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DYNISCO

# 7000 Series Capillary Rheometers & LabKARS Software

Version: 2.4



User Guide

This manual was produced using *Doc-To-Help*®, by WexTech Systems, Inc.



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## Notice

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Thank you to all Dynisco Polymer Test Systems employees for their input into this manual.

Comments or suggestions for possible improvement to the manual are appreciated and may be sent to the following location:

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# Contents

<b>INTRODUCTION .....</b>	<b>7</b>
1. <i>How to use this manual .....</i>	7
2. <i>Typographic Conventions.....</i>	7
3. <i>How to Contact DYNISCO POLYMER TEST.....</i>	8
<b>GETTING STARTED.....</b>	<b>9</b>
1. <i>Uncrating and Setup.....</i>	9
2. <i>Bench Requirements and Placement.....</i>	9
3. <i>Unpacking the Rheometer.....</i>	9
4. <i>Power connection .....</i>	10
5. <i>Computer Connection.....</i>	10
6. <i>Laser Micrometer connection (optional).....</i>	10
7. <i>Model 7000.....</i>	10
8. <i>Model 7001 .....</i>	10
9. <i>Model 7002.....</i>	10
10. <i>Load Cell assembly (models 7000 &amp; 7001).....</i>	11
11. <i>Connecting the Barrel Pressure Transducer plug (models 7001 &amp; 7002).....</i>	11
12. <i>Powering up the Rheometer.....</i>	12
13. <i>Software installation.....</i>	12
14. <i>E-mail Software Upgrades .....</i>	13
16. <i>Rheometer Firmware and Firmware Upgrades .....</i>	14
17. <i>Die Swell Laser Installation .....</i>	15
<b>SAFETY .....</b>	<b>17</b>
1. <i>Use Gloves - Its Hot! .....</i>	17
2. <i>Electrical Hazard .....</i>	17
3. <i>Calibration Thermometers use Mercury (Models 7000, 7001 &amp; 7002) .....</i>	17
4. <i>Pinch Points.....</i>	18
5. <i>Fumes from Materials .....</i>	18
6. <i>Before Testing Safety Check.....</i>	18
7. <i>Purging the rheometer.....</i>	19
8. <i>Putting the Die(s) in the Barrel .....</i>	19
9. <i>Loading the Sample .....</i>	19
10. <i>Starting the Purge.....</i>	20
11. <i>Cleaning Up.....</i>	20
<b>TEST DRIVE.....</b>	<b>22</b>
1. <i>Time Sweep Test .....</i>	22
2. <i>Starting Lab KARS and setting up the rheometer.....</i>	22
3. <i>Performing the test .....</i>	22
4. <i>The Second Test: A Rate Sweep.....</i>	23
5. <i>Cleaning Up a really Big Mess.....</i>	23
<b>LAB KARS.....</b>	<b>25</b>

LABKARS 7000 and 7001.....	25
LABKARS 7002.....	25
LABKARS OPERATIONS.....	25
<i>PC Requirements &amp; Port Settings</i> .....	26
<i>Starting LabKARS or LabKARS Set-Up</i> .....	28
<i>Creating a LabKARS Icon</i> .....	28
<i>Security/Password Protection</i> .....	29
<i>LabKARS Security Access Levels</i> .....	29
<i>Database Record Manipulation Keys</i> .....	30
<i>Before The Start of any Run</i> .....	30
<i>Moving around LabKARS</i> .....	31
SET UP TEST PAGE.....	31
<i>Setting up a new test</i> .....	32
<i>Data Point Setup</i> .....	33
<i>Data Acquisition Mode</i> .....	35
<i>Start Position</i> .....	35
<i>Die Selection</i> .....	36
<i>Generic Prerun Check List</i> .....	36
<i>Test Setup Example</i> .....	37
HAMA LASER MICROMETER.....	38
<i>Starting up and testing the laser</i> .....	38
<i>HAMA Laser Calibration</i> .....	40
RHEOMETER CONTROL SCREEN .....	40
<i>Control Buttons</i> .....	41
<i>Real-time Data during the Test</i> .....	42
<i>Test Automation Server</i> .....	43
PLOTTING AND ANALYZING YOUR DATA .....	44
<i>Plotting</i> .....	44
<i>Merging</i> .....	44
<i>Single Run Analyze / Database</i> .....	45
<i>Plotting</i> .....	46
<i>From Database/ Filters Option</i> .....	46
<i>From Single Run/Modify Screen</i> .....	46
<i>Modifying Plots using the Plot Toolbar</i> .....	46
<i>Appending and Overlaying Plots</i> .....	48
<i>Copying Plots</i> .....	48
<i>Saving Plots</i> .....	48
<i>Global Preferences (SQC, NNI, I.V. and Bagley)</i> .....	48
<i>SQC Plots</i> .....	48
<i>NNI</i> .....	49
<i>I.V. Preferences</i> .....	50
<i>Misc.</i> .....	50
<i>Bagley Correction</i> .....	50
<i>Plot RMS Error</i> .....	51
LABKARS REPORTS .....	51
EXPORTING DATA TO EXCEL.....	53
<i>C. Selecting Load Cell or Pressure Transducer</i> .....	54
<i>D. Time Sweep</i> .....	54
<i>E. Shear Rate Sweep</i> .....	55
<i>F. Shear Stress Sweep</i> .....	56
<i>G. Combining Time and Rate Runs</i> .....	56
<i>H. Accuracy and Reproducibility</i> .....	57
Precision .....	57
Accuracy.....	57
<b>FORMULAS USED IN DATA ANALYSIS .....</b>	<b>58</b>

<i>Apparent Shear Rate Calculation:</i> .....	58
<i>Apparent Shear Stress Calculation:</i> .....	58
<i>Apparent Shear Viscosity Calculation:</i> .....	59
<i>Volumetric Flow Rate Calculation:</i> .....	59
<i>Mass Flow Rate Calculation:</i> .....	60
<b>INTRODUCTION TO RHEOLOGY .....</b>	<b>61</b>
<i>A. Geometries of Deformation</i> .....	61
<i>B. Newton's Law</i> .....	63
<i>C. Effect of Shear Rate on Viscosity</i> .....	66
<i>D. Effect of Time on Viscosity</i> .....	66
<i>E. Effect of Temperature on Viscosity</i> .....	66
<i>F. Effect of Fillers on Viscosity</i> .....	67
<i>G. Effect of Moisture on Viscosity</i> .....	67
<i>H. Equations and Corrections</i> .....	67
<i>I. Pressure Profile</i> .....	68
<i>J. Why use a Pressure Transducer?</i> .....	69
<i>K. Common Assumptions</i> .....	69
<i>L. Weissenberg-Rabinowitsch Correction</i> .....	69
<i>M. Corrected Shear Stress</i> .....	70
<i>N. Extrudate Swell</i> .....	71
<i>O. PVC</i> .....	71
1. <i>System Configuration</i> .....	72
<b>MAINTENANCE / CALIBRATION.....</b>	<b>72</b>
2. <i>Force Calibration</i> .....	73
A. <i>Calibrating the Calibration Unit</i> .....	73
B. <i>Calibrating the rheometer load cell with the calibration unit</i> .....	74
3. <i>Temperature Calibration</i> .....	78
4. <i>Pressure Transducer Calibration</i> .....	81
A. <i>ZERO SHIFT</i> .....	81
5. <i>RCAL</i> .....	84
6. <i>Dimensional Verification</i> .....	85
7. <i>Die Diameter</i> .....	85
8. <i>Die Length</i> .....	85
9. <i>Barrel Diameter</i> .....	86
10. <i>Plunger Tip Diameter</i> .....	86
11. <i>Lubrication</i> .....	86
12. <i>Barrel Drawing</i> .....	86
<b>EXTRAS.....</b>	<b>87</b>
<i>Measurement of Melt Density</i> .....	87
<i>The Non-Newtonian Index</i> .....	87
<b>TROUBLE SHOOTING .....</b>	<b>89</b>
1. <i>Frequently Asked Questions (FAQs)</i> .....	89
2. <i>When your Control material goes out of control</i> .....	92
<b>PARTS LIST .....</b>	<b>93</b>
<i>DIES and ORIFICES</i> .....	93
<i>Die design and configuration</i> .....	96
<i>Supplied Hand Tools</i> .....	97
<i>LCR 7000 SPARE PARTS LIST</i> .....	98

NAVIGATING THE KEYPAD .....	102
<i>The Keypad</i> .....	102
<i>Keypad Definitions</i> .....	102
<b>CONVERSION FACTORS .....</b>	<b>105</b>
<b>EXAMPLE RUNS .....</b>	<b>106</b>
1. <i>Polyethylene-</i> .....	106
<i>Terephthalate I.V. Test</i> .....	106
2. <i>Acrylonitrile-</i> .....	107
<i>Butadiene-</i> .....	107
<i>Polystyrene</i> .....	107
<i>(ABS)</i> .....	107
3. <i>Nylon</i> .....	108
4. <i>Glass filled Polyurethane</i> .....	109
<b>SUPPORT VENDORS .....</b>	<b>110</b>
1. <i>Pin Gages</i> .....	110
2. <i>Cleaning Patches</i> .....	110
3. <i>Bore Gages</i> .....	110
4. <i>NIST Standard Reference Material</i> .....	110
5. <i>Hg spill kits</i> .....	111
6. <i>Thermometers</i> .....	111
A. <i>Fixing a Thermometer when the Mercury has separated</i> .....	111
<b>RUNNING A GREY POLYPROPYLENE STANDARD TEST .....</b>	<b>112</b>
<i>PROCEDURE</i> .....	112
<b>RAM RATE / SHEAR RATE TABLE .....</b>	<b>118</b>
<b>REFERENCE BOOKS .....</b>	<b>119</b>
1. <i>Process and Theory</i> .....	119
2. <i>General Introduction to Polymers</i> .....	120
3. <i>Theoretical</i> .....	120
<b>GLOSSARY OF TERMS .....</b>	<b>121</b>
<b>INDEX .....</b>	<b>123</b>

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# Introduction

## 1. How to use this manual

This manual describes the setup procedure and basic operation of the *DYNISCO POLYMER TESTS 7000* series capillary rheometers. It provides the complete description of resources at your command when operating your rheometer. 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 rheometers at our in-house polymer testing labs.

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

## 2. Typographic Conventions

*Italics*: Rheological items which, have defining equations presented in this manual, are shown in italics.

***Bold Italics***: These are parameters that 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 keypad.

**BOLD ALL CAPITALS**: This indicates an actual key found on the rheometer keypad. Usage is as follows: Press the **ESC** button to stop the rheometer movement.

Underlined Items: Underlined items head paragraphs or sections that 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 *DYNISCO POLYMER TEST*'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.

### **3. How to Contact DYNISCO POLYMER**

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

- Rheometer name and model number
- Rheometer serial number (on back panel of the unit)
- Computersystem make and model
- Current version of software (LabKARS "About" page found on the main operating page of the software)

Should you wish to contact us directly, Contact Dynisco Customer Service at 508-541-9400, Fax 508-541-6206. Should you wish to comment or query in writing address it to:



DYNISCO POLYMER TEST, Customer Service  
38 Forge Parkway  
Franklin, MA 02038-3134  
United States

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# Getting Started

## 1. Uncrating and Setup

The instrument arrives in a crate weighing approximately 770 kg (350 lb.) measuring 165 X 63.5 X 61 cm (65" X 25" X 24"). Save the crate until you are fully satisfied with the operation of your instrument. We recommend that you save the shipping crate for future shipments of the rheometer back to Dynisco for upgrades, service and/or calibration, or for your own facility or rheometer relocation.

Remove the lid to the crate, labeled TOP, by removing the wood screw fasteners. Remove only the shipping restraints for the boxes shipped inside the crate and then remove the boxes. Stand the crate up, with the instrument still inside, so that the instrument is right side up. **Remove all instrument-shipping restraints taking care that the instrument does not shift and fall out of the crate.** Walk the instrument out of the box, "Caution, there may be a shipping restraint under the instruments feet". Once fully removed, check for any shipping damage and contact the factory if necessary.

## 2. Bench Requirements and Placement

Typical laboratory benches are too high for efficient use of the rheometer. 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 (desktop 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 rheometer. 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 the carpet.

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 check with 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.

## 3. Unpacking the Rheometer

Most of the rheometer 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 rheometer is possible. These items include the Load Cell (models 7000 & 7001), Interface cables for the Load Cell and Pressure Transducers (models 7001 & 7002), Cable connection for PC (Computer), printer (if applicable), Laser Micrometer (if applicable), and AC Power connections.

**\* Keep the wooden crate in storage in case you ever need to move or ship the rheometer back to Dynisco or another location.**

#### 4. Power connection

First find the surge protector/power box. This is an extension cord 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 this thickest cord to the back of the rheometer base 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 (if used) can be connected to any remaining outlet.

#### 5. Computer Connection

The data processing system consists of a PC and the LabKARS software package. Connecting the interface cable, from the back of the PC (Com. port #1), to the back of the rheometer, makes the link between the PC COM1 port and the rheometer. On the back panel of the rheometer you will find a female DB9 connector for a serial PC connection. Recommended PC requirements are:

- CPU/Speed: Pentium II / 400 Mhz or better & 1 free serial com
- RAM: 32 MB
- Hard Drive Space: 2 GB or more
- Laser option: available ISA for Windows 2000
- Monitor: 17-inch
- Display Properties: High Color 16-Bit & 1024 x 768 screen area

#### 6. Laser Micrometer connection (optional)

If you have purchased the HAMA Laboratories PA02L High Resolution Laser Micrometer (available for models 7000, 7001, and 7002), you will find a digital I/O board, and interface cable, and the laser micrometer itself. The digital I/O board base address switch is preset at 2E0h. If this address is already assigned to some other device in your computer, you must set this switch for a different address. Reference the HAMA Laboratories User's Guide. Please note that if the PC was purchased with the rheometer the digital board for the Laser Micrometer will already be installed.

**CAUTION: BE SURE THE PC IS NOT POWERED UP WHEN CONNECTING THE INTERFACE CABLE FROM THE DIGITAL CARD TO THE LASER MICROMETER DAMAGE TO THE UNIT MAY OCCURE.**

#### 7. Model 7000

This model is a single bore rheometer and has Load Cell measurement capability only. You will need to install the Dynisco Load Cell to the rheometer crossbar and connect the coiled interface cable from the back of the rheometer base to the Load Cell. (See Load Cell Assembly Section for more details).

#### 8. Model 7001

This model is a single bore rheometer that has simultaneous Load Cell and/or PT measurement capability. You will need to install the Dynisco Load Cell to the cross bar, and install the Dynisco PT into the rheometer barrel. Connect the coiled interface cable from the rheometer drive system at the top of the rheometer to the Load Cell, and another coiled interface cable from the back of the rheometer base to the PT. (See Load Cell Assembly and Pressure Transducer Installation sections bellow for more details).

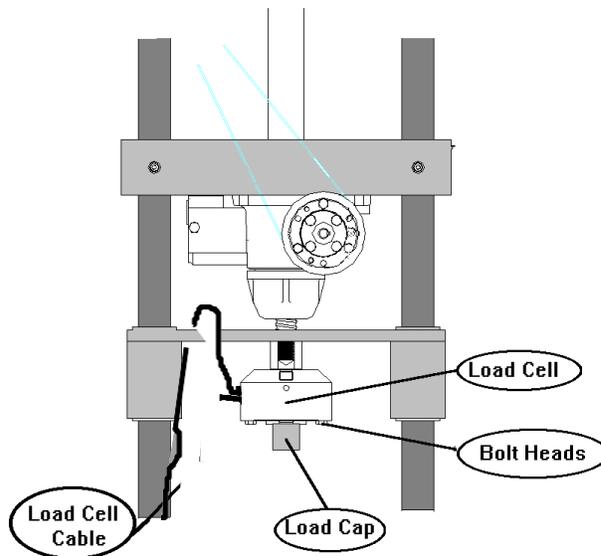
#### 9. Model 7002

This model rheometer has a dual bore and Twin Pressure Transducers for twin die measurement capability. Bagley and entrance pressure loss measurements can be

performed in one test using this instrument. Install both of the Dynisco PT's into the rheometer barrel and connect the coiled interface cable from the rheometer base to the PT's. (See Pressure Transducer Installation Section for more details). Be sure Sensor #1 is attached to barrel 1, and Sensor #2 is attached to barrel 2.

## 10. Load Cell assembly (models 7000 & 7001)

Models 7000 & 7001 have Load Cell measurement capability. The Load Cell is a round, blue unit with a 6-pin BENDIX connector. You will need to mount the Load Cell to the crosshead and connect it to the rheometer's coiled interface cable. 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 that has the exposed bolt heads (active end). The rheometer plunger will fit into the load cap when a test is underway. Connect the load cell to the cross-head of the rheometer as follows: Screw the open end onto the bolt in the center of the crosshead located between the two-rheometer posts and directly above the barrel hole. A two handed snug tightening is all that is required. Connect the coiled interface cable to the Load Cell.



## 11. Connecting the Barrel Pressure Transducer plug (models 7001 & 7002)

For models with the PT measurement option, you will need to confirm the pressure transducer port is plugged and the PT Sensor is turned OFF if a PT is not being used, or a pressure transducer is inserted into the port(s). If a PT is not being used to test, apply a light coat of anti-seize to the threads of the PT Port Plug and insert into the PT port. Only hand-tighten the Port Plug until the plug warms up to the barrel temperature. Once the PT or the plug has had time to heat, snug it tight with a wrench. Press the "SETUP" button on the front control panel of the rheometer. Use the Smart Keys on the rheometer display to move Left or Right through the menus until the sensor you wish to turn off (Sensor #1, Sensor #2 or both) is shown on the control panel display. Press the "Edit" Smart Key and scroll through the sensor selections until the display reads "Device Not Entered" and press the "Select" smart key. The sensor that you selected is now turned off.

## 12. Powering up 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. Be sure the safety shield is down and turn on the power to the main power box. Then turn on the rheometer by pressing down the top half of the black power switch, located 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 rheometer has reached its **Park Position** and stops moving. In this process the rheometer is resetting its Position based on the upper limit switch stop. If it is stopped during this process the rheometer position will be incorrect. Simply shut the rheometer off wait for 10 seconds then turn it back on. Let the rheometer equilibrate at test temperature for a minimum of 20 minutes before performing any tests. The rheometer display will read, "READY" when temperature is stable and ready to test.

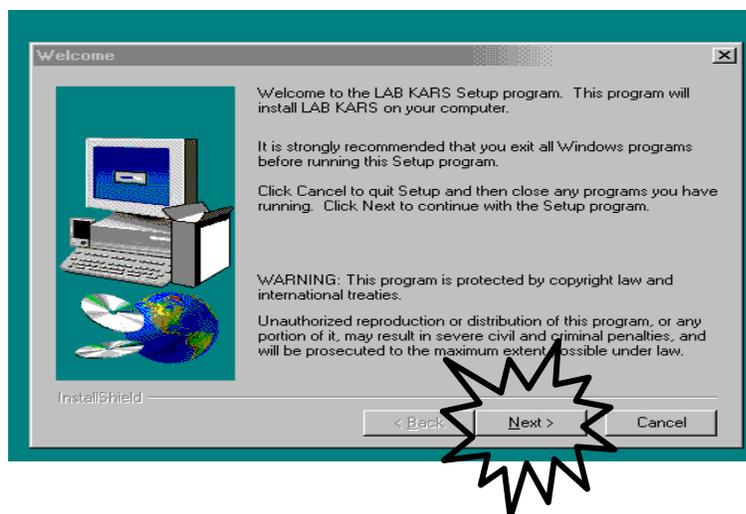
**IF NOTHING HAPPENS:** If nothing happens when you turn the rheometer on, check that the **EMERGENCY STOP** button is pulled out (be sure to press **RESET** before pulling out the emergency stop button) and all power connections have been made. If still nothing happens proceed to the troubleshooting section.

## 13. Software installation

If you purchased your PC from *DYNISCO POLYMER TEST* the LabKARS software will already be installed on your computer. An installation disks will also be included in your package for file or reinstallation should it be necessary. If you have accidentally erased the program or have purchased your own computer for the rheometer system, insert the LAB KARS CD-Rom disk into the PC's CD-Rom drive. Under WINDOWS START, Run, type X:\Setup where X= the designation of your PC's CD-Rom drive. The Install Shield will launch and guide you through the installation process. Accept all defaults if you are installing the software for the first time. If you are upgrading to another version of LabKARS you will be asked by Install Shield if you wish to proceed with a Complete Install or Upgrade.

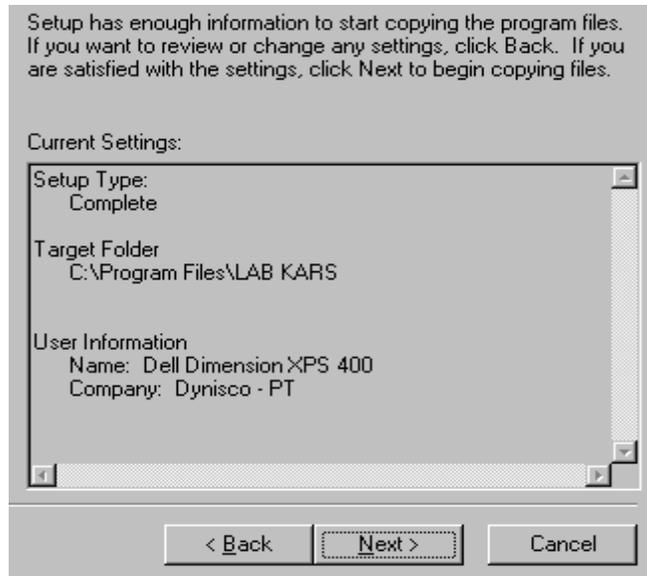
**Caution: Before altering any version of LabKARS software, we recommend that a backup of your current LabKARS and Databases be made in another directory before conducting any upgrades or changes to the software.**

Select the Compact Install option and accept all defaults.



Press the NEXT button when finished. Next you will be asked to choose a directory for LabKARS. The install default is set to install LabKARS onto your computers hard drive. Dynisco recommends saving LabKARS to any other drive as backup only and not for general everyday use.

Install Shield will ask you to double-check your settings before completing the setup process. If your information is accurate press the NEXT button to start copying the LabKARS files.



Once the LabKARS files are copied, you will need to restart your computer in order to finish the install process and enter into the LabKARS program.

**CAUTION: When restarting your PC for the first time, your computer may detect a Software Error in LabKARS. You will be asked to choose an option "A", "B" or "C". Choose option "A. View Settings" to clear up this error and start Windows™.**

## 14. E-mail Software Upgrades

If you receive your LabKARS upgrade via E-Mail from Dynisco Polymer Test. The KARS upgrade will consists of application files and may be in ZIP format depending on your email restrictions.

Name	Size	Type	Modified
EXAMPLE	2KB	WinZip File	10/17/1998 7:00 AM

Before installing the application file:

Rename your current LabKARS application file to “LabKARS\_First” or “LabKARS\_old”

Proceed to the attached application file:

- If the upgrade is zipped, unzip the exe. in your e-mail and copy and paste all files into your LabKARS directory and rename it LabKARS

- If it is not zipped, copy all files to your LabKARS directory and overwrite the existing files.

Name	Size	Type	Modified
Bagley	1,172KB	Application	02/06/2001 10:32 AM
LabKars	1,202KB	Application	02/06/2001 10:18 AM
LabReport	1,152KB	Application	02/02/2001 11:57 AM
UpdateRD	672KB	Application	02/02/2001 11:52 AM

You should now be able to run the latest version of LabKARS

## 16. Rheometer Firmware and Firmware Upgrades

Firmware is the programming embedded into the computer chips (EPROMS etc.) found inside the rheometer. The firmware governs most of the rheometer behavior and all of its communications to outside devices such as a PC or a directly connected printer. Software (like LabKARS) runs on your PC and helps you save and analyze your data.

Turn the rheometer power off and unplug instrument.

Remove four (4) Phillips head screws from each side of instrument (eight (8) screws total).

Swing forward keypad panel mount and remove top cover.

Locate LSB and USB chips (fig 1.1).



FIGURE 1.1

Remove the USB and LSB by gently prying loose with a flat-blade screwdriver. Begin prying the firmware ICs from left side switching from left to right side to extract the firmware IC evenly. This will prevent socket damage and bent firmware IC leads.

