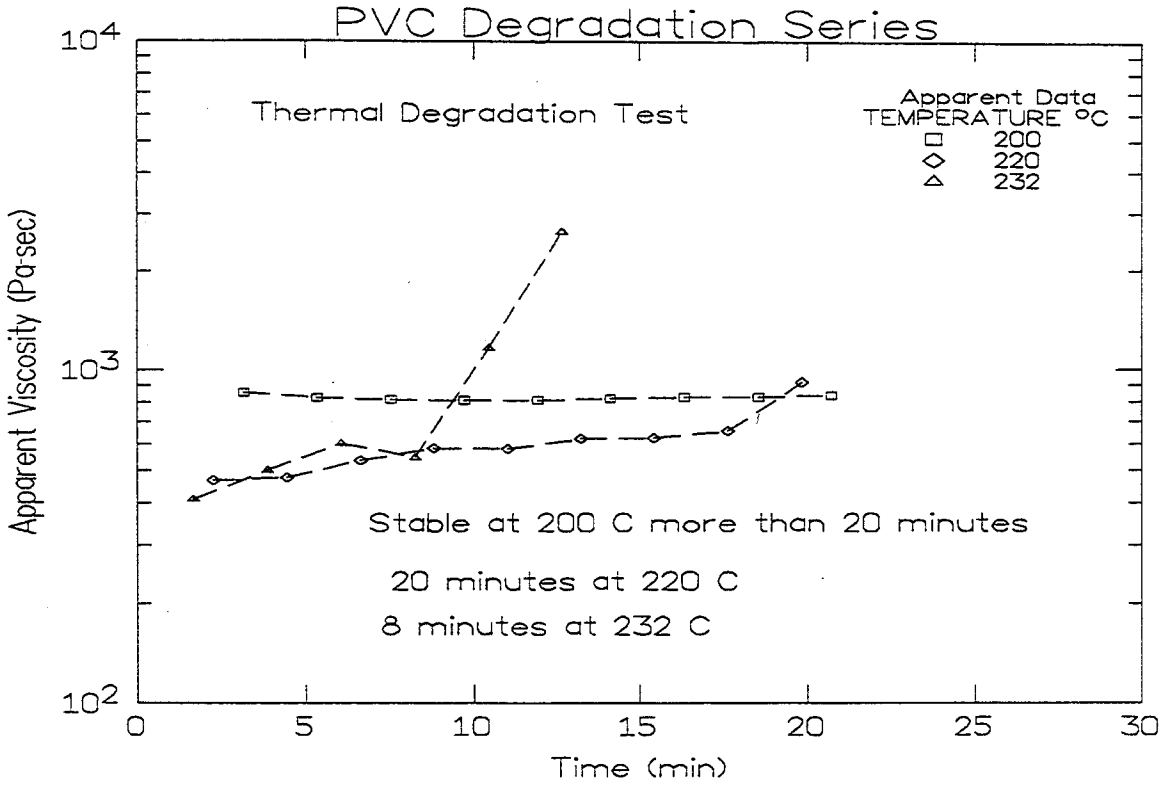
F. Time Sweep

1. Insure Stability prior to Rate Sweep

It is often convenient to run a time sweep on a material to assess the materials tendency to degrade at test temperature before collecting data as a function of rate.

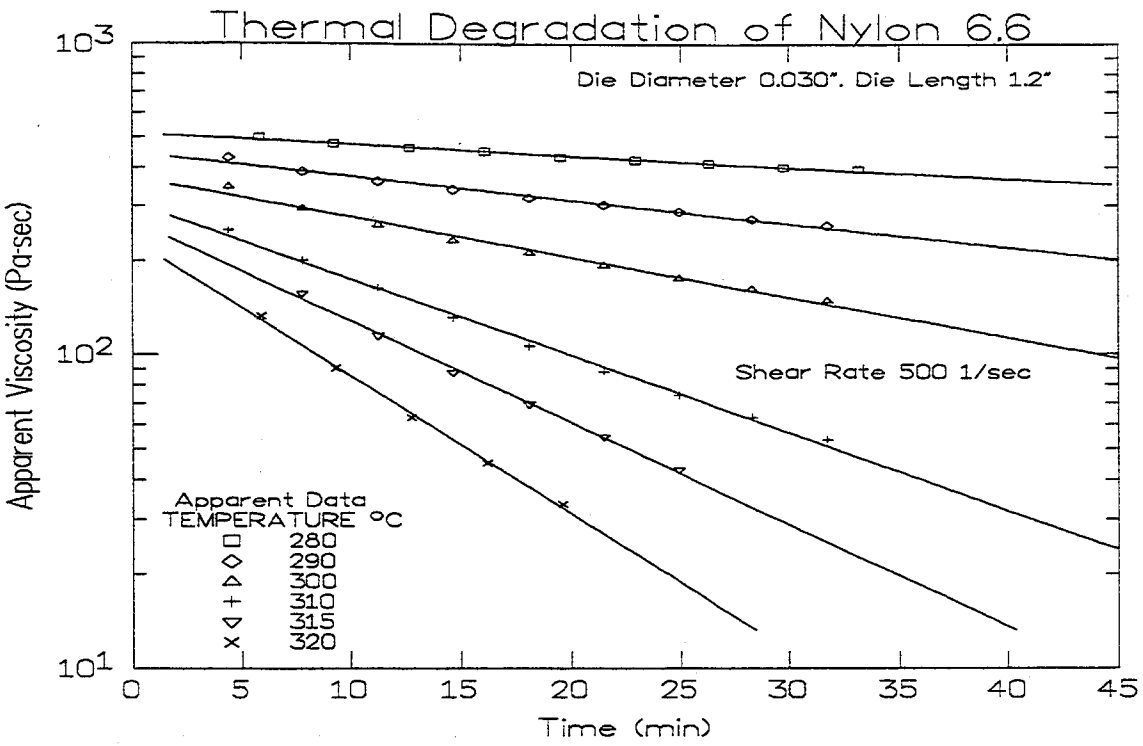
2. PVC Example

PVC is a particularly interesting material to run since when its stabilizer package fails the material crosslinks fairly quickly thereafter showing a marked increase in viscosity. Comparing run made at different temperatures gives insight into the lifetime of the material in an extruder at various temperatures.



Note the test at 232 °C does not extend much past 13 minutes this is because the test was stopped, the die removed and the sample purged before it could degrade any further in the barrel.

3. Nylon 6,6 Example



4. A Standard Time Run Procedure

Galaxy V ONLY

Setting a standard TIME SWEEP rheometer *PROGRAM*

Note press **EDIT** on the front panel of the rheometer to start the process

Machine Responses	**PRESS THESE KEYS**	Comments
Melt Time = XXX.X	**"NO" 2 4 0 "YES"**	XXX.X is any given number, in seconds.
MATL ID= ????????	**"NO" ASK "YES" SKIP SKIP SKIP + YES SKIP SKIP - YES YES**	NO clears ++ makes "B" appear. YES accepts value.
TEMP.= XXX.X	**"NO" 2 3 0 "YES"**	NO clears, 230C entered. YES accepts value.
ORIF. DIA.=.X.XXX	**"NO" 1 0 0 0 "YES"**	Assumes capillary die is 1mm in diameter
ORIF. LEN.=XX.XX	**"NO"2 0 0 0 "YES"**	Assumes capillary die is 20mm in length
SAMPL LEN=X.XXXX	**"NO" "YES"**	Set sample rate to zero for rate run
RATE #1= XXX.XX	**NO" 0 0 5 0 0 "YES"**	Set Ram Rate to 5mm/min
RATE #2= XXX.XX	**NO" 0 0 5 0 0 "YES"**	Set Ram Rate to 5mm/min
RATE #3= XXX.XX	**NO" 0 0 5 0 0 "YES"**	Set Ram Rate to 5mm/min
RATE #4= XXX.XX	**NO" 0 0 5 0 0 "YES"**	Set Ram Rate to 5mm/min
RATE #5= XXX.XX	**NO" 0 0 5 0 0 "YES"**	Set Ram Rate to 5mm/min
RATE #6= XXX.XX	**NO" 0 0 5 0 0 "YES"**	Set Ram Rate to 5mm/min
RATE #7= XXX.XX	**NO" 0 0 5 0 0 "YES"**	Set Ram Rate to 5mm/min
RATE #8= XXX.XX	**NO" 0 0 5 0 0 "YES"**	Set Ram Rate to 5mm/min
RATE #9= XXX.XX	**"NO" "YES"**	Zero rate means no more speeds
MELT FORCE=XXXX	**"NO" 1 0 0 0 "YES"**	1000N Pre-Start Force to Pack
TERM FORCE=XXXX	**"NO" 3 3 0 0 "YES"**	3300N Safety Overload
START POS.=XXX.XX	**"NO" 1 6 0 0 0 "YES"**	Begin test at 160mm from top
POS. #1 = XXX.XX	**NO" 1 7 0 0 0 "YES"**	Force acquired at 170mm position
POS. #2 = XXX.XX	**NO" 1 7 5 0 0 "YES"**	Force acquired at 175mm position
POS. #3 = XXX.XX	**NO" 1 8 0 0 0 "YES"**	Force acquired at 180mm position
POS. #4 = XXX.XX	**NO" 1 8 5 0 0 "YES"**	Force acquired at 185mm position
POS. #5 = XXX.XX	**NO" 1 9 0 0 0 "YES"**	Force acquired at 190mm position
POS. #6 = XXX.XX	**NO" 1 9 5 0 0 "YES"**	Force acquired at 195mm position
POS. #7 = XXX.XX	**"NO" 2 0 0 0 "YES"**	Force acquired at 200mm position
POS. #8 = XXX.XX	**NO" 2 0 5 0 0 "YES"**	Force acquired at 205mm position
PARK POS.=XXX.XX	**NO" 0 5 5 0 0 "YES"**	Crosshead Park 55mm position
TEST DELAY=XXX.XX	**"NO" 1 2 0 0 "YES"**	Pause for 120 sec between points
RUN# START	**"NO" "YES"**	Clears RUN# to zero
PROGRAM ENTERED		

Galaxy III & IV

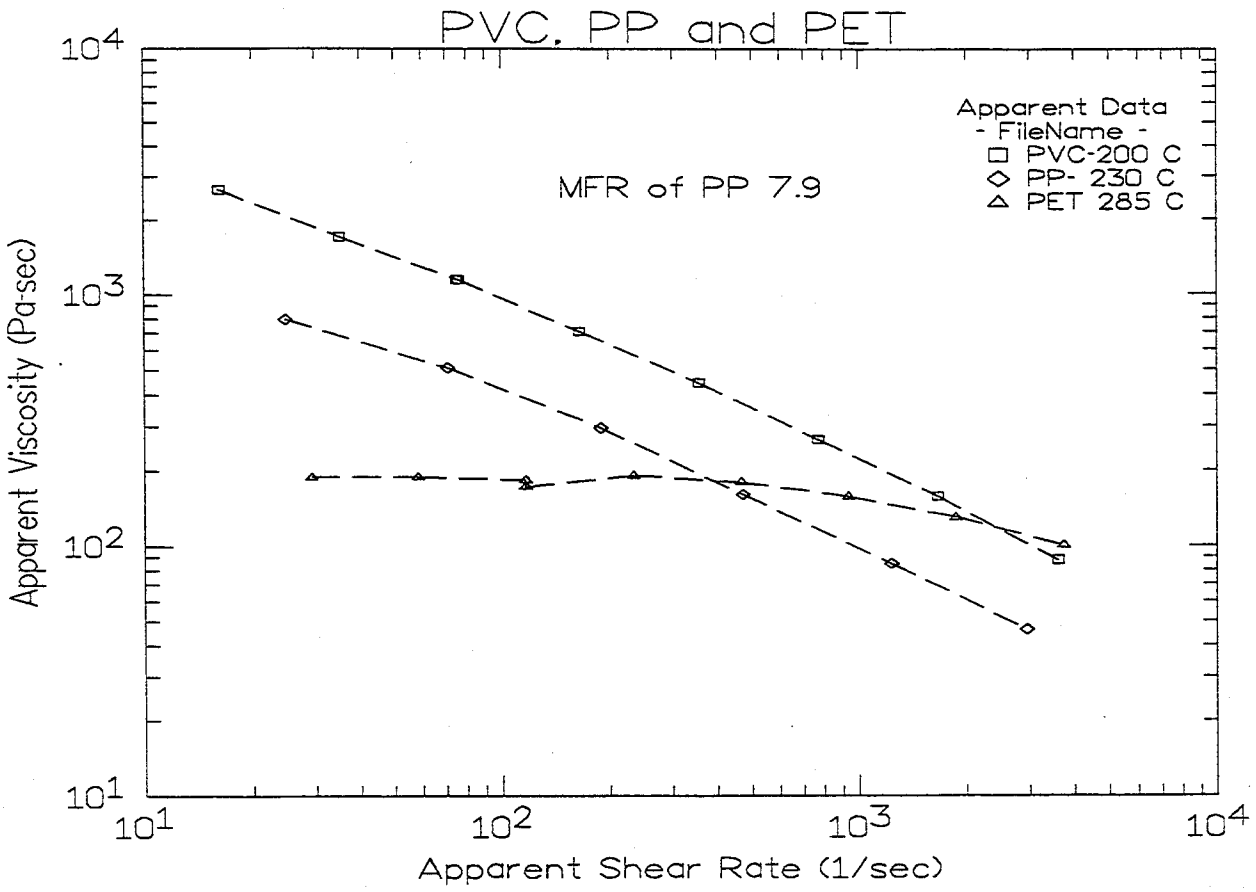
Setting a standard TIME SWEEP rheometer *PROGRAM*

Note press **EDIT** on the front panel of the rheometer to start the process

Machine Responses	**PRESS THESE KEYS**	Comments
Melt Time = XXX.X	**"NO" 2 4 0 "YES"**	XXX.X is any number value given in seconds
MATL ID= ????????	**"NO" + "YES" . SKIP SKIP SKIP + YES SKIP SKIP - YES YES**	NO clears, remainder sets, ID to "ASK" PC then asks for ID
TEMP. = XXX.X	**"NO" 1000 "YES" 230**	NO clears, Enter the Temperature Required
ORIF. DIA. =X.XXX	**"NO" 1000 "YES"**	Assumes your capillary die is 1mm in diameter
ORIF. LEN. =XX.XX	**"NO" 2000 "YES"**	Assumes die length is 20mm
SAMPL LEN=X.XXXX	**"NO" "YES"**	Set to 0.0 for rate control run
RATE #1= XXX.XX	**"NO" 00500 "YES"**	Set Ram Rate to 5mm/min
RATE #2= XXX.XX	**"NO" 00500 "YES"**	Set Ram Rate to 5mm/min
RATE #3= XXX.XX	**"NO" 00500 "YES"**	Set Ram Rate to 5mm/min
RATE #4= XXX.XX	**"NO" 00500 "YES"**	Set Ram Rate to 5mm/min
RATE #5= XXX.XX	**"NO" 00500 "YES"**	Set Ram Rate to 5mm/min
RATE #6= XXX.XX	**"NO" 00500 "YES"**	Set Ram Rate to 5mm/min
RATE #7= XXX.XX	**"NO" 00500 "YES"**	Set Ram Rate to 5mm/min
RATE #8= XXX.XX	**"NO" 00500 "YES"**	Set Ram Rate to 5mm/min
RATE #9= XXX.XX	**NO YES**	0.0 rate means no more rates
MELT FORCE=XXXX	**"NO" 1000 "YES"**	1000N pre-start force to pack
TERM FORCE=XXXX	**"NO" 3300 "YES"**	3300N safety overload
START POS.=XXX.XX	**"NO" 1000 "YES"**	Begin test at 100mm
POS. #1 = XXX.XX	**"NO" 10500 "YES"**	Force acquired at 105mm position
POS. #2 = XXX.XX	**"NO" 11000 "YES"**	add 5mm for others
POS. #3 = XXX.XX	**"NO" 11500 "YES"**	
POS. #4 = XXX.XX	**"NO" 12000 "YES"**	
POS. #5 = XXX.XX	**"NO" 12500 "YES"**	
POS. #6 = XXX.XX	**"NO" 13000 "YES"**	
POS. #7 = XXX.XX	**"NO" 13500 "YES"**	
POS. #8 = XXX.XX	**"NO" 14000 "YES"**	Finished entering positions
PARK POS.=XXX.XX	**"NO" 01200 "YES"**	Crosshead Park 12mm position
TEST DELAY=XXX.XX	**"NO" 1 2 0 YES**	Pause for 120 between pts
S.I., Pascal	**YES**	Accept S.I. units
RUN# START	**NO YES**	Clears RUN# to zero

G. Shear Rate Sweep

1. PVC, PP, PET Example Rate Sweep Data



Note the Newtonian-like character of the PET at 285 °C. The PP shows some signs of falling toward a constant viscosity at lower rates but the PVC looks very powerlaw like (straight line) throughout the sweep.

2. A Standard Rate Run Procedure

Galaxy V

Setting a Rate Sweep rheometer *PROGRAM*

Note press **EDIT** on the front panel of the rheometer to start the process

Machine Responses	PRESS THESE KEYS	Comments
Melt Time = XXX.X	**"NO" 3 6 0 "YES"**	XXX X is any given number in seconds.
MATL ID= ????????	**"NO" ASK "YES"**	NO clears, + + makes "B" appear, YES accepts value
TEMP.= XXX.X	**"NO" 2 3 0 "YES"**	NO clears, 230C entered, YES accepts value
ORIF. DIA.=X.XXX	**"NO" 1000 "YES"**	assumes capillary die is 1mm in diameter
ORIF. LEN.=XX.XX	**"NO" 2000 "YES"**	assumes capillary die 20 mm in length
SAMPL LEN=XX.XXX	**"NO" "YES"**	Set sample rate to zero for rate run
RATE #1= XXX.XX	**"NO" 30000 "YES"**	Set Ram Rate to 300 mm/min
RATE #2= XXX.XX	**"NO" 1000 "YES"**	Set Ram Rate to 100 mm/min
RATE #3= XXX.XX	**"NO" 03000 "YES"**	Set Ram Rate to 30 mm/min
RATE #4= XXX.XX	**"NO" 01000 "YES"**	Set Ram Rate to 10 mm/min
RATE #5= XXX.XX	**"NO" 00300 "YES"**	Set Ram Rate to 3 mm/min
RATE #6= XXX.XX	**"NO" 00100 "YES"**	Set Ram Rate to 1mm/min
RATE #7= XXX.XX	**"NO" 00040 "YES"**	Set ram rate to 0.4 mm/min
RATE #8 = XXX.XX	**"NO" 01000 "YES"**	Set ram rate to 10 mm/min (again)

RATE #9 = XXX.XX	"NO" "YES"	Zero rate means no more speeds
MELT FORCE=XXXX	"NO" 1000 "YES"	1000N Pre-Start Force to Pack
TERM FORCE=XXXX	"NO" 3300 "YES"	3300N Safety Overload
START POS.=.XX.XX	"NO" 14000 "YES"	Begin test at 140 mm from top
POS. #1 = XXX.XX	"NO" 17500 "YES"	Force acquired at 175 mm position
POS. #2 = XXX.XX	"NO" 19000 "YES"	Force acquired at 190 mm position
POS. #3 = XXX.XX	"NO" 19800 "YES"	Force acquired at 198 mm position
POS. #4 = XXX.XX	"NO" 20400 "YES"	Force acquired at 204 mm position
POS. #5 = XXX.XX	"NO" 20800 "YES"	Force acquired at 208 mm position
POS. #6 = XXX.XX	"NO" 21000 "YES"	Force acquired at 210 mm position
POS. #7 = XXX.XX	"NO" 21100 "YES"	Force acquired at 211 mm position
POS. #8 = XXX.XX	"NO" 21700 "YES"	Force acquired at 217 mm position
PARK POS.=XXX.XX	"NO" 05500 "YES"	Crosshead Park at 55 mm position
TEST DELAY=XXX.X	"NO" "YES"	No Pause between points
RUN# START	"NO" "YES"	Clears RUN# to zero
PROGRAM ENTERED		

Galaxy III & IV

Setting a Rate Sweep rheometer *PROGRAM*

Note press **EDIT** on the front panel of the rheometer to start the process

Machine Responses	PRESS THESE KEYS	Comments
Melt Time = XXX.X	NO 3 6 0 YES	XXX X is any number value given in seconds
MATL ID= ????????	"ENTER YOUR MAT ID"	NO clears YES accepts final word
TEMP.= XXX.X	NO 2 3 0 YES	NO clears, 230 °C entered, YES accepts value
ORIF. DIA.=.XXXX	NO 1000 YES	assumes your capillary die is 1mm diameter
ORIF. LEN.=X.XXX	NO 2000 YES	assumes capillary die length is 20 mm
SAMPL LEN=XX.XXX	NO YES	Set SAMPLE LEN to 0.0 for rate run
RATE #1= XX.XXX	NO 25000 YES	Set Ram Rate to 250 mm/min
RATE #2= XX.XXX	NO 05000 YES	Set Ram Rate to 50 mm/min
RATE #3= XX.XXX	NO 00600 YES	Set Ram Rate to 6 mm/min
RATE #4= XX.XXX	NO 00100 YES	Set Ram Rate to 1mm/min
RATE #5= XX.XXX	NO 00030 YES	Set Ram Rate to 0.3 mm/min
RATE #6= XX.XXX	NO 00600 YES	Set Ram Rate to 6 mm/min (again)
RATE #7= XX.XXX	NO YES	0.0 rate means no more rates
MELT FORCE=XXXX	NO 1000 YES	1000N pre-start force to pack
TERM FORCE=XXXX	NO 3300 YES	3300N safety overload
START POS.=X.XXX	NO 10000 YES	Begin test at 100 mm
POS. #1 = X.XXX	NO 13500 YES	Force acquired at 135 mm position
POS. #2 = X.XXX	NO 15200 YES	Force acquired at 152 mm position
POS. #3 = X.XXX	NO 16000 YES	Force acquired at 160mm position
POS. #4 = X.XXX	NO 16250 YES	Force acquired at 162.5 mm position
POS. #5 = X.XXX	NO 16300 YES	Force acquired at 163 mm position
POS. #6 = X.XXX	NO 17800 YES	Force acquired at 178 mm position
PARK POS.=X.XXX	NO 01200 YES	Crosshead Park 12 mm position
TEST DELAY=XXXX	NO YES	No Pause between pts
S.I., Pascal	YES	Accept S.I. units
RUN# START	NO YES	Clears RUN# to zero
PROGRAM ENTERED		

H. Shear Stress Sweep

1. Constant Stress vs. Constant Rate

If testing in the non-Newtonian region constant stress test will show the effects of the chemical breakdown of the material plus the effect of increasing shear rate on the viscosity. That is, constant stress tests on degrading material tested in the non-Newtonian region have a viscosity vs. time curve which falls faster than the corresponding viscosity vs. time data collected at constant rate.

2. A standard Stress Run Procedure

Galaxy V

Setting a Constant Stress rheometer *PROGRAM*

Note press **EDIT** on the front panel of the rheometer to start the process

Machine Responses	PRESS THESE KEYS	Comments
Melt Time = XXX.X	NO 3 6 0 YES	XXX X is any number value given in seconds
MATL ID= ????????	"ENTER YOUR MAT ID"	NO clears YES accepts final word
TEMP.= XXX.X	NO 2 3 0 YES	NO clears, 230 °C entered, YES accepts value
ORIF. DIA.=.XXXX	NO 1000 YES	assumes your capillary die is 1MM DIAMETER
ORIF. LEN.=X.XXX	NO 2000 YES	assumes capillary die length is 20 MM
SAMPL LEN=XX.XXXX	NO 02500 YES	Set SAMPLE LEN to 2.5 MM
FORCE *#1= XX.XXX*	NO 70000 YES	Set Load to 700N
FORCE *#2= XX.XXX*	NO 70000 YES	Set Load to 700N
FORCE *#3= XX.XXX*	NO 70000 YES	Set Load to 700N
FORCE *#4= XX.XXX*	NO 70000 YES	Set Load to 700N
FORCE *#5= XX.XXX*	NO 7000 YES	Set Load to 700N
FORCE *#6= XX.XXX*	NO 70000 YES	Set Load to 700N
FORCE *#7= XX.XXX*	NO YES	0.0 load means no more loads
MELT FORCE=XXX.X	NO 0700 YES	700N pre-start force to pack
TERM FORCE=XXX.X	NO 3300 YES	3300N safety overload
START POS.=X.XXX	NO 130005 YES	Begin test at 130 MM
POS. #1 = X.XXX	NO 14000 YES	Speed acquired at 140-142.5 position
POS. #2 = X.XXX	NO 15000 YES	Speed acquired at 150-152.5 position
POS. #3 = X.XXX	NO 16000 YES	Speed acquired at 160-162.5 position
POS. #4 = X.XXX	NO 17000 YES	Speed acquired at 170-172.5 position
POS. #5 = X.XXX	NO 18000 YES	Speed acquired at 180-182.5 position
POS. #6 = X.XXX	NO 19000 YES	Speed acquired at 190-192.5 position
PARK POS.=X.XXX	NO 05500 YES	Crosshead Park 55 MM position
TEST DELAY=XXXX	NO 1 8 0 YES	3 minute Pause between pts
S.I., Pascal	YES	Accept S.I. units
RUN# START	NO YES	Clears RUN# to zero
PROGRAM ENTERED		

Galaxy III & IV

Setting a Constant Stress rheometer *PROGRAM*

Note press **EDIT** on the front panel of the rheometer to start the process

Machine Responses	PRESS THESE KEYS	Comments
Melt Time = XXX.X	NO 3 6 0 YES	XXX X is any number value given in seconds
MATL ID= ????????	"ENTER YOUR MAT ID"	NO clears YES accepts final word
TEMP. = XXX.X	NO 2 3 0 YES	NO clears, 230 °C entered, YES accepts value
ORIF. DIA.=XXXX	NO 1000 YES	assumes your capillary die is 1mm diameter
ORIF. LEN.=X.XXX	NO 2000 YES	assumes capillary die length is 20 mm
SAMPL LEN=XX.XXX	NO 02500 YES	Set SAMPLE LEN to 2.5 mm
FORCE *#1= XX.XXX*	NO 70000 YES	Set Load to 700N
FORCE *#2= XX.XXX*	NO 70000 YES	Set Load to 700N
FORCE *#3= XX.XXX*	NO 70000 YES	Set Load to 700N
FORCE *#4= XX.XXX*	NO 70000 YES	Set Load to 700N
FORCE *#5= XX.XXX*	NO 70000 YES	Set Load to 700N
FORCE *#6= XX.XXX*	NO 0 70000 YES	Set Load to 700N
FORCE *#7= XX.XXX*	NO YES	0.0 load means no more loads
MELT FORCE=XXXX	NO 0700 YES	700N pre-start force to pack
TERM FORCE=XXXX	NO 3300 YES	4400N safety overload
START POS.=X.XXX	NO 10000 YES	Begin test at 100 mm
POS. #1 = X.XXX	NO 11000 YES	Speed acquired at 110-112.5 position
POS. #2 = X.XXX	NO 12000 YES	Speed acquired at 120-122.5 position
POS. #3 = X.XXX	NO 13000 YES	Speed acquired at 130-132.5 position
POS. #4 = X.XXX	NO 14000 YES	Speed acquired at 140-142.5 position
POS. #5 = X.XXX	NO 15000 YES	Speed acquired at 150-152.5 position
POS. #6 = X.XXX	NO 16000 YES	Speed acquired at 160-162.5 position
PARK POS.=X.XXX	NO 01200 YES	Crosshead Park 12 mm position
TEST DELAY=XXX.X	NO 1 8 0 YES	3 minute pause between pts
S.I., Pascal	YES	Accept S.I. units
RUN# START	NO YES	Clears RUN# to zero
PROGRAM ENTERED		

I. Combining Time and Rate Runs

One of the difficulties in getting accurate viscosity vs. rate data can be that the material degrades during the test. ASTM D3835 states that when a material is unstable (degrades >5% of initial value) then each shear rate must be run after a given residence time in the barrel. That is each rate requires a separate charge of the barrel so that all data are compared at the same residence time in the barrel. There are other ways.

If only three or four unique rates are run during a test some can be repeated at various times. In this way an estimate of the degradation effects and shear rate effects can be made at the same time. Indeed, interactions between rate and time can only be studied this way. You may wish to review a paper given on this at a recent RETEC[2].

Constant shear rate vs. time runs may be collected at a number of different shear rates for each charge of material then merge together. Taking data off these graphs at constant time allows isochronal (constant time) curves of viscosity vs. shear rate to be obtained. Extrapolation to viscosity vs.

[2]Reilly,J.F. & P.A.Limbach. "Correlating Melt Rheology of PET to solution IV", RETEC 1992, Atlanta, Ga.

rate curves at zero time can also be accomplished with this technique. How the viscosity vs. rate curves are affected by time at constant temperature can be evaluated.

J. Accuracy and Reproducibility

Precision

In our inhouse labs we have found the same machine day-to-day reproducibility of the apparent shear viscosity in shear rate ranges of 10 to 1000 to be about 1.5 to 2.0% (1 standard deviation over the mean viscosity value, pooled estimate). The variation tends to increase very quickly at lower rates. This variation value is the same range found by 17 labs participating in the D3835 round robin testing in 1991.[3] This 2% value is used in the reduced chi square estimate (RCS) shown with the fitted information in KARS. The RCS tells how many standard deviation the fitted curve is away from the typical standard deviation of a point.

The machine to machine reproducibility (for all capillary rheometers in general, according to ASTM) is significantly higher and the latest values are included in ASTM D3835 precision and bias statement found at the end of the ASTM document. This value is on the order of 8%. See ASTM method D3835 (attached) for a graphical display of round robin results.

Accuracy

The PRT'S on Kayeness Inc. machines are calibrated to NIST traceable standard temperature boxes which are recalibrated every 6 mo. by NIST. Die diameters are checked with hardened, class X gage pins (+/- 20 millionths accuracy). Die lengths are checked with micrometers for flat dies. For those with entrance cones a matching cone is inserted and the overall length measured. Capillary length is back calculated through trigonometry . Piston tips and length are check with hand micrometers or laser micrometers. Force cells are calibrated using a calibration cell which is calibrated with dead weights that are NIST traceable. Accurate die diameter measurements, particularly at very small die diameters, is the most critical item for achieving accurate results. NIST does provide standard reference materials for melt indexers which can be used on rheometers though they tend to be expensive. ASTM also has a group of standard materials for those who wish to participate in round robins, all are welcome to contact the current chairperson of D20.30.08 to get involved. Many of these materials are used as Indexer standards as well and can be used as a good bridge in going from Indexer to more controlled capillary rheometry .

[3]Report on file at ASTM headquarters, 1916 Race St., Philadelphia, Pa. 19103-1187 (215) 299-5529

V. Data Analysis

A. KARS

The Kayeness Advanced Rheometry Software or KARS is a software program that runs on an IBM PC or 100% compatible platform. KARS will accept and analyze data from the rheometer, doing the typical Rabinowitsch and Bagley corrections if desired. Printing, plotting, curve fitting and some application specific items like predicting Intrinsic solution viscosity (I.V.) can also be performed. KARS has it's own separate manual and will not be covered here. Most of the graphs produced for this manual came directly from KARS via the capturing of plotter output which can be imported directly by the WORD® processing program used to produce this manual.

B. Cogswell Analysis[4]

Using a Quattro© compatible spreadsheet worksheet (available from Kayeness) you can easily perform a typical Cogswell type analysis for elongational viscosity, shear modulus as well as the other shear viscosity calculation normally obtained in a Cogswell type analysis[1]. You need to collect data with a very short L/D die and a die longer than 16:1 L/D and acquire swell data on the extrudates to perform this analysis.

VI. Maintenance

A. NIST Traceability

You can assure yourself of NIST traceability if you have a service contract on your machine with Kayeness. After a calibration certification papers with the NIST traceability reference numbers will be provided along with the before and after calibration values of the machine (for Force, temperature etc.). Please contact Kayeness or your local representative for more information about this service.

B. Force Calibration

1. Calibrating the Calibration Unit

The unit which can be easily calibrated with dead weights then put in series with the rheometers load cell must be calibrated to assure proper calibration of the rheometer. To calibrate the calibration unit:

Obtain a weight or set of weights which are at least 10% of the calibration load cells total capacity. Know the force exerted by these weights to ± 0.5%.

Un-screw the phenolic ends if they are currently on the calibration unit and screw the round aluminum platform into the side of the calibration load cell which has the bolt heads exposed.

---

[4]F.N. Cogswell, Polymer Melt Rheology: Aguide for Industrial Practice",John WIley &Sons (PRI), New York, 1981.

Connect the calibration load cell (1000 lbs) to the AD 4321 weighing indicator display unit. Plug in the calibration unit and allow it to warm up for 15 minutes.

Open the bottom right cover on the unit to expose the dip switches and the calibration (CAL) switch. The calibration switch is the furthest switch on the right.

Push the calibration switch up and press the **STANDBY/OPERATE** button.

Press **STANDBY/OPERATE** again and you should see CAL appear then the display will go blank.

Being sure there is nothing on the platform and the load cell is sitting flat press the **ZERO** button. The unit will display CAL-1.

Press **STANDBY/OPERATE** and . . . . . should appear momentarily on the display unit. Then a series of zero will appear. It is now time to enter in the value of the calibration weights. Pressing the **GROSS/NET** key will increment the current digit which is flashing. Move to the left digits by pressing **ZERO** and move to the right digits by pressing **TARE.** Enter the value of the calibration weights. (e.g. 100 lbs)

Next place the weights onto the level platform. It is important that the load be applied to a level platform for the proper calibration. Press **STANDBY/OPERATE.**

Move the calibration switch back down to it's normal position then press **STANDBY/OPERATE** you should see the calibration weight appear if the weights are still in place. Remove the weights and you should see zero appear. If you do not, repeat the procedure checking for errors.

Read the Model:AD-4321A/B instruction manual that came with the calibration display unit for more details. Note that the dip switches are set so that when the unit first turns on it goes directly to showing the force. Page 12 of the calibration manual covers the calibration procedure. Earlier pages cover the setting of dip switches and the resolution of the device. The resolution is set for the maximum allowed using this cell. Using this resolution requires that the unit not be brought over about 2640 N (600 lbs) or the display will go blank.

2. Calibrating the rheometer load cell with the calibration unit

Once assured of the calibration unit's accuracy, calibrate the rheometer's force unit by doing the following (explicit details follow below):

Make the load cell the active device by pressing + at the rheometer
Zero the rheometer force reading.

Put attachment bobs on calibration unit.
Zero calibration unit.
Move the cross head down until it is about 6" from the swing arm.
Insert the calibration cell up into load cap and press DN.
Press **RESET** when calibration cell is 1/4" from hitting swing arm.
Press the **EMERGENCY STOP** button.
Turn the top large pulley counterclockwise until 400 lbs is read on calibration unit.
Adjust the fine gain until both units match.
Turn pulley clockwise and remove CAL cell.
Press **RESET** then pull **EMERGENCY STOP** button out.
Press **UP**

Details:

Zero the rheometer force reading:

Machines equipped with barrel pressure transducers

Machines equipped with pressure transducers automatically set the force zero when *Auto F Zero* is set ON and the **Delete** key on the front panel is pressed. Make the load cell the active device by pressing + at the rheometer prior to pressing the **Delete** key. Zeroing is complete.

You can verify that Auto force zero is ON by pressing **SHIFT** then the **PRNT** keys at the rheometer. Press **YES** until Auto F Zero appears. If it shows ***Auto F Zero ON*** press **RESET** to leave it set ON. If it shows ***Auto F Zero OFF*** press **NO** then **YES** then **RESET** to turn Auto zeroing ON. The machine will Once set ON it will remain in effect even if the unit is shut off.

Machines equipped with load cell only

Remove the top cover of the rheometer. Through a slot on the top left of the clear plastic electrical protection cover find a series of small adjustment screws. There are five adjustment screws, the middle is labeled OFFSET this adjusts the offset or Zero on the Force cell. To show the active force value on the front panel of the rheometer press **TEST** on the rheometer front panel. Press **NO** to the front panel display of ***TEMP. TEST?*** then press **YES** when the front panel displays ***FORCE TEST?***

Being certain you are adjusting the third screw from the left turn the screw clockwise to increase the zero force value or counter-clockwise to decrease it's value until you read about 5.0 N (about 1.0 lbs) on the front display. Setting the value to zero is **incorrect**! The electronics only allows value greater than or equal to zero to be displayed. That is if the display reads zero it could really be any negative load value like -10, -15 etc.

Put attachment bobs on calibration unit.

Small phenolic caps, similar to the one that sits on top of the plunger, attach to each end of the calibration load cell after removing the round loading platform. This isolates the calibration cell from the heat of the machine. The most accurate calibration will be done at normal test temperatures.

Zero calibration unit.

With the calibration cell in a vertical position (the same position it will be in when placed in the load cap where the plunger normally goes) on the table and no external loads applied press **ZERO** on the calibration display units front panel. Remember the calibration unit is the reference, use its reading to judge where you are in terms of load on the system. If your rheometer force calibration is grossly in error you could overload your equipment if you trust the rheometer front panel force display before it is calibrated.

Move the cross head down until it is about 150 mm from the swing arm.

Press DN on the rheometer's front panel and allow the cross head to come down until there is about 150 mm clearance between the cross head and the swing arm. This is enough room to allow you to slip the calibration load cell into the upper load cap and hold it there. Press RESET to stop the cross head motion downward or push up on the cell with about 10-15N of force. If the cross head has come down too far press **UP** and the cross head will move quickly upward. Press **RESET** when it has moved up enough to insert the calibration load cell.

Insert the calibration cell up into load cap and press DN
Press RESET when the calibration cell is 10 mm from hitting swing arm.
Press the EMERGENCY STOP button.

Put the calibration load cell in series with the current cell by putting the top phenolic bob into the rheometers load cell cap. Be sure to keep the calibration load cell in the same relative position as when it was zeroed (i.e. don't flip it over.) While holding the load cell up in the load cap press **DN,** press **RESET** when the bottom phenolic bob comes within 10 mm (½") of the swing arm. **DO NOT LET THE MACHINE DRIVE THE TWO LOAD CELLS TOGETHER!** Press **RESET** at anytime to stop the machines movement.

Once stopped within a 10 mm (½") of the swing arm press in the big red **EMERGENCY STOP** button. The emergency stop button is like the clutch on a car it disengages the drives system.

Turn the top large pulley counterclockwise until 1800N is read on the calibration unit.