Replace the old LSB chip with the new chip labeled LSB. Repeat the same procedure for the MSB chip (fig 1.2).



FIGURE 1.2

Replace U3 (if supplied) with chip labeled U3 v2.0 in the same manner as the USB and LSB chips.

Confirm proper firmware orientation by replacing the firmware ICs with their notches matching the socket notches (notches point to the right). Reassemble unit by replacing keypad panel mount and top cover.

Restore power and test the unit.

## 17. Die Swell Laser Installation

### Installation of the PCI-DI024H board into the LabKARS computer:

**Caution: Installation or removal the PCI-DI024H board while power to the computer is on can damage the computer!**

- 1.) Turn off power to the computer, rheometer and all attached equipment.
- 2.) Remove the computer chassis cover.
- 3.) Select an unoccupied PCI accessory slot and remove the corresponding blank plate from the I/O connector panel.
- 4.) Insert and secure the board in the selected slot.
- 5.) Replace the computer cover.

### Installation of the Die Swell Laser to the LCR 7000 Series Rheometer:

- 1.) Swing the rheometer barrel swing arm out to ease installation of the Die Swell Laser.
- 2.) Attach the Die Swell Laser bracket by placing the (2) ¼-20 UNC Socket Head Cap Screws through the holes in the black hinge and square stainless steel spacer and into the tapped holes on the right collar of the Rheometer
- 3.) Tighten both Socket Head Cap Screws securely.
- 4.) Pivot the Die Swell Laser into operating position. If pivot tension is too low, the “Phillips” head screw on the back of the hinge can be tightened to provide the desired pivot tension.

Caution: Connecting or disconnecting the Die Swell Laser while computer power is on can damage the PCI-DI024H board and/or Die Swell Laser!

- 5.) Install the supplied cable to the Die Swell Laser and computer PCI board port.
- 6.) Restore power to computer, rheometer and all attached equipment.

### **Configuration of computer to recognize Die Swell Laser:**

- 1.) Turn computer “ON”
- 2.) When Windows recognizes new PCI hardware place InstaCal Computer Boards Inc. CD-ROM into drive.
- 3.) Follow instructions to complete driver installation.
- 4.) Run Setup.exe off of the CD-ROM to install InstaCal software.
- 5.) Launch InstaCal software via <Start Programs> <Computer Boards ><InstaCal> selection.
- 6.) Select “Installation” option.  
InstaCal should recognize new board automatically and save it as Board#1
- 7.) Exit program after board is detected.
- 8.) Open LabKARS folder using Windows Explorer. The LabKARS folder may be located in the “Program Files” folder.
- 9.) Scroll down to DPT.INI file and open it.
- 10.) Scroll down to [Machine] section.
- 11.) Change “LaserConnected=0” to “LaserConnected=1”
- 12.) Save DPT.INI file and exit Windows Explorer.
- 13.) Launch LabKARS.
- 14.) Laser Die Swell should appear between Temperature and Position  
If “Laser” icon is disabled go back to step #8  
If Laser data does not appear click on “Laser” icon and verify that board type is correct. Should be PCI type.

---

# Safety

## 1. Use Gloves - Its Hot!

Gloves and a long sleeve shirt (or lab coat) are required to prevent burns. Dies die nut holders and piston rods become extremely hot. They are designed to transfer heat quickly to the sample being tested, and unfortunately this means they will transfer heat very quickly to you as well. Even brief contact with a hot item can cause a serious burn. The barrel swing arm and connecting lower barrel cover may also get fairly hot. However, at barrel temperatures lower than 350 °C these may 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 *DYNISCO POLYMER TEST* rheometer contains high voltages inside the rheometer base. DO NOT remove this cover unless you are instructed to do so by a *DYNISCO POLYMER TEST* representative or are experienced with high voltage devices. **Be sure the outlet used to power the rheometer is properly grounded.**

## 3. Calibration Thermometers use Mercury (Models 7000, 7001 & 7002)

If a thermometer is used to calibrate the temperature on the rheometer, it will contain about 8 grams of mercury. 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, they provide an accurate and effective means of calibration. Keep the thermometer in a safe place where 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 all 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 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 rheometer 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 rheometer can be brought to bear. When the down button (T) is pressed on the rheometer a 2 to 3 LB safety load limit on the plunger will stop the movement of the crosshead / plunger (this is true for models with Load Cell only). Any item between the cross head and the lower swing arm which does not affect the load reading will not be "seen" by the rheometer and will be crushed! The down button (T) should be used for routinely moving the crosshead down to do force calibrations or to check motor operation. The operator should be sure the area between the crosshead and the rheometer swing arm is clear prior to any rheometer movement. The large red **EMERGENCY STOP** button, when pressed, will stop any rheometer 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. To have the crosshead return to the Park Position, power the rheometer down and then power it back up after about 1 second (confirm the emergency stop button is pulled out). Once powered up, the crosshead will return to the Park Position.

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

#### 5. Fumes from Materials

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

#### 6. Before Testing Safety Check

- Is the 120V power outlet properly grounded? (220 V Europe/Asia)
- Is the guide bushing in place?
- Are no objects on the swing arm (eg: beaker)?
- Is the exhaust hood or snorkel operational?
- Does the operator have safety glasses?
- Does the operator have high temp gloves?
- Is he wearing arm protection long sleeves or lab coat?
- Has the protective oil been wiped out of barrel?
- Are the safety shields installed?

## 7. Purging the rheometer

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 rheometer. Use rheometer-cleaning tool #2 (pictured in the Parts List Section of this manual) to run the patches up and down the full length of the barrel. The die and die holder nut should always be removed when cleaning the rheometer barrel. Repeat the process with fresh patches until the patches come out clean.

Turn the rheometer power switch “ON”. If you hear a continuous beeping sound, turn the rheometer off and be sure you have connected the load cell correctly, as described under the Getting Started section. If the rheometer's front panel lights up but there is no movement on the crosshead, shut the rheometer off, pull out the **EMERGENCY STOP** button, then turn the rheometer back on. Be sure the rheometer safety shield is closed before attempting to move the rheometer crosshead. If the rheometer cross head moves up then down and stops it is operating properly and you may continue. If the rheometer is not working correctly proceed to the trouble shooting section.

At 230° C purge the rheometer with Polyethylene (PE) or Polypropylene (PP). A purge is simply a charge of material run through the rheometer 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.

To set the rheometer up for a purge at 230°C:  
Press **EDIT** on the front panel of the rheometer  
Press the scroll left (<< --) or scroll right (-- >>) key until SET POINT appears  
Press **EDIT** and type 230  
Press **ENTER**  
Press **ESC** twice to return to the main display screen  
Press **SAVE** to keep changes to setup

## 8. Putting the Die(s) in the Barrel

The rheometer is shipped with a die nut (Tool #11, See Parts List Page \*\*) 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 it from seizing in the barrel. To switch or install the die, remove the die nut, drop the die into the die nut then screw the nut into the bottom of the barrel. Uses the die wrench to just snug the die nut into place. **DO NOT OVER TIGHTEN.** As the die heats it will tighten. See Page \*\*.

## 9. Loading the Sample

Turn the knob 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. Use the PYREX® beaker to scoop pellets from the sample you wish to run. Get the phenolic 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 one 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 contents, 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. The rheometer will completely pack the sample at the start of the test.

**Note:** Do Not 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 and hard to clean.

Add the remaining material and pack the sample 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. Be sure NOT to leave the beaker on the swing arm.

## 10. Starting the Purge

Place the plunger into the barrel, giving it a clockwise twist (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 when heating, especially if overfilling or degassing occurs) then close the swing arm. Tighten the knob to fix the swing arm in position. Let the material heat up for about three minutes or so. Time is not critical here; we merely want to give the material enough time to melt so we don't over force the rheometer trying to push it out cold. After about 3 minutes of time, press the SHIFT than T (down key) on the front panel of the rheometer to start the purge. The display will read **BARREL PURGE** and purge material from the barrel. The ram will continue to increase speed until all the material is purged. The plunger is always driven to the bottom limit switch setting, which is located about 3 mm (1/8 ") above where the piston and die will contact. To abort the Purge mode, press the QUIT key. Pressing the SHIFT than .6. (up key) will move the ram back to the Park Position.

## 11. Cleaning Up

Loosen the knob 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 and 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 reheat before the next test.

We recommend cleaning the barrel between all test runs. For materials that degrade clean both the barrel and the die immediately at the end of the run. You can clean the die by first running a cleaning drill bit up into the die while it is in the rheometer and still hot. This will make it much easier to get the drill bit in, for final cleaning, 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. Using a 100% 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 a 120° or 180° tapered entrance. Run the supplied drill bit into the die. Remove material that collects in the drill 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 will be the wait for the temperature to stabilize. Minimizing the time the die stays out of the

rheometer will help to increase the number of tests you can run. When the temperature on the front display is stable, you will hear a “beep” and the display will read “READY”. You can now begin your next test. Loading material will cause a small temperature change even after temperature set point is achieved. The melt time should provide ample time to get the temperature to set point before the first data point is collected.

Always leave the rheometer clean. If the rheometer is going to be idle at room temperature, for an extended period of time, you may wish to coat the barrel with a light rheometer oil to prevent rusting. The oil will need to be purged from the rheometer 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.*

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# Test Drive

## 1. Time Sweep Test

At this point the rheometer should be powered up and connected to the PC. If not, please review the previous chapters. We suggest that initial tests should be made using 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 cause a dramatic lowering of the viscosity of the material (e.g. PET, Nylon, PBT, PEEK and others). The first test you should perform on the rheometer is a standard thermal degradation run or a Time Sweep. A thermal degradation run is used to see how the viscosity of the material changes with time at a constant temperature. Typically, the longer the material is melted at a specific temperature, the more the material may degrade. This type of run is very simple. We ask the rheometer to move at only one speed: 35 mm/min. (ca. 1.4"/min.) and take measurements at positions in the barrel that are 8.7 mm apart (15 seconds per measurement). Additionally, we ask the rheometer to pause (stop plunger movement) for 15 seconds after each data point is taken.

## 2. Starting Lab KARS and setting up the rheometer.

If LabKARS isn't already running on your PC now is a good time to start it. Click on START/ LAB\_KARS or go to PROGRAMS/ LAB KARS/ LAB\_KARS. Once the program launches, click on the DOWN button to see if the crosshead starts to move. This will confirm a communication link between the PC and the rheometer. (If the proper link is established, continue to the Setup page of the LabKARS software.

**See the LabKARS section of this manual for more details on operating the LabKARS software.**

## 3. Performing the test

Load the barrel as described on Page 19 under the purging section. Do not press the SHIFT T (down key) to purge the rheometer of its contents as before. Instead, click on **RUN** button on the software's Rheometer Control Page. Fill in the Sample ID and Lot number for the materials you are testing and press the OK button.

The test will run automatically from here on. When the test is finished the rheometer will purge the remaining material automatically. The cross head will then move back up to the park position and the data will be transferred to the PC.

#### 4. The Second Test: A Rate Sweep

Clean the Rheometer, and set up for the second test. This test should be a standard shear rate test or a rate sweep test. It is called a rate sweep test because it shows how the viscosity of a material, flowing through a die, changes with the flow rate. For most polymers the faster the flow rate the lower the viscosity. In this simple run we ask the rheometer to move through 10 unique speeds. It makes measurements at specified positions in the barrel (more about how these are obtained later). We want the rheometer to perform this test as quickly as possible, so we set no time delay between each measurement point. In LabKARS, go to the Material List work sheet. Once in the Setup screen of LabKARS, click on the Control Polymer, Row 2 and press the Send button. This sends the setup details to the rheometer. The display on the rheometer should read “#2 Control Polymer”. Verify that this is indeed the correct program by clicking on the Test Conditions work sheet and reviewing Program #2 “Control Polymer”.

Load the barrel as described previously on Page 19. Once the plunger is in place, the swing arm is fully closed, and the plunger guide bushing is in place, click the **RUN** button on the Rheometer Control screen of LabKARS and fill in your test sample information and click on “OK” to start the test. The test will automatically acquire the data, purge the barrel of remaining material, move back to park position and send the data to the PC.

##### What's happening during the run:

When **RUN/OK** is pressed on the Rheometer Control Page, the rheometer starts an internal timer which keeps track of the total test time, the **Melt Pause Delay**, and the **Melt Time**. The rheometer will now adjust speed and reach the **start position** to achieve the allotted melt pause delay. The melt time and piston position are displayed. Once the melt pause delay expires the test starts.

#### 5. 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 slop. This can be very difficult to remove. We have tried several methods including solvents but found a tap and die set for the die holder nut or wire brushes work 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 entirely up into the barrel 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 excess material. The inside of the die nut can also be clogged with material. For short dies running Nylon, this is common. Included with your spare parts kit 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 also an excellent cleaning method.

Oven cleaner (Easy-Off®) sprayed onto parts and left overnight will do an excellent job of cleaning degraded material off the piston shaft, the outside of the die and on the die holder nut.

**Caution: Be careful not to breathe in oven cleaner vapors.**

If running at high temperatures (<300 °C) you may wish to coat the outside of the die and Pressure Transducer 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 during the test 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 affect your results. Some customers have used a brass wire

flywheel to clean piston tips between runs at high temperatures. Should you also choose 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 DYNISCO POLYMER TEST rheometers). Material leakage past the tip should also be less than 0.1 g.

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# LAB KARS

## LABKARS 7000 and 7001

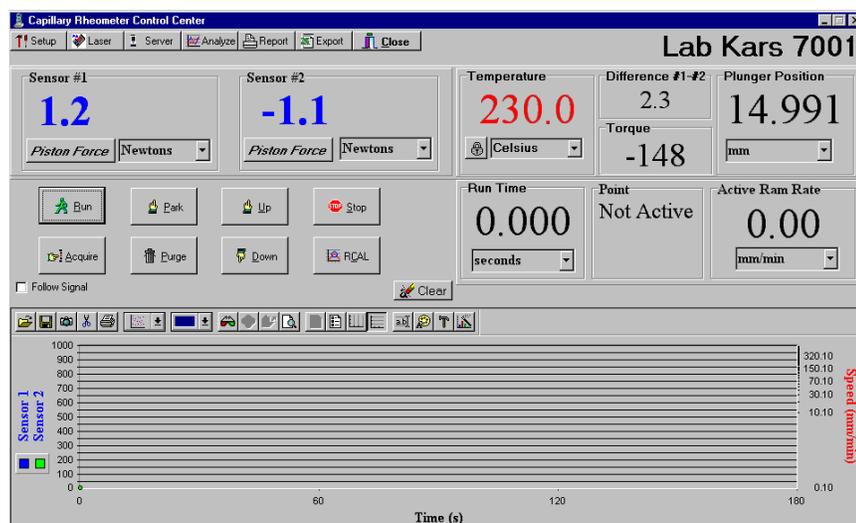
This version of software is aimed at users controlling operations and collecting data with the LCR7000 or LCR 7001 Capillary Rheometer.

## LABKARS 7002

This version of software is aimed at users controlling operations and collecting data with the two-barrel LCR7002 Capillary Rheometer.

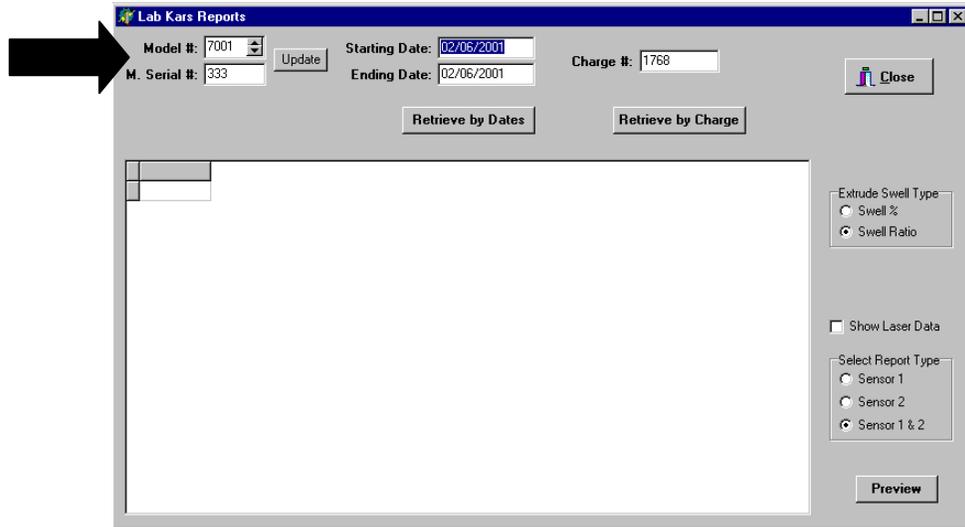
## LabKARS Operations

There are two versions of the LabKARS software designed to run the three different 7000 series rheometers (LCR 7000 & 7001, or LCR 7002). Be sure that you are working with the correct version of LabKARS necessary for operating the LCR model you are using to run your tests. Check the upper right hand screen of the Rheometer control page to find out what version you are working with.



If you need to change the version of LabKARS you are working with, click on the Reports Icon in the tool bar at the top of the page. In the upper Left hand corner of the Reports screen, change the Model Number and Serial Number to the rheometer you are working

with then click on the Update button to the right. Be sure to close down and Restart LabKARS to refresh the system after you have made your changes.

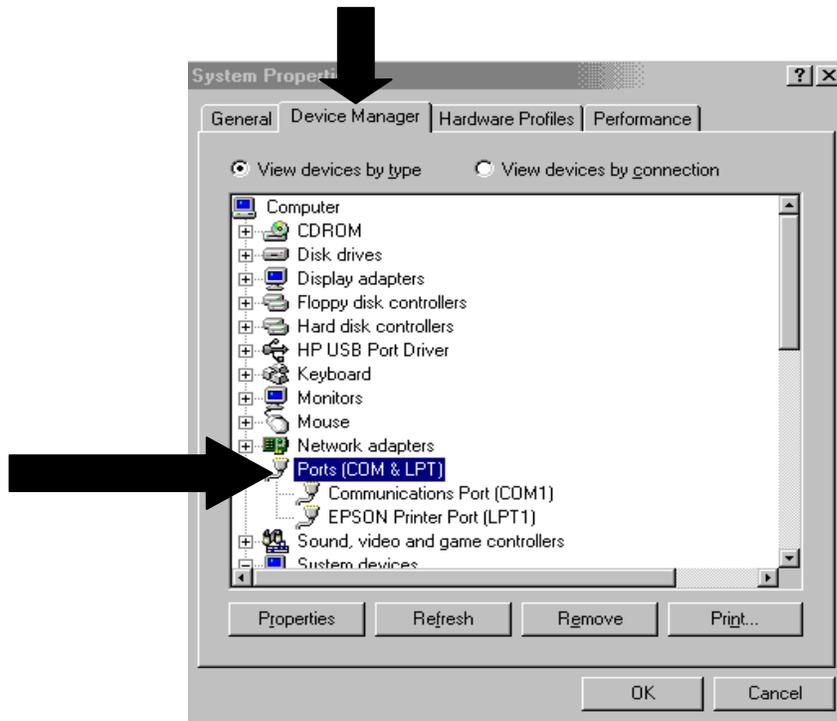


## PC Requirements & Port Settings

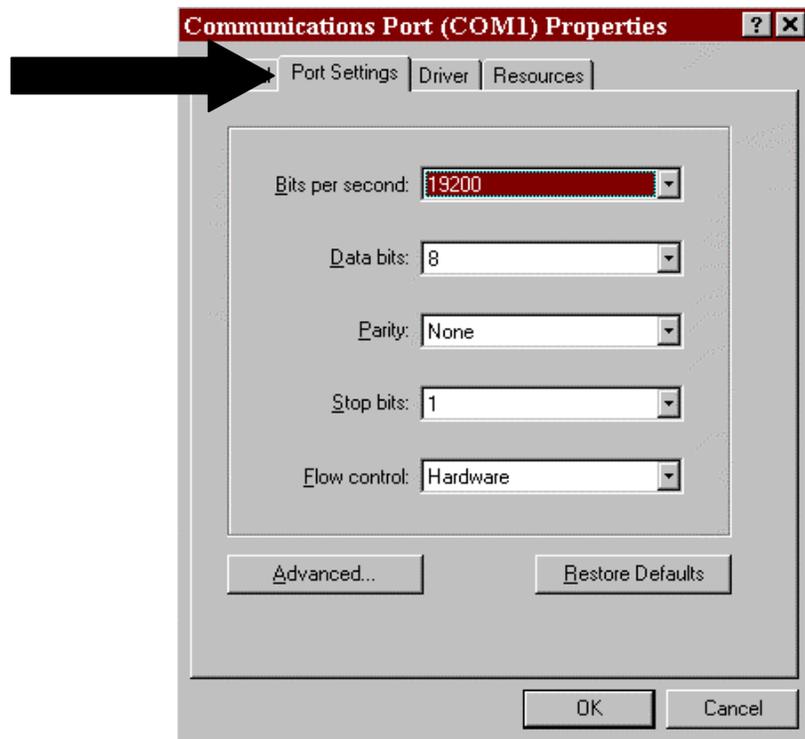
You need to be operating a minimum Pentium II™ Processor running at least 400mhz with Microsoft Windows 98™ or better to run LabKARS.

**Caution: Dynisco Polymer Test Recommends DELL Dimension PC's for the operation of the LabKARS software. Dynisco Polymer Test MAY NOT support the LabKARS software if installed on PC's of other manufacturers.**

It is important that the flow control setting for your Com. ports be set to Hardware. From Control Panel, go into System / Device Manager / Port settings / Com1.

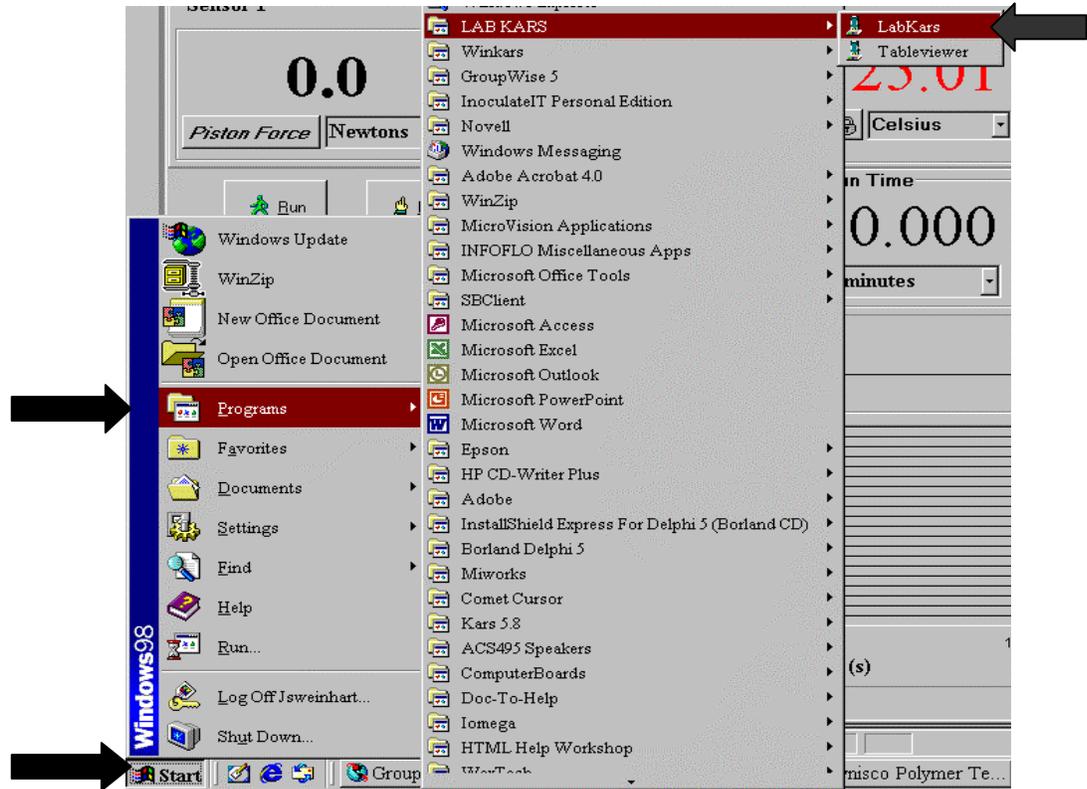


Port Settings, Bits per second is set to 19200. Data bit is set to eight (8). Parity is None. Stop Bits should be set to two (2). Flow Control verifies that the setting is set to Hardware.