With the EMERGENCY STOP button pressed in, rotate the top pulley counterclockwise to move the cross head down. Position the calibration cell so that it is sitting flat and centered over the top of the barrel hole. Watch the calibration display unit and keep turning until you see about 1800N on the calibration display unit. **DO NOT EXCEED 2400 N**

In the same bank of calibration screws as the force zero offset adjust the fine force gain (screw on far left) until the calibration unit and the rheometer force unit match. The rheometer display should read 1.0-1.2N higher than the

calibration display unit to account for the plunger weight. (e.g. If the calibration display unit reads 1832.6 the rheometer front display should read 1833.8)

Turn the pulley clockwise by hand until you can remove the calibration cell. Hold the calibration cell so it does not fall off the rheometer. **Important step** Press **RESET** then and only then pull the EMERGENCY STOP button back out. Press **UP** on the rheometer to return it to park position and you are ready for your next test.

3. Calibrating without the calibration unit

Without the calibration unit the load cell currently being used for measurements on the rheometer must be removed from the cross head and checked directly with dead weights. Due to the ease of stripping threads, excessive twisting cables and awkwardness of handling large loads we do not recommend this. An aluminum platform has been provided for this purpose. We suggest placing the load on the floor and being sure the platform is on a level surface before attempting the calibration. Adjust the zero offset (see above for location) with no weights on the platform then adjust the fine gain to achieve the calibration weight. We suggest using a minimum of 10% of the load cells full scale capacity to achieve a calibration.

C. Temperature Calibration

One Temperature Reference

To perform a temperature calibration go into EDIT and set the temperature of the machine to the temperature of the calibration thermometer. Allow the machine to come within a few degrees of the thermometer's nominal value **then** remove the set screw which covers the thermometer well and carefully insert the thermometer into the thermometer well. DO NOT REMOVE THE PRT SENSOR . The thermometer well is just in front of the PRT probe and can be accessed by removing the small Allen head screw that keeps it free from debris that would fall in during normal operations.

Allow the system to equilibrate for a minimum of 20 minutes after placing the thermometer into the machine. Read the thermometer and check the thermometer's calibration certificate to determine if any adjustments are needed to determine the true temperature. For example, the thermometer shows the mercury column to be at 230.4 C the calibration certificate says this thermometer reads 0.1 C high so the true temperature is 230.3 and this is what we want the front panel of the rheometer to display.

Remove the front top cover of the rheometer and locate the slot in the clear plastic sheet on the LEFT side of the machine (not the slot on the top, that's for force calibration). There are four adjustment screws from the top down they are labeled OFFSET, GAIN, COMP and EYE.

To perform the calibration we must first disconnect the temperature control system so it does not change the temperature of the machine as we are trying to calibrate it! To "lock" the temperature in place simply press down both the RED and

BLACK buttons together on each side of the rheometers front LED display. Hold the buttons down until you see the current temperature reading change to some new number. After letting go of the buttons you should see a dot flashing on the left side of the LED display (a dot on the right side may also be flashing). If you do not see any flashing dot press down both the RED and BLACK and release only after you see the number on the front panel change. Only when the flashing dot appears should you turn the OFFSET screw on the left side of the rheometer until the FRONT display matches the true temperature as determined from your thermometer and its calibration certificate. Once set correctly press either the BLACK or RED button down to start the system controlling again. The flashing dot should disappear once the RED or BLACK button is pressed.

After about 15 minutes the rheometer should come back to controlling temperature and the rheometer and thermometer readings should agree.

Carefully remove the hot thermometer and place it on some cotton patches to cool. Store it in a protective box along with its calibration certificate.

The pre-1994 ASTM specification D3835 (and D1238 Melt indexer) calls for calibration of temperature based on the melt temperature. Kayeness has performed a number of studies comparing the temperature measured via probes (thermocouples, PRT's) in the melt, probes of similar size to the barrel without melt, and thermometers in barrel wells. Generally the agreement between these probes is within a few tenths of a degree and requires great care in the their measurement. At higher temperatures >300 °C it is not practical or cost effective to make such measurements in the melt. We suggest routine calibration with thermometers in the barrel for general temperature maintenance. We suggest you read the latest ASTM specification for temperature calibration included with this manual.

Two Temperature References

Remove the front stainless steel top cover of the rheometer and locate the slot in the clear plastic sheet on the LEFT side of the machine (not the slot on the top, that's for force calibration). There are four adjustment screws from the top down they are labeled OFFSET, GAIN, COMP and EYE.

The two point temperature calibration can be performed using an ice bath or two certified (NIST traceable) thermometers which cover the range of testing being performed.

Using an ICE BATH

If using an ice bath go into **EDIT** and set the **TEMP.** to 0.0 only then can the PRT be removed from the rheometer. Unscrew the two fasteners which hold the top barrel insulator in position and remove the PRT. Place the PRT temperature probe from the rheometer into a distilled ice / distilled water mixture which has been well stirred. Allow the system to equilibrate for 10 minutes then without performing the temperature "lock" (as is needed when the rheometer is controlling the

temperature) turn the OFFSET screw until the front display reads 0.0. Wipe the PRT dry and insert it back into the machine. Set the machine temperature to match the calibration thermometer's nominal value (e.g. 230 C).
The second part of the calibration is exactly the same as the single point calibrations described above **except** now the second temperature is adjusted by turning the **GAIN** screw NOT the offset screw. Perform the following actions and refer back to the single point calibration where needed but don't adjust the offset screw.

- Set the rheometer to the thermometer temperature.
- After the rheometer is at temperature insert the thermometer.
- Determine true temperature based on the thermometer.
- "Lock" the temperature and adjust the GAIN screw as needed to match true temperature.
- Press the RED button to unlock the system.

Using two reference thermometers
The lower temperature calibration of one of the two reference thermometers is exactly the same as doing a one temperature reference calibration and can be done by following the procedure described above. The higher temperature thermometer of the pair is done almost exactly the same. The only difference is when the adjustment to match the display to the front panel is made, the GAIN screw is used for the second thermometer rather than the offset adjustment. Be careful to place the thermometers into the thermometer well ONLY when the rheometer barrel temperature is within a degree or two of the thermometer's nominal temperature value.

DO NOT ADJUST THE COMP SCREW!. This corrects for the non-linearity of the voltage vs. temperature curves for the PRT Kayeness uses and is set at the factory. If you do change this setting it will need to be readjusted at the Kayeness factory.

D. Pressure Transducer Calibration

ZERO SHIFT
The main change in a pressure transducer's output with temperature is the zero shift or sometimes called the zero offset. As temperature increases the pressure transducer reading will increase even when the pressure remains exactly the same. The *change* in the transducers output for a given *change* in pressure is not affected very much by temperature (provided the electronics housing is near ambient). This results in the need for calibration of the zero point only but, it must be at the required test temperature.

RCAL
With the addition of the pressure transducer comes an automatic check of the

electronics system. A precision resistor is placed into the circuit and acts like a known pressure on the system. From the systems response to this simulated measurement we can assess the current calibration of the machine. The value of the pressure signal obtained when measuring the precision resistor is known as the RCAL (Resistance CALibration) value. The current RCAL value can be seen by pressing **TEST** on the rheometer's front key pad and then pressing **NO** until the words RCAL TEST appear in the display. Pressing **YES** will cause the current RCAL value to be displayed for the active device.

The machine always performs the ZERO SHIFT and RCAL calibrations together when the **DELETE** button is pressed on the front panel of the rheometer. To be done correctly the machine should be warmed up for at least twenty minutes after being first turned on and locked on to the current test temperature. The pressure transducer should be in the barrel and also warmed up for twenty minutes. For the most accurate measurements, the pressure transducer reservoir should be clean so that previously tested material is not influencing the readings.

Calibrate the Pressure Transducer by pressing **DELETE** on rheometer key pad ONLY when:

- Pressure reservoir cleaned.
- Pressure transducer in barrel and snug (100 in-LB torque).
- Cross head is at the Park position (Up and away from plunger).
- Machine is "locked in" at test temperature.
- PT is active sensing device (Press - on front panel)

The Rcal value for pressure transducers should be set as follows:

Nomal Range	English	SI
(psi)	(lbs)	(N)
10,000	933	4151
20,000	1867	8303
30,000	2800	12454

E. Dimensional Verification

1. Die Diameter

Check the die by doing the following:

A quick visual inspection of the die:

The die bore and its finish are critical, there should be no visible tool marks. Top and bottom surfaces should be flat and smooth.

Clean the Die.

The die should be extremely clean!, Clean **cold** die with drill bits and solvent if needed. (Any die that has been in use will need it!). For Indexer dies use the small cleaning brush supplied. Do not clean the die with the Go No-Go pins!

Select the Go No-Go pin needed:
For example, for a nominally 0.030" die choose the ASTM 0.030" pin set. If you have a 1.0 mm die choose the ISO 1.0 mm Go, No-GO gage. The Indexer checking pins should be 0.0823" (GREEN) and 0.0827" (RED) in diameter.

Carefully attempt to put the green side of the gage into the top end of the die. The pins are hardened steel but can we worn down. They are made to 0.0010 mm tolerance, do not wear them down by forcing them into a die. The pin should go **all the way through** the die **without** being forced. Remove the pin and enter through the bottom side of the die as well. If the pin will not go **all the way through** either end of the die it Fails to meet the specifications. Verify you have the correct nominal dimension and reject the die. (DO NOT USE THIS DIE). CHECK BOTH ENDS OF THE DIE WITH THE PIN!

If the green side of the gage goes through both ends of the die then attempt to put the red side pin into the die, be sure to check both ends. If the RED pin enters more than 1 mm into the die on either end the die is rejected. Verify you have the correct nominal dimension and reject the die (DO NOT USE THIS DIE). Getting to this point without rejecting the die means is has passed ASTM specifications listed in ASTM D3835-90 for capillary rheometer dies or D1238 specs for melt indexers.

(SKIP this for INDEXER DIES, ASTM & ISO Indexer die specs are identical!) The die may also be good enough to meet the ISO specifications. This check can be made by selecting the ISO equivalent Go No-Go pin (for a nominal die dimension of 0.030" the ISO 0.03" gage would be used). Check to be sure the green side goes through both ends as before and the red side goes in less than 1 mm on each end. If it passes the die is a more valuable commodity and should be marked as meeting ISO specs. If it has not passed the ISO pins but passed the ASTM pins it should be marked as meeting ASTM specs.

2. Die Length

If the die has a flat entrance angle a hand held micrometer can be used to measure the die length. For dies with an entrance angle other than flat a small precision ball can be mounted to a dial indicator which is only slightly larger in diameter than the die capillary diameter. Small errors in the cone angle will affect the die length measurement very little if the ball is close to the capillary size. Be sure the entrance cone and faces of the die are extremely clean before measuring.
For reference standard melt Indexer dies should be (8.000 ± 0.025 mm) in length. Diameter (2.095 ± 0.005 mm). Be sure the micrometer is zeroed correctly.

3. Barrel Diameter

The support vendor list shows the address for a bore gage manufacturer. Once the barrel is extremely clean, all dimensional measurements are to be

made at room temperature it can be checked using a bore gage. Be sure to get or make a long enough extension so the entire barrel can be checked. The barrels are 9.550 ± 0.005 mm leaving the factory.

4. Plunger Tip Diameter

Clean the barrel tip with a piece of bronze wool or alike and use a hand held micrometer to verify the room temperature diameter of 9.515 to 9.520 mm. This is on the low side of the ASTM specification (but still within barrel - plunger tolerance specifications) as we have found larger tips can lead to seizing in the barrel at higher temperatures.

F. Lubrication

Galaxy V

There are two grease fittings on the Galaxy V machine. One on the lower right side of the worm gear reducer system (just behind main pulley) and one behind the left pole on the swing arm. These should be lubricated about once a year (G.P. #2 grease).

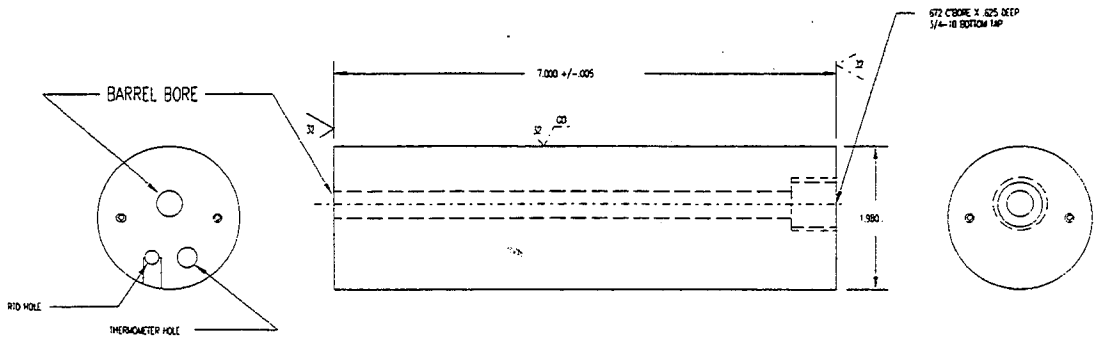
The poles which guide the crosshead-load cell assembly can become dry and cause a squeaking sound. A short spray of WD-40 or similar lubricant will remedy this.

Galaxy III & IV

There is one grease fitting on the Galaxy IV or III machine located on the left pole directly behind the swing arm. This should be lubricated about once a year. (G.P. #2 grease).

G. Barrel Drawing

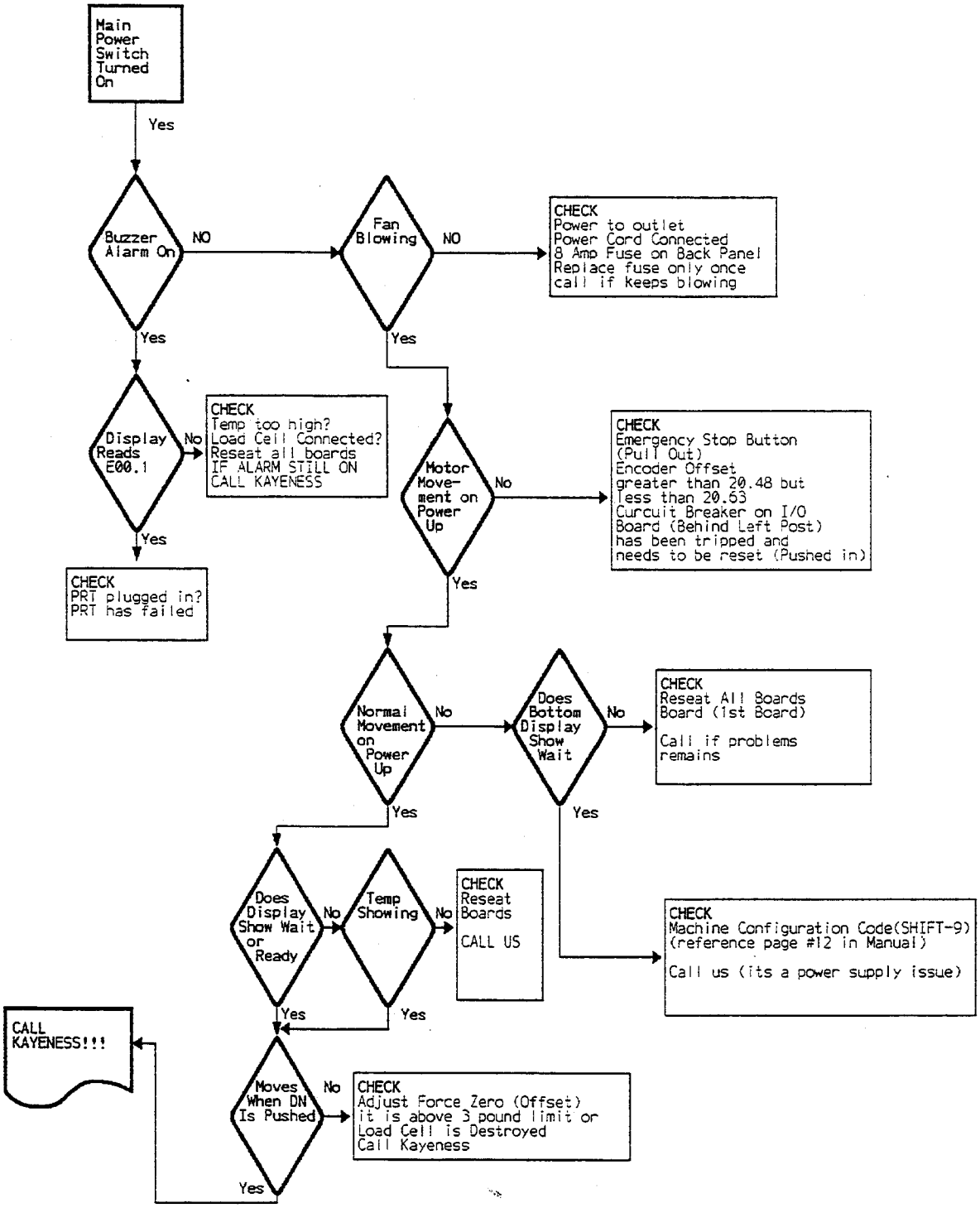
The drawing below may be helpful for understanding placement of the die in the barrel.



VII. Trouble Shooting

A. Logic Diagram

The flow chart below runs through some basic trouble shooting that can be done to get your machine functioning properly. If you have no experience with electrical items or are unsure of any issue dealing with the inner workings of the machine please call us before proceeding, we will be happy to assist you. A Kayeness technician may request an electrical technician at your site to make some voltage checks that require a voltmeter and removal of the machine top safety cover. Don't make this check yourself. For experienced electrical technicians: When reseating boards, always unplug the equipment first and touch the cabinet for ground before touching the boards. Always hold the boards by the edges.


Main Power Switch Turned On
Yes
Buzzer Alarm On
NO
Fan Blowing
NO
CHECK
Power to outlet
Power Cord Connected
8 Amp Fuse on Back Panel
Replace fuse only once
call if keeps blowing
Yes
Yes
Display Reads E00.1
No
CHECK
Temp too high?
Load Cell Connected?
Reseat all boards
IF ALARM STILL ON
CALL KAYENESS
Yes
CHECK
PRT plugged in?
PRT has failed
Motor Move-ment on Power Up
No
CHECK
Emergency Stop Button
(Pull Out)
Encoder Offset
greater than 20.48 but
less than 20.63
Curcuit Breaker on I/O
Board (Behind Left Post)
has been tripped and
needs to be reset (Pushed in)
Yes
Normal Movement on Power Up
No
Does Bottom Display Show Wait
No
CHECK
Reseat All Boards
Board (1st Board)
Call if problems
remains
Yes
Yes
Does Display Show Wait or Ready
No
Temp Showing
No
CHECK
Reseat
Boards
CALL US
CHECK
Machine Configuration Code(SHIFT-9)
(reference page #12 in Manual)
Call us (its a power supply issue)
Yes
Yes
CALL KAYENESS!!!
Moves When DN Is Pushed
No
CHECK
Adjust Force Zero (Offset)
it is above 3 pound limit or
Load Cell is Destroyed
Call Kayeness
Yes

B. Parts List and How to Order

Call Kayeness or your local representative for the latest pricing information; orders can also be placed at that time. A purchase order number will be required.