## Starting LabKARS or LabKARS Set-Up

LabKARS should already be installed on your PC upon receipt. To start the program, click START and choose LabKARS or go to PROGRAMS and choose LAB\_KARS. The LabKARS workbook will open and the main Homepage will appear. If you are using an existing computer to run your rheometer you should have a compact disks containing the Install application for the LabKARS software. To install the software, insert the disk into your CD drive and type X:\ setup (where X:\ is your CD-Rom disk drive) at the WINDOWS™ Run prompt. Install shield™ will guide you through the installation process.



## Creating a LabKARS Icon

Open the Windows™ home page and Right click on the Home Page. Using the mouse move the cursor to "New Shortcuts" and double click. You will receive the "Browse" menu; double click on "All Files" chose "LabKARS" from your C:\ Drive. Double click on "NEXT" then "Finish". Your computer will then create a shortcut icon for running LabKARS.



## Security/Password Protection

Your LabKARS Security protection allows you to lock out certain functions of the LabKARS program from different users. If you wish to remove from certain operators the ability to make test changes, or if you wish to limit their freedom to move around the program, by assigning a lower access level to an operator you can control who can use certain functions of the program.



When you start LabKARS for the first time you will be prompted for a password. You can begin by using the default password “POLYTEST” to get started. The default password will only allow you to login TEN times before it will expire. Once you have logged in for the first time, click on the “Add User” button to add yourself and new operators.

You will be asked to enter your user name, password and security level. Different levels of security will allow only certain functions of the program to be accessible.



## LabKARS Security Access

**Security Level 1000** Complete access to all operations of the LabKARS software. Access to Passwords, user names, all options and functions of the program.

**Security Level 900 & 800** Not accessible to software server and user names and passwords, all other functions are open to Level 800 and 900 users.

**Security Level 700** Not a valid user.

**Security Level 600** Not Accessible to software server or users names and passwords, can change test temperatures, melt times, start positions and dies but not ram rates and or shear rates in test setups, can not change any test data or information in analyze.

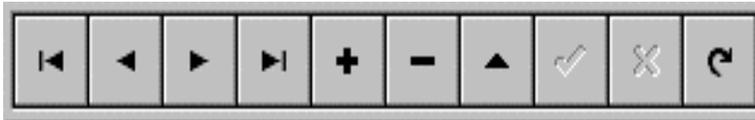
**Security Level 500** Not accessible to software server, laser or users names and passwords, can access LabKARS setup page and select different test setups but can not change any testing information in setup, can not change any test data or information in Analyze.

**Security Levels 400-200** Not accessible to any software server, setup, laser or analyze functions.

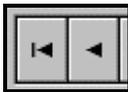
**Security Level 100** Not a valid user.

## Database Record Manipulation Keys

Common to most LabKARS screens are the record **Manipulation Keys** shown below.



These keys allow the user to navigate and edit data base tables



Page up to beginning of database table and page down one record, Respectively



Page down one record and page down to bottom of table, Respectively



Add, Delete and Edit a record, respectively



Cancel change of record or undo, refresh fields, refresh table

## Before The Start of any Run

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

Setup #	Program Name	Start Pos.	Temperature	Melt Time	Sensor1 ID	Die
1	LLDPE	100	190	360	LC-103N	CX300-33
2	7523 Control Grey PP	100	230	300	LC-103N	CX300-33
3	Polycarbonate	100	300	300	LC-103N	CX300-33

The term 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 of the load cell. All positions displayed and requested are therefore the distance away from the HOME position. When the rheometer 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 rheometer then moves to the PARK position set by you for sample loading. Since the HOME position is used as a reference for all other positions it is important not to interrupt the rheometer when it first starts up and finds the HOME position.

**RUN #:** This is the run number the rheometer includes with the data. Normally when a new test is started this value should automatically update to the next corresponding number. This value is automatically incremented every time the **RUN than OK** button is pressed. The run number is part of the extension name for file saved with LabKARS (e.g. SampleID.K01 or SampleID.D01). The automatic incrementing of the RUN # allows the file name to be unique.

**Point Position:** 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.

## Moving around LabKARS

Any time you enter LabKARS you will enter at the LabKARS Rheometer Control page. From the control page you can move freely in and out of any one of the operating pages of LabKARS by clicking on the icon of your choice.

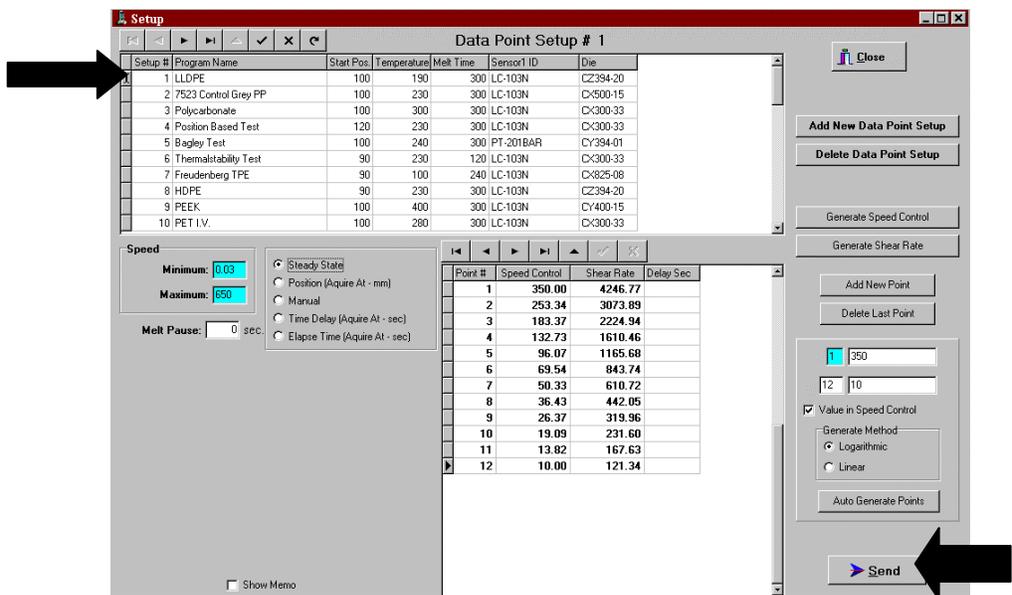


You can always return back to the Rheometer Control page at anytime simply by clicking on the Close icon in each of the screen.

**CAUTION: LabKARS is a Microsoft™ based operating package, DO NOT highlight and / or delete large blocks of data. Damage to the software may occur.**

## Set up Test Page

Enter the "Setup" page by clicking on the Setup icon located in the tool bar the LabKARS Rheometer Control page. The Setup page contains ten preset testing conditions for some standard industry materials. Choose the test for the material you are running or change the testing conditions to your liking. Highlight the test you wish to run and click on the "Send" icon to send the testing conditions to the rheometer and move directly into the Rheometer Control Screen to start the test. All preset test conditions are set up with standard shear rate curves and can be altered at any time.

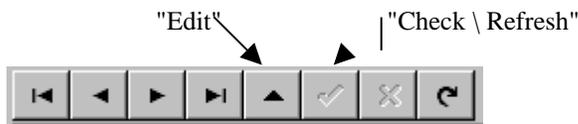


## Setting up a new test

To create a new test setup, click on the "Add New Data Point Setup" icon on your Setup page. Be sure to fill in all the blanks to complete your test setup. Be sure to include the Program Name, Melt Temperature, Melt time, Data Point Setup, Die or Dies used and Acquisition mode.

**Add New Data Point Setup**

You can make Program Name anything you wish as long as it's no more than twenty-five characters. You will have to activate the "Edit" mode of the LabKARS setup page to make changes to the test setup. Simply click on the "Up" arrow at the top of the page to edit. Be sure to click on the "Check" button to refresh the screen and save your changes.



There are four important control variables, which govern the start of a test: **Melt Pause**, **Start Position**, **Delay**, **Temperature**, **Melt Time**, **Sensor1 ID** and **Die size**.

Setup #	Program Name	Start Pos.	Temperature	Melt Time	Sensor1 ID	Die
1	LLDPE	100	190	360	LC-103N	CX300-33
2	7523 Control Grey PP	100	230	300	LC-103N	CX300-33
3	Polycarbonate	100	300	300	LC-103N	CX300-33

**Melt Time:** The **Melt Time** is the minimum amount of time the material will be exposed to test temperature in the barrel before the first rate begins. The front panel of the rheometer shows a count down time for the **Melt Time**, it shows MT XXX where XXX is the number of seconds remaining in the **Melt Time**.

**Park Position:** The **Park Position** is the position where the ram remains idle and the position to which the ram returns after completing a run or sample purge.

**Melt Pause Delay:** The **Melt Pause Delay** is the amount of time the plunger will stay idle at the start position. For low viscosity polymers this is set to zero or kept it to a minimum due to the fact that the polymer may run out of the barrel while the plunger is idle. For polymers that are higher in viscosity this is set close to the melt time to allow the polymer to “relax” prior to starting the test.

**Melt Pause:**  sec.

**Start Position:** The **Start Position** is the position where the first programmed ram rate begins.

## Data Point Setup

In the middle of the “Setup” screen you’ll see a table listing your Data Points. LabKARS will automatically create your Data Point Setup for you using a Linear or Logarithmic fit to Ram Rate or Shear Rate, or you may also create your Data Point Setup individually one point at a time.

Point No	Speed Control	Shear Rate	Delay Sec
1	300.00	9294.17	
2	159.18	4931.45	
3	84.46	2616.61	
4	44.81	1388.37	
5	23.78	736.66	
6	12.62	390.87	
7	6.69	207.39	
8	3.55	110.04	
9	1.88	58.39	
10	1.00	30.98	

To create a new set of data points for your test you can use the Auto Generate function of you Setup page. Type in the Ram Rate for your first data point in the box to the right of the "1" data point. In the box below the "1" type in how many data points you want to acquire during your test. The Right of your number of data points, type in the Ram Rate of your last data point. Select weather you would like a Logarithmic or Linear fit to your data points and click the Auto Generate icon.

If you want to setup you test profile according to Shear Rates, Click on the "Check" mark in the "Value in Speed Control" to uncheck the indicator box. Now you may type in the first Shear Rate to the last Shear Rate you want to acquire during your test.

**EXAMPLE:** This test will acquire fifteen data points at Ram Rates from 300mm/min to 0.5mm/min in a logarithmic method.

**EXAMPLE:** This test will acquire ten data points at Shear Rates from 10,000 1/s to 2 1/s in a linear method.

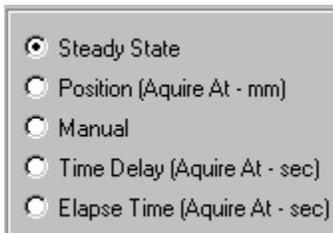
If you would like to setup your data points one at a time in your own profile. Delete all the existing data points in the data point setup buy using the "Delete Last Point" button.

Then add each point one by one using the "Add New Point" button until you are satisfied with the profile of your test.

If you are going to giving each point a Ram Rate you will have to click the "Generate Shear Rate" button to complete the data point information. If you are setting up your data points individually buy Shear Rate, you will have to click on "Generate Speed Control" for each point to complete the data point information.

## Data Acquisition Mode

You will need to specify the Mode of acquiring your data during the test. This is done by selecting the Mode of Acquisition on your Test Setup page. Your choices of acquisition are *Steady State*, *Position Based*, *Manual*, *Time Delay* and *Elapse Time*.



**Steady State/Manual** - In the *Steady State/ Manual* mode, real-time data is sent to the computer from the instrument. Noise is filtered from the data and when a steady state algorithm determines that the received data is steady, the computer grabs the average force values, stores it and proceeds to the next point. The Manual mode is also active at the same time with this setting, this gives the operator the option to press the "Acquire" icon to take the data reading and move onto the next point.

**Position:** 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. Positions will only be entered for ram rates other than zero.

**Manual Mode** allows the operator to decide when to acquire the viscosity reading of the sample. During the test, when the **End Point** key or "Acquire" icon is pressed the position and sample viscosity value at that instant is stored in a position value associated with that rate in the database. If **Manual Mode** is used and the operator does not click on Acquire," the rheometer will continue to push on the sample at the first ram rate until Acquire is pressed or the rheometer runs out of material in the barrel.

**Point Delay:** The **Point Delay** stops the 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.

## Start Position

You will need to set the test Start Position to complete your test setup procedure. **Start Position** is the position where the first programmed rate begins. In controlled stress runs it is where the first programmed load begins.

Set your Start Position depending on the amount of data points you will be collecting during your test. The more data points, the more barrel charge you'll need to complete your test.

Most test Start Positions will range from a barrel position of 90mm to 130mm. Dynisco Polymer Test recommends a barrel Start Position of 100mm as an average standard for your test.

## Die Selection

In the test setup "Edit" mode double click on the Die box in the Test Setup you wish to change. Find the Die size in submenu that you wish to use during your test. Double click on the die Stock number of the die to apply it to your Test Setup. If you know the Die Stock number you would like to use, simply type it in the Die box of your Test Setup

STOCK	DIAMETER	LENGTH	ConsAngle	Last/VerifiedDate	SpeedtoRate	ForcetoStress	WIDTH	TYPE
V160-03	0.406	1.22	30	9/18/01	181.1836	1.1633	0	0
X125-10	0.318	3.18	120		379.9696	0.3490		
X150-60	0.381	22.86	120	1/1/95	219.8898	0.0582	0	
X150-80	0.381	30.48	120	1/1/95	219.8898	0.0436	0	
X200-03	0.508	1.52	120		92.7660	1.1663		
X200-15	0.508	7.62	120		92.7660	0.2327		
X200-20	0.508	10.16	120	1/1/95	92.7660	0.1745	0	0
X200-40	0.508	20.00	120	1/1/95	92.7660	0.0886	0	0
X200-50	0.508	25.40	120	10/24/01	92.7660	0.0698	0	0
X200-60	0.51	30.48	120	6/6/00	92.7700	0.0600	0	0
X250-20	0.635	12.70	120		47.4962	0.1745		
X250-24	0.635	15.24	120	12/27/01	47.4962	0.1454	0	0
X300-01	0.762	0.76	120	1/1/95	27.4862	3.4899	0	
X300-10	0.762	7.62	120	5/29/01	27.4862	0.3490	0	0
X300-15	0.762	11.43	120	1/1/95	27.4862	0.2327	0	0
X300-20	0.762	15.24	120	1/1/95	27.4862	0.1745	0	0

If the die size you are looking for is not found in the preset die list of dies on the "Set up Page". Using the "+" button you may add a new line to the die list for a new die. Fill in the Dynisco STOCK number in the Stock number block. Click on the "Auto Calculate" button at the top of the page to automatically calculate the information of your new die. Once you have filled in all the information for your new die, be sure to click on the "Check \ Refresh" button to save your changes to the die list.

**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. Or Die Diameter:** This is the orifice diameter or die diameter. It is assumed the die is uniform in diameter through its entire length. For accurate viscosity measurements the die diameter is the most critical dimension. (Refer to appendix).

**Remember the smaller the orifice of the die the higher the shear rates observed during the test.**

## Generic Prerun Check List

- Immediately prior to pressing RUN
- Plunger in the rheometer?
- Guide Bushing in place?
- Swing arm fully closed?
- Area between crosshead and swing arm clear?
- Die Nut tight? Pressure transducer tight?
- PC (LabKARS) or printer ready?
- Prior to loading Sample
- Is material properly prepared (dried, mixed, check for contaminates)
- Rheometer on 20 minutes?

Correct Die in rheometer?

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.)

Are die diameters within spec. (passed G0-No Go gage, ASTM, ISO, DIN?)

Force calibration and R-Cal 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?

## Test Setup Example

The screenshot shows the 'Setup' window for 'Data Point Setup # 1'. A black arrow points to the 'Setup # 1' column header. The main table lists 10 test setups with columns for Setup #, Program Name, Start Pos, Temperature, Melt Time, Sensor1 ID, and Die. Below this, the 'Speed' section is configured for 'Steady State' mode with a 'Melt Pause' of 0 seconds. A 'Data Point Setup' table is visible, showing 12 points with columns for Point #, Speed Control, Shear Rate, and Delay Sec.

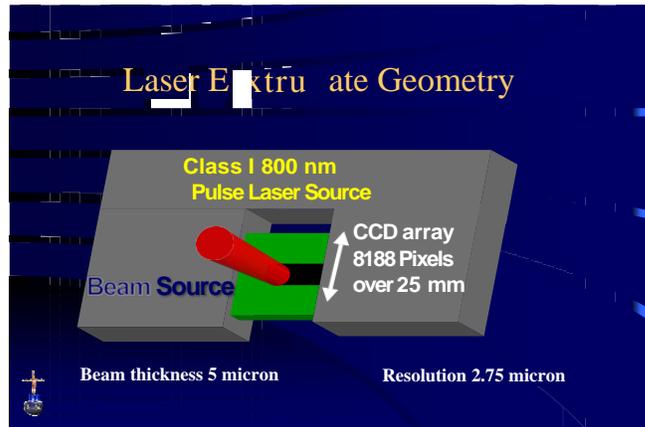
Setup #	Program Name	Start Pos	Temperature	Melt Time	Sensor1 ID	Die
1	LLDPE	100	190	300	LC-103N	CZ394-20
2	7523 Control Grey PP	100	230	300	LC-103N	CX500-15
3	Polycarbonate	100	300	300	LC-103N	CX300-33
4	Position Based Test	120	230	300	LC-103N	CX300-33
5	Bagley Test	100	240	300	PT-201BAR	CY394-01
6	Thermalstability Test	90	230	120	LC-103N	CX300-33
7	Freudenberg TPE	90	100	240	LC-103N	CX825-08
8	HDPE	90	230	300	LC-103N	CZ394-20
9	PEEK	100	400	300	LC-103N	CY400-15
10	PET I.V.	100	280	300	LC-103N	CX300-33

Point #	Speed Control	Shear Rate	Delay Sec
1	350.00	4246.77	
2	253.34	3073.89	
3	183.37	2224.94	
4	132.73	1610.46	
5	96.07	1165.68	
6	69.54	843.74	
7	50.33	610.72	
8	36.43	442.05	
9	26.37	319.96	
10	19.09	231.60	
11	13.82	167.63	
12	10.00	121.34	

This test is setup is Test Setup#1. It is for LLDPE with a Start Position in the rheometer barrel of 100mm. The test Temperature is 190°C. The Melt Time is 300 seconds and the piston will reach the start position 60 seconds BEFORE the end of the Melt Time (Melt Pause). Sensor #1 on the rheometer is a 10 Kn Load Cell and LabKARS is calculating the data using the dimensions of an Z394-20 Capillary Die. The rheometer will collect 12 data points from 4,246 1/s down to 121 1/s under Steady State mode with a 0 second delay between each data point.

# HAMA Laser Micrometer



## How it works:

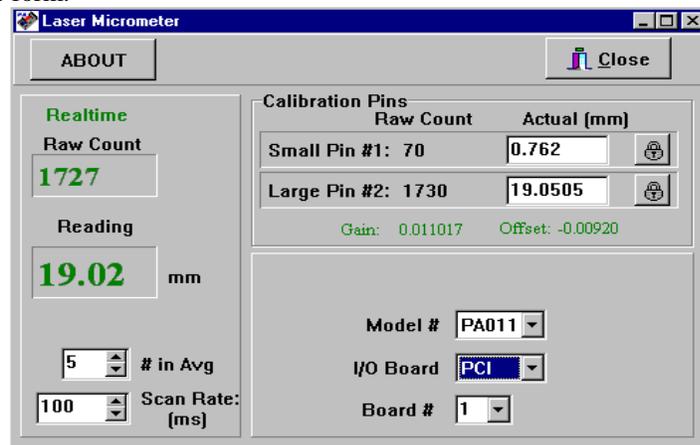
The laser sends out a UV pulse that is picked up by a series of diodes 8188 to be exact. When the light is blocked the diode registers no light and the side of an object from edge to edge can be determined.

## Starting up and testing the laser

If a HAMA LASER micrometer is attached and you wish to collect real time extrudate swelling measurement, this option should be set to ON. This option signals the PC to talk to the LASER micrometer and acquire data at the same time the load data is acquired.

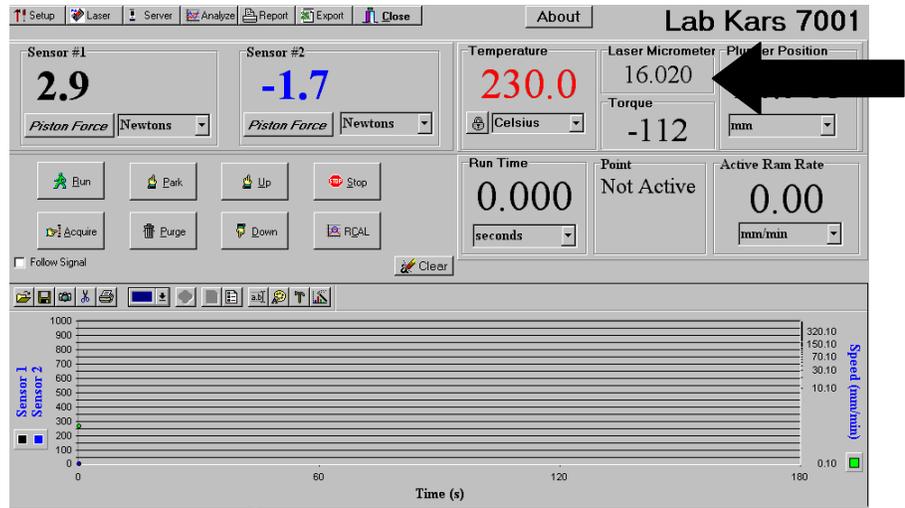


The program LCRComm.exe talks to the laser. When LCR Comm is running (you will see a “DYNISCO Communication” item on the windows task bar). Click on it to activate the form.

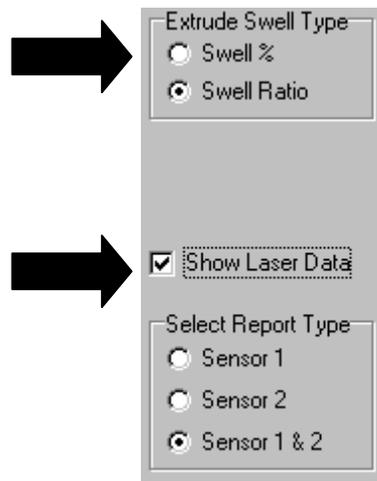


Once the Laser screen is open you should run through the Laser calibration procedure in the next section of this manual. When you have finished the calibration procedure leave the Laser Screen open and return to the Rheometer Control screen to run your test.

If you would like to monitor the Laser measurements during the test, double click on the differences box on the Rheometer Control Screen to display the actual readings during the test.



You can report your Laser die swell data in "Swell %" or "Swell Ratio" by going into LabKARS "Reports". Type the Charge number you would like to report in the "Charge #" box and click the "Retrieve by Charge" button. Selection the "Show Laser Data" box and "Exradate Swell Type" and click the "Preview" button to view the data and print the information.



The Green scaled reading is the extrudate diameter in millimeters. Stick something in the laser beam (it won't hurt your finger) and see if the scaled value changes. If it does the laser has been configured correctly. If no changes appear, go into **Control panel > System > Device Manager > (Data Acquisition)>** Chose the acquisition card for the laser then Resources **Input / Output Range**. The first number displayed is the hexadecimal address of the laser. This value **MUST** be identical to the I/O Card Address setting in the HAMA laser micrometer form shown above. If the value does not match, enter the correct value and then press the button next to it with the exclamation point surround by a circle.

If you can not find the acquisition card information under the Device Manager area, find and install the CD or diskettes PA /PAS terminal disk and install it on the machine, after the laser I/O card has been installed. You can also run this software to test the laser hardware and machine setup.

## HAMA Laser Calibration

Loosen the knob on the rheometer swing arm and swing the arm so that the Laser Micrometer is accessible

In LabKARS, on the RUN screen upper left-hand corner, click on the red numerals in “X-D: xxx.x mm” This opens the Laser Micrometer Calibration window in LabKARS.

Insert the Pin Gage with the smaller pin in the laser window. (Note that the Calibration Pin Gage has a serial number plate-it must face outwards when inserting the gage into the micrometer.)

Before proceeding, make certain that the “Diameter” mode is selected on the left side of the Calibration window.

The smaller pin is 0.762 mm +/- 0.001 mm in diameter. In the Calibration window upper right corner, click on the “Actual [mm]” box for Gage Pin #1 and enter “0.762” then click on the lock icon for that gage pin. The micrometer is now calibrated to the smaller pin.

Remove the Pin Gage and flip it around so that the larger pin is in the laser window.

The larger pin is 19.050 mm +/- 0.001 mm in diameter. Click on the “Actual [mm]” box for Gage Pin #2 and enter “19.050” then click on the lock icon for that gage pin.

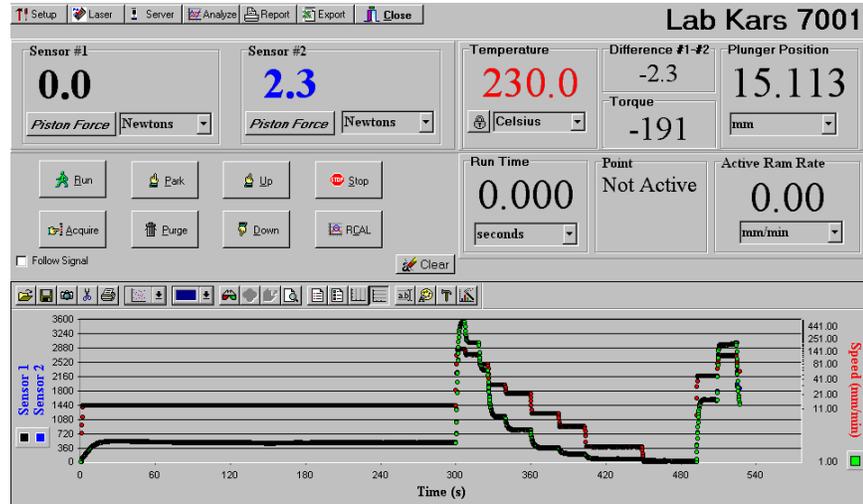
### **Caution: Clicking the lock icon for the large pin without first flipping the Pin Gage around to the large pin will cause the program to issue a “Fatal Error”!**

As a double check, remove the Pin Gage and flip it around so that the smaller pin is in the laser window. Observe the “Scaled Reading”. It should concur with the value in the “Actual [mm]” box for Gage Pin #1.

Laser Micrometer Calibration is now complete. Close the calibration window and return to “RUN” mode.

## Rheometer Control Screen

The Rheometer Control screen is the run / test monitor screen for the rheometer. From this screen you can control the movement and operation of rheometer or simply monitor the real time data of the test.



## Control Buttons

The rheometer crossbar and test run can be controlled manually from the operation buttons at the top of the rheometer control page.

When your ready to start your test simply press the RUN button, you will be asked to enter a Run Sample, Lot Number, Operator ID and Material Name before starting the test.



**Park Position:** The *Park Position* is the position from which the ram remains idle and the position to which the ram returns after completing a run or sample purge. To move the crossbar to the Home Position, press the PARK button.



To take the current data point and move onto the next data point on either Steady State or Manual modes press the ACQUIRE button.



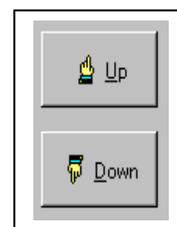
To abort a test and purge the barrel, press the PURGE button.



RCAL will rezero the rheometer sensors to a zero value for the LC Load Cell and or PT Pressure Transducer.



To manually move the crossbar Up or Down press the UP or DOWN buttons.



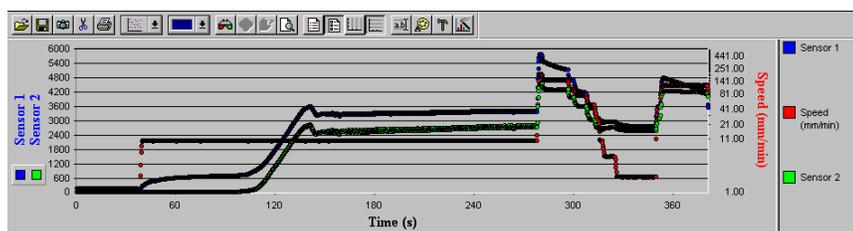
To stop the movement of the crossbar or the test, simply press the STOP button.



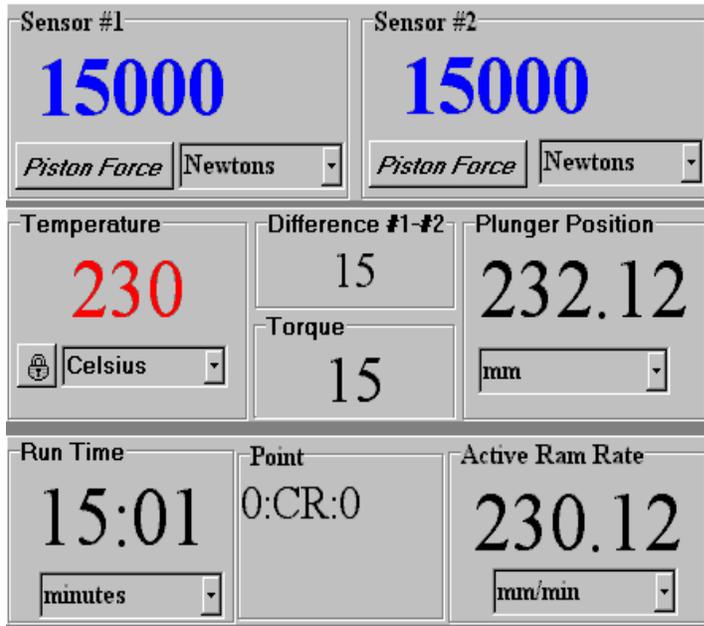
## Real-time Data during the Test

Real-time Test Data can be viewed on the Rheometer Control screen during the actual test.

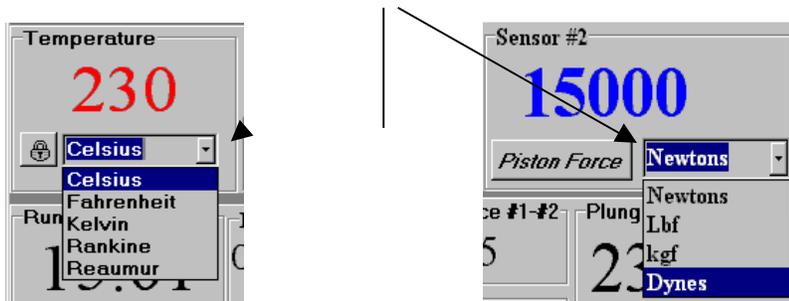
Real-time Data graphs will be developed on the Rheometer Control page as the test progresses. These graphs can be saved for future analysis or reports when the test is completed.



- *Sensor #1* is the actual force on the Load Cell.
- *Sensor #2* is the actual force on the Pressure Transducer.
- *Temperature* is the actual barrel temperature.
- *Plunger Position* is the actual position of the end of the Piston Tip.
- *Run Time* is the amount of time elapsed since the start of the run.
- *Active Ram Rate* is the actual speed at which the plunger is traveling.
- *Difference* is the difference between the Load Cell value and the Pressure Transducer value.
- *Torque* is the actual force applied to achieve Shear Rate.
- *Point* is the most recent point collected during the test.



The above mention values of the Control Screen can be changed to readout values comfortable to the operator or geographical location of the company. For example; the Temperature readout can be changed to read temperature in Celsius, Fahrenheit, Kelvin, Rankine or even Reaumur. This feature is also available for the Pressure, Time and Speed / Rates values of the Real Time Data.

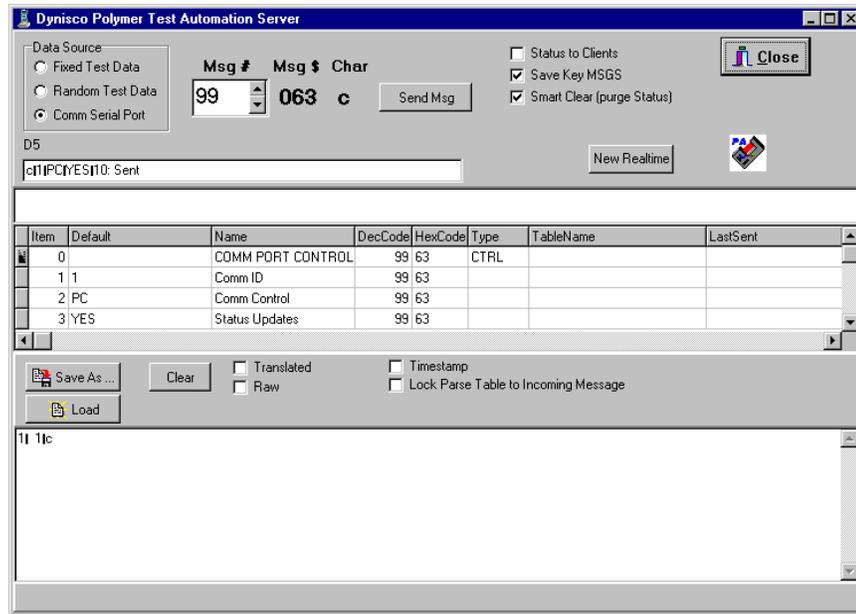


### Test Automation Server

The Test Automation Server is a diagnostic page used by Dynisco's software engineers to trouble shoot an / or maintain the LabKARS software.



**Do Not make any changes to this page at any time unless directed by authorized Dynisco personnel.** Simply keep the Automation Server page closed or minimized at all times to eliminate any confusion.



## Plotting and Analyzing your Data

Once a test run is completed you will want to analyze and Plot your data into a graph to easily analyze your test. You will need to enter the LabKARS database by clicking on the "Analyze" button at the top of you Rheometer Control screen.



### Plotting

- Browse the list of experiments to find the data you want to plot
- **Highlight** the charges you wish to plot and press the PLOT cloud. This will plot your charges on screen for you
- Select the printer icon to print the plot to your printer

If you select the PLOT cloud at the top of the screen, LabKARS will attempt to plot all of the data runs in the database.

**NOTE:** You can filter the data runs down to several experiments, and then select the plot button to merge them all into one plot.

### Merging

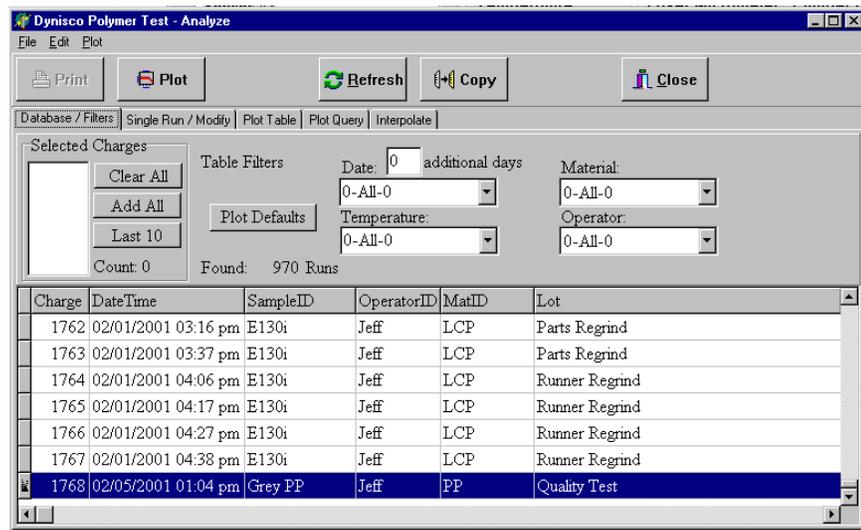
The easiest way to merge runs is to filter the data down to the experiments or charges you wish to merge. Once this is accomplished, select the Append cloud to merge them all together.

Another merging technique is as follows:

- Copy & Plot run files that you want to merge into. This will bring up & plot the data of the files you choose.

- Switch to Extract Options.
- Filter the data so that there is only one data run showing in the database list
- Select the append button and the data is overlaid on the first data run plot.
- Change the dates in the upper Left-hand corner to select runs form specific dates.

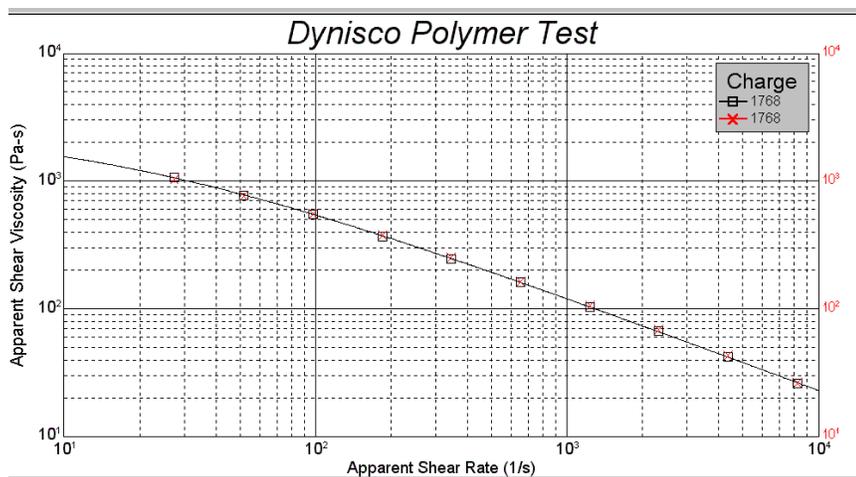
## Single Run Analyze / Database



Single Run/Modify gives the user the ability to modify any attribute about a run. Highlighted and framed data can be changed. More critical information like the temperature, packing forces and die attributes cannot be changed.

- Click on any editable field to bring up the database on that attribute.
- Select, add or delete a value for that attribute.
- Modify forces, position times and rate by clicking on them.
- Select plot to view the data for the particular run you are viewing.
- Modify, add or delete comments in the comment field by clicking in it.
- Advance to the next table, or experiment, by selecting the page tabs at the bottom of the screen.

## Plotting



Plotting is simply accomplished by clicking the plot button on the LabKARS Database page. If the Plot cloud is clicked from LabKARS Database screen, the entire set of displayed experiments is merged and plotted. From the plot screen, select either the printer icon or File-Print to output to your printing device.

### From Database/ Filters Option

- Browse the list of experiments to find the data you want to plot
- **Highlight** the charge you wish to plot. This will transfer the data to the LabKARS Data screen then press the PLOT cloud.
- Select the printer icon from the plot toolbar to print it to your printing device

If you select the PLOT button at the top of the screen, WinKARS will attempt to plot all of the data runs in the database.

**NOTE:** You can filter the data runs down to several experiment, and then select the plot button to merge them all into one plot.

### From Single Run/Modify

- Browse the list of experiments to find the data you want to plot using the



toolbar

- Select the printer icon to print it to your printing device

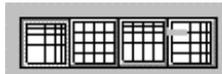
### Modifying Plots using the Plot



The plot toolbar provides the user with easy graph manipulation tools. Following are their explanations.



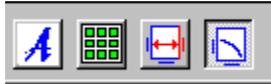
Print the displayed plot



Format plot as either Log Log, Lin Lin, Lin Log or Log Lin, respectively

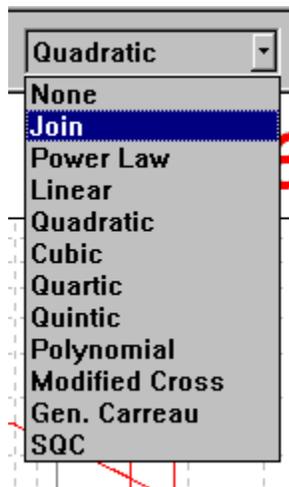


Enable or disable Rabinowitsch correction

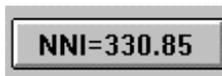


Add annotation, move legend, swap the graph axes

Set the curve type, Select from eight different curve types



Show curve fitting parameters and display database of data being plotted.



Perform Non-Newtonian Index calculations on the experiment(s)

- Click on any title or legend to change its parameter
- Click on the title to add a new title to the chart
- Click on the axis label to select new axis labels
- Click on the legend and select what data you want to plot.

To add annotations to the chart, click on the Annotate Plot icon in the toolbar of the plot window. You will be prompted for a text entry. Enter the desired annotation and click on the OK button. To move the annotation, click and drag it.

## Appending and Overlaying Plots

The easiest way to merge runs is to filter the data down to a limited number of experiments or charges. Once this is accomplished, select the Append cloud in the LabKARS Database screen to merge them all together.

## Copying Plots

Plots can be copied to the clipboard by selecting Edit-Copy plot to clipboard from the plot windows. Once the plot is in the clipboard, it can be pasted into any OLE compliant Windows software package.

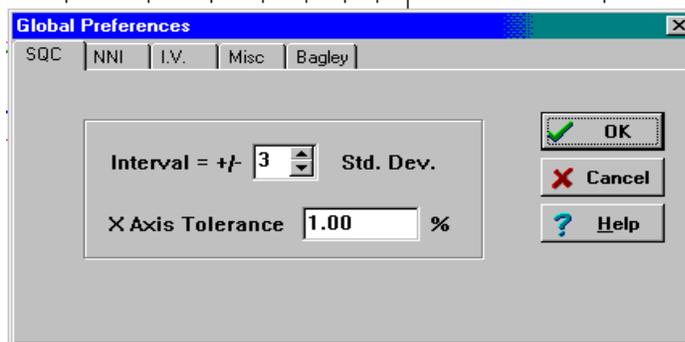
Plots can also be cloned. From the plot windows, select File-Clone. This will open up a new plot display window and create a copy of the original plot in it.

## Saving Plots

From the plot display windows, select File-Save. You will be prompted for a file name with a .dB extension.

## Global Preferences (SQC, NNI, I.V. and Bagley)

From a plot display, click on *File* and *Global Preferences*. A screen will display which looks like the following:



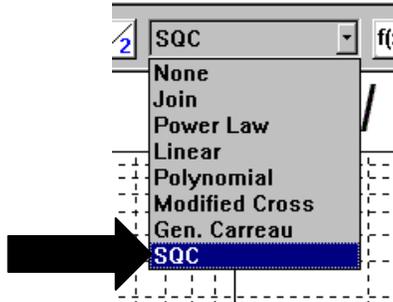
## SQC Plots

SQC allows the user to reject data not within predetermined limits. An interval of standard deviation acts as a filtering device for data.

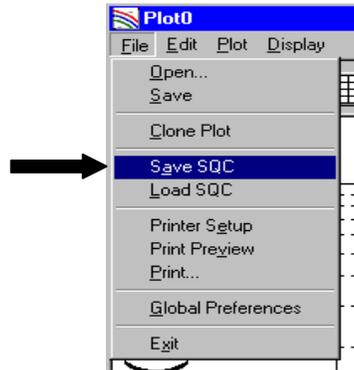
An X axis tolerance disregards data that is not within a certain percentage of the known good data or other data points. For example, a multiple plot which versus time on the X axis will not consider data +/- 1% of the average of the other points. Therefore, if a data point took 1 more minute to obtain than the other data points, it will be ignored.

**To create a new SQC for your favorite material** simply run three or more consecutive runs of your sample. Plot as many good runs of your sample as you can. The more data runs plotted the better, this will give you a true upper and lower control limit for your materials performance.

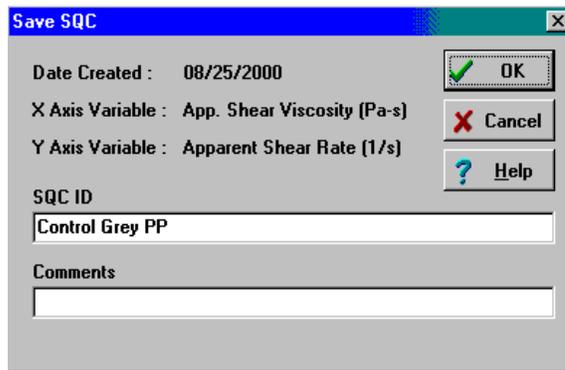
From the plot curve menu, select the "SQC" plot to give you upper and lower limits of your data.



Go to the "File" menu and click on the "Save SQC" to save the data setting of your plot.



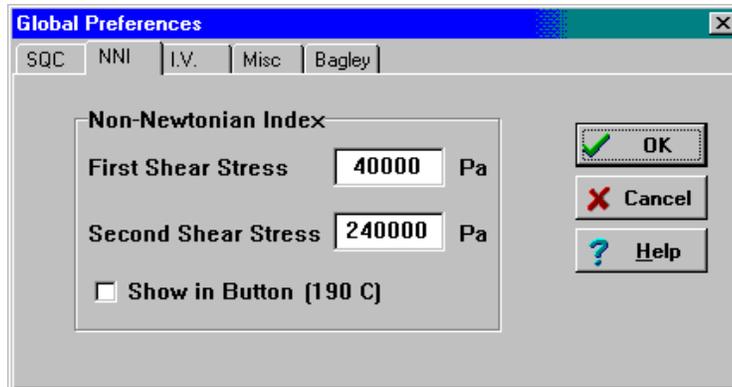
You will be asked to give your SQC a name. Try to include some of the test settings such as Test Temperature, Die size, and Material name and number in the SQC name. Add any notes or comments regarding your data tests, such as Drying conditions, Lot numbers, and maybe supplier and date of manufacturing so you will remember these conditions later.



Now the next time you test your material you can apply the SQC to your data plot to be sure your material and rheometer are still in specification. Use your SQC option in Global Preferences to adjust your standard deviation of your control limits.

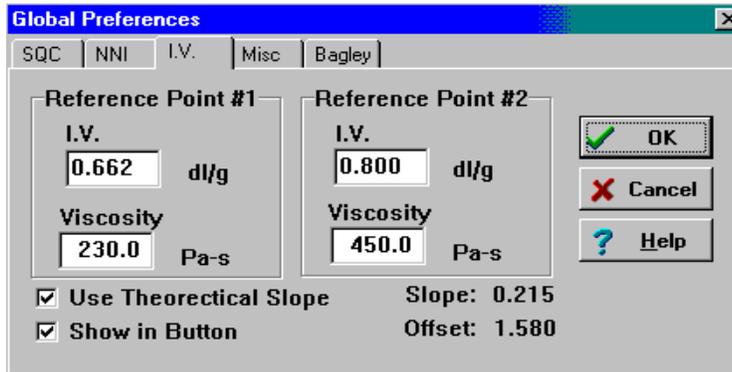
## NNI

Non-Newtonian Index is a 2nd order polynomial fit to the log shear stress versus the log shear rate data. Shear rates at the specific stresses,  $\sigma_1$  and  $\sigma_2$ , are determined from the fit. This function is valid only at temperatures of 190°C and 250°C.



## I.V. Preferences

In order to calculate intrinsic viscosity a sample of **known I.V.** must be run with the same chemical backbone (i.e. same type, homopolymer or copolymer) as the unknown I.V. sample. After the rheometer run an ETA0 value (limiting viscosity at low shear rates is calculated from a modified cross fit of the results this value is entered into the next parameter).



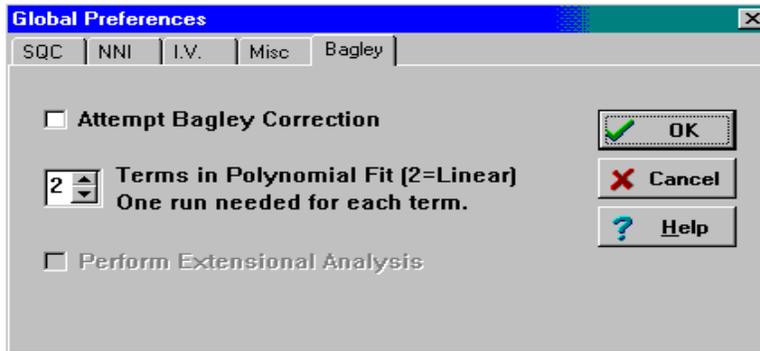
The I.V. screen allows the user to enter the known I.V. values at different viscosity. If you select Use Theoretical Slope, the first point is the I.V. that you enter, and only 1 I.V. test is performed. When Use Theoretical Slope is not selected, a slope is calculated and a curve is generated to predict the Intrinsic Viscosity.

## Misc.

Use miscellaneous to select the curve fit you wish to use.