DIES and ORIFICES

Order Name	Capillary Diameter	ength/ Diam.	Entr. Angle	Capillary Length	Capillary Length	leaning Drill #	Total Length	Capillary Dia	No-Go Pin
	(in)		deg.	(in)	(mm)	# Eng.)	(mm)	(mm)	Note 1
Z295-1C	0.02953	1	180	0.02953	0.75000	70	0.7500	0.75	P295
Z295-16	0.02953	16	180	0.47244	12.00000	70	12.0000	0.75	P295
Z295-33	0.02953	33.3	180	0.98327	24.97500	70	24.9750	0.75	P295
Z295-40	0.02953	40	180	1.18110	30.00000	70	30.0000	0.75	P295
X300-1C	0.03000	1	120	0.03000	0.76200	69	3.2917	0.762	P30
X300-16	0.03000	16	120	0.48000	12.19200	69	14.7217	0.762	P30
X300-20	0.03000	20	120	0.60000	15.24000	69	17.7697	0.762	P30
X300-33	0.03000	33.3	120	1.00000	25.40000	69	27.9297	0.762	P30
X300-40	0.03000	40	120	1.20000	30.48000	69	33.0097	0.762	P30
Z394-16	0.03937	16	180	0.62992	16.00000	61	16.0000	1.0	P394
Z394-20	0.03937	20	180	0.78740	20.00000	61	20.0000	1.0	P394
Z394-30	0.03937	30	180	1.18110	30.00000	61	30.0000	1.0	P394
Y400-01	0.04000	1	90	0.04000	1.01600	61	5.2705	AMP	P40
Y400-15	0.04000	15	90	0.60000	15.2400	61	19.4945	AMP	P40
X400-1C	0.04000	1	120	0.04000	1.01600	61	3.4723	1.016	P40
X400-16	0.04000	16	120	0.64000	16.25600	61	18.7123	1.016	P40
X400-20	0.04000	20	120	0.80000	20.32000	61	22.7763	1.016	P40
X400-30	0.04000	30	120	1.20000	30.48000	61	32.9363	1.016	P40
Z492-16	0.04921	16	180	0.78740	20.00000	3/64	20.0000	1.25	P492
Z492-20	0.04921	20	180	0.98425	25.00000	3/64	25.0000	1.25	P492
Z492-25	0.04921	25	180	1.23031	31.25000	3/64	31.2500	1.25	P492
X500-20	0.05000	20	120	1.00000	25.40000	56	27.7830	1.27	P50
X600-20	0.06000	20	120	1.20000	30.48000	53	32.7897	1.524	P60
Z825-4	0.08250	3.82	180	0.31496	8.00000	1/16	8.0000	MI Die	0051-55
X825-11	0.08250	11	120	0.90750	23.05050	1/16	25.1952	2.0955	0051-55

Note 1: Pin gauges are designed to ASTM Spec. or ISO Spec. ±.0.005 mm, (similar after '94, previous to '94 ASTM spec. was larger)

Note full die entrance angle are given (not half angles): X=120°, Y=90°, W= 60°, Z=180°; thus a 120° angle 0.040 diameter 15:1 L/D is a X400-15

Older 2052 Capillary Rheometer

ITEM	DESCRIPTION
0053-04	Paper Spool Rod
0053-13	Computer Dust Cover
2052-101	Toggle Switch/Hinds Power
2052-102	Exhaust Fan - Paramotor
2052-104	Switch - P/P-Crouse/Hinds Safety
2052-105	Switch Lens - Crouse Hinds
2052-109	Pilot Lamp - C & H - 28 V.
2052-110	Bridge Sensor W/ Mount
2052-120	Touch Keyboard
2052-121	Main Computer Board
2052-122	Sub Computer Board
2052-33	Top Guard - Bronze Tuffak
2052-47	Gate Holder
2052-48	Safety Gate - Bronze Tuffak
2052-49	Micro switch
2052-62	Heat Chamber Cover G7 (Large White)
GP-1020	10 Turn Helipot - Beckman - R50K
GP-1022	10 Turn Helipot - Beckman - R1K
GP-1040	10 Turn Counting Dial
GP-1120	Pomona Plug - Black
GP-1214	Switch- SPST - Solder - Carling (HEAT)
GP-1450	Neon Pilot Light -IDI- W/ Clip
GP-1606	Power Supply - Heavy Duty - 5 VDC
GP-1615	Power Supply - (± 15 V)
GP-1620	Power Supply (6431)
GP-7520	Printer - PL 12M W/ PCB 12 Clamp
GP-7520P	Replacement Print Head - Only
GP-7521	Thermal Printer Paper (3/Pkg)
GP-7705	Digital Temp. Indicator
GP-7707	Over Temperature Cutoff/Alarm
GP-7708	Over Force Cutoff Alarm
GP-7720	Temp. Controller
7051-100	Mother Board
7051-101	Computer Board
7051-102	Analog Temp Board
7051-103	Phase Control Board
7051-104	Main Computer Board
7051-105	Digital Display Board
7051-110	Touch Panel
8052-121	Rheometer Servo Board

GALAXY V, IV and III SPARE PARTS LIST

PART #	DESCRIPTION
MOST COMMON ITEMS	
0051-80	LARGE POWDER FILL FUNNEL
2052-46	PISTON TOP INSULATOR
2052-66	PISTON TIPS
2052-66G	PISTON TIPS W/GROOVE
2052-66R	TFE PISTON TIP O-RINGS (DOZ)
2052-152	O-RING INSTALLATION TOOL
2052-72	POLYIMIDE ORIFICE INSULATOR (BOTTOM)
2052-66LL	1/2" Long piston tip needs short rod
2052-66LLS	1/2" Long piston tip for corrosion resist. barrels
0051-43LL	Short piston rods for Long tips
2052-73	BRONZE ORIFICE HOLDER NUT
GP-1752	PLATINUM RTD
2052-42	LOAD CELL-500 KG.
2052-45I	LOAD CAP
0051-36	S.S. CHARGING TOOL
0051-40	S.S. PATCH CLEANING ROD
0051-43	PISTON ROD - ONLY
0051-44	PISTON GUIDE BUSHING
0051-47	BORE BRUSH HANDLE
0051-48	BRONZE BORE BRUSHES
2052-100	CAPACITOR - SPRAGUE
2052-106	RELAY-MIDLAND ROSS
2052-107	RESISTOR .5 OHM 1%
2052-108	MAIN POWER TRANSISTOR - MJ11032
2052-111	MAIN POWER TRANSISTOR - MJ11033
2051-151	PULL CLIP (PISTON REMOVAL)
2051-156	T-WRENCH HANDLE
2051-157	SOCKET
2052-160	CERAMIC INSULATION BLANKET
2052-200	COMPLETE 4 TRANSISTOR PWR BLOCK ASSY.
2052-300	COMPLETE PISTON ASSEMBLY: 0051-43,44 2052-46,66
2052-22	MOTOR W/GLASS ENCODER (GAL V)
9052-22	MOTOR W/GLASS ENCODER (GAL III)
2052-32	WORM JACK W/ MICRO SWITCH BOX
2052-42	LOAD CELL-500 KG.
9052-42	LOAD CELL 1000 LBS. W/B101 BASE
9052-42	LOAD CELL 1000 LBS. W/ADAPTERS
9052-43	LOAD CELL 2000 LBS. W/B101 BASE
2052-43	FLANGE BEARING - FAF
8052-75	HEAT CHAMBER - TOP INSULATOR (BROWN)
2052-64A	INSULATOR SLEEVE (BARREL MOUNT)

2052-70	BARREL HEATER (SINGLE ZONE)
2052-7DZ	BARREL HEATER (DUAL ZONE)
2052-77	THERMISTOR COVER
7051-45	POLYIMIDE FILL TUBE (LARGE WHITE)
8052-62	HEAT CHAMBER COVER G7 (HI TEMP)
8052-65	HEAT CHAMBER (BARREL - STD)
8052-141	HEAT CHAMBER (BARREL) NON LINED HIGH FORCE
8052-141H	HEAT CHAMBER (BARREL) LINED HIGH FORCE
2052-65H	HEAT CHAMER (BARREL) LINED
8052-79	TOOL RACK
GP0300	LOADING BEAKER (PYREX)
GP-1111	PLUG-MIDGET-HUBBEL
GP-1122	POMONA PLUG -RED
GP-1400	FUSE HOLDER
GP-1410	FUSE - BUZZ - 8 AMP (MAIN)
GP-1428	CIRCUIT BREAKER - 8 AMP
GP-1510	TRANSFORMER 120/24 V.
GP-3004	LEVELING FEET - 3/8-16
GP-7820	RECTIFIER - 35 A.-BRIDGE

C. When your Control material goes out of control

Before a machine leaves the factory it is checked with a control material using standard SQC/SPC techniques. When our data indicates a machine is not performing to previous standards we have always found the answer to be one of the items below.

- Force calibration OK
- Temperature calibration OK
- The program conditions are exactly the same as before
- The Force is zeroed correctly
- Your using the same die as before, dimensions OK.
- Piston Tip Dimension is OK (generally 09.525 > Tip OK > 9.515 mm)
- The die is very clean: if high temp material run previously heat die up to previous sample temperature and re-clean.
- The guide bushing is free to move up and down the Plunger rod.
- The material was dried as before (if necessary)
- The material is exactly the same as before.
- Verify correction scheme used is as before (i.e. Rabinowitsch on or off etc.)
- Machine ID value OK? (See **SHIFT-9**)
- Check if piston rod is bent.
- Put machine temporarily on a filtered cleaner powder supply.
- Clean PRT well. Poor contact between PRT and barrel can cause poor temperature control.

- Is bottom limit switch set correctly (3 mm of plunger movement when at bottom limit switch?)

  Has a previously run material affected the barrel (EVOH particularly nasty)? Heat machine to 400° and bake off coating layer).

VIII. HELP Utilities

A. Rheometer Keypad Functions

Keys on the front rheometer pad are never pressed two at a time like a capital "A" is shift-A on a PC keyboard. **SHIFT PRNT** will denote that the **SHIFT** key is pressed first and released then the **PRNT** key is pressed and released.

SHIFT RUN : This re-sends data and test conditions from the current program over to the PC. If you experience any difficulties in the transfer pressing **RESET** on the rheometer will clear the buffer on the PC and you can try **SHIFT RUN** again.

SHIFT DN : This causes to query ***PURGE?*** if you then press **YES** the rheometer crosshead will move down slowly increasing speed until it reaches maximum speed or the force becomes 1/2 the terminal force currently set on the program. (Note: this means if you are having trouble purging you can increase your terminal force and this may help). If a purge will not occur (Force too high) relieve the pressure on the system by pressing **UP** then **RESET** when the load cap has cleared the plunger bob. Then remove the die and die holder nut. Press **SHIFT DN** then YES to complete the purge.

SHIFT PRNT : This allows access to a number of options which are detailed on a separate page. They include ***Manual Mode***, ***PC attached***, ***Auto Force Zero***, Direct Rheometer output to a Printer and is the logical place for new features to be added.

Using the printer with KARS

When using the KARS software package the printer should be connected directly to the PC. Typically this is done parallel to centronics cable which is supplied with the printer.

- To get the proper symbols and graphical output a Okidata Microline 320 should be set for EPSON emulation mode with character set II active. Other Epson compatible printers will work though some may not support the graphics characters created using the KARS software package.

Connecting Directly to the Rheometer

If you wish to connect the printer directly to the rheometer (No PC) connect your printer to the parallel port at the back of the rheometer. This is a female DB25 25 pin connector. Using **SHIFT PRNT** set the printer to ***ON*** and ***PC Attached*** to option to ***OFF***. Not setting the options correctly will cause the rheometer to stall as it tries to communicate with a device it believes is attached.

B. Conversion Factors (Viscosity, Temp)

Temperature

To Convert From	To	Use Formula
°C	°K	$T_k = T_c + 273.15$
°F	°C	$T_c = (T_f - 32)/1.8$

Viscosity

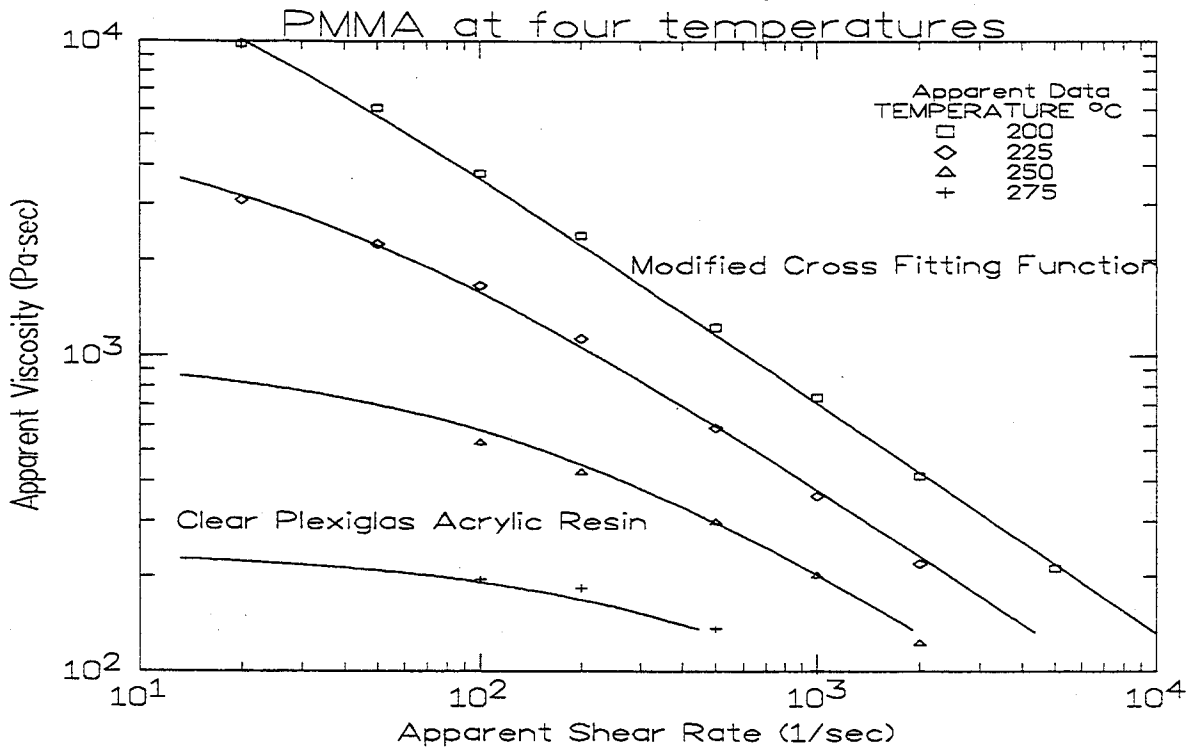
To Convert From	To	Multiply By
Poise	Pa-sec	0.10
centi-Poise	Pa-sec	0.001
centi-Stokes	m^2/sec	1e-6
lbf-sec/ft^2	Pa-sec	4.788026E+01

Pressure or Stress

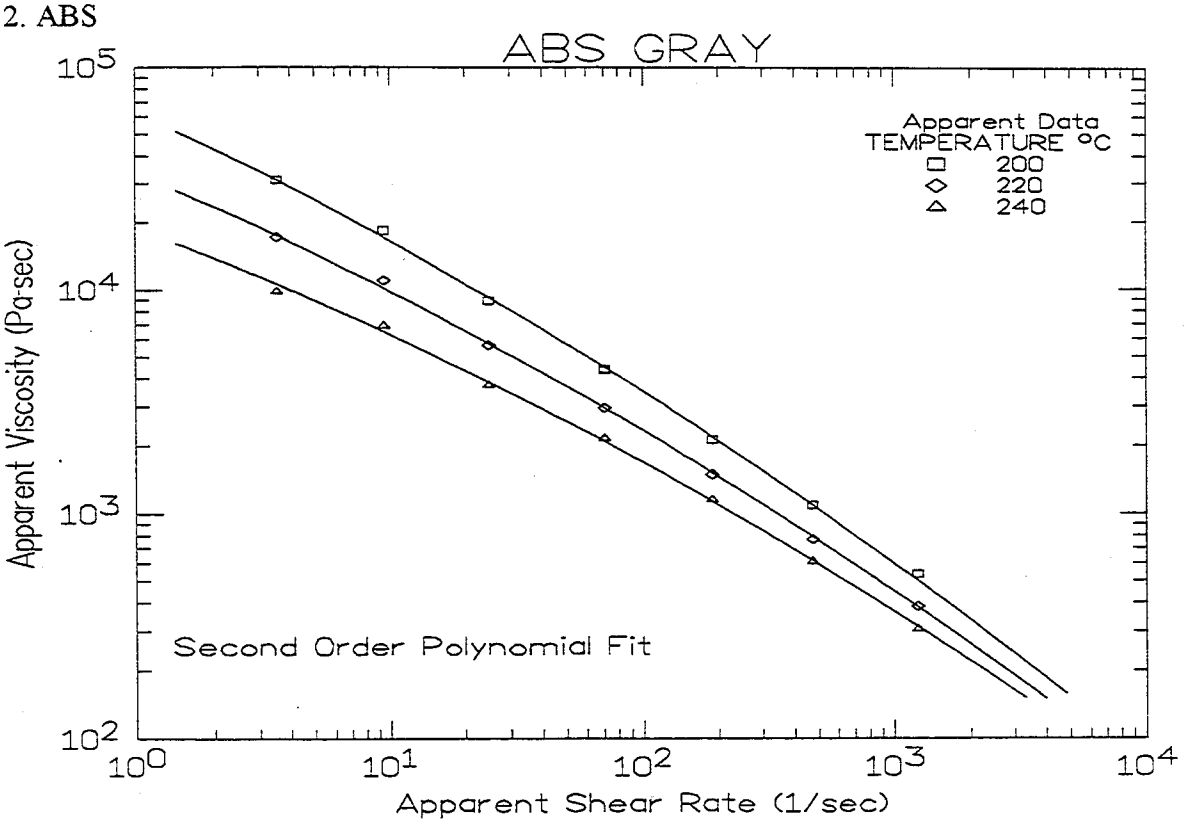
To convert From	To	Multiply By
Psi	Pa	6.894757E+03
lbf/in^2	Pa	6.894757E+03
Atm (STD)	Pa	1.01325E+05
Atm(1 kgf/cm2)	Pa	9.80665E+4
bar	Pa	1.0E+5
dyne/cm2	Pa	0.10
ksi (kip/in2)	Pa	6.894757E+6