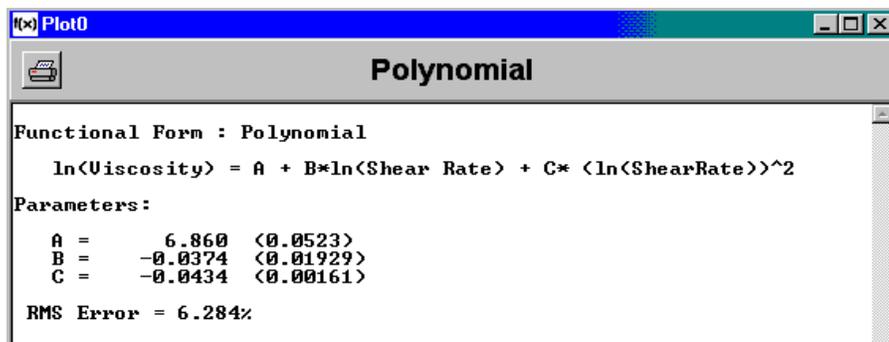
## Bagley Correction

When material is run at the same temperature with several capillaries the static (with respect to changing die length) pressures in the system can be removed from the calculation of viscosity producing more accurate shear viscosity values. K.A.R.S. automatically does the correction when sufficient data exists.



## Plot RMS Error

The RMS or root mean square of the data is considered as well. The lower the RMS the tighter the fit of a data series. A lower number here means more discriminating data.

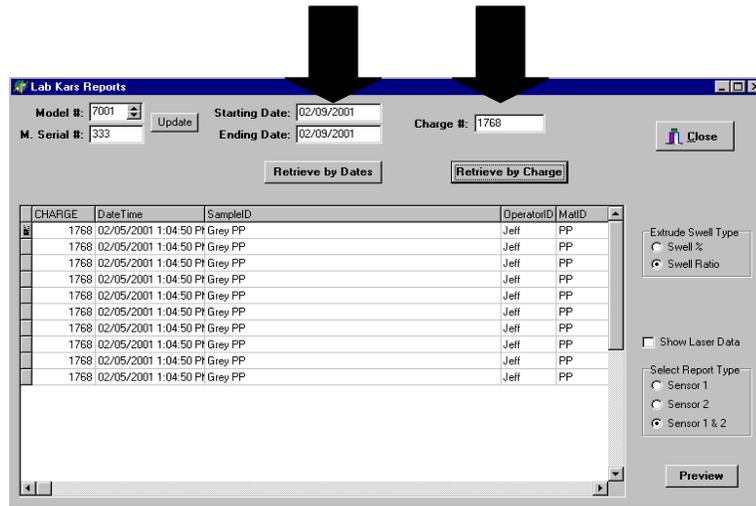


## LabKARS Reports

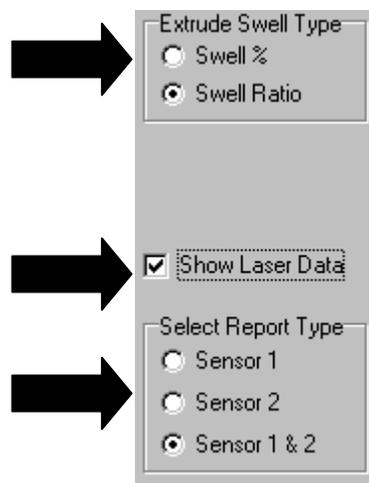
LabKARS Reports page is designed to condense the raw data of your test into a one-page sheet suitable for reports and / or material data files. Click on the "Reports" icon in the Rheometer Control tool bar to enter the reports page.



Once you have entered the "Reports" page you can retrieve test data from the database either by Date or by Charge Number. Simple type in the Starting Date and Ending Date of the tests you wish to report. Click the "Retrieve by Dates" button to extract all the test data from the database for those dates. If you know exactly what "Charge Number" you would like to report, type the charge number in the "Charge #" box and click the "Retrieve by Charge" button.



Before going to the "Preview" screen to print your data reports, verify your Reports defaults for displaying your Hama Laser data and sensor report information. You can report your data as "Sensor #1" information only "Sensor #2" information only, or "Sensor 1 & 2" together by selecting one of the "Select Report Type" options. You can also report your Laser die swell data in "Swell %" or "Swell Ratio" by selection the "Show Laser Data" box and "Extradate Swell Type".



Once you have retrieved the Test data that you would like to report, Click on the "Preview" button to view the data in report form and print the report.

#### Capillary Rheometry Results

Date: 02/05/2001 1:04:50 PM		Workorder: 1768		Start Position: 100 mm							
Operator: Jeff		Sample ID: Grey PP		Melt Time: 300 s							
Die: CX000-33		Temperature: 230 C		RCAL 1: 99.87 %							
Sensor 1: LC-103N		Points: 10		RCAL 2: 100 %							
Sensor 2: PT-142BAR-A		Lot #: Quality Test		Machine #, Serial #: 7001, 333							
Point #	Force (N)	Force (N)	Position (mm)	RamRate (mm/min)	Time (min)	Shear Stress 1 (kPa)	Shear Stress 2 (kPa)	Shear Rate 1 (1/s)	Shear Viscosity1 (Pa-s)	Shear Viscosity2 (Pa-s)	Extrudate Swell Ratio
1	2044	2093	120.4	900	5.07	216.51	221.72	6245.87	26.26	26.89	1.58
2	1752	1805	138.4	159.2	5.18	185.63	191.20	4375.20	42.43	43.70	1.58
3	1474	1520	147.7	84.5	5.29	156.17	161.05	2321.48	67.27	69.37	1.55
4	1224	1259	158.5	44.8	5.53	129.66	133.37	1231.77	105.26	108.28	1.37
5	1009	1031	163.8	23.8	5.76	106.95	109.26	653.57	163.63	167.17	1.40
6	817	836	171.1	12.6	6.34	86.60	88.62	346.78	249.74	255.56	1.34
7	651	664	176.4	6.7	7.13	68.95	70.39	184.00	374.71	382.53	1.34
8	512	518	178.7	3.6	7.79	54.28	54.91	97.63	596.01	562.41	1.29
9	385	384	180.4	1.9	8.68	40.74	40.67	51.80	786.51	785.12	1.14
10	281	274	181.2	1	9.48	29.76	28.99	27.49	1082.81	1054.86	.25

Once you have viewed your data and you are satisfied with the results you can use the Reports buttons at the top of the page to move from one Charge Number to another. You can send your Data Sheet to the printer for a file copy. You can Zoom In and Out on your report, you can save the report to another file or directory.



## Exporting Data to Excel

At times you may want to export your test information into Microsoft Excel for plotting or graphing the data to your own reports of quality system. By clicking on the "Export" button on the Rheometer Tool Bar, you can select Test Data or Charge Numbers and export that tests information to Excel.



Once you are in the LabKARS "Export" page you will see the contents of your database. Simply select the Charge Number you want to export to Excel by clicking on it or typing the Charge Number in the "Charge #" box at the bottom of the screen and clicking on the "Locate" button.

Charge	DateTime	SampleID	OperatorID	MatID	Lot	Die	Die2	Temperature	Di
1755	02/01/2001 01:07	ppr E130i	Jeff	LCP	Virgin Sample	C2394-20	C2394-20	350	C2
1756	02/01/2001 01:20	ppr E130i	Jeff	LCP	Virgin Sample	C2394-20	C2394-20	350	C2
1757	02/01/2001 01:43	ppr E130i	Jeff	LCP	Virgin Sample	C2394-20	C2394-20	350	C2
1758	02/01/2001 01:52	ppr E130i	Jeff	LCP	Virgin Sample	C2394-20	C2394-20	350	C2
1759	02/01/2001 02:07	ppr E130i	Jeff	LCP	Virgin Sample	C2394-20	C2394-20	350	C2
1760	02/01/2001 02:46	ppr E130i	Jeff	LCP	Parts Regrind	C2394-20	C2394-20	350	C2
1761	02/01/2001 03:06	ppr E130i	Jeff	LCP	Parts Regrind	C2394-20	C2394-20	350	C2
1762	02/01/2001 03:16	ppr E130i	Jeff	LCP	Parts Regrind	C2394-20	C2394-20	350	C2
1763	02/01/2001 03:37	ppr E130i	Jeff	LCP	Parts Regrind	C2394-20	C2394-20	350	C2
1764	02/01/2001 04:06	ppr E130i	Jeff	LCP	Runner Regrind	C2394-20	C2394-20	350	C2
1765	02/01/2001 04:17	ppr E130i	Jeff	LCP	Runner Regrind	C2394-20	C2394-20	350	C2
1766	02/01/2001 04:27	ppr E130i	Jeff	LCP	Runner Regrind	C2394-20	C2394-20	350	C2
1767	02/01/2001 04:38	ppr E130i	Jeff	LCP	Runner Regrind	C2394-20	C2394-20	350	C2
1768	02/05/2001 01:04	ppr Grey PP	Jeff	PP	Quality Test	CX300-33	CX300-33	230	C2
1769	02/06/2001 10:21	ppr Grey PP	Jeff	PP	Quality Test	CX300-33	CX300-33	230	C2

Starting Row #: 10      Charge #: 1769      Locate

You will have to set the "Starting Row #" for Excel so the software will know where to locate the data in the Excel spread sheet. If you Export more than one Charge Number to Excel and don't adjust the Starting Row #, the program will overlay the second charge over the first and you will not be able to view your data.

It is good practice to back-up your database frequently.

## C. Selecting Load Cell or Pressure Transducer

If you need to change the pressure transducer or load cell to conduct your test, you will have to let the rheometer know what size PT or Load Cell is being used. If you are not testing using a pressure transducer, you will need to confirm the pressure transducer port is plugged and the PT Sensor is turned OFF. If a PT is not being used to test, apply a light coat of anti-seize to the threads to the PT Port Plug and insert into the PT port. Only hand-tighten the Port Plug until the plug warms up to the barrel temperature. Once the PT or the plug has had time to heat, snug it tight with a wrench.

To turn off a sensor or to select another pressure transducer or load cell. Press the "SETUP" button on the front control panel of the rheometer. Use the Smart Keys on the rheometer display to move Left or Right through the menus until the sensor you wish to change or turn off (Sensor #1, Sensor #2 or both) is shown on the control panel display. Press the "Edit" Smart Key and scroll through the sensor selections until the display reads "Device Not Entered" or you locate the sensor device you wish to use, and press the "Select" smart key. The sensor that you selected is now active.

### To Select a Transducer Step by Step

1. Press the "SETUP" key on the front panel of the rheometer
2. Scroll Left or Right through the Setup menus using the Arrow Smart Keys.
3. Locate setup menu "Sen Chanel#1" or "Sen Chanel #2" NOTE: Sensor #1 will always be the Load Cell on the LCR 7000 and 7001.
4. When you have located the sensor you wish to change press the "Edit" Smart Key on the rheometer display
5. Using the Arrow Smart Keys scroll Left or Right to locate the proper sensor you wish to install.
6. Press the "SELECT" Smart Key to activate the sensor.

The pressure transducer or Load Cell you selected is now active.

1. Insures stability prior to Rate Sweep

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

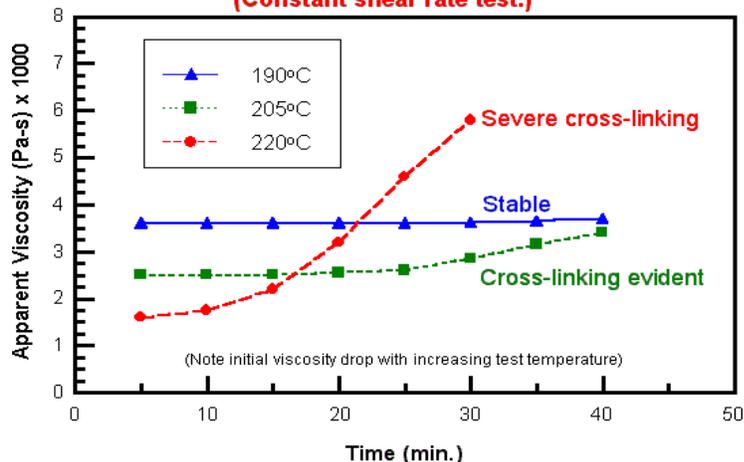
2. PVC Example of a Time Sweep.

PVC is a particularly interesting material to run since, when its stabilizer package fails, the material cross-links fairly quickly and shows a marked increase in viscosity. Comparing runs made at different temperatures gives insight into the stable lifetime of a material in an extruder at various temperatures.

## D. Time Sweep

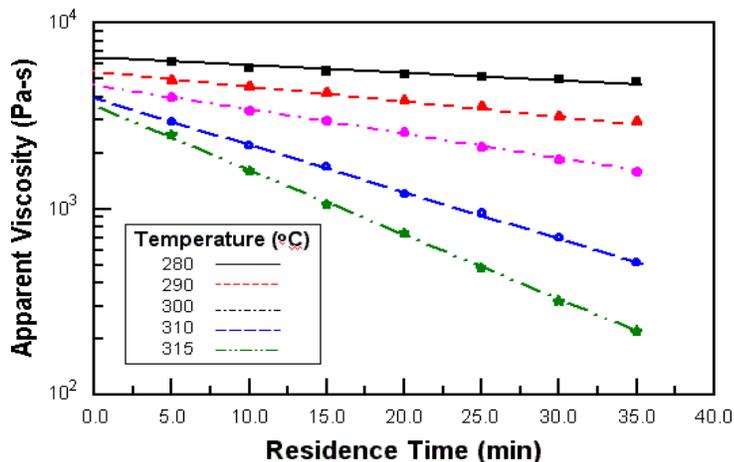
## Cross-Linking of PVC

(Constant shear rate test.)



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.

## Nylon 6/6 Thermal Degradation

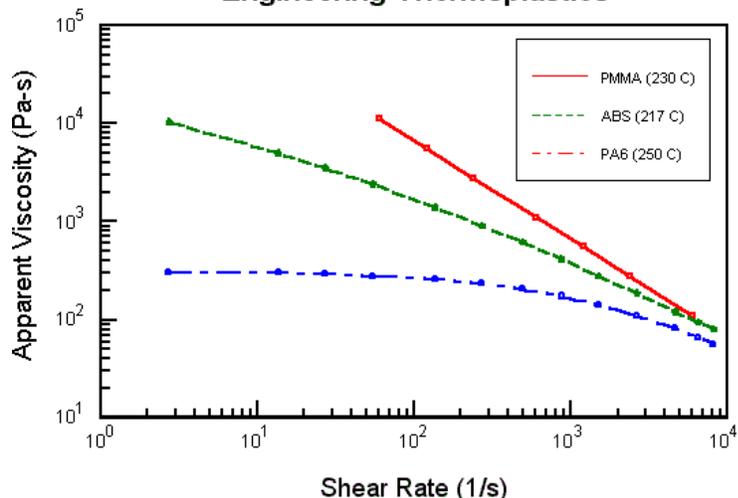


### E. Shear Rate Sweep

A shear rate sweep is a test where variable speeds (mm/min) or shear rates (sec<sup>-1</sup>) are applied to the polymer. In doing so we can assess the rheological behavior of the material at various shear rates.

1. PVC, PP, PET Example Rate Sweep Data

### Viscosity Curves for Some Engineering Thermoplastics



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

#### F. Shear Stress Sweep

Constant Stress vs. Constant Rate

Constant stress tests on degrading material tested in the non-Newtonian region have viscosity vs. time curves, which fall faster than the corresponding viscosity vs. time data, collected at constant rate.

#### G. Combining Time and Rate Runs

One of the difficulties in acquiring accurate viscosity vs. rate data can arise from material degradation during the test. ASTM D3835 states that when a material is unstable (degrades >5% from the initial value) 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. However, there are other methods.

If only three or four unique rates are run during a test, some rates 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.

Constant shear rate vs. time runs may be collected at a number of different shear rates for each charge of material then merged together. Data, taken from these graphs at constant time, allow isochronal (constant time) curves of viscosity vs. shear rate to be obtained. Extrapolation to viscosity vs. rate curves at zero time can also be accomplished with this technique. How the viscosities vs. rate curves are affected by time at constant temperature can then be evaluated.

## **H. Accuracy and Reproducibility**

In our in-house lab we have found a single rheometer 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 in the same range as found by 17 labs participating in the D3835 round robin testing in 1991.<sup>1</sup> 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 deviations the fitted curve is away from the typical standard deviation of a point.

### ***Precision***

The rheometer-to-rheometer reproducibility (for all capillary rheometers in general, according to ASTM) is significantly higher than that for a single instrument. 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 for a graphical display of round robin results.

### ***Accuracy***

The PRT'S on DYNISCO POLYMER TEST Inc. rheometers 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). An accurate die diameter measurement, particularly at very small die diameters, is the most critical item for achieving accurate results. 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 checked using hand or laser micrometers. Force cells are calibrated using a calibration cell, which is calibrated with dead weights that are NIST traceable.

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 robin testing. 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.

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## Formulas Used in Data Analysis

### Apparent Shear Rate Calculation:

#### Formula for Shear Rate

$$\dot{\gamma} = \frac{4R_b^2 S_p}{R_c^3} = \frac{4R_b^2 (\text{Distance/Time})}{R_c^3} = \frac{8D_b^2 (\text{Distance})}{D_c^3 (\text{Time})}$$

Typical Units are 1/sec or sec<sup>-1</sup>.

Where:

R<sub>b</sub> = radius of the barrel

R<sub>c</sub> = radius of the capillary (die)

S<sub>p</sub> = speed of the plunger

D<sub>b</sub> = diameter of the barrel

D<sub>c</sub> = diameter of the capillary (die)

Example: When the plunger moves at a rate of 20 mm/min (or 20/60 mm/sec), the Die diameter is 1mm, and the Barrel diameter is 9.545 mm

$$\text{then: } \dot{\gamma} = \frac{8(9.545)^2(20)}{(1)^3(60)} = 243 \text{ 1/sec.}$$

Note all length units cancel out.

### Apparent Shear Stress Calculation:

#### Formula for Shear Stress

$$\tau = \frac{F_p R_c}{2 \pi R_b^2 L_c} = \frac{F_p D_c}{\pi D_b^2 L_c}$$

Typical Units are Pascals (Pa) or megaPascals (Mpa).

Where:

R<sub>b</sub> = radius of the barrel

R<sub>c</sub> = radius of the capillary (die)

D<sub>B</sub> = diameter of the barrel

D<sub>c</sub> = diameter of the capillary (die)

F<sub>p</sub> = the load on the plunger (Ram)

Example: For a Ram load of 800 Newtons, die of diameter 1.0 mm and length 20 mm.

$$\text{Example: } \tau = \frac{800 (1.0)}{\pi (9.545)^2 (20)} = 0.1398 \left( \frac{N}{mm^2} \right) = 139800 \left( \frac{N}{m^2} \right)$$

$$= 139800 \text{ Pa}$$

## Apparent Shear Viscosity

### Formula for Shear Viscosity

$$\eta = \frac{\tau}{\dot{\gamma}} = \frac{D_c^4 F}{8\pi D_b^4 S_p L} = \frac{D_c^4 F (\text{Time})}{8\pi D_b^4 L (\text{Distance})}$$

Typical Units Pascal seconds (Pa-sec) or Poise (10poise=1 Pa-sec).

Where:

$S_p$  = speed of the plunger

$D_b$  = diameter of the barrel

$D_c$  = diameter of the capillary (die)

Example:

$$\eta = \frac{(1.0)^4 (800)(60)}{8\pi (9.545)^4 (20)(20)} = 575 \text{ Pa - sec}$$

## Volumetric Flow Rate Calculation:

### Formula for Volumetric Flow Rate

$$Q = \pi R_b^2 S_p = \frac{\pi D_b^2 \text{Distance}}{4 \text{Time}}$$

Where:

$R_b$  = radius of the barrel

$R_c$  = radius of the capillary (die)

$S_p$  = the speed of the plunger

$D_b$  = diameter of the barrel

$D_c$  = diameter of the capillary (die). (Assumes no leakage.)

Example: If the plunger moves a distance of 10 mm in 4.5 seconds

$$\text{therefore: } Q = \frac{\pi (9.545)^2 (10)}{4(4.5)} = 159 \text{ mm}^3/\text{sec or } 9.541 \text{ cm}^3/\text{min}$$

**Mass Flow Rate  
Calculation:**

**Formula for Mass Flow Rate**

$$MFR = \frac{\text{Mass of material collected}}{\text{Time Period}}$$

Example: If 22.31 grams of material is collected over 120 seconds then:

$$MFR = \frac{22.31}{120} = 0.186 \text{ g/sec or } 11.16 \text{ g/min or } 111.6 \text{ g/(10 minutes)}$$

NOTE: Melt Flow (also MFR) ASTM D1238 is a mass flow rate measurement made at specific conditions of stress, die dimensions and temperature, which differ from material to material. It typically uses units of g/(10 minutes).

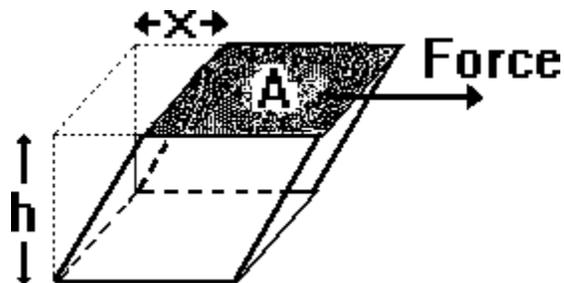
Note2: Strictly speaking all calculation shown above are "apparent values" since they assume Newtonian behavior and that the entire pressure drop occurs in the die (no entrance or exit effects).

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# Introduction to Rheology

## A. Geometries of Deformation

### Simple Shear Deformation



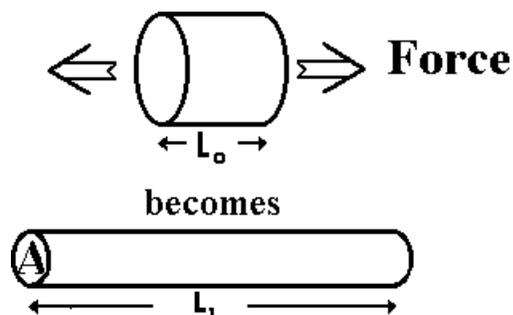
$$\text{Shear Stress} = \tau = \text{Force} / \text{Area} = F / A$$

$$\text{Shear Strain} = \gamma = \text{Displacement} / \text{Gap} = x / h$$

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

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

### Extensional Deformation



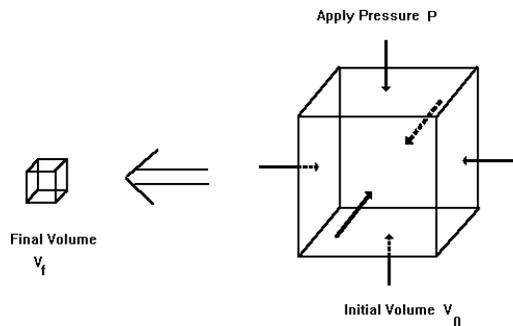
$$\text{Extensional Stress} = F/A = \sigma = \frac{F e^{t\dot{\epsilon}}}{\pi R_0^2}$$

$$\text{Extensional Strain} = \epsilon = \frac{L_1 - L_0}{L_0} = \ln \frac{L_1}{L_0}$$

$$\text{Extensional Strain Rate} = \dot{\epsilon} = \left( \frac{1}{L} \right) \left( \frac{\Delta L}{\Delta t} \right)$$

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, such as those seen in the rheometer, contain a combination of the three fundamental deformation geometries shown above. When the molten material is compressed in the barrel it undergoes a volumetric compression, which increases its density slightly. Material flowing from the barrel into the entrance of the die experiences an extensional deformation. (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 stationary (more on this assumption later), yet the material in the center of the capillary is moving very fast. (The die wall is like the bottom plate in the simple shear geometry and the center of the stream is similar to the top plate.) Capillary rheometers are designed primarily to measure the shear viscosity of a material. With some additional measurements, extensional viscosity can also be estimated. Limited volumetric data (PVT) can be collected for the molten material using a solid die, special sealing plunger and pressure transducer.

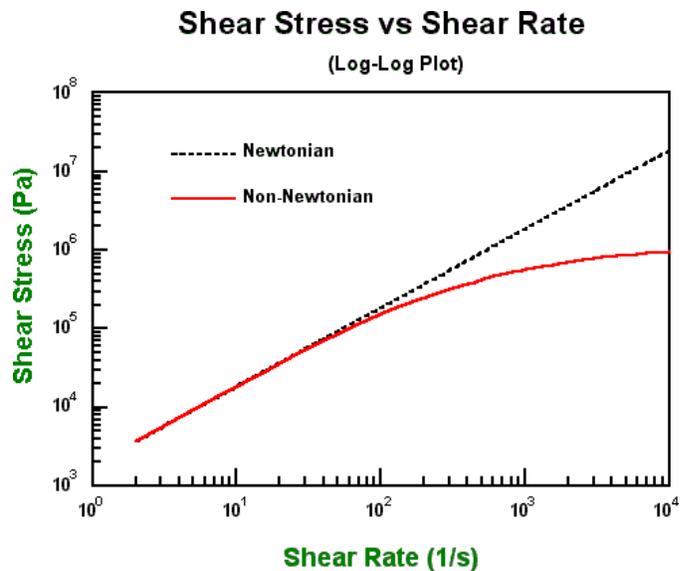
## B. Newton's Law

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

Shear Stress
Newtonian Viscosity
Shear Rate

Newton's law states that the shear stress on a material is directly proportional to the rate at which it is sheared. Using the simple shear deformation model 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 next Figure. The slope at any given point on the curve is the viscosity of the material at that shear rate.

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

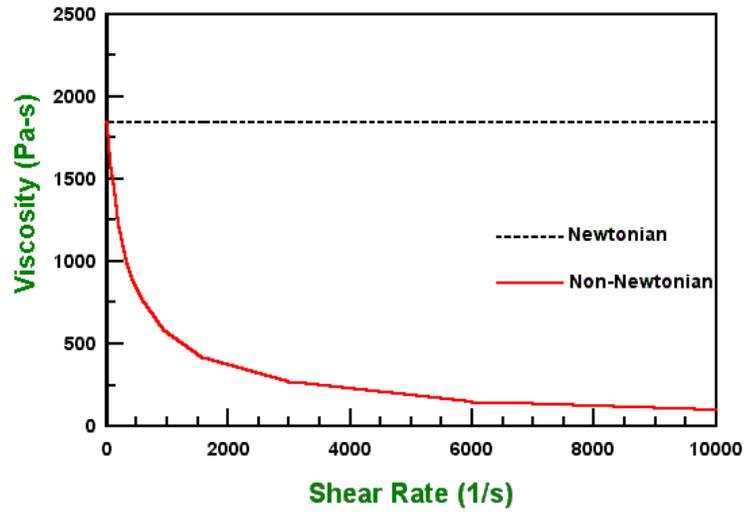


The Newtonian region for materials on a log-log plot of shear stress vs. shear rate is where the slope is equal to 1.0. For most polymers, the slope falls to less than 1.0 as the shear rate increases.

It is often easier to see if the material is behaving like a Newtonian material by 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 shown in the examples below.

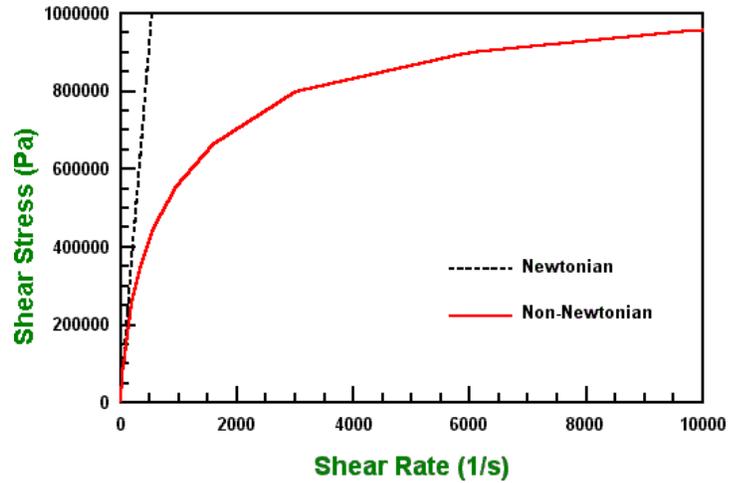
### Viscosity vs Shear Rate

(Linear-Linear Plot)

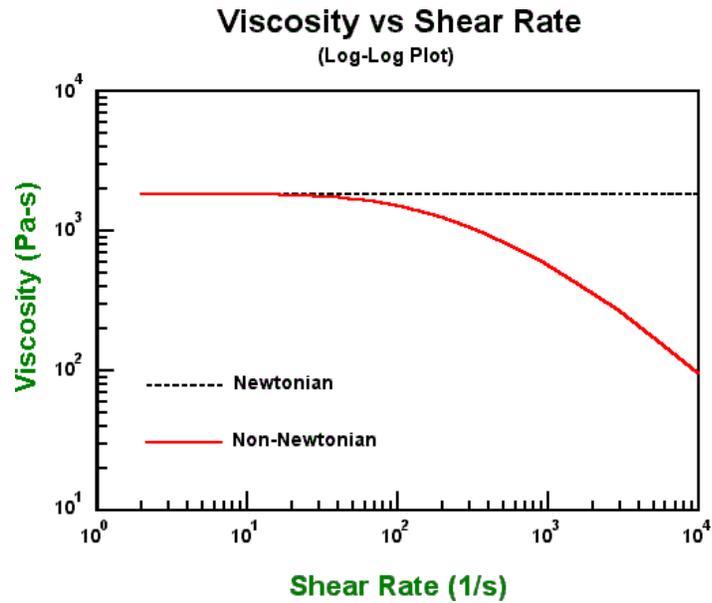


### Shear Stress vs Shear Rate

(Linear-Linear Plot)



or the same data the precipitous viscosity drop can be displayed more clearly in the log-log plot shown below.



Below is a list of some common materials and their viscosities at their 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. This means that their viscosity decreases with increasing shear rate. For a given rheometer and die, the viscosity is directly proportional to the force on plunger divided by speed (It is a force to speed ratio.). The shear rate for a fixed die geometry and rheometer depends only on the speed of the plunger. If you double the speed of the plunger for a Newtonian material, you will double the force on the plunger. For pseudoplastic materials, doubling the plunger speed will not double the force. The force will be lower. The force needed to push the plunger will increase, but not as much as we would predict based upon Newtonian behavior. (See the graphs under Newton's Law for more insight into this behavior.)

When the long chain polymer molecules flow they tend to orient in the direction of the flow. This orientation (alignment) makes the flow process easier and results in a decrease in viscosity. Given enough time after deformation, the polymer chains will return to their original random configuration (provided the material is stable for that period of time). If deformed again the effect of shear rate thinning will then be replicated.

#### **Dilatant**

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

#### **Thixotropic**

If a material is thixotropic, its viscosity decreases with time. Given enough time polymeric materials will tend to orient themselves, making flow easier. This effect is reproducible. Given enough time to recover, the material will demonstrate the same time dependence when retested. If a material degrades chemically to a lower molecular weight this will be a non-reversible effect.

#### **Rheopectic**

Rheopectic materials generally increase in viscosity with time. Crosslinking or further polymerization can cause this increase in viscosity to occur.

### D. Effect of Time on Viscosity

### E. Effect of Temperature on Viscosity

In general, polymeric materials tend to decrease in viscosity with an increase in temperature, provided they are chemically stable. (Unstable materials may increase in molecular weight through polymerization or crosslinking, producing a rise in viscosity. Alternatively, it can break down via chain scission causing a larger than expected drop in viscosity with temperature.)

A common method of representing the dependence of the viscosity on temperature is through the use of the Arrhenius Equation, where the log of the viscosity (often the zero shear viscosity value) vs.  $1/\text{Temperature}$  is fitted to a straight line. Sometimes a log (viscosity) vs. Temperature curve may be used. Both give similar good curve fits. LabKARS uses the Arrhenius Equation in its curve fitting models.

The WLF (Williams-Landel-Ferry) dependence is also used sometimes. It is found to be particularly useful at temperatures closer to the glass transition temperature of the material.

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

## F. Effect of Fillers on

Generally, inorganic fillers such as carbon black, CaCO<sub>3</sub>, TiO<sub>2</sub> etc., increase the viscosity of polymeric materials based on their volume fraction concentration. They also tend to reduce extrudate swell (rocks don't swell).

## G. Effect of Moisture on

Many materials adsorb (surface) and absorb (internal) moisture. While at room temperature this often has little effect on the material, at elevated temperature even small amounts of moisture can have dramatic effects on the viscosity of the material. This is especially true for condensation polymers. (For example, it is known that the fiber strength of PET is a strong function of the moisture in the material before the spinning process.)

## H. Equations and Corrections

### Viscosity is Shear Stress over Shear Rate:

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

Where:  $\eta$  is viscosity,  $\tau$  is the shear stress and  $\dot{\gamma}$  is 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:

$\tau$  = shear stress

$F$  = force acting on surface area  $A$ .

$F_p$  = force on the plunger

$r_c$  = radius of the capillary

$D_c$  = diameter of the capillary

$R_b$  = radius of the barrel

$L_c$  = length of the capillary

$\Delta P$  = pressure drop across the capillary

### Apparent Shear Rate:

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

Where:

$\dot{\gamma}$  = shear rate

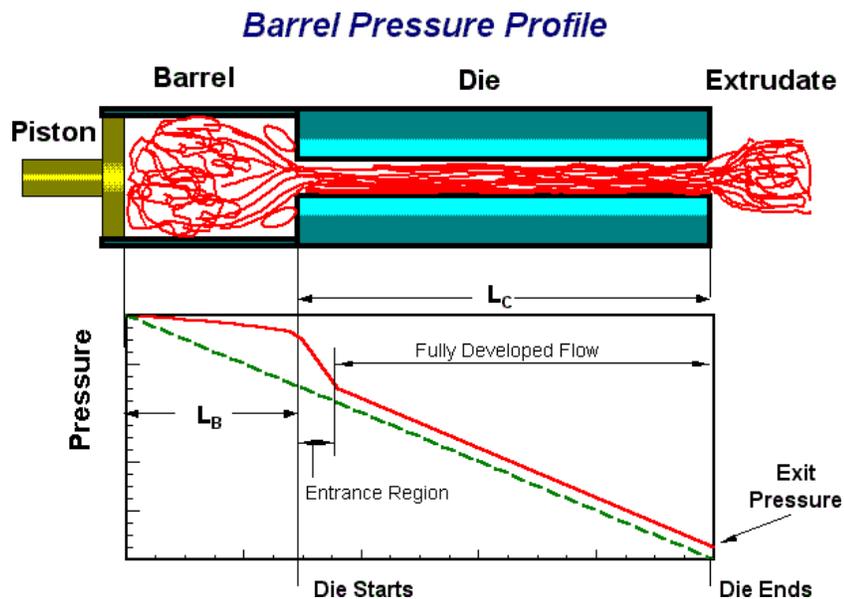
S = speed of the plunger

Q = volumetric flow rate.

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

Using the above expressions for shear stress and shear rate the viscosity reduces to the equation shown above. Physically the viscosity is proportional to the force on the plunger divided by the speed of the plunger. As can be seen in the equation, viscosity is strongly dependent on barrel and die radius.

## I. Pressure Profile



In the above diagram of the pressure vs. position it can be seen that, within the die the pressure falls steeply as the exit is approached. Even within the barrel there is a 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 quite steep 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 as a result of the shearing action within the die. (Note that the exit pressure is typically non-zero.) All of these pressure effects, the barrel pressure drop, the entrance pressure drop, frictional effects and the exit pressure are all part of the

total pressure in addition to the pressure drop in the die. In the previous equations we assumed that we were only measuring the pressure drop in the die. If we use this assumption, we over-estimate the viscosity because of the extra pressures in the system. We can correct for this 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. These data are plotted as pressure versus the L/D of the dies for a series of different shear rates and the lines extrapolated to L/D=0. The pressure at this point represents the extraneous pressures, which can be subtracted so the true viscosity can be calculated. (See the section Corrected Shear Stress.)

## J. Why use a Pressure Transducer?

### Advantages of a pressure transducer

- a) Frictional effects of plunger tip are removed.
- b) The barrel Pressure drop from plunger tip to the pressure sensor is removed.
- c) Some improvements in precision can be gained.

### Disadvantages of a pressure transducer

- a) Pressure reservoir requires flow into it for the pressure sensor to stabilize.
- b) Increased thermal losses from pressure probe.
- c) The pressure reservoir needs cleaning for materials that degrade.
- d) The device itself is inherently less accurate than a load cell.
- e) Reduced dynamic range.
- g) Must be removed when hot to prevent damage to the device. (Polymer may adhere to the diaphragm and cause it to fail if removed cold.)

## K. Common Assumptions

1. Fully developed, steady-state, laminar flow is achieved.
2. There are no radial or circumferential velocity components
3. There are no body forces (inertial effects etc.).
4. Viscosity is independent of pressure
5. Isothermal conditions are achieved.

## L. 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 Newtonian material, with a constant flow rate, the velocity of the melt stream is at a maximum in the center of the die. The velocity then falls to zero at the capillary wall. The change in this parabolic velocity profile, as we move from the center of the die to the wall, represents the shear rate for that position in the die. At high flow rates, polymeric materials show a much larger change in velocity near the wall of the die compared to Newtonian materials. Thus at higher shear rates, the calculated apparent shear rate (equation shown above) is an underestimate of the true shear rate. This results in an over estimation of the viscosity.

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 = the tangent slope of the log true shear stress vs. log apparent shear rate curve at the apparent shear rate being corrected.

•

$\dot{\gamma}$  = the true shear rate

•

$\dot{\gamma}_a$  = 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.

## M. Corrected Shear Stress

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

Use a minimum of two dies (though preferably three or more) having the same entrance angle and same diameter (D), but with 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 5 or less 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 ( $P_0$ ) or Force axis ( $F_0$ ). To obtain the true shear stress use the following equation:

$$\tau = \frac{(P - P_0) D}{4 L} = \frac{(F - F_0) D}{4 L A_B}$$

Where:

$\tau$  = the true shear stress

P = melt pressure

$P_0$  = the intercept obtained for a given shear rate from the above described plot (see Figure 1).

D = die diameter

L = die length.

For plunger force measuring devices:

F = force on the plunger

$F_0$  = the intercept force on the Bagley plot described above.

$A_B$  = 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 the 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 non-linear changes in the pressure vs.  $L/D$  plots due to the effects of pressure or viscous heating on the viscosity. In such cases one should use only the data from shorter capillaries, which do not exhibit the effect.

LabKARS 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 straightline conditions are not met.

## **N. Extrudate Swell**

Extrudate swell (sometimes called “die swell”) is defined as the ratio of the 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

## **O. PVC**

PVC is unlike most other polymeric materials. This unusual behavior results from the degree of particle fusion and the fact that many crystallites exist at typical test conditions. PVC behaves more like a lightly crosslinked rubber than a true homogeneous melt. As a result of this, it may exhibit behavior, which can be directly opposite to that of most true polymer melts. It may show an increase in extrudate swell with temperature and viscosity values that are very dependent upon how (temperature and 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 affects your results.

---

# Maintenance / Calibration

## 1. System Configuration

### System Initialization and Configuration

Connect power cord (GP1102 for 115V or GP1102-230V for 230V) to power input module (EH0402) at rear of machine.

Plug in power cord to receptacle observing proper voltage.

Confirm the emergency stop switch on the front panel is in its outward position.

Power up system by moving the power switch from the side marked "0" to the side marked "1".

Allow system to boot and self test.

Press the SYS key located on the keypad.

Press the CAL key located at center smart key.

Press SHIFT-SYS simultaneously.

Press → once (using right side smart key) SERIAL SELECT will appear at top of display.

Press SELECT smart key.

Press the CLR key. Using number keypad at front right side of machine enter the serial 3 digit serial number of the machine and press ENTER.

Press → once (using right side smart key) MODEL SELECT will appear at top of display.

Press SELECT smart key.

Press the right smart key (→) until the model (e.g. 7000) is viewed above the ⬠SELECT⬠ smart key.

Press the SELECT smart key.

Press the ENTER smart key

Press ESC key on keypad twice.

## 2. Force Calibration

### A. Calibrating the Calibration Unit

An LC unit can be easily calibrated with dead weights and then put in series with the rheometer's load cell. It must, however, be calibrated to assure proper calibration of the rheometer. To calibrate the calibration unit:

Obtain a weight or set of weights with an equivalent of at least 10% of the calibration load cell's total capacity. The force exerted by these weights should be known to within  $\pm 0.5\%$ .

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 that has exposed bolt heads.

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 dipswitches 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.

With nothing on the platform and the load cell sitting flat, press the **ZERO** button. The unit will display CAL-1.

Press **STANDBY / OPERATE** and ..... Should appear momentarily on the display unit, followed by a series of zeros. It is now time to enter in the value of the calibration weights. Pressing the **GROSS / NET** key will increment the current digit that 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)

Now place the calibration 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 to its normal position and 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 see the correct values, repeat the procedure checking for errors.

**Note:** Read the Model: AD-4321A/B instruction manual that comes with the calibration display unit for more details. Note that the dipswitches 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 dipswitches 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 loaded to over 2640 N (600 lbs) or the display will go blank.

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

Details:

Zero the rheometer force reading:

***B. Calibrating  
the rheometer  
load cell with  
the calibration  
unit***

SKIP TO 3.2 FOR 7002

Load cell (LC) configuration and calibration.

Connect load cell sensor.

Carefully align 6 pin bendix female connector of the 7052-37 to the 6 pin male connector of the load cell.

Enter device into library.

Press **SYS** on keypad.

Press **DEVICE** smart key.

Press **LC** smart key.

Press **→** until desired LC# (load cell sensor number) is viewed on display.

Press **NEW** smart key.

Press **→** until desired load cell sensor is viewed on display.

Press **ENTER** smart key.

Press **→** . LC UNIT is displayed and should read: N.

Press **→** . LC CAPACITY is displayed and should read: LC capacity.

Press **→** . LC SCALING is displayed and should read: 2.000 mV/V.

Press **→** . GAIN is displayed and should read: 128.

Press **→** . LC R-CAL is displayed and should read: 72.920%.

Press **→** . LC SEQUENCE is displayed and should be blank.

Press **ESC** once on keypad.

Press **SAVE** smart key.

Perform step 3.1.2.5 –3.1.2.15 for any other load cells that may be present on the sales order. Before 3.1.2.5 is performed perform 3.1.2.1 –3.1.2.3 then press → until LC#2 is shown. The next load cell after LC#2 would be LC#3, etc. If a duplicate load cell (a load cell of the same range) is to be included with the order the LC SEQUENCE option needs to be indexed alphabetically (A, B, C, etc.). Also, label the load cell sensors with the sequence character to differentiate them.

Activate main load cell sensor.

**Note:** The main load cell sensor is the load cell that is going to be calibrated and tested on the machine.

Press **SETUP** key on keypad.

Press → four times.

Press **EDIT** smart key.

Press → until load cell sensor of interest is displayed.

Press **SELECT** smart key.

Press **ESC** key on keypad.

R-CAL

**Note:** Step 3.1.4 is an exercise to test that the load cell is properly electrically connected.

Press **SYS** on keypad.

Press **TEST** smart key.

Press → once and CH1 TEST DISPLAY should appear at top of display.

Press **SELECT** smart key.

Press **RCAL** smart key.

Auto Identification

Press **SYS** on keypad.

Press **TEST** smart key.

Press → once and CH1 TEST DISPLAY should appear at top of display.

Press **SELECT** smart key.

Press **ID** smart key.

Compare the ID value displayed for the attached load cell sensor with that of the typical value in Table 3.1. The ID value should be within  $\pm 5\%$  of the typical value.

Table 3.1: Typical Load Cell ID values

Load Cell P/N	Load Cell Capacity	Typical Value
7052-41	1.5 KN	155.5
	2.5 KN	744
7052-42	5 KN	2526
7052-43	10 KN	7068
	300 lbf	119.31
	1000 lbf	597.7
	2000 lbf	1593.8

Calibrate load cell sensor.

Press **ESC** twice on keypad.

Press **SYS** on keypad.

Press **→** once. CHAN #1 SENSOR should appear on screen.

Press **SELECT** smart key.

Press **SINGLE** smart key.

Press **R-CAL** key on keypad.

Press **ENTER** smart key.

Press **CLR** key on keypad.

Enter typical value from Table 3.2 below. Locate load cell capacity then enter the typical value.

Table 3.2: Typical Load cell R-CAL values

Load Cell P/N	Load Cell Capacity	R-CAL Value
7052-41	1.5 KN	1093.8
	2.5 KN	1823
7052-42	5 KN	3646
7052-43	10 KN	7292
	300 lbf	218.76
	1000 lbf	729.2
	2000 lbf	1458.4

Press **ENTER** smart key.

Record offset and gain.

Press **SAVE** smart key.

Confirm LC calibration.

Assemble the electronic calibration system.

Connect the power cord to the rear of the calibration display.

Connect patch cord (has silver connector and bronze) to the rear of the calibration display.

Connect the calibration load cell to the bronze end of the patch cord.

Attach phenolic insulators to both sides of the calibration load cell. **Note:** The phenolic bobs isolate the calibration load cell from thermal damage due to the heat of the barrel. The most accurate calibration will be done at the highest test temperature.

Press **SYS** key on keypad.

Press **CAL** smart key.

Press **→** once. CHANNEL #1 SENSOR should appear at the top of the display.

Press **SELECT** smart key.

Press **SINGLE** smart key.

Rotate, if necessary, the calibration load cell such that the bolt heads are facing up.

Insert the calibration load cell in series with the machine load cell by placing the calibration load cell phenolic bob into the load cap of the machine load cell. Recall the calibration load cell has the bolt heads facing up.

Lower the crosshead to engage the two load cells. Press **ENTER** and the **T** on the machine keypad simultaneously to increase the down speed of the crosshead. The calibration load cell should be sitting flat and centered over the barrel hole. As the load cells near each other release the **ENTER** key and use **T**. The down arrow key alone will cause the crosshead to move at a safe slow speed.

Zero the calibration unit by pressing **TARE** on the front of the display. This needs to be completed before there is force placed on the calibration load cell.

Continue lower the crosshead until both load cells come in contact and note the force read on the rheometer display and force read on the calibration display. The values should be within 0.01%.

### 3. Temperature Calibration

Press **SYS** key and **CAL** smart key. Select RTD Sensor. Select **DOUBLE**.

Press **STABLE** smart key.

Enter 0.4

Press **CLR** key on keypad.

Use the number pad on the right side of the keypad to enter **0.4**

Press **ENTER** smart key.

Insert wieland plug labeled 354 C into TS3 (RTD) located at left rear of motherboard (4051-100F).

Press **SYS** key and **CAL** smart key. Select RTD Sensor. Select **DOUBLE**.

Press **STABLE** smart key.

Enter 354

Press **CLR** key on keypad.

Use the number pad on the right side of the keypad to enter **354**.

Press **ENTER** smart key.

Heat unit to 190° C.

Reboot system by switching machine off and on.

Press **EDIT** smart key.

Press **EDIT** smart key. “**SET POINT=\_\_\_**” will appear on display.

Enter 190.

Press **CLR** keypad button.

Use the number pad on the right side of the keypad to enter **190**.

Press **ENTER** smart key.

Press **ESC** key on keypad twice.

Press **SAVE** smart key. The system will now ramp to 190° C.

Insert temperature probe into barrel.

**Note:** Ensure probe is completely seated in the barrel.

Install die and die holder nut in barrel.

Insert 12.7 mm [1/2”] ceramic spacer.

Place long insulator on probe.

Place short aluminum spacer on probe.

Place probe in barrel.

Allow unit to stabilize at 190° C for 45 minutes.

Calibrate unit at 190° C.

Press **SYS** key and **CAL** smart key to enter calibration mode.

Select RTD Sensor in the Calibrate Select mode.

Press **SELECT** smart key.

Select **SINGLE**. Press **STABLE** smart key. Press **ENTER** then press **SAVE**. This sets offset to 0.

Allow unit to re-stabilize for 10 minutes.

Press **SYS** key and **CAL** smart key. Select RTD Sensor. Select **DOUBLE**.

Press **STABLE** smart key.

Enter reading displayed on the BTP 1000A display.

Use the number pad on the right side of the keypad to enter the temperature. Enter the numbers in order as they appear on the BTP 1000A display, also pressing the decimal where necessary. NOTE: numbers are entered by just pressing the appropriate number key.

Press **ENTER** smart key.

Heat unit to 350° C.

Press **EDIT** smart key.