C. More Example Runs

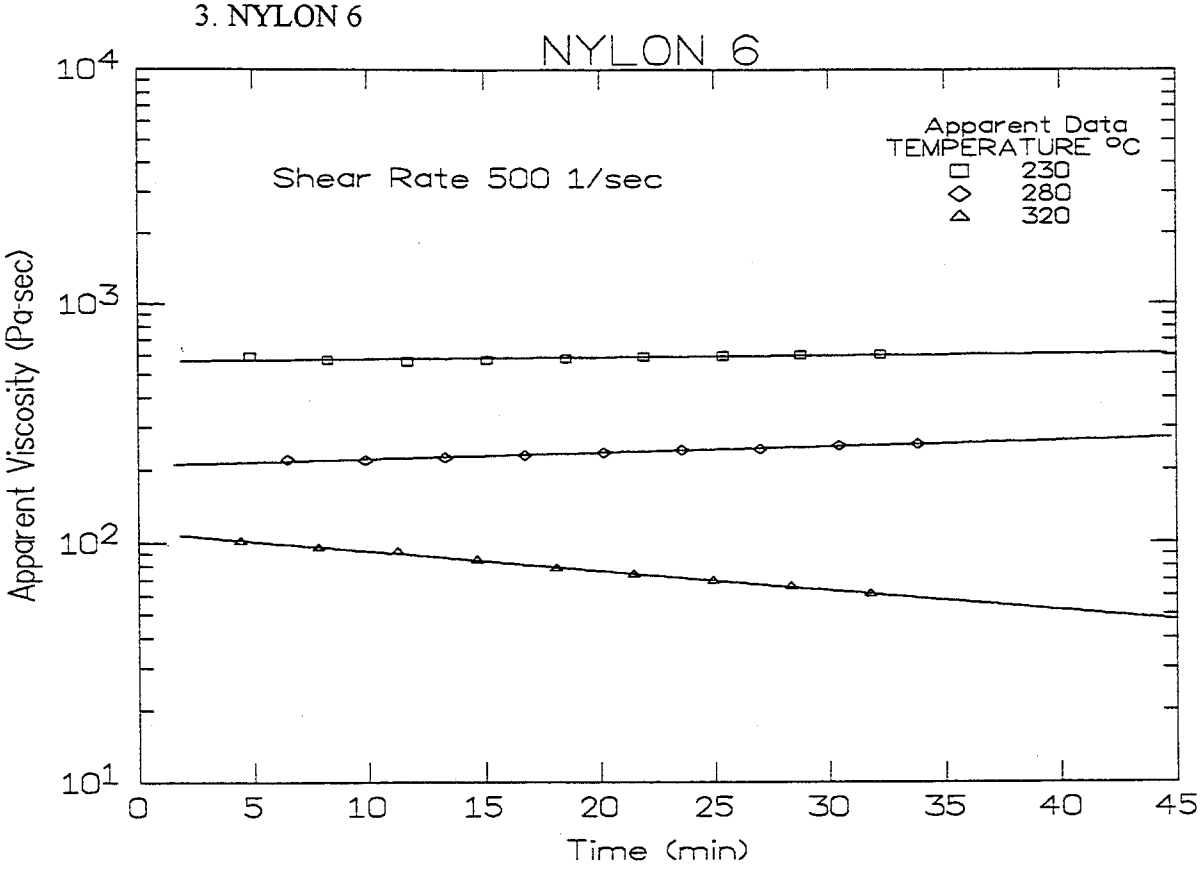
1. PMMA (Multi Temp)



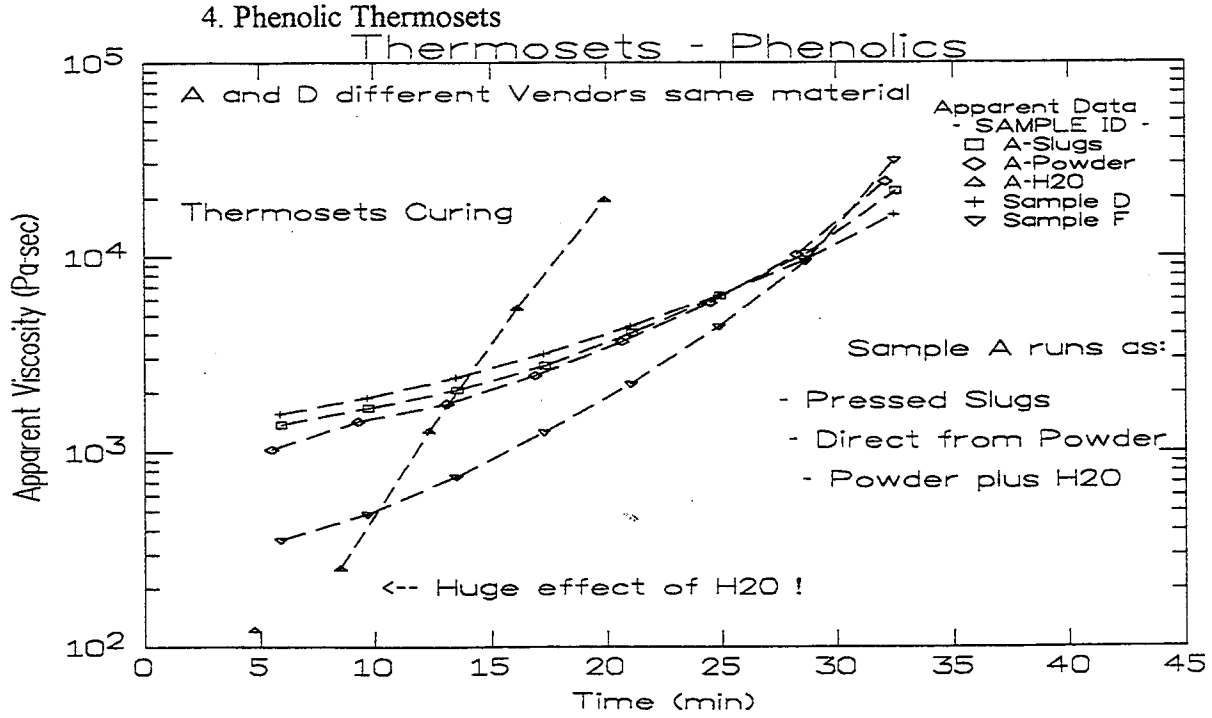
2. ABS


ABS GRAY
Apparent Data
TEMPERATURE °C
200
220
240
Second Order Polynomial Fit
Apparent Viscosity (Pa-sec)
Apparent Shear Rate (1/sec)
10^5
10^4
10^3
10^2
10^0
10^1
10^2
10^3
10^4

3. NYLON 6



4. Phenolic Thermosets



D. Support Vendors

1. Pin Gages

Calibration or Certification of Cylindrical Pins
Zero Check
POB 903
Thomaston, Connecticut 06787
Tel:203-283-5629
FAX: 203-283-4113
Contact: Louise.

Meyer Gage Company
230 Burnham St.
South Windsor, CT 06074
(203) 528-6526
Ask for Class X pins (ISO specs are ± 0.0002 of nominal, ASTM specs (D3835) are ± 0.0003 of nominal)

2. Cleaning Patches
Skyline Center Inc.
POB 3064
Clinton, IA 52732
(319) 243-4065
(800) 747-4065 Extension-4065
FAX (319) 243-9901

3. Bore Gages
Inspex Corp
664 Bussee Hwy
Park Ridge, IL 60068
(708) 825-2200, Fax 825-0825
Order Diameter Probe #029 Probe, N-6 Needle, 0.0001 dial indicator, 8mm holder, #029 ring (0.375"), 0.315" x 10" Depth Extension

4. NIST Standard Reference Materials (SRM)
For example: Standard Material 1476 is a branched polyethylene with (a MFR of 1.19 ± 0.01) as of 1992 cost about $255 for 50 grams.

SRM Catalog number is NIST Special Publication 260
To order: (301) 975-6776 Fax (301) 948-3730

5. Hg spill kits
Mercury Clean Up Spill Kits
Mercon Products: distributed by Fisher Scientific
Unit 8, 7551 Vantage Way

Delta, B.C.
Canada V4G 1C9
Tech Assistance (800)926-8999
(604) 940-0975 or call Fisher Scientific

PRINCO Instruments Inc. (Accepts Hg for Recycle)
1020 Industrial Highway
Southampton, PA 18966
(215) 355-1500

E. ASTM Capillary Rheometry D3835

The following capillary rheometer test method is reprinted , with permission, from the Annual Book of ASTM Standards, copyright American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103

Designation: D 3835 – 93a

An American National Standard

Standard Test Method for Determination of Properties of Polymeric Materials by Means of a Capillary Rheometer[1]

This standard is issued under the fixed designation D 3835; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes measurement of the rheological properties of polymeric materials at various temperatures and shear rates common to processing equipment. It covers measurement of melt viscosity, sensitivity, or stability of melt viscosity with respect to temperature and polymer dwell time in the rheometer, die swell ratio (polymer memory), and shear sensitivity when extruding under constant rate or stress. The techniques described permit the characterization of materials that exhibit both stable and unstable melt viscosity properties.

1.2 This test method has been found useful for quality control tests on both reinforced and unreinforced thermoplastics, cure cycles of thermosetting materials and other polymeric materials having a wide range of melt viscosities.

1.3 The values stated in SI units are to be regarded as standard. The inch-pound units given in parentheses are for information only.

1.4 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

NOTE 1—There is no similar or equivalent ISO standard.

2. Referenced Documents

2.1 *ASTM Standards:*

D 618 Practice for Conditioning Plastics and Electrical Insulating Materials for Testing[2]

D 1238 Test Method for Flow Rates of Thermoplastics by Extrusion Plastometer[2]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[3]

2.2 *ANSI Standard:*

B46.1 Surface Texture[4]

3. Terminology

3.1 *Descriptions of Terms Specific to This Standard:*

3.1.1 *apparent values*—viscosity, shear rate, and shear stress values calculated assuming Newtonian behavior and that all pressure drops occur within the capillary.

3.1.2 *critical shear rate*—the shear rate corresponding to the critical shear stress (1/s).

3.1.3 *critical shear stress*—the value of the shear stress at which there is a discontinuity in the slope of log shear stress versus log shear rate plot or periodic roughness of the polymer strand occurs as it exits the rheometer die (MPa).

3.1.4 *delay time*—the time delay between piston stop and start when multiple data points are acquired from a single charge(s).

3.1.5 *melt time*—the time interval between the completion of polymer charge and beginning of piston travel(s).

3.1.6 *percent extrudate swell*—the percentage change in the extrudate diameter relative to the die diameter.

3.1.7 *shear rate*—rate of shear strain or velocity gradient in the melt, usually expressed as reciprocal time such as second^{-1} (s^{-1}).

3.1.8 *shear stress*—force per area, usually expressed in pascals (Pa).

3.1.9 *swell ratio*—the ratio of the diameter of the extruded strand to the diameter of the capillary (die).

3.1.10 *viscosity*—ratio of shear stress to shear rate at a given shear rate or shear stress. It is usually expressed in pascal seconds (Pa·s).

3.1.10.1 Viscosity determined on molten polymers is sometimes referred to as melt viscosity.

3.1.10.2 Viscosity determined on materials exhibiting non-Newtonian flow behavior is referred to as apparent viscosity unless corrections are made as specified in Section 11.

3.1.11 *zero shear viscosity*, η_0—the limiting viscosity as the shear rate falls to zero.

4. Significance and Use

4.1 This test method is sensitive to polymer molecular weight and molecular weight distribution, polymer stability—both thermal and rheological, shear instability, and additives such as plasticizers, lubricants, moisture reinforcements, or inert fillers, or combination thereof.

4.2 The sensitivity of this test method makes the data useful for correlating with processing conditions and aids in predicting necessary changes in processing conditions. Unlike Test Method D 1238, which makes a one-point measure at a shear rate typically below processing conditions, this test method determines the shear sensitivity and flow characteristics at processing shear rates, and therefore can be used to compare materials of different compositions.

5. Interferences

5.1 Relatively minor changes in the design and arrange-

---

[1] This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.30 on Thermal Properties (Section D20.30.08).

Current edition approved Oct. 15, 1993. Published December 1993. Originally published as D 3835 – 79. Last previous edition D 3835 – 93.

[2] *Annual Book of ASTM Standards*, Vol 08.01.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

ment of the component parts have not been shown to cause differences in results between laboratories. However, it is important for the best interlaboratory agreement that the design adhere closely to the description herein; otherwise, it should be determined that modifications do not influence the results.

5.1.1 *Temperature*—The effect of temperature variation on output rate, Q, or resultant pressure, P, the other variables remaining constant, is given approximately by:

(*A*) For a constant-stress rheometer:

$$\% \text{ error in } Q = \frac{dQ}{Q} \times 100$$

$$= \frac{E^*}{RT^2} dT \times 100$$

(*B*) For a constant-rate rheometer:

$$\% \text{ error in } P = \frac{dP}{P} \times 100$$

$$= \frac{E^*}{RT^2} dT \times 100$$

where:

E^* = an energy of activation,
R = gas constant (8.3 J/K·mol), and
T = absolute temperature, K.

For some thermoplastics $dT = 0.2$ K will produce up to 5 % error in Q or P. Therefore, the temperature control should meet the requirements specified in 6.1.5.

5.1.2 *Force and Output Rate*—The output rate varies approximately as the pressure, P, raised to some power, b, greater than unity. Over a range of output rates, b may not be constant. The effect of pressure variation on output rate, the other variables remaining constant, is given by:

$$\% \text{ error in } Q = \frac{dQ}{Q} \times 100 = b \frac{dP}{P} \times 100$$

Thus a 0.5 % error in pressure measurement implies an error of $b/2$ % in output rate. As the value of b can range from 1 to 3, a corresponding error in Q of 0.5 to 1.5 % could result from this 0.5 % error in P. It is therefore necessary that the precision of the force and output rate measurements be within 1.0 % of the absolute values.

5.1.3 *Capillary Dimensions*—The output rate and force vary with $r^3 + bL - b$, where b is as defined in 5.1.2, r is the capillary radius, and L the length of land. The error that arises in Q due to variations only in r and L is given by:

$$\% \text{ error in } Q = \frac{dQ}{Q} \times 100 = b \frac{dP}{P} \times 100$$

$$= (3 + b)\frac{dr}{r} \times 100 - b \frac{dL}{L} \times 100$$

As the value of b can range from 1 to 3, the resultant error in Q due to a variation in r of ±0.5 % can be 2 to 3 %, and the resultant error in Q due to variation in L of ±0.5 % can be 0.5 to 1.5 %. If Q is being held constant, similar variations in r and L can result in an error of 1.0 to 2.0 % and 0.5 %, respectively, in P.

6. Apparatus

6.1 *Rheometer*—Any capillary rheometer is satisfactory in which molten thermoplastic can be forced from a reservoir through a capillary die and in which temperature, applied force, output rate, and barrel and die dimensions can be controlled and measured accurately as described below. Equipment that operates under constant stress or constant rate has been shown to be equally useful.

6.1.2 *Barrel*—The barrel (Note 1) shall have a smooth, straight bore between 6.35 and 19 mm in diameter. Well(s) for temperature sensor(s) shall be provided as close to the barrel inside wall as possible. The barrel bore should be finished by techniques known to produce approximately 12 rms or better in accordance with American National Standard B46.1.

NOTE 2—Cylinders with Rockwell hardness, C scale, greater than 50 have shown good service life when used at temperatures below 300°C.

6.1.3—The capillary (Note 3) shall have a smooth straight bore that is held to within ±0.00762 mm (±0.0003 in.) in diameter and shall be held to within ±0.025 mm (±0.001 in.) in length. The bore and its finish are critical. It shall have no visible drill or other tool marks and no detectable eccentricity. The capillary bore shall be finished by techniques known to produce about 12 rms or better when measured in accordance with American National Standard B46.1. Dies having a flat (180°) inlet angle and die length to diameter ratios greater than or equal to 20 are recommended. Other inlet angles may be used, but comparisons should be made using only dies with identical inlet cones. The inlet cone shall expand from the capillary at fixed angle to a diameter no less than 50 % of the barrel diameter.

NOTE 3—Hardened steel, tungsten carbide, Stellite, and Hastelloy are the most generally used capillary materials. The capillary shall have a diameter such that the ratio of barrel diameter, D, to capillary diameter, d, is normally between 3 and 15. The length-to-diameter ratio of the capillary shall normally be between 15 and 40. Smaller ratios of L/D may be used in selected situations, but are more likely to result in the necessity of applying large corrections to the data (1, 2).[5]

6.1.3.1 The precision with which capillary dimensions can be measured is dependent upon both the capillary radius and length. With capillaries of diameter smaller than 1.25 mm (0.050 in.) the specified precision is difficult. Due to the extreme sensitivity of flow data to capillary dimensions, it is most important that both the capillary dimensions and the precision with which the dimensions are measured are known and reported.

6.1.4 *Piston*—The piston shall be made of metal of a hardness of Rockwell hardness, C scale, of greater than 45. The land of the piston shall be 0.0254 ± 0.007 mm (0.0010 ± 0.0003 in.) smaller in diameter than the barrel and at least 6.35 ± 0.13 mm (0.250 ± 0.005 in.) in length. Alternative piston-barrel-sealing methods (o-rings, split seals, multilands, etc.) outside these tolerances may be used, provided

[5] The boldface numbers in parentheses refer to the list of references at the end of this test method.

there is less than 0.1 g of material going past the sealing device. Machines that measure plunger force must demonstrate that piston-tip frictional effects are less than 1 % over the range of force measurement, or correct for this effect. Demonstration of low frictional force is not required for pressure-measurement devices; however, adequate seals are still needed for proper flow-rate calculations. Above the land, the piston shall be relieved at least 0.25 mm (0.010 in.) less than the barrel diameter. The finish of the piston foot shall be 12 rms when measured in accordance with American National Standard B46.1.

6.1.5 Make provisions for heating and temperature control systems such that the apparatus maintains the temperature of a fluid, at rest, in the barrel to within ± 0.2°C of the set temperature (see Note 4). Due to shear heating and chemical or physical changes in the material, it may not be possible to hold this degree of control during an actual test. In such a case, the temperature shall be reported with each data point collected. The temperature specified shall be the temperature of the material 6 min after a full charging of the barrel measured in the center of the barrel 12.7 mm above the top of the die.

NOTE 4—A high melt-flow-rate polypropylene >20 (g/10 min) has been found useful for calibrations of control probes.

6.1.6 The temperature sensing device in the apparatus shall be calibrated by the following method. A traceable temperature sensor shall be inserted into the rheometer barrel containing a typical charge of material (see Note 5). The combined accuracy of the sensor and display unit shall be 0.1°C or better. The reference unit shall display temperature to 0.1°C or better. The sensor shall be positioned such that it acquires the average temperature centered vertically at 12.7 mm above the top of the die and centered radially within the barrel. For large sensor (for example, large bulb thermometers) elements provisions shall be made to avoid direct contact of the sensing element with the die or barrel wall. Proper insulation or immersion levels, or both, should be adhered to, as required, for sufficient accuracy. Charging the barrel with typical material can be omitted if it has been demonstrated that for the sensor in question the steady-state temperature in air results are statistically equivalent (95 % confidence limits) to the standard charge temperature results. The controlling point temperature device should be calibrated to within ±0.1°C of the reference temperature sensor after steady-state temperature has been achieved. Subsequent temperature checks of the controlling temperature probe should not exceed ±0.2°C of the reference probe temperature.

NOTE 5—Any type of temperature sensor (thermometer, RTD, optic probe, etc.) is allowed under 6.1.6 provided it is traceable and falls within the element size restriction and positioning requirements.

7. Test Specimen

7.1 The test specimen may be in any form that can be introduced into the bore of the cylinder such as powder, beads, pellets, strips of film, or molded slugs. In some cases it may be desirable to preform or pelletize a powder. In the case of preformed plugs, any application of heat to the sample must be kept to a minimum and shall be held constant for all specimens thus formed.

8. Conditioning

8.1 Many thermoplastic materials do not require conditioning prior to testing. Materials that contain volatile components, are chemically reactive, or have other unique characteristics are most likely to require special conditioning procedures. In many cases, moisture accelerates degradation or may otherwise affect reproducibility of flow-rate measurements. If conditioning is necessary, see the applicable material specification and Practice D 618.

9. Procedural Conditions

9.1 Typical test temperature conditions of several materials are given below. These are listed for information only. The most useful data are generally obtained at temperatures consistent with processing experience. The shear stress and shear rate conditions applied should also closely approximate those observed in the actual processing.