Press **ENTER** smart key. **SET POINT:** will appear on display.

Enter 350.

Press **CLR** keypad button.

Use the number pad on the right side of the keypad to enter **350**.

Press **ENTER**.

Press **ESC** key on keypad twice.

Press **SAVE** smart key. The system will now ramp to 350° C.

Allow unit to stabilize at 350° C for 45 minutes.

Calibrate unit at 350° C.

Press **SYS** Key and **CAL** smart key.

Select RTD Sensor in the Calibrate Select mode.

Press **→** once.

Press **SELECT** smart key.

Select **DOUBLE** smart key.

Press **STABLE** smart key.

Press **CLR** key on keypad.

Enter reading displayed on the BTP 1000A display.

Press **CLR**.

Use the number pad on the right side of the keypad to enter the temperature. Enter the numbers in order as they appear on the BTP 1000A display, also pressing the decimal where necessary. **Note:** Numbers are entered by just pressing the appropriate number key.

Press **ENTER** smart key.

Record OFFSET and GAIN numbers. Press SAVE smart key.

Temperature calibration verification.

Allow machine to re-stabilize at 350° C. for 15 minutes. Record temperature.

Change temperature set point to 300° C. Repeat steps 2.13.1 through 2.13.5 replacing **350** with **300**.

Allow machine to re-stabilize at 300° C for 45 minutes. Record temperature.

Change temperature set point to 230° C. Repeat steps 2.13.1 through 2.13.5 replacing **350** with **230**.

Allow machine to re-stabilize at 230° C for 45 minutes. Record temperature.

Change temperature set point to 190° C. Repeat steps 2.13.1 through 2.13.5 replacing **350** with **190**.

Allow machine to re-stabilize at 190° C for 45 minutes. Record temperature.

Reset temperature set point to polymer testing temperature (230° C).

Change temperature set point to 23° C. Repeat steps 2.13.1 through 2.13.5 replacing **190** with **230**.

## 4. Pressure Transducer Calibration

### A. 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.

With the addition of the pressure transducer comes an automatic check of its 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 rheometer. 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 keypad 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.

Pressure transducer (PT) configuration and calibration

**Note:** The PT sensor for the 7000 and 7001 is designated for sensor channel 2. The PT sensors for the 7002 are designated for sensor channel 1 and 2. Therefore, when initializing the first PT for the 7002, substitute sensor channel #2 with sensor channel #1 in the steps below up until step 3.3.1.2.

Connect PT sensor.

Carefully thread one end (8 pin bendix female connector) of the 7052-56 onto the 8 pin connector located at left rear (facing from front). **CAUTION: Do not force connection and be sure to align all pins.**

Carefully thread the free end (8 pin bendix female connector) of the 7052-56 onto the 8 pin connector of the mounted PT. Use same caution as mentioned in 3.2.6.1.

Enter device into library.

Press **SYS** on keypad.

Press **DEVICE** smart key.

Press **PT** smart key.

Press **→** until desired PT# is viewed on display.

Press **NEW** smart key.

Press → until desired PT sensor is viewed on display.

Press **ENTER** smart key.

Press → . PT UNIT is displayed and should read: bar.

Press → . PT CAPACITY is displayed and should read: PT capacity.

Press → . PT SCALING is displayed and should read: 3.33000 mV/V.

Press → . GAIN is displayed and should read: 128.

Press → . PT R-CAL Press → . PT R-CAL is displayed and should read:  
84.000%.

Press → . PT SEQUENCE is displayed and should be blank.

Press **ESC** once on keypad.

Press **SAVE** smart key.

Perform step 3.2.7.1 - 3.2.7.15 for any additional PTs. If a duplicate PT (a pressure transducer of the same range) is to be included with the order the PT SEQUENCE option needs to be indexed alphabetically (A, B, C, etc.). Also, label the PT sensors with the sequence character to differentiate them.

Activate PT sensor.

**Note:** The main pressure transducer sensor is the load cell that is going to be calibrated and tested on the machine.

Press **SETUP** key on keypad.

Press → five times.

Press **EDIT** smart key.

Press → until PT sensor of interest is displayed.

Press **SELECT** smart key.

Press **ESC** key keypad.

R-CAL

**Note:** Step 3.1.4 is an exercise to test that the PT is properly electrically connected.

Press **SYS** on keypad.

Press **TEST** smart key.

Press → twice and CH2 TEST DISPLAY should appear at top of display.

Press **SELECT** smart key.

Press **RCAL** smart key.

Auto Identification  
Press **SYS** on keypad.

Press **TEST** smart key.

Press **→** once and CH1 TEST DISPLAY should appear at top of display.

Press **SELECT** smart key.

Press **ID** smart key.

Compare the ID value displayed for the attached PT sensor with that of the nominal value in Table 3.3. The ID value should be within  $\pm 5\%$  of the nominal value.

**Table 3.3: Nominal Pressure Transducer ID values**

PT P/N	PT Capacity	Typical Value
8052-150 MID	50 bar	4.92
8052-150 MID	200 bar	60.68
8052-151 MID	700 bar	351.68
8052-152 MID	1000 bar	594.5
8052-153 MID	1400 bar	983.64
	2000 bar	1806
	250 psig	50.525
	10000 psig	3951
	30000 psig	23745

Calibrate PT sensor.

Press **ESC** twice on keypad.

Press **SYS** on keypad.

Press **→** twice. CHAN #2 SENSOR should appear on screen.

Press **SELECT** smart key.

Press **SINGLE** smart key.

Press **R-CAL** key on keypad.

Press **ENTER** smart key.

Press **CLR** key on keypad.

Enter typical value from Table 3.4 below. Locate PT capacity then enter the typical value.

**Table 3.4: Typical Pressure Transducer R-CAL values**

PT P/N	PT Capacity	Typical Value
8052-150 MID	50 bar	42
8052-150 MID	200 bar	168
8052-151 MID	700 bar	588
8052-152 MID	1000 bar	840
8052-153 MID	1400 bar	1176
	2000 bar	1680
	250 psig	210
	10000 psig	8400
	30000 psig	25200

Press **ENTER** smart key.

Record offset and gain.

Press **SAVE** smart key.

Press **ESC** key on keypad until the home screen on the machine display is viewed.

#### **SKIP TO STEP 4 FOR 7000 AND 7001**

Carefully thread one end (8 pin bendix female connector) of the 7252-56 onto the 8 pin connector located at left rear (facing from front). **CAUTION: Do not force connection and be sure to align all pins.**

Carefully thread the free end (8 pin bendix female connector) of the 7252-56 onto the 8 pin connector of the mounted PT. Use same caution as mentioned in 3.3.1.1.

Repeat step 3.2.7 – 3.2.10.

## **5. RCAL**

The rheometer always performs the ZERO SHIFT and RCAL calibrations together when the **R-CAL** button is pressed on the front panel of the rheometer. To be performed correctly the rheometer should be warmed up for at least twenty minutes 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 will not influence the read.

Calibrate the Pressure Transducer by pressing **R-CAL** on rheometer key pad ONLY when:

- The pressure reservoir is clean
- The pressure transducer is mounted in barrel and snug (100 in-LB torque)
- The cross head is at the Park position (Up and away from plunger)
- The rheometer is "locked in" at test temperature
- The PT is an active sensing device, Press R-CAL on panel

## 6. Dimensional Verification

Check the die by doing the following:

Perform a quick visual inspection of the die. The die bore and finish of the bore 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 the **cold** die with drill bits and solvent if needed. (Any die that has been in use will need it!). 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.

## 7. Die 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 be 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 if it does not pass. (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 if it does not pass this test. (DO NOT USE THIS DIE). Getting to this point without rejecting the die means it has passed ASTM specifications listed in ASTM D3835-90 for capillary rheometer dies or D1238 specs for melt indexers.

The die may also be good enough to meet the ISO specifications. This check can be made using 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 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.

## 8. 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, which is only slightly larger in diameter than the die capillary diameter, can be mounted to a dial indicator. 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.

## 9. 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 to the gage so the entire barrel can be checked. The barrels are specified at  $9.550 \pm 0.005$  mm leaving the factory.

## 10. Plunger Tip Diameter

Clean the barrel-tip with a piece of bronze wool or a like material. 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), since we have found that larger tips can cause seizing in the barrel at higher temperatures.

## 11. Lubrication

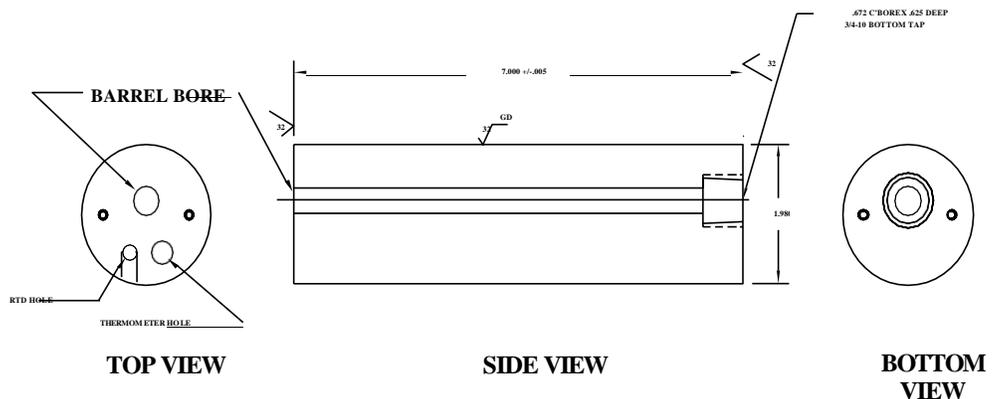
### Galaxy

There are two grease fittings on the Galaxy V rheometer. One is located on the lower right side of the worm gear reducer system (just behind main pulley) and the other 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 stick-slip sliding (which may cause measurement errors) and a squeaking sound. A short spray of WD-40 or similar lubricant will remedy this.

## 12. Barrel Drawing

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



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## Extras

### Measurement of Melt Density

To get apparent melt density of a material, its volume at test temperature and the mass of that volume must be known. We can get the volume of material by running the rheometer over a given distance at constant speed. The rheometer extrudes a known volume of material and then we measure its mass. The following test for extrudate swell is suggested:

1. Pick the longest die you have. This will allow easier cutting of the extrudate strand as it exits the die.
2. Set the rheometer to 360 sec melt time, all rates at 12 mm/min and a melt force of 890 N, and delay- time 120 sec. Set the start position to 6.0 inches and the first location to 8.0 inches, set all remaining positions to 8.1
3. When the rheometer reaches the start position, wipe the die clean and remove all material directly under the die.
4. It will take four minutes to go from start position to 8.0 inches when the plunger hits 8.0 it will stop. Collect all the extrudate that has come out from the start position to the 8.0 inch position. Using a balance obtain the mass of this material. Let the material cool before weighing as hot material can affect the balance reading.
5. The volume extruded from the rheometer is (independent of speed) 1.82 cc per inch of stroke or 3.64 cc (cm<sup>3</sup>) for the test described.
6. Divide the mass of material by 3.638 to get the apparent melt density in g/cc when using the test described above.

Note: The melt indexer has a looser piston to barrel clearance tolerance. This allows more material to go by the plunger tip on the melt index and may cause the density obtained using an indexer to be *slightly* less than that from the rheometer.

### The Non-Newtonian Index

The Non-Newtonian Index is defined as follows:

A standard rate sweep is performed at 190°C at rates from 0.05 to 500 mm/min. A 2nd order polynomial is then fit to the log shear stress vs. log shear rate data. Shear rates at the specific stresses  $\sigma_1$  and  $\sigma_2$  are determined from the fit. (A fit of rate vs. stress can also be done.)

$$\sigma_1 = 4 \times 10^4 \text{ Pa} \quad \sigma_2 = 24 \times 10^4 \text{ Pa}$$

In some cases, a higher temperature (250°C) is used. An experimentally determined conversion factor (2.30) is used to convert the shear rate at 250°C to that at 190 °C.

$$\dot{\gamma}_1(2 \times 10^4, 190\text{C}) = \dot{\gamma}_1(2 \times 10^4, 250\text{C}) / 2.30$$

$$\dot{\gamma}_2(24 \times 10^4, 190\text{C}) = \dot{\gamma}_2(24 \times 10^4, 250\text{C}) / 2.30$$

Additionally, a low stress  $\sigma_3$  is sometimes used, along with the higher temperature, to avoid melt fracture. In this case another correction is made to convert back to the  $\sigma_2$  level. (Note the 1.83 applies only to the higher stress level.)

$$\sigma_3 = 20 \times 10^4 \text{ Pa}$$

$$\dot{\gamma}_2(24 \times 10^4, 190\text{C}) = \dot{\gamma}_2(20 \times 10^4, 250\text{C}) 1.83 / 2.30$$

$$\frac{\dot{\gamma}_2(24 \times 10^4, 190\text{C})}{\dot{\gamma}_1(4 \times 10^4, 190\text{C})} = NNI \quad \text{A unitless value.}$$

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# Trouble Shooting

## 1. Frequently Asked Questions (FAQs)

1. “The rheometer doesn't respond when I press UP, DN or RUN, but the front display shows the temperature is 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 rheometer responds when pressing the DN button.

If, when you press DN, the rheometer still doesn't go down. Check the readout on Sensor # 1 to see if any force is being seen by the sensor. Be sure the safety shield is in the down position. Recal the sensors and return the cross head to the park position.

2. “When I pressed RUN the rheometer appeared to start, but now it just sits there.”

The rheometer is trying to communicate with the Personal Computer (PC). It must communicate with it before the test starts. Either: the software is not running on the PC, or the cable connection or communications port on the PC is faulty. Check the PC attached option (under Setup) is it set to *ON*. Check your connection between the rheometer and the PC. Check your Communication Port settings to section “LabKARS Setup” of this manual.

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

Check your test setup to see if the manual mode is selected. When the manual mode is on, data is *ONLY* collected if and when the *AQUIRE* button is pressed. To set the test to *RUN* automatically, shut manual mode off select the *Steady State* option in the test setup page of the software. By setting the positions you wish the rheometer 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 rheometer 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 PRT sits in may be contaminated with material. Set the rheometer temperature to 0.0 and then (and only then) remove the temperature probe (PRT), wipe it clean if necessary and use a long drill bit to remove any material in 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 on the assumption that all material exits through the die. Check to see if the tip is within ASTM specifications (between 0.3751 - .3745). High temperature rheometers 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. *Why can't I see the data I ran yesterday in Analyze? Data is missing?*

It is possible that a filter in Database / Filters was left on. Verify that the filters are all set to 0-ALL-0.

It is possible that an entry that is being referenced by Capdata was deleted from a database table. To verify this, go into Maintain and review Capdata.db. If for example, Charge 556 references a number 12 in the customer column and Customer.db has NO entry 12 then you would need to change Customer reference 12 in Capdata.db to a reference that exists in the Customer.db table.

7. *I am using a multiple rate test but the first rate never started.*

It is possible that you never reached your starting position. Try increasing your Packing Force or changing the starting position to one you will reach prior to the melt time expiring.

8. *The ram just purges the barrel after the Melt time expires.*

Verify the settings specified in section 1.2 Windows OS settings.

Verify that the Trend is not set to "0" zero in Run.

Verify that the setup you are using has the correct machine specified.

9. I used the over pack function in RUN and now the ram returns to the uppermost park position not what I previously set it for?

Once the OVERPACK function is used the machine automatically adopts a park position of 5.0mm. To return to the previous park position, just press the EDIT key at the machine, press the left/right arrow until PARK POS. is displayed and then press EDIT, type in the desired park position, press ENTER to accept and then press ESC. Now press the "up"  key or HOME in the RUN screen and the ram should return to the desired park position.

10. How can I tell what some of the buttons do without referring to the manual or the notes?

Holding the mouse pointer over any key will give a brief description of its' function.

11. "My software runs very, very slowly."

LabKARS on a 400mhz PC or better normally should respond well. 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 LabKARS 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 DYNISCO POLYMER TEST supplied cables or carefully follow the cabling diagrams found in the utilities.

12. "I've over forced the rheometer 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. Remove the die by unscrewing the die nut holder. Take the die and nut holder completely out and purge the rheometer by pressing SHIFT then DN then YES, as usual, to purge.

13. "The X-Head of the rheometer will only move Up but not Down and / or the test will not start when I press the RUN button."

If the Load Cell is run to the bottom of the rheometer barrel before the test is finished the drive motor will over force itself and throw up an error. You will not see the error on your PC but the Load Cell will only be allowed to move Up and not Down.

You should first try to toggle the power switch Off and then back On to reset the drive system. Be sure the Load Cell and / or Pressure Transducers are connected to the rheometer before resetting the machine. If this does not work you may have to adjust the Torque of the drive motor inside the base of the rheometer.

Start your LabKARS software and open the Rheometer Control Page. Check the Torque Load reading in the little box in the upper right hand corner of the screen. If it is not reading approximately Zero +/- 100, you may have to make an adjustment. Remove the back cover of the rheometer base and locate the Gold box with the Green LED just behind the right post. Along side the Green LED you will see a very small screw, this screw is the torque adjustment for the rheometer drive system. Using a very small screwdriver (**CAUTION: There is power being applied to the rheometer when this adjustment is being made. DO NOT come in contact with any other items in the rheometer base**) adjust the torque setting by turning the screw in or out. Watch the torque reading on the screen of your PC and try to get it as close to Zero as you can. Then Toggle the power button Off and On to reset the system. This should restore the unit to its working order, if the rheometer still does not working properly please contact Dynisco Customer Service.

## Questions? Please feel free to contact Dynisco at

**(508) 541-9400 or fax (508) 541-6206**

### **2. When your Control material goes out of**

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

- Force calibration OK?
- Temperature calibration OK?
- The program conditions are exactly the same as before?
- The Force is zeroed correctly?
- Are you using the same die as before? Dimensions OK?
- Is the Piston Tip Dimension OK? ( 09.525 > Tip OK > 9.515 mm)
- Is the die very clean? (If high temp material was run previously, heat the die up to the previous sample temperature and re-clean.
- Is the guide bushing free to move up and down the Plunger rod?
- Was the material dried as before? (If necessary)
- Is the material exactly the same as before?
- Verify correction scheme used is as before (i.e. Rabinowitsch on or off etc.)
- Is the piston rod bent?
- Put rheometer temporarily on a filtered cleaner powder supply.
- Is the PRT well clean? 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?)

# Parts List

## DIES and ORIFICES

Call DYNISCO POLYMER TEST or your local representative for the latest pricing information. Orders can also be placed at that time. A purchase order number will be required.

New Name	DIE	STOCKNO	DIAMETE		ConeAngl		WIDTH	SpeedtoRate	ForcetoStress
			R	LENGTH	e				
CSlit Die L	532	Slit Die L	2.748	36.860	180			0.586043451	0.260176555
CSlit Die S	533	Slit Die S	1.976	21.730	180			1.57622852	0.317346691
CUnknown	1	Unknown	1.000	1.000	1	1		12.1613	3.48985
CX125-10	546	X125-10	0.318	3.175	120			379.9695662	0.348985
CX150-60	510	X150-60	0.381	22.860	120	0		219.8897953	0.058164167
CX150-80	506	X150-80	0.381	30.480	120	0		219.8897953	0.043623125
CX200-15	530	X200-15	0.508	7.620	120			92.76600738	0.232656667
CX200-20	500	X200-20	0.508	10.160	120	0		92.76600738	0.1744925
CX200-3C	547	X200-3C	0.508	1.520	120			92.76600738	1.166344605
CX200-40	512	X200-40	0.508	20.000	120	0		92.76600738	0.08864219
CX2520	529	X2520	0.635	12.700	120			47.49619578	0.1744925
CX300-10	150	X300-10	0.762	7.620	120	0		27.48622441	0.348985
CX300-15	151	X300-15	0.762	11.430	120	0		27.48622441	0.232656667
CX300-1C	519	X300-1C	0.762	0.762	120	0		27.48622441	3.48985
CX300-20	152	X300-20	0.762	15.240	120	0		27.48622441	0.1744925
CX300-25	153	X300-25	0.762	19.050	120	0		27.48622441	0.139594
CX300-30	10	X300-30	0.762	22.860	120	0		27.48622441	0.116328333
CX300-33	22	X300-33	0.762	25.400	120	0		27.48622441	0.1046955
CX300-35	25	X300-35	0.762	26.670	120	0		27.48622441	0.09971
CX300-40	26	X300-40	0.762	30.480	120	0		27.48622441	0.08724625
CX394-10	110	X394-10	1.000	10.000	120	0		12.1613	0.348985
CX394-20	7	X394-20	1.000	20.000	120	0		12.1613	0.1744925
CX394-30	8	X394-30	1.000	30.000	120	0		12.1613	0.116328333
CX394-40	9	X394-40	1.000	40.000	120	0		12.1613	0.08724625
CX394-5	520	X394-5	1.000	5.000	120	0		12.1613	0.69797
CX400-05	50	X400-05	1.016	5.080	180	0		11.59575092	0.69797
CX400-10	51	X400-10	1.016	10.160	180	0		11.59575092	0.348985
CX400-20	4	X400-20	1.016	20.320	120	0		11.59575092	0.1744925

CX410-15	528	X410-15	1.040	15.620	120		10.81135142	0.232358771
CX410-20	505	X410-20	1.041	20.828	120	0	10.76780748	0.1744925
CX500-16	543	X500-16	1.270	11.900	120		5.937024472	0.372446176
CX500-20	501	X500-20	1.270	25.400	120	0	5.937024472	0.1744925
CX600-05	517	X600-05	1.524	7.620	180	0	3.435778051	0.69797
CX825-40	531	X825-40	2.096	8.382	120		1.321651662	0.8724625
CX825-40	540	X825-40	2.096	83.820	120		1.321651662	0.08724625
CXMI	511	XMI	2.095	8.000	120	0	1.322598178	0.913904469
CY0052CIL	504	Y0052CIL	0.051	0.051	90	0	92766.00738	3.48985
CY125-08	513	Y125-08	3.170	25.430	90	0	0.381770367	0.435030456
CY300-33	23	Y300-33	0.762	25.400	90	0	27.48622441	0.1046955
CY300-5	503	Y300-5	0.762	3.810	90	0	27.48622441	0.69797
CY394-1C	109	Y394-1C	1.000	1.000	90	0	12.1613	3.48985
CY394-20	6	Y394-20	1.000	20.000	90	0	12.1613	0.1744925
CY400-01	526	Y400-01	1.016	1.016	90		11.59575092	3.48985
CY400-30	52	Y400-30	1.016	30.480	90	0	11.59575092	0.116328333
CY484-41	541	Y484-41	1.229	50.800	90		6.551252724	0.084429639
CZ197-20	28	Z197-20	0.500	10.000	180	0	97.2904	0.1744925
CZ197-6.35	542	Z197-6.35	0.500	3.175	180		97.2904	0.549582677
CZ2951-4	507	Z2951-4	0.750	2.862	180	0	28.82678519	0.914530922
CZ295-40	527	Z295-40	0.749	29.972	180		28.9424005	0.087211319
CZ300-01	509	Z300-01	0.762	0.762	180	0	27.48622441	3.48985
CZ300-10	200	Z300-10	0.762	7.620	180	0	27.48622441	0.348985
CZ300-15	201	Z300-15	0.762	11.430	180	0	27.48622441	0.232656667
CZ300-20	202	Z300-20	0.762	15.240	180	0	27.48622441	0.1744925
CZ300-25	203	Z300-25	0.762	19.050	180	0	27.48622441	0.139594
CZ300-30	204	Z300-30	0.762	22.860	180	0	27.48622441	0.116328333
CZ300-33	24	Z300-33	0.762	25.400	180	0	27.48622441	0.1046955
CZ300-40	206	Z300-40	0.762	30.480	180	0	27.48622441	0.08724625
CZ394-10	100	Z394-10	1.000	10.000	180	0	12.1613	0.348985
CZ394-15	518	Z394-15	1.000	15.000	180	0	12.1613	0.232656667
CZ394-1C	502	Z394-1C	1.000	1.000	180	0	12.1613	3.48985
CZ394-20	2	Z394-20	1.000	20.000	180	0	12.1613	0.1744925
CZ394-20	30	Z394-20	1.000	20.000	180	0	12.1613	0.1744925
CZ394-30	3	Z394-30	1.000	30.000	180	0	12.1613	0.116328333
CZ394-40	104	Z394-40	1.000	40.000	180	0	12.1613	0.08724625
CZ394-5	508	Z394-5	1.000	5.000	180	0	12.1613	0.69797
CZ400-01	521	Z400-01	1.016	1.016	180		11.59575092	3.48985
CZ400-15	5	Z400-15	1.016	15.240	180	0	11.59575092	0.232656667
CZ420-9.5	548	Z420-9.5	1.067	10.130	180		10.01121397	0.367588346
CZ787-2.5	536	Z787-2.5	1.999	4.990	180		1.522445026	1.398038106
CZ787-20	544	Z787-20	2.000	40.000	180		1.5201625	0.1744925
CZ825-4	522	Z825-4	2.096	8.382	180		1.321651662	0.8724625
CZ295-30	549	Z295-30	0.749	30.000	180	0	28.9424005	0.087129922
CZ591-30	550	Z591-30	1.501	30.000	180	0	3.59615105	0.174608828
CZ700-30	551	Z700-30	1.770	30.000	180	0	2.193108928	0.20590115

CZ492-20	553	Z492-20	1.24968	24.9936	180	0	6.231370067	0.1744925
CY400-15	554	Y400-15	1.016	15.24	90	0	11.59575092	0.232656667
CZ690-12	555	Z690-12	1.7526	21.0312	180	0	2.25907984	0.290820833
CY400-20	556	Y400-20	1.016	20.32	90	0	11.59575092	0.1744925
CY300-20	557	Y300-20	0.762	15.24	90	0	27.48622441	0.1744925
CZ156-30.5	558	Z156-30.5	0.39624	12.08532	180	0	195.4812273	0.114421311
CZ787-15	564	Z787-15	1.99898	29.9847	180	0	1.522490723	0.232656667
CX825-11	565	X825-11	2.0955	23.0505	120	0	1.321651662	0.317259091
CY295-16	570	Y295-16	0.7493	11.9888	90	0	28.90765109	0.218115625

Note 1: Pin gauges are designed to ASTM or ISO Specifications.  $\pm 0.005$  mm, (They are similar after '98. Previous to '98 ASTM specification was larger)

Note 2: Full die entrance angles 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



## Supplied Hand Tools



1. Charging tool for compacting pellets into barrel. (Relief area to allow air to escape during packing.)



2. Cleaning tool for barrel. Use with two patches for proper friction.



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



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. Tip is removable. Tip. Sliding guide bushing



5. Clean out drill bit and drill bit vise for cleaning capillary dies.  
Bit Vise Specify die size to select correct bit.