	Typical Test Temperature, °C
Acetals	190
Acrylics	230
Acrylonitrile-butadiene-styrene	200
Cellulose esters	190
Nylon	235 to 275
Polychlorotrifluoroethylene	265
Polyethylene	190
Polycarbonate	300
Polypropylene	230
Polystyrene	190 to 230
Poly(vinyl chloride)	170 to 205
Poly(butylene terephthalate)	250
Thermoplastic Elastomer (TES) Unsaturated	150 to 210
Thermoplastic Elastomer (TES) Saturated	180 to 260

10. Procedure

10.1 Select test temperature shear rates and shear stress in accordance with materials specifications (see the ASTM document for the specific material) and within the limitations of the testing equipment.

10.2 Before beginning determinations, inspect the rheometer and clean it if necessary, as described in 10.11 (see Note 6). Ensure that cleaning procedures or previous use have not changed the dimensions. Make frequent checks to determine the die diameter and to ensure that it is within the tolerances given in 6.1.3. A go/no-go pin with the smallest pin (green) being the low end of the specification (for example, 0.99238 mm for a nominal 1-mm diameter die) and the largest pin (red) being the largest end of the specification (for example, 1.00762 mm for a nominal 1.0-mm diameter die) is effective for checking die diameter. The go (green) pin should go effortlessly all the way into the die from both ends. The no-go (red) pin should not enter more than 1 mm in either end of the die. All errors in pin production should be in the direction of making the specification tighter.

NOTE 6—Experience has shown that an initial purge of the rheometer with the test material is often good practice after periods of equipment inactivity and when changing material types. Purging is also effective at reducing the variability of unstable materials (PVC); it is important, however, that both the barrel and die be cleaned after the purge prior to running the sample.

10.3 Replace the die and piston in the barrel and allow the assembled apparatus to reach thermal equilibrium.

10.4 Remove the piston, place on an insulated surface, and charge the barrel with the sample until the barrel is filled to within approximately 12.5 mm (0.5 in.) of the top. Manually tamp the charge several times during the loading to minimize air pockets. Charging should be accomplished in not more than 2 min.

10.5 Place the piston in the barrel, start the melt time timer, and immediately apply a load that imparts a constant stress on the polymer, or start the piston moving at a constant rate. Extrude, at least, a small portion of the barrel charge. Stop the piston movement until the full melt time has expired.

NOTE 7—There may be cases where 6 min of preheat time may not be sufficient or desirable. Longer preheat periods are permissible and often useful, as are shorter preheat times when proved to be sufficient or necessary due to thermal degradation.

NOTE 8—Running first rates that correspond to forces that exceed the nominal packing force used to charge the sample often results in lower operator-to-operator variability on subsequent rates that correspond to forces lower than the packing force. Additionally, running from higher to lower rates (or stress) tends to reduce the time necessary to achieve steady-state.

10.6 Reactivate the piston to start extrusion. After the system has reached steady-state operation, record the force on the piston and the data necessary to calculate the output rate, Q. The criterion used for steady-state determination should be reported with the data.

10.7 If the specific material being tested has previously been demonstrated thermally stable at the current test temperature, any combination of shear rates or shear stress may be applied, provided data is taken under steady-state conditions.

10.8 If the rheological thermal stability of the material has not been determined, one must perform either of the following:

(*1*) Run a constant rate test (or a constant shear stress test in the Newtonian region) with sufficient delay time to cover the expected time for the subsequent multi-point shear rate or shear stress run and collect a minimum of four data points. If the viscosity of the material changes by more than 0.5 % (higher or lower) per minute at any point along the viscosity-time curve, the material is considered thermally unstable rheologically from that point on. Subsequent tests must be performed before this time is reached. If tests must be performed at times exceeding the thermal stability time limit, they must be made at constant time. This requires a new sample to be charged for each rate or stress point collected.

(*2*) Run a multiple rate or multiple stress level test, or both, in a manner that both rate effects and time effects can be estimated within the same run. The minimum requirements for such a test would be that, at least, one condition (rate or stress) must be repeated and the time difference between them be equal to, at least, half the total test time. Should a 0.5 % change or greater be observed in the viscosity per minute, the rate data should be considered confounded with the time dependence and so noted. The user may then wish to revert back to the previous method to explore the nature of the thermal instability.

10.9 If the percent extrudate swell is desired, measure the extrudate diameter using any NIST traceable device capable of measuring diameters to within ± 0.5 %. If measured after cutting a piece of extrudate away from the die, measure the diameter 6.25 mm away from the die exit.

10.9.1 Scanning devices measuring extrudate diameter during a test that are operating at ambient temperature should have the measurement being made 25 mm away from the die exit. At least 8 independent samplings should be used to report an average extrudate diameter. The associated real time shear viscosity data should be collected within 2 s of the real time extrudate measurement. At extrudate exit speeds of less than approximately 200 mm/min, the extrudate should be cut such that its total length is approximately 50 mm at the time of measurement.

10.10 Discharge the remainder of the specimen and remove the capillary from the barrel. Clean the piston and capillary thoroughly and swab out the barrel with cotton cloth patches or a brush softer than the barrel, in the manner of cleaning a pistol barrel. The capillary may be cleaned by dissolving the residue in a solvent. The method of pyrolytic decomposition of the residue in a nitrogen atmosphere is useful only on capillaries made from materials that will not themselves be softened or oxidized by the pyrolysis operation. Place the die in a tubular combustion furnace or other device for heating to 550 ± 10°C and clean with a small nitrogen purge through the die. In certain cases where materials of a given class having similar flow characteristics are being tested consecutively, interim capillary cleaning may not be required. In such cases, however, the effect of cleaning upon viscosity determinations must be shown to be negligible.

11. Errors and Corrections (See Refs (4) through (9))

11.1 In some cases it is necessary to have more exacting rheological data from capillary rheometry measurements. In this event, data may be reported in different terms than given in Section 3. For example, true shear rates, corrected for non-Newtonian flow behavior and true shear stresses, corrected for end effects or kinetic energy losses, may be calculated. In such cases, the exact details of the mode of correction must be reported. The application of these corrections is discussed in the references at the end of this test method.

11.2 *Capillary Calibration*—No completely satisfactory method for determining capillary inside diameter has yet been developed. Since apparent viscosity varies with the fourth power of r, it is desirable to know this value within ±0.00762 mm (0.0003 in.).

11.3 *Piston Friction*—This is caused by contact of the piston with the barrel. Normally the frictional force is negligible compared to the pressure drop through the capillary. When significant, the frictional force should be subtracted from the force reading.

11.4 *Polymer Back Flow*—The clearance between the plunger and the barrel may permit a small amount of melt to flow back along the piston instead of through the capillary. This causes the real shear rate to be lower than that calculated from the piston velocity. Usually this error is negligible. However, in some cases, particularly when slow piston speeds are run at high loads, a back-flow correction may be necessary. This is evidenced by material exuding past the top of the land on the piston. This material should be scraped from the plunger, weighed, and compared to the

weight of the capillary extrudate for the same time period to determine the percent back-flow error. A second method for determining the magnitude of this error consists in measuring the rate of capillary extrudate and comparing this with the actual piston displacement rate, taking into account the change in fluid density.

11.5 *Melt Compressibility*—Some fluids are compressible to a significant degree. As shear rate at the capillary wall is calculated from the piston displacement rate, an error is introduced by the drop in hydrostatic pressure (and in fluid density) along the capillary. As the hydrostatic pressure diminishes along the capillary, the fluid density decreases and the flow rate increases. This results in an increase in shear rate down the capillary. If the compressibility or the equation of state for the material under study is known, this correction can easily be made; for example, using a published equation of state for polystyrene (3), a compressibility correction chart can be made for this material.

11.6 *Barrel Pressure Drop*—It is assumed in most work that the pressure drop in the rheometer barrel is negligible compared to the pressure drop through the capillary. This is not true for short capillaries of large diameter. Under isothermal conditions, the pressure drop of Newtonian materials varies as

$$\frac{\Delta P_1}{\Delta P_2} = \left(\frac{L_B}{L_C}\right)\left(\frac{R}{r}\right)^4$$

where L_B refers to the rheometer barrel length and L_C to the capillary length. When the pressure drop in the barrel is significant, it should be subtracted from the overall pressure drop of the system in order to calculate shear stress.

11.7 *Determining True Shear Stress*—The correction method according to Bagley will be used to calculate true stress. To obtain the true shear stress, perform the following procedure: Using a minimum of two dies (although preferably three or more) having the same entrance angle and same diameter (D) yet of differing capillary lengths (L), collect steady-state flow data on shear rate and test pressure (or plunger force). At least one L/D ratio should be less than 10, and at least one should be greater than 16. Prepare a plot of pressure (or plunger force) versus the length to diameter (L/D) ratio of the dies used. For points at constant apparent shear rate, draw the best straight line through the data and determine the intercept with the pressure axis (P_c) or force axis (F_c). Obtain true shear stress using the following equation:

$$\tau = \frac{(P - P_c)D}{4L} = \frac{(F - F_c)D}{4L\,A_B}$$

where:

τ = the true shear stress,
P = the melt pressure,
P_c = the intercept obtained for a given shear rate from the above described plot (see Fig. 1),
D = the die diameter, and
L = the die length.

For plunger force measuring devices, F is the force on the plunger, F_c is the intercept force on the Bagley plot described above, and A_B is the cross sectional area of the barrel. Devices that measure plunger force must acquire data for a given shear rate (a given line on the graph) at the same position in the barrel for the various dies used. In this way barrel pressure drop effects will be removed along with the other stationary pressures in the system when the Bagley correction is performed.

NOTE 9—When using very long dies, there may be non-linear changes in the pressure versus L/D plots due to the effects of pressure on viscosity or viscous heating. In such cases use only the data from shorter capillaries which do not exhibit the effect.

NOTE 10—The Bagley correction may be performed using computer programs. If it is performed in such a manner, inherent in the computer program will be code assessing the validity of the assumption of having straight lines in the Bagley plot. Users will be warned that the Bagley

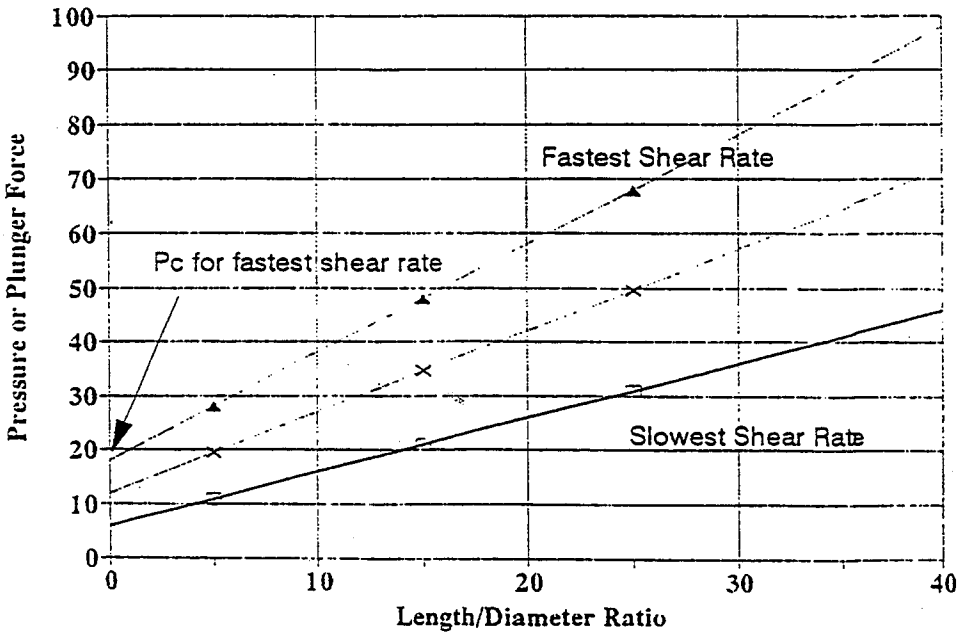


FIG. 1 Bagley Correction

correction is not valid under such circumstances where the straight line conditions are not met.

11.8 *Determining True Shear Rate*—The Weissenberg Rabinowitsch shear rate correction accounts for the fact that the true shear rate is often larger than the apparent shear rate for non-Newtonian materials. The true shear rate can be calculated using the following equation:

$$\dot{\gamma} = \frac{(3n + 1)}{4n}\dot{\gamma}_a$$

where:

n = the tangent slope of the log true shear stress versus log apparent shear rate curve at the apparent shear rate being corrected,
$\dot{\gamma}$ = the true shear rate, and
$\dot{\gamma}_a$ = the apparent shear rate described under 12.1 (see Fig. 2).

The stationary pressure correction (Bagley entrance correction) should always be performed prior to the Rabinowitsch correction.

12. Calculation

12.1 Perform calculations using the following equations:

$$\text{Shear stress, Pa} = \frac{Pr}{2L} = \frac{Fr}{2\pi R^2 L}$$

$$\text{Shear rate, s}^{-1} = \frac{4Q}{\pi r^3} = \frac{4V}{\pi r^3 t}$$

$$\text{Viscosity, Pa·s} = \frac{p\pi r^4}{8LQ} = \frac{Fr^4 t}{8R^2 LV}$$

where:

P = pressure by ram, Pa,
F = force on ram, N,
r = radius of capillary, m,
R = radius of barrel, m,
L = length of capillary, m,
Q = flow rate, m^3/s,
V = volume extruded, m^3, and
t = extrusion time, s.

12.1.1 The equations given in 12.1 yield true shear rate and true viscosity for Newtonian fluids only; for non-Newtonian fluids, the apparent shear rate and viscosity are obtained. (See Section 11.)

12.2 Calculate swell ratio and percent memory as follows:

$$\text{swell ratio} = \frac{\text{strand diameter}}{\text{capillary diameter}}$$

$$\%\ \text{extrudate swell} = \frac{\text{strand diameter} - \text{capillary diameter}}{\text{capillary diameter}} \times 100$$

13. Report

13.1 Report the following information:

13.1.1 *Information Other Than Flow Data:*

13.1.1.1 Description of the material being tested,

13.1.1.2 Description of the rheometer used,

13.1.1.3 Temperature at which the data were obtained and the precision of the temperature measurement (°C),

13.1.1.4 Diameter, d, and the length to diameter ratio, L/d, of the straight section, and the precision of these measurements (mm),

13.1.1.5 Die-entry-cone maximum diameter and angle,

13.1.1.6 Statement as to any preconditioning which the sample has undergone, and

13.1.1.7 Melt time and dwell times (s).

13.2 *Flow Data*—These data should be reported in tabular or graphical form, stating either "apparent values," "Rabinowitsch corrected," "Bagley corrected," or "Rabinowitsch and Bagley corrected." If no die-wall slippage was assumed in the Rabinowitsch correction, it should be noted. Corrections of other types should be noted if greater than 1 %.

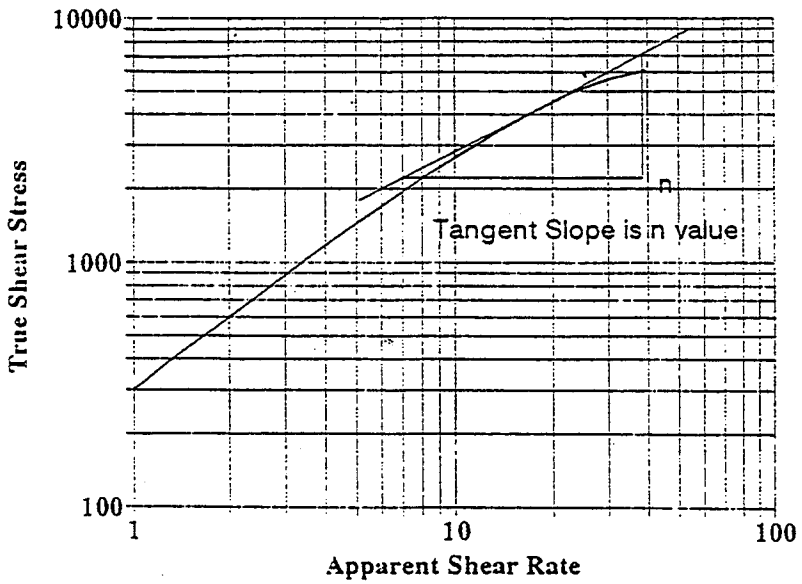
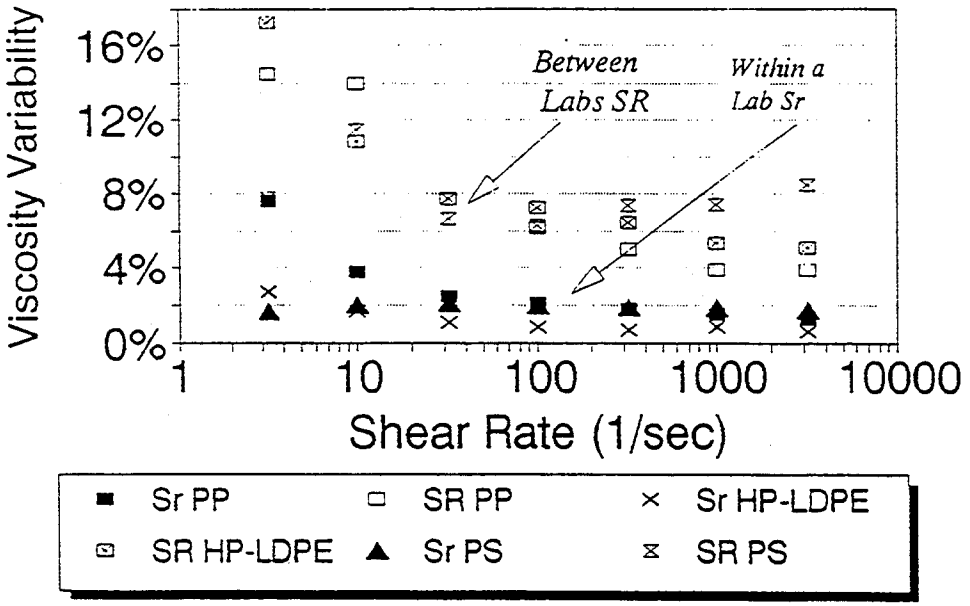


FIG. 2 Weissenberg Rabinowitsch Correction for True Shear Rate

TABLE 1 Summary of Round Robin for Test Method D 3835 Conducted in 1992

			Points Used		Points Used		Points Used		Points Used		Points Used		Points Used		Points Used		Points Used
PP	Set Rate, 1/s	3162	...	1000	...	316	...	100	...	32	...	10	...	3.2	...	100	...
	Stress, kPa	150	...	103	...	70	...	44	...	24	...	12	...	5	...	43	...
	Viscosity, Pa·s	47.3	...	103.2	...	222.3	...	440.5	...	734.5	...	1249.2	...	1544.9	...	434.9	...
	S_R, Pa·s[A]	1.8	13	4.0	13	11.2	12	27.3	13	56.5	13	174.3	12	223.1	7	30.7	10
	S_r, Pa·s[B]	0.6	36	1.6	36	4.0	33	9.4	36	18.3	36	47.3	33	117.0	18	8.5	30
	S_r/average, %	1.28	...	1.52	...	1.81	...	2.13	...	2.49	...	3.79	...	7.57	...	1.95	...
	S_R/average, %	3.89	...	3.91	...	5.05	...	6.20	...	7.69	...	13.95	...	14.44	...	7.05	...
HP-LDPE	Set Rate, 1/s	3162	...	1000	...	316	...	100	...	32	...	10	...	3.2	...	100	...
	Stress, kPa	320	...	214	...	139	...	83	...	48	...	28	...	15	...	82	...
	Viscosity, Pa·s	101.2	...	213.7	...	439.3	...	834.0	...	1509.1	...	2837.3	...	4651.6	...	817.9	...
	S_R, Pa·s[A]	5.2	13	11.4	13	28.3	12	60.3	13	116.3	13	307.7	12	802.5	8	46.4	10
	S_r, Pa·s[B]	0.7	36	1.8	36	2.9	33	7.2	36	16.5	36	48.8	33	125.7	21	5.1	30
	S_r/average, %	0.65	...	0.84	...	0.66	...	0.86	...	1.09	...	1.72	...	2.70	...	0.63	...
	S_R/average, %	5.10	...	5.32	...	6.45	...	7.23	...	7.71	...	10.85	...	17.25	...	5.67	...
PS	Set Rate, 1/s	3162	...	1000	...	316	...	100	...	32	...	10	...	3.2	...	100	...
	Stress, kPa	299	...	220	...	163	...	121	...	84	...	59	...	37	...	118	...
	Viscosity, Pa·s	94.5	...	220.3	13	516.9	...	1207.6	...	2638.0	...	5928.4	...	11 696.5	...	1179.0	...
	S_R, Pa·s[A]	8.0	13	16.4	35	38.0	12	76.0	13	175.7	13	679.7	12	907.3	8	76.1	10
	S_r, Pa·s[B]	1.6	35	4.2	...	9.9	32	23.9	35	55.6	35	122.7	32	197.6	21	18.4	29
	S_r/average, %	1.74	...	1.90	...	1.92	...	1.98	...	2.11	...	2.07	...	1.69	...	1.56	...
	S_R/average, %	8.51	...	7.43	...	7.35	...	6.30	...	6.66	...	11.47	...	7.76	...	6.45	...

[A] S_R = between laboratory standard deviation.
[B] S_r = within-laboratory standard deviation (pooled estimate).



NOTE—By material, within-laboratory and laboratory-to-laboratory.

FIG. 3 Variability versus Shear Rate

13.2.1 Log shear stress versus log shear rate,

13.2.2 Log viscosity versus log shear stress or log shear rate,

13.2.3 Log viscosity versus the reciprocal of the absolute temperature at a constant shear stress or shear rate,

13.2.4 Log viscosity versus the temperature in degrees Celsius at a constant shear stress or shear rate,

13.2.5 Log critical shear stress or log critical shear rate versus the reciprocal of the absolute temperature, and

13.2.6 Log critical shear stress or log critical shear rate versus the temperature in degrees Celsius.

13.3 Individual data obtained at a single set of test conditions should include the following information:

13.3.1 Shear stress, τ, Pa,

13.3.2 Shear rate, $\dot{\gamma}$, s^{-1},

13.3.3 Intrinsic melt viscosity (see Appendix), η_a, Pa·s,

13.3.4 Melt viscosity stability, δ (%/min),

13.3.5 Percent extrudate swell or swell ratio.

13.4 *Visual Observation*—In cases where observation is possible, gloss character or melt fracture and distortion of the monofilament may be noted at or above a certain shear stress. These values may correspond to a critical shear stress. The data shall be reported separately as "visual" critical shear stress. In addition, the general color of the extrudate at the conditions of test or the dwell time at which a distinct color change occurs, or both, can be noted.

14. Precision and Bias[6]

14.1 *Precision:*

14.1.1 Figure 3 and Table 1 are based on a round robin conducted in 1992 in accordance with Practice E 691, involving materials tested by 13 laboratories. Three materials were used in the round robin: polypropylene copolymer, polystyrene, and low density polyethylene. Each material was prepared by a single source and underwent no additional conditioning (drying, etc.) prior to testing. The number of measurements made by a given laboratory is noted in the tables. Typically each laboratory ran three tests per material. It should be noted that the full scale capacity of the pressure transducer or load cell and the proper die selection can significantly affect the ability to measure at low rates (low stresses).

NOTE 11—The following explanations of r and R are intended to present a meaningful way of considering the approximate precision of this test method. The data in Table 1 should not be rigorously applied to the acceptance or rejection of material, as those data are specific to the round robin and may not be representative of other lots, conditions, materials, or laboratories. Users of this test method should apply the principles outlined in Practice E 691 to generate data specific to their laboratory and materials or between specific laboratories.

14.1.2 *Concept of r and R*—If S_r and S_R have been calculated from a large enough body of data, and the test result of interest is that obtained from a single viscosity versus rate sweep, then the following applies:

14.1.2.1 *Repeatability (r)*—Comparing two results for the same material, obtained by the same operator using the same equipment on the same day, the two test results should be judged not equivalent if they differ by more than the r value, where $r = 2.8\ S_r$.

14.1.2.2 *Reproducibility (R)*—Comparing two results for the same material, obtained by different operators using different equipment on different days, the two test results should be judged not equivalent if they differ by more than the R value, where $R = 2.8\ S_R$.

14.2 Any judgment pertaining to the repeatability or reproducibility would have an approximate 95 % (0.95) probability of being correct.

14.3 *Bias*—There are no recognized standards by which to estimate the bias of this test method.

15. Keywords

15.1 capillary; plastics; polymers, rheology; thermal flow; viscosity

[6] Supporting data giving results of the interlaboratory tests have been filed at ASTM Headquarters. Request RR: D-20-1076.

REFERENCES

(1) Tordella, J. P., *Transactions of the Society of Rheology*, Vol 1, p. 203, 1957.

(2) Philippoff, W., and Gaskins, F. H., *Transactions of the Society of Rheology*, Vol 2, p. 263, 1958.

(3) Wellman, R. E., deWitt, R., and Ellis, R. B., *Journal of Chemical Physics*, Vol 44, p. 3070, 1966.

(4) Rabinowitsch, R., *Zeitschrift Fuer Physikalische Chemie*, Vol A145, p. 1, 1929.

(5) Bagley, E. B., and Birks, A. M., *Journal of Applied Physics*, Vol 31, p. 556, 1960.

(6) Poiseuille, J. L. M., *C.r. Held. Acad. Sci. Seance Paris*, Vol 11, p. 961, 1840.

(7) Landel, C. E., Moser, B. G., and Bauman, A. J., *Proceedings of the Fourth International Congress of Rheology*, Vol 2, p. 663, 1965.

(8) Tordella, J. P., "Unstable Flow of Molten Polymers," *Rheology*, Vol 5, edited by R. R. Eirich, Academic Press, New York, N.Y., 1969.

(9) Metzer, A. P., and Knox, J. R., *Transactions of the Society of Rheology*, Vol 9, No. 1, p. 13, 1965.

APPENDIX

(Nonmandatory Information)

X1. PROCEDURE FOR DETERMINATION OF INTRINSIC MELT VISCOSITY AND MELT FLOW STABILITY

X1.1 Measure the melt viscosity at constant conditions after at least four dwell times in the barrel.

X1.2 Plot the four or more melt viscosity values on semilogarithmic paper with viscosity plotted on the log scale and dwell time on the linear scale (see Figs. X1.1 and X1.2). In most cases these data will fall on a straight line. A single data point that does not fall on the line drawn through the other data points can be attributed to polymer heterogeneity or test techniques and can be discarded.

X1.3 Draw a straight line through the data and extrapolate to the *y* axis (corresponding to dwell time =v 0). The melt viscosity value thus defined by the intercept of the data line should be recorded as *intrinsic melt viscosity*. This parameter has been found to correlate with polymer molecular weight average, as defined by solution techniques for linear polymers.

X1.4 Calculate the slope of the best fit line to obtain the rate of change of the viscosity as a function of time at a specified temperature. This rate shall be called the *melt viscosity stability*, *S*, of the material at the conditions of test (Notes X1.1 and X1.2).

NOTE X1.1—The total dwell times for viscosity measurement should be selected according to the stability of the material. A highly unstable material can be accurately characterized for its stability factor in relatively short times (for example, 10 min). A material exhibiting small changes in viscosity may require 20 to 30 min dwell times to accurately define the rate of viscosity change.

NOTE X1.2—In the case of materials such as PVC, the material often exhibits stable flow for an initial period of time until the stabilizer becomes ineffective and unstable flow commences. In cases such as this, the dwell time at which unstable flow initiates can be determined and the effectiveness of the stabilizer can thus be defined.

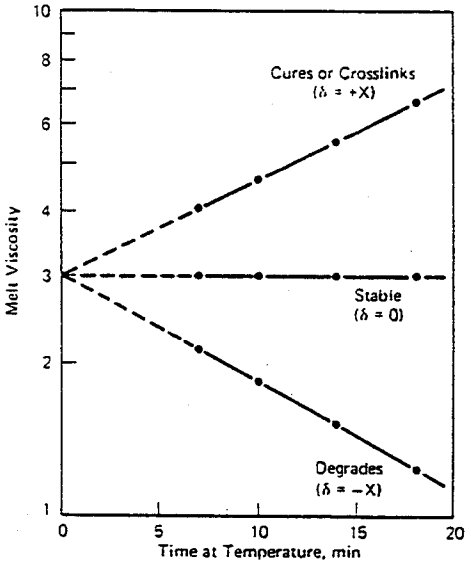


FIG. X1.1 Determination of Intrinsic Melt Viscosity and Stability Factor, δ

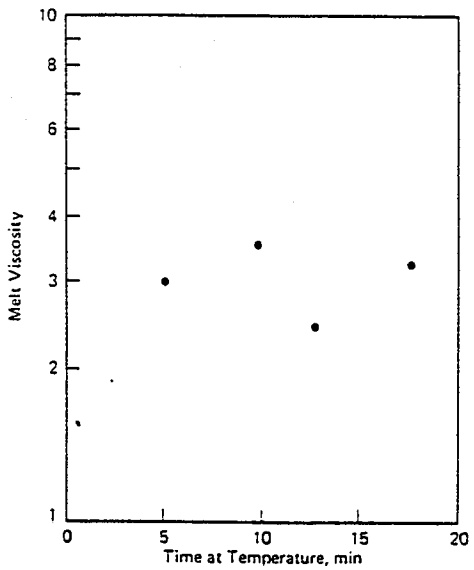


FIG. X1.2 Example of Flow Data Obtained on Heterogeneous Material

F. ISO Capillary Rheometry ISO/TC 61 SC 5

Not available at print time. Drafts have been issued.

G. How to mimic a MCR (ACR)

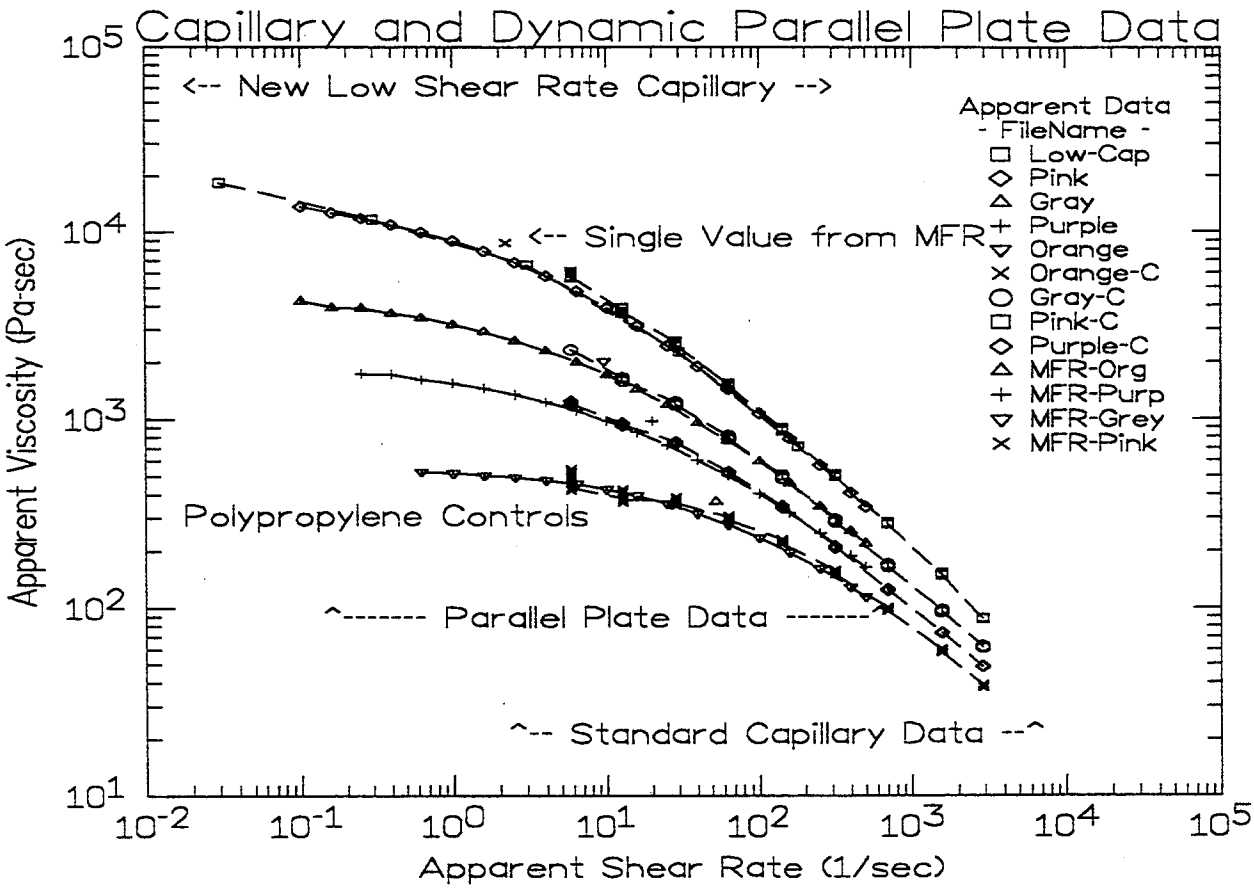
The Monsanto capillary rheometer is similar to the Kayeness instrument in many respects. To perform tests in a manner similar to the MCR machine the user must be under stress control mode and set positions that are similar to the ones used on the MCR.

Since MCR uses English units here is and English example
Constant Stress rheometer *PROGRAM*
Note press **EDIT** on the front panel of the rheometer to start the process

Machine Responses	PRESS THESE KEYS	Comments
Melt Time = XXX.X	NO 222 YES	XXX X is any number value given in seconds
MATL ID= ????????	ASK	NO clears YES accepts final word
TEMP.= XXX.X	NO 190 YES	NO clears, 190 °C entered, YES accepts value
ORIF. RAD.=.XXXX	NO 0125 YES	assumes your capillary die is 0.0125 Radius
ORIF. LEN.=X.XXX	NO 0625 YES	assumes capillary die length is 0.625 inch
SAMPL LEN=XX.XXXX	NO 075 YES	Set SAMPLE LEN to 0.75 inch
FORCE *#1 = XX.XXX*	NO 0100 YES	Set Load to 100 lb.
FORCE *#2= XX.XXX*	NO 0100 YES	Set Load to 100 lb.
FORCE *#3= XX.XXX*	NO 0100 YES	Set Load to 100 lb.
FORCE *#4= XX.XXX*	NO 0100 YES	Set Load to 100 lb.
FORCE *#5= XX.XXX*	NO YES	0.0 load means no more loads
MELT FORCE=XXX.X	NO 150 YES	150 LB force to pack
TERM FORCE=XXX.X	NO 750 YES	750 Lb. safety overload
START POS.=X.XXX	NO 4500 YES	Begin test at 4.5 "
POS. #1 = X.XXX	NO 475 YES	Speed acquire at 4.75-5.5 position
POS. #2 = X.XXX	NO 575 YES	Speed acquired at 5.75-6.5 position
POS. #3 = X.XXX	NO 675 YES	Speed acquired at 6.75-7.5 position
POS. #4 = X.XXX	NO 775 YES	Speed acquired at 7.75-8.5 position
PARK POS.=X.XXX	NO 20 YES	Crosshead Park 2.0 inch position
TEST DELAY=XXXX	NO 280 YES	200 sec Pause between pts
S.I., Pascal	YES	Accept S.I. units
RUN# START	NO YES	Clears RUN# to zero
PROGRAM ENTERED		

H. Comparison with Parallel Plate Results

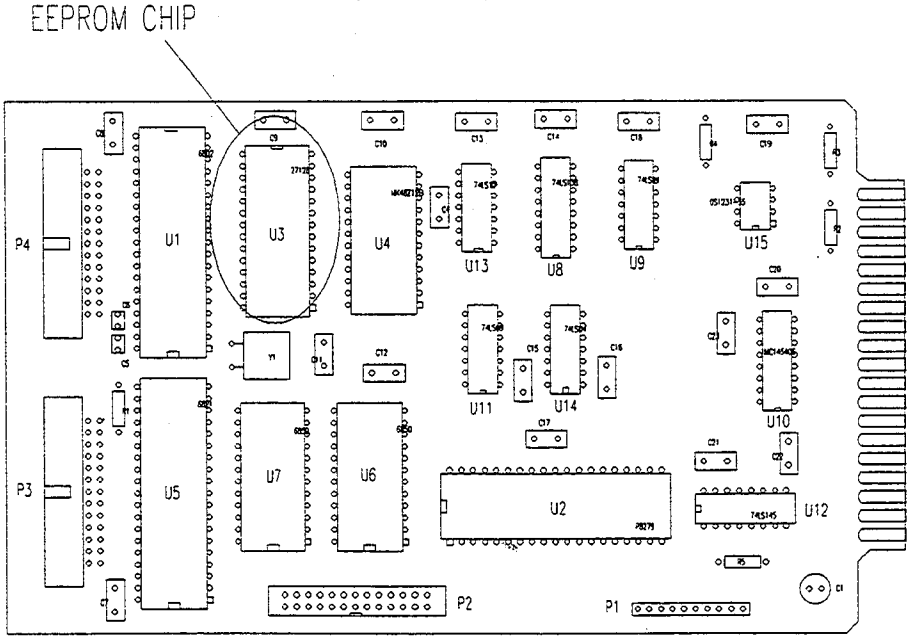
The graph below shows multiple runs on standard Kayeness capillary rheometers and a special low shear unit designed to run at very slow speeds (with fairly long dies) and a Rheometrics RDA® 700 parallel plate rheometer. The capillary data is uncorrected. The materials are a series of polypropylene copolymers produced by HIMONT Inc. which range in melt flow rate at 230 °C (standard conditions) from 21.1 to 0.8 g/10 minutes. The single viscosity value and associated shear rate for the melt flow measurements are also shown on the graph.


Capillary and Dynamic Parallel Plate Data
<-- New Low Shear Rate Capillary -->
<-- Single Value from MFR
Polypropylene Controls
^------- Parallel Plate Data -------
^-- Standard Capillary Data --^
Apparent Data
- FileName -
Low-Cap
Pink
Gray
Purple
Orange
Orange-C
Gray-C
Pink-C
Purple-C
MFR-Org
MFR-Purp
MFR-Grey
MFR-Pink
Apparent Viscosity (Pa-sec)
Apparent Shear Rate (1/sec)
10^5
10^4
10^3
10^2
10^1
10^-2
10^-1
10^0
10^1
10^2
10^3
10^4
10^5

I. Installing a new EPROM (firmware)

The programs that control the rheometer are contained on an Erasable Programmable Read Only Memory (EPROM) computer chip. To install a new EPROM perform the firmware update in the exact order listed.
Note: The static electricity in your body can and will damage many of the electronic components in the rheometer. The earth ground provided by the power cord will drain static back to earth ground. To eliminate any possible shock hazard we will have you touch the vertical stainless pole to drain the static from your person and then unplug power cord from the rear of the instrument. It is important that you do not walk around the room or building looking for tools as this is the best way to build up static in your body. On a dry day or low humidity environment with the right shoes and floor you can build up several thousand volts. The current is minimal which is why it does not harm you but the high voltage will destroy the sensitive electronic circuits. The EPROM is also easily damaged by static, do not remove it from it's protective foam or container until ready to install.

a. Grasp upright poles to remove static from you
b. Unplug power cord from rear of instrument
c. Remove large front stainless cover, remove lexan shield under stainless cover
d. Above keyboard to right of center remove single screw to allow keyboard/display panel to hinge forward, towards you
e. There may be an aluminum shipping restraint located at the lower left edge of the printed circuit boards, remove and save.
f. Locate (main computer bd) printed circuit board closest to you,



The position of the EPROM chip as shown above. The third item from the top left labeled U3 (they are ceramic chips and will normally have a white

tag or label denoting version or model) is the chip to be replaced. Note the notch which lies on the top edge of the chip. It is critical that the new chip be installed with the notch at the top. Insert a small screw driver between the chip and the socket which holds it in place. If necessary bend the small round capacitor just above the chip gently out of the way. Gently twist and pry the chip out of its socket being careful not to bend the legs. Once the chip is almost free grab the two ends and pull the chip straight away from its socket. Between the chip and its socket lies a thin flat capacitor called a Roger's Cap. Leave the Roger's Cap in place. If it is removed place it's legs in the four corners of the socket and note the notch position must be up just like the EPROM chip. (Note the socket also has a notch at the top). Rotate the new EPROM so its notch faces up; then start all the right side legs partially into their sockets. Pushing the body of the IC slightly to the right will move the left side legs into position so that they will go into their socket holes. While supporting the board from behind push the chip to the right and down. The pins of the new IC's are usually spread apart for automatic insertion equipment, this could make them difficult to install by hand. It is possible to straighten the legs by holding the IC at the ends and rolling it against the counter top with firm pressure.

Note: Inspect the chip for any pin (legs) which may have been bent or did not go into their socket hole. If such an error is found remove the chip straighten the pins legs if necessary and try again. Remember, the legs will only survive being bent a few times at best. If all looks OK put the machine back together in the reverse order it was disassembled. Be sure that all protective covers and shields are in their proper place before reconnecting power and turning the machine back on. Once turned on the machine should move to find the HOME position then move to the park position. If the machine does not move after a few seconds the EPROM was probably not installed correctly. Power the machine down and check for incorrect installation. Call Kayeness Inc. if no obvious error can be found.

J. Fixing a Thermometer when the Mercury has separated

Mercury Separation in Thermometer

Before using any thermometer it should be examined very carefully for mercury separation in the main mercury column, expansion chamber, contraction chamber and bulb. Mercury separation in the bulb will usually show as small bubbles. All the mercury must be united. If a thermometer does not read zero at the ice point mercury separation is typically the cause.

There is no known method to completely insure that the mercury will not

separate in a thermometer when the thermometer is subjected to shock. This can occur either in transit or by improper storage and handling in reuniting a separated mercury column. Remember that the thermometer contains only two fluids, mercury and gas. The object is to get all the liquid below the gas or, conversely all the gas above the liquid.

Cooling Method for Reuniting Mercury Thermometers

This method is the easiest to use and is the method PRINCO recommends. In a small Dewar flask or thermos bottle mix powdered dry ice with Methanol or Acetone. Holding the thermometer vertical, immerse about 3/4 of the lower section of the bulb into the mixture. DO NOT immerse the capillary or funnel section that is above the bulb into the mixture. The main portion of the mercury will retreat into the bulb, and the separated portion will follow.

Occasionally the separated portion may cling to the walls of the funnel portion of the bulb. When all the mercury, including the separated portion, has retreated into the bulb, remove the thermometer from the dry ice mixture. The mercury should go together. Stand the thermometer in a vertical position and allow the mercury to rise into the capillary of its own accord. DO NOT TOUCH THE BULB WITH YOUR HANDS!

If you are unsuccessful repeat the cooling method except this time gently tap (do not bounce) the thermometer bulb vertically on a desk pad after removing from the flask.

NOTE: Where possible, thermometers should be stored in a vertical position.

K. Answers to Common Questions

1. The machine doesn't respond when I press UP, DN or RUN, but the front display shows the temperature OK and says READY.

Most likely the EMERGENCY stop button is depressed. Press the stop button then pull the red EMERGENCY stop button out and see if the machines response when pressing the DN button.

If when you press DN the machine still doesn't go down, go into TEST mode by pressing TEST then press NO. FORCE TEST should appear; then press YES,. If a value greater than 2.0 appears it is causing the problem. Adjust the OFFSET screw on the force board to get the force value of 0.2 to 0.3 as the offset (0.25 is the weight of the

plunger)

2. I pressed RUN the machine appeared to start but now it just sits there saying F0 on the front display.

   The machine is trying to communicate with the Personal Computer (PC) because it must communicate with it before the test starts. Either: 1) the software is not running on the PC; 2) the cable connection or communications port on the PC is faulty; 3) the PC attached option (under SHIFT PRNT) is set *ON* when it should be set *OFF* because the machine is not attached to a PC.

3. I was running a test and the plunger came down and emptied the barrel of material but no data was collected.

   Press SHIFT then PRNT you will see the manual mode is set to YES. When the manual mode is on data is ONLY collected if and when the END button is pressed. Set the test to RUN automatically by shutting manual mode off and by setting the positions you wish the machine to collect data or RUN manual and press END when the force values are steady (ca. +/- 1%).

4. The temperature used to be very stable on the machine now it seems to vary more than +/- 0.2 degrees when just sitting there. (not loading sample or cleaning barrel)

   The hole that the temperature measurement probe sits in may be contaminated with material. Set the current machine temperature to 0.0 then and only then remove the temperature probe (PRT), wipe it clean if necessary and use a long drill bit to remove any material down the PRT measurement hole.

5. A large amount of material (more than 1/4" up the plunger) has gone past the Plunger tip is this OK?

   NO. Shear rates calculated from plunger speed and forces measured depend greatly on the assumption that all material exits through the die. Check to see the tip is within ASTM specs that is between 0.3751 - .3745, high temperature machines will tend to be on the low side of this specification (bigger gaps when measured cold). If the tip is worn replace it, if it is OK and you still see significant material going past the tip you may wish to try using an O-ring tip or one of other special designs available for low viscosity (or high pressure) applications.

6. How do I check the temperature?

   You received with the machine a mercury thermometer. This thermometer comes with a calibration certificate from KAYENESS comparing it to our NIST traceable reference box. Any deviation from our NIST traceable box will be noted and supplied with the thermometer. Set the machine to the temperature of the reference thermometer (see

thermometer markings). Let the machine come to temperature. Only after the machine is stable insert the thermometer gently down the thermometer well which lies under an Allen screw cap, directly adjacent to the PRT probe, on the top of the rheometer barrel.

7. My software runs very, very slowly.

KARS on a 286 or better normally is very peppy. If not the most common reason is that it is trying to communicate through a communication port to a device it thinks is present. Often this means the plotter cable is wired incorrectly so KARS thinks a laser swell device is present. When it doesn't get any data from the device it thinks is present it slows everything down trying to get data. Solution: Use Kayeness supplied cables or follow the cabling diagrams found in the utilities.

8. I've overforced the machine and now it's taking forever to purge or just won't purge.

Press UP so the plunger is not putting pressure on the material in the barrel, then remove the die by unscrewing the die nut holder. Take the die and nut holder completely out then purge the machine by pressing SHIFT then DN then YES, as usual, to purge.

L. Example Programs (English Units).

Setting a Time Sweep rheometer *PROGRAM*
Note press **EDIT** on the front panel of the rheometer to start the process

Machine Responses	**PRESS THESE KEYS**	Comments
Melt Time = XXX.X	**NO 2 4 0 YES**	XXX.X is any number value given in seconds
MATL ID= ????????	**NO A S K YES YES**	NO clears, + makes "A' appear, ES accepts letter, YES accepts all
TEMP. = XXX.X	**NO 2 3 0 YES**	NO clears, 230 °C entered, YES accepts value
ORIF.RAD. =.XXXX	**NO 0 2 0 YES**	Assumes your capillary die is 0.020 inch radius (0.04 Diam.)
ORIF. LEN. =X.XXX	**NO 0 8 0 YES**	Assumes die length is 0.8"
SAMPL LEN=X.XXXX	**NO YES**	Set to 0.0 for rate control run
RATE #1= XX.XXX	**NO 0 0 3 YES**	Set Ram Rate to 0.3"/min
RATE #2= XX.XXX	**NO 0 0 3 YES**	Set Ram Rate to 0.3"/min
RATE #3= XX.XXX	**NO 0 0 3 YES**	Set Ram Rate to 0.3"/min
RATE #4= XX.XXX	**NO 0 0 3 YES**	Set Ram Rate to 0.3"/min
RATE #5= XX.XXX	**NO 0 0 3 YES**	Set Ram Rate to 0.3"/min
RATE #6= XX.XXX	**NO YES**	0.0 rate means no more rates
MELT FORCE=XXX.X	**NO 1 5 0 YES**	150 LB pre-start force to pack
TERM FORCE=XXX.X	**NO 7 5 0 YES**	750 LB safety overload
START POS.=X.XXX	**NO 5 YES**	Begin test at 5 inches
POS. #1 = X.XXX	**NO 5 3 YES**	Force acquired at 5.3 position
POS. #2 = X.XXX	**NO 5 6 YES**	add 0.3" for others
POS. #3 = X.XXX	**NO 5 9 YES**	
POS. #4 = X.XXX	**NO 6 2 YES**	
POS. #5 = X.XXX	**NO 6 5 YES**	Finished entering positions
PARK POS.=X.XXX	**NO 2 0 YES**	Crosshead Park 2.0 inch position
TEST DELAY=XXXX	**NO 1 2 0 YES**	Pause for 120 secs between pts
S.I., Pascal	**YES**	Accept S.I. units
RUN# START	**NO YES**	Clears RUN# to zero
PROGRAM ENTERED		

Setting a Rate Sweep rheometer *PROGRAM*

Note press **EDIT** on the front panel of the rheometer to start the process

Machine Responses	PRESS THESE KEYS	Comments
Melt Time = XXX.X	NO 360 YES	XXX X is any number value given in seconds
MATL ID= ????????	NO ++ YES YES	NO clears, ++ makes "B' appear, YES accepts value
TEMP.= XXX.X	NO 230 YES	NO clears, 230 °C entered, YES accepts value
ORIF. RAD.=.XXXX	NO 020 YES	assumes your capillary die is 0.02" radius
ORIF. LEN.=X.XXX	NO 080 YES	assumes capillary die length is 0.8"
SAMPL LEN=X.XXXX	NO YES	Set SAMPLE LEN to 0.0 for rate run
RATE #1= XX.XXX	NO 12 YES	Set Ram Rate to 12.0 "/min
RATE #2= XX.XXX	NO 02 YES	Set Ram Rate to 2.0 "/min
RATE #3= XX.XXX	NO 0024 YES	Set Ram Rate to 0.24 "/min
RATE #4= XX.XXX	NO 0004 YES	Set Ram Rate to 0.04 "/min
RATE #5= XX.XXX	NO 00012 YES	Set Ram Rate to 0.012 "/min
RATE #6= XX.XXX	NO0024 YES	Set Ram Rate to 0.24 "/min (again)
RATE #7= XX.XXX	NO YES	0.0 rate means no more rates
MELT FORCE=XXX.X	NO 175 YES	175 LB pre-start force to pack
TERM FORCE=XXX.X	NO 750 YES	750 LB safety overload
START POS.=X.XXX	NO 5 YES	Begin test at 5 inches
POS. #1 = X.XXX	NO 67 YES	Force acquired at 6.70 position
POS. #2 = X.XXX	NO 745 YES	Force acquired at 7.45 position
POS. #3 = X.XXX	NO 775 YES	Force acquired at 7.75 position
POS. #4 = X.XXX	NO 785 YES	Force acquired at 7.85 position
POS. #5 = X.XXX	NO 787 YES	Force acquired at 7.87 position
POS. #6 = X.XXX	NO 82 YES	Force acquired at 8.20 position
PARK POS.=X.XXX	NO 20 YES	Crosshead Park 2.0 inch position
TEST DELAY=XXXX	NO YES	No Pause between pts
S.I., Pascal	YES	Accept S.I. units
RUN# START	NO YES	Clears RUN# to zero
PROGRAM ENTERED		

Setting a Time Sweep rheometer *PROGRAM*

Note press **EDIT** on the front panel of the rheometer to start the process

Machine Responses	**PRESS THESE KEYS**	Comments
Melt Time =XXX	**NO 360 YES**	XXX.X is any number value given in seconds
MATL ID= ????????	**NO + YES YES**	NO clears, + makes "A' appear, YES accepts letter, YES accepts all
TEMP.= XXX.X	**NO 230 YES**	NO clears, 230 ^{o}C entered, YES accepts value
ORIF. RAD.=.XXXX	**NO 020 YES**	Assumes your capillary die is 0.02" radius, 0.04 diameter
ORIF. LEN.=X.XXX	**NO 080 YES**	Assumes die length is 0.8"
SAMPL LEN=X.XXXX	**NO YES**	Set to 0.0 for rate control run
RATE #1= XX.XXX	**NO 003 YES**	Set Ram Rate to 0.3"/min
RATE #2= XX.XXX	**NO 003 YES**	Set Ram Rate to 0.3"/min
RATE #3= XX.XXX	**NO 003 YES**	Set Ram Rate to 0.3"/min
RATE #4= XX.XXX	**NO 003 YES**	Set Ram Rate to 0.3"/min
RATE #5= XX.XXX	**NO 003 YES**	Set Ram Rate to 0.3"/min
RATE #6= XX.XXX	**NO YES**	0.0 rate means no more rates
MELT FORCE=XXX.X	**NO 150 YES**	150 LB pre-start force to pack
TERM FORCE=XXX.X	**NO 750 YES**	750 LB safety overload
START POS.=X.XXX	**NO 5 YES**	Begin test at 5 inches
POS. #1 = X.XXX	**NO 53 YES**	Force acquired at 5.3 position
POS. #2 = X.XXX	**NO 56 YES**	add 0.3" for others
POS. #3 = X.XXX	**NO 59 YES**	
POS. #4 = X.XXX	**NO 62 YES**	
POS. #5 = X.XXX	**NO 65 YES**	Finished entering positions
PARK POS.=X.XXX	**NO 20 YES**	Crosshead Park 2.0 inch position
TEST DELAY=XXXX	**NO 120 YES**	Pause for 120 secs between pts
S.I., Pascal	**YES**	Accept S.I. units
RUN# START	**NO YES**	Clears RUN# to zero
PROGRAM ENTERED		

M. Plunger Speeds Necessary to Achieve Shear Rates for given Die Diameters

Apparent Shear Rate	RAM RATE ("/min) needed for shear rate at left for Die Diameters of:								
	0.0820	0.0600	0.0500	0.0400	0.0300	0.0250	0.0200	0.015	0.0120
0.1	0.003								
0.2	0.006	0.002							
0.5	0.015	0.006	0.003						
1.0	0.029	0.011	0.007	0.003					
2	0.059	0.023	0.013	0.007	0.003				
5	0.146	0.057	0.033	0.017	0.007	0.004	0.002		
10	0.293	0.115	0.066	0.034	0.014	0.008	0.004		
20	0.585	0.229	0.133	0.068	0.029	0.017	0.008	0.004	
50	1.463	0.573	0.332	0.170	0.072	0.041	0.021	0.009	0.005
100	2.925	1.146	0.663	0.340	0.143	0.083	0.042	0.018	0.009
200	5.850	2.292	1.326	0.679	0.286	0.166	0.085	0.036	0.018
500	14.625	5.729	3.316	1.698	0.716	0.414	0.212	0.090	0.046
1,000	29.250	11.459	6.631	3.395	1.432	0.829	0.424	0.179	0.092
2,000		22.918	13.263	6.790	2.865	1.658	0.849	0.358	0.183
5,000				16.976	7.162	4.145	2.122	0.895	0.458
10,000					14.324	8.289	4.244	1.790	0.917
20,000					28.647	16.578	8.488	3.581	1.833
50,000							21.220	8.952	4.584
100,000								17.904	9.167
200,000									18.334
500,000									
1,000,000									
	Kayeness Galaxy V Based on machine speeds min and max shear rates obtainable (0.005 - 24 "/min)								
Diameter	0.0820	0.0600	0.0500	0.0400	0.0300	0.0250	0.0200	0.0150	0.0120
Min SR	0.17	0.44	0.75	1.47	3.49	6.03	11.78	27.93	54.54
Max SR	820	2094	3619	7068	16755	28953	56550	134045	261807

The following equation can also be used, where S is speed in "/min $\dot{\gamma}$ is the desired shear rate, D_c is the die diameter

$$S = \dot{\gamma} D_c^3 (53.05)$$

N. General Capillary Rheometer Reference Books

The Dynisco Injection Molders Handbook, Tony Whelan and John Goff 1991
The Kayeness Practical Rheology Handbook, Tony Whelan and John Bryson 1991
The Dynisco Extrusion Processors Handbook, Tony Whelan and David Dunning 1991

Melt Rheology and it's role in Plastics Processing,
Dealy & Wissburn, Van Nostrand Reinhold, 1990

Modern Plastic Encyclopedia
(Get Latest Issue)
McGraw-Hill, POB 423
Highstown, NJ 08520
(Has data bank on material, + lots of good intro. articles)

Polymer Melt Rheology
(A guide for industrial practice)
F. N. Cogswell,
George Godwin Limited in association with The Plastics Rubber Institute,
Wiley & Sons, New York & Toronto (C) 1981

Plastics Engineering
Chap 3 Rigid PVC Rheology Chap 4 Rigid PVC Extrusion
Edited by I. Luis Gomez. (Monsanto, Springfield, MA)
Marcel Dekker, Inc. New York and Basel

Polymer Extrusion
Chris Rauwendaal
Hanser, 1986

Principles and Applications of Rheology
A. G. Frederikson
Prentice Hall, N.Y. 1969

Rheological Techniques
R.W. Whorlow
Ellis Horwood limited
Merket Cross House,
Cooper St., Chichester, West Sussex
PO19 1EB, England
USA. Halsted Press division of John Wiley & Sons, N.Y.

Polymer Structure, Properties and Applications
R.D. Deanin,

Boston, Cahners Books, a division of Cahners Publishing Co. 1972

General Introduction to Polymers
Textbook of Polymer Science
F.W. Billmeyer, Jr.
Wiley-Interscience, New York 1962

Theoretical
Computer Programs for Rheologists
G. Gordon,M. Shaw, SPE, Hanser 1994

Viscoelastic Properties of polymers
J. D. Ferry, Wiley & Sons, 1980

Properties of Polymers
D. W. Van Krevlen,
Elsevier, New York 1990. Third Ed

INDEX