6. Sample filling funnel. Fits into counter sink on top of



7. PYREX  beaker used for sample filling of pellets or powder.



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 (model 7000 & 7001).



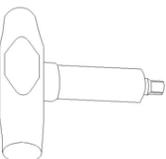
9. Breaker Bar Die nut removal tool only.



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



11. Rheometer Die. Made of Tungsten Carbide. (Don't drop hot die into solvent or on a hard surface!) See table in appendix for ordering information.



12. T-Handle torque wrench die nut installation tool only

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 barrel 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).

## LCR 7000 SPARE PARTS LIST

PART #                      DESCRIPTION  
MOST COMMON ITEMS

### LCR 7000 Series Rheometer Spare Parts List

<u>Part Description</u>	<u>LCR 7000</u>	<u>LCR 7001</u>	<u>LCR 7002</u>
<b><u>Barrels:</u></b>			
Replacement Barrel	8052-141	6052-65PT	7252-65PT
Replacement Barrel / Haynes Lined	8052-141H	6052-65HPT	7252-65HPT
Replacement Barrel / w/ PT	8052-141P	N/A	N/A
Replacement Barrel / Lined, w/PT	8052-141HP	N/A	N/A

### **Barrel Heaters:**

Barrel Heater 115 V	2052-70DZ	7052-70PT	7252-70PT
Barrel Heater 115 V W/PT	2052-70TZ	N/A	N/A
Barrel Heater 230 V	2052-70DZHV	7052-70HVPT	7252-70HVPT
Barrel Heater 230 V W/PT	2052-70TZHV	N/A	N/A

### **Barrel Insulators:**

Barrel Flange Insulators Upper	8052-142MU	8052-142MU	8052-142MU
Barrel Flange Insulators Lower	8052-142ML	8052-142ML	8052-142ML
Lower Barrel Insulator	2052-72M	6052-72	7252-72
Upper Barrel Insulator	9052-75M	6052-75	7252-75
Heat Chamber Cover	8052-62M	8052-62M	7252-62M

## **Barrel Plunger Parts:**

Plunger Top Insulator	2052-46	2052-46	2052-46
Piston Tip	2052-66	2052-66LL	2052-66LL
Piston Tip / Lined Barrel	2052-66H	2052-66LLS	2052-66LLS
Plunger Rod (only)	0051-43	6052-15	6052-15
Plunger Guide Bushing	0051-44	6052-14	6052-14
Complete Plunger Assembly	2052-300	6052-300	6052-300
Complete Plunger/Lined Barrel	2052-300H	6052-300LLS	6052-300LLS

## **Crosshead Drive Parts:**

Ball Screw Jack w/ Limit switch	4052-32N	4052-32N	4052-32N
Flange Bearing, crosshead	2052-43	2052-43	2052-43
Motor	7052-22	7052-22	7052-22
Gearbelt	7052-89	7052-89	7052-89

## **Electronic Parts:**

Capacitor	GP1514	GP1514	GP1514
Transformer	GP1512	GP1512	GP1512
Temp Board 115 V	4051-101F	4051-101F	4051-101F
Temp Board 230 V	4051-101H	4051-101H	4051-101H
Main Board	7052-100F	7052-100F	7052-100F
Sensor Board	7052-140F	7052-140F	7052-140F
Power Supply	EHO400	EHO400	EHO400
Fuse 115 V Unit	GP1410	GP1410	GP1410
Fuse 230 V Unit	GP1405	GP1405	GP1405
Rectifier	GP7820	GP7820	GP7820

## **Exterior Parts:**

Leveling Foot	GP3004	GP3004	GP3004
KeyPad	7052-110	7052-110	7052-110
E-stop KeyPad Adder	7052-120	7052-120	7052-120
Front Base Top Plate	7052-01	7052-01	7052-01
Rear Base Top Plate	7052-04	7052-01	7052-01

### **Load Cells:**

Load Cell 1.5 kN	7052-41	7052-41	N/A
Load Cell 5 kN	7052-42	7052-42	N/A
Load Cell 10 kN	7052-43	7052-43	N/A
Load Cell 15 kN	7052-49	7052-49	N/A
Load Cap	7052-45I	7052-45I	N/A

### **Pressure Transducers:**

Pressure Transducer 50 Bar	8052-150MID	8052-150MID	8052-150MID
Pressure Transducer 200 Bar	8052-150M1ID	8052-150M1ID	8052-150M1ID
Pressure Transducer 700 Bar	8052-151MID	8052-151MID	8052-151MID
Pressure Transducer 1000 Bar	8052-152MID	8052-152MID	8052-152MID
Pressure Transducer 1400 Bar	8052-153MID	8052-153MID	8052-153MID
Pressure Transducer 2000 Bar	8052-153MHID	8052-153MHID	8052-153MHID
Pressure Transducer Port Cleaning Kit	8052-155	8052-155	8052-155

### **RTD's, Platinum:**

RTD, Platinum	7052-13S	7052-13	7052-13
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### **Tools:**

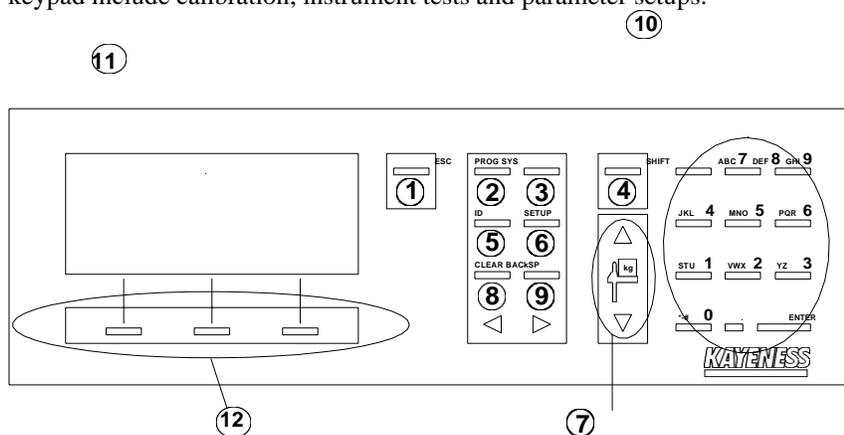
Charging Tool	7052-86	7052-86	7052-86
Patch Cleaning Tool	7052-87	7052-87	7052-87
Bore Brush handle	0051-47	0051-47	0051-47
Bore Brushes (5)	0051-48	0051-48	0051-48
Orifice Drill Bit Vice	0051-38C	0051-38C	0051-38C

Large Powder Fill funnel	<b>0051-80</b>	<b>0051-80</b>	<b>0051-80</b>
Loading Beaker, 20 ml Pyrex	<b>GP0301</b>	<b>GP0301</b>	<b>GP0301</b>
Pull Clip	<b>2052-151</b>	<b>2052-151</b>	<b>7252-151</b>
T-Wrench Handle	<b>2052-156</b>	<b>2052-156</b>	<b>2052-156</b>
Orifice Removal Socket	<b>2052-157</b>	<b>2052-157</b>	<b>2052-157</b>
Orifice Holder Nut	<b>2052-73</b>	<b>2052-73</b>	<b>2052-73</b>
Orifice Cleaning Tool	<b>8052-960</b>	<b>8052-960</b>	<b>8052-960</b>
Tool Rack	<b>6052-16</b>	<b>6052-16</b>	<b>6052-16</b>
Barrel Thread Bottom Tap	<b>8052-93</b>	<b>8052-93</b>	<b>8052-93</b>
Round Orifice File	<b>8052-94</b>	<b>8052-94</b>	<b>8052-94</b>
Never Seize Compound	<b>8052-95</b>	<b>8052-95</b>	<b>8052-95</b>
Wire Brush for Die Nut Cleaning	<b>8052-96</b>	<b>8052-96</b>	<b>8052-96</b>
Cleaning Patches Pack of 100	<b>GP0104</b>	<b>GP0104</b>	<b>GP0104</b>
Complete Tool Kit	<b>D7052_Tool</b>	<b>D7052_Tool</b>	<b>D7052_Tool</b>
Note: Orifice Holder Nut not included			

## Navigating the Keypad

The keypad provides all of the means necessary for programming the Dynisco Polymer Test Melt Rheometer. Other functions that can be performed on the keypad include calibration, instrument tests and parameter setups.

### The Keypad



### Keypad Definitions

**1** ESC (ESCAPE) - Backs out one level from any mode. Quits a test totally

**2** PROG (PROGRAM) - Browse and select a preprogrammed program

**3** SYS (SYSTEM) - Enter self calibration modes or hardware diagnostic modes for testing unit.

**4** SHIFT - Toggle between numerical and alphanumeric input

**5** ID (IDENTIFICATION) - Enter operator and sample identifications

**6** SETUP - Change system parameters

**1** ESC (ESCAPE)

**7** CHROSSHEAD UP/DOWN - Raise and lowers crosshead

**8** CLEAR - Clears present value in editing modes

**9** BACKSP (BACKSPACE) - Destructive backspace

**10** NUMERAL/ALPHA INPUT - Allows for numerical and alphanumeric character input.

**11** DISPLAY - 4 X 20 instrument display

**12** SMART KEYS - Main Keys used for browsing, selecting and entering values and parameters

The ESC key can be used in any screen. During an EDIT function, ESC backs out one level. During a sample run, ESC aborts the test.

## **2** PROG (PROGRAM)

Pressing the PROG key lets the user browse through a list of preprogrammed test parameters. From this mode the user can browse through a list of programs by using the UP or DOWN SMART KEY button and then select the desired program by pressing the ACTIVE SMART KEY button.

## **3** SYS (SYSTEM)

In this mode the user can choose to calibrate, test or quit by selecting the appropriate CALIB, TEST or QUIT SMART KEY, respectively.

\*\*\* Depending on what instrument model you have, prompts may vary \*\*\*

**CALIB**- Select RTD Electronics or Sensor.

Offset calibrations by toggling prompt with UP or DOWN QUICK KEYS and using SELECT to start the calibrations. Please reference **Configuring the Instrument sections (Page) or Appendix #** for more detailed operations of calibrations.

**TEST**- Select either RTD Display, Printer or Display

Brightness tests by toggling prompts with the UP or DOWN SMART KEYS and using SELECT to start the test. Please reference **Configuring the Instrument sections (Page)** for more detailed operations of tests.

## **4** SHIFT

The shift key is used when inputting numeric and alphanumeric characters in the sample, operator or program identification screens. Pressing SHIFT, then the appropriate numeric/alpha key enters the alpha input mode. This mode provides a selection of alpha characters above the SMART KEYS that correspond to the numeric/alpha key pressed. When in the alpha mode it is not necessary to press the SHIFT key to select alpha input every time. Just press the appropriate numeric key to select different alpha characters. Press SHIFT to re-enter the numeric input mode.

## **5** ID (IDENTIFICATION)

Define your operator and sample identifications for each program by pressing the ID keypad. Toggle between operator and sample by pressing the right SMART KEY. Press the ENTER SMART KEY to enter a new identification for the desired parameter.

## **6** SETUP

Setup provides a series of system settings pertaining to your test. After pressing SETUP, scroll through the list of settings using the UP or DOWN SMART KEYS. Select the EDIT SMART KEY to change the parameter accordingly. Please reference **Configuring the Instrument Section** for a detailed description of each parameter

## **7** CROSSHEAD UP/DOWN

These keys will manually operate the CROSSHEAD/LOAD CELL up and down.

## **8** CLEAR

The CLEAR key will clear any active value displayed in the EDIT PROGRAM mode (EDIT SMART KEY then EDIT SMART KEY again) or any text entry. The clear key will also clear the PROGRAM number in the VIEW PROGRAM mode (EDIT SMART KEY) where a new program number can be entered.

## **9** BACKSP (BACKSPACE)

The BACKSP key performs a destructive backspace function in text editing screens.

## **10** NUMERAL/ALPHA INPUT

Using combinations of the SHIFT key and the NUMERAL/ALPHA INPUT keys, either numbers or letters can be entered in the SAMPLE, OPERATOR and PROGRAM identification modes. Pressing SHIFT then the appropriate numeric/alpha key enters the alpha input mode. This mode provides a selection of alpha characters above the SMART KEYS that correspond to the numeric/alpha key pressed. Press SHIFT to re-enter the numeric input mode.

A space can be entered by pressing the **3, YZ** keypad.

## **11** DISPLAY

Your instrument uses a 4 line vacuum fluorescent display, VFD, to display information. Brightness can be adjusted in the SYSTEM TEST mode.

## **12** SMART KEYS

Dynisco Polymer Test provided three keypads below the VFD, to allow for quick navigation of the machine interface. The functions of these keys change depending on the mode of operation. Each SMART KEY is clearly and concisely labeled to assist you in programming your instrument.

# Conversion Factors

## 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 <sup>2</sup> /sec	1e-6
lbf-sec/ft <sup>2</sup>	Pa-sec	4.788026E+01

## Pressure or Stress

To convert From	To	Multiply By
Psi	Pa	6.894757E+03
lbf/in <sup>2</sup>	Pa	6.894757E+03
Atm (STD)	Pa	1.01325E+05
Atm(1 kgf/cm <sup>2</sup> )	Pa	9.80665E+4
Bar	Pa	1.0E+5
dyne/cm <sup>2</sup>	Pa	0.10
ksi (kip/in <sup>2</sup> )	Pa	6.894757E+6

# Example Runs

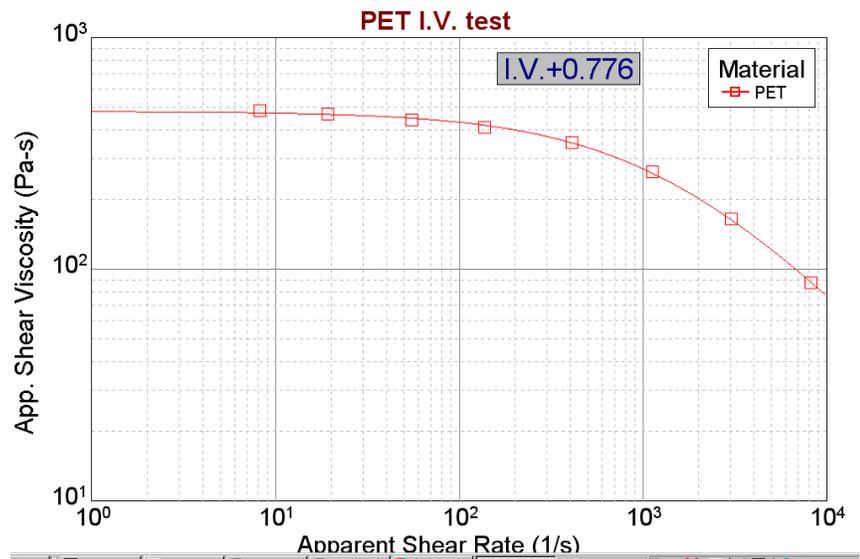
## 1. Polyethylene- Terephthalate I.V. Test

**Charge #:** 3254

<b>Date:</b> 12-Apr-00 13:10	<b>Sample ID:</b> <input type="text" value="Sample"/>	<b>Temperature:</b> 285 C
<b>Operator:</b> Jeff	<b>Workorder:</b> <input type="text" value="3254"/>	<b>Packing Force:</b> 1500 N
<b>Material:</b> PET	<b>Lot#:</b> <input type="text" value="Eastman"/>	<b>Start Position:</b> 120 mm
<b>Supplier:</b> Bill Rousseau	<b>Die:</b> X300-40	<b>Melt Time:</b> 360 s
<b>Customer:</b> KONA Hot Runner	<b>Delay Time:</b> 0 s	<b>Die Diameter:</b> 0.762 mm
<b>Device:</b> LC 9 kN, 2000 lb		<b>Die Length:</b> 30.480 mm
<b>Comments:</b> <input type="text" value="PET I.V. for 6-7 IV"/>		<b>RCAL:</b> 5621 N
		<b>PTS:</b> 9

Point	Force (N)	Pos (mm)	Ram (mm/min)	Time (min)	Stress (Pa)	Rate (1/s)	Visc. (Pa-s)	X-D (mm)
1	8317	144.8	300	6.1	725629	8245.9	88	.81
2	5739	158.2	110	6.2	500708	3023.5	165.6	.75
3	3413	171	41	6.5	297772	1126.9	264.2	.68
4	1670	180.4	15	7.1	145702	412.3	353.4	.61
5	649	184.2	5	7.9	56623	137.4	412	.5
6	279	185.9	2	8.8	24342	55	442.8	.55
7	104	187	.7	10.4	9074	19.2	471.6	-.05
8	46	187.5	.3	11.8	4013	8.2	486.7	-.14

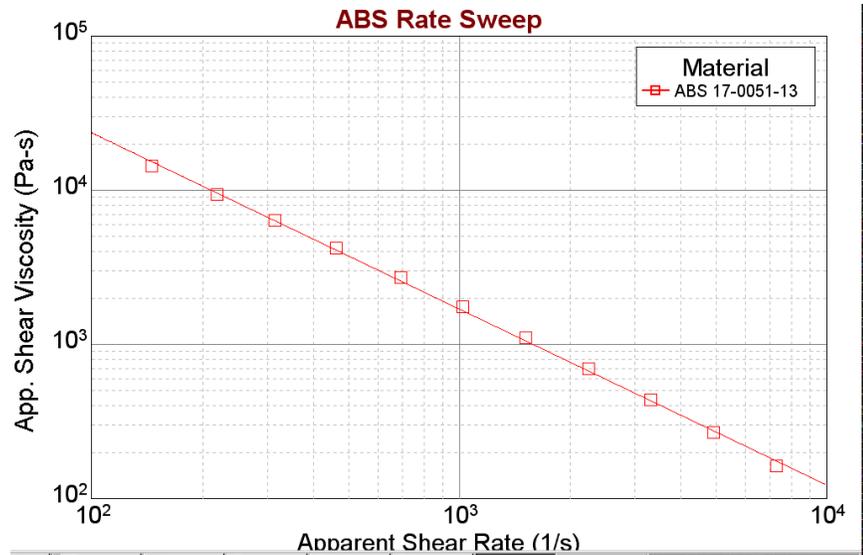


2. Acrylonitrile-  
Butadiene-  
Polystyrene  
(ABS)

Charge #: 3157								
Date:	07-Mar-00 10:08	Sample ID:	17-0051-13 ABS	Temperature:	280	C		
Operator:	Don	Workorder:	3157	Packing Force:	3626	N		
Material:	ABS 17-0051-13	Lot#:	1234	Start Position:	100	mm		
Supplier:	Bill Rousseau	Die:	Z394.20	Melt Time:	240	s		
Customer:	KONA Hot Runner	Delay Time:	0	s	Die Diameter:	1.000	mm	
Device:	PT 1400 Bar	RCAL:	5532	N	Die Length:	20.000	mm	
Comments:	Nylon6/6			PTS:	12			

Point	Force (N)	Pos (mm)	Ram (mm/min)	Time (min)	Stress (Pa)	Rate (1/s)	Visc. (Pa-s)	X-D (mm)
1	6908.4	124.5	600	4	1205467	7296.8	165.2	5.04
2	7647.3	143	405	4.1	1334398	4925.3	270.9	5.04
3	8360.5	158.8	274	4.1	1458846	3332.2	437.8	5.04
4	9088.8	169.2	185	4.2	1585932	2249.8	704.9	5.04
5	9750.7	173.9	125	4.2	1701416	1520.2	1119.2	5.04
6	10306.7	180.1	84	4.3	1798444	1021.5	1760.5	5.04
7	10791.8	191.9	57	4.5	1883080	693.2	2716.5	5.04
8	11236	199	38	4.7	1960597	462.1	4242.5	5.04
9	11551.8	204.9	26	4.9	2015702	316.2	6374.9	5.04
10	11810.2	209.6	18	5.2	2060789	218.9	9414.1	5.04
11	12058	214.5	12	5.6	2104029	145.9	14417.5	5.04
12	8372.6	220.3	274	5.6	1460956	3332.2	438.4	5.04

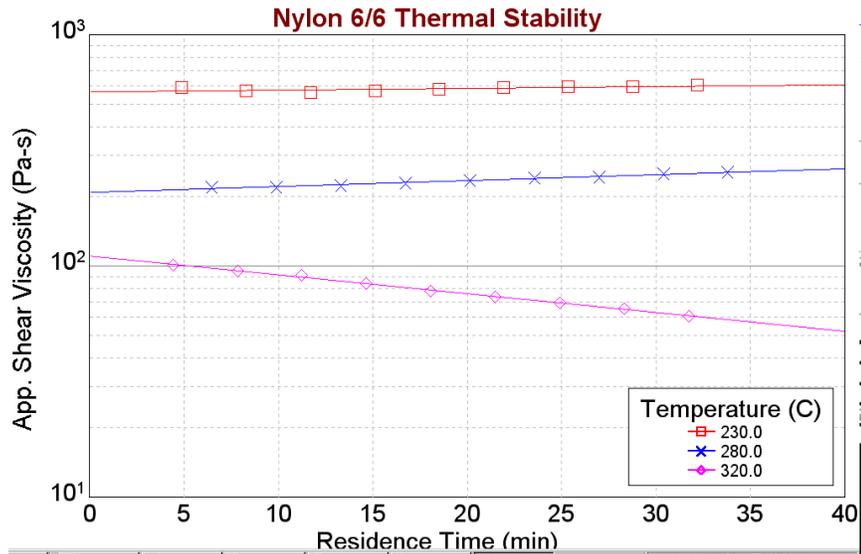


### 3. Nylon

**Charge #: 271**

<b>Date:</b> 07-May-92 18:45	<b>Sample ID:</b> PA6-280T	<b>Temperature:</b> 280 C
<b>Operator:</b> Jeff	<b>Workorder:</b>	<b>Packing Force:</b> 200 N
<b>Material:</b> Nylon 6	<b>Lot#:</b>	<b>Start Position:</b> 4 mm
<b>Supplier:</b> Bill Rousseau	<b>Die:</b> Unknown	<b>Melt Time:</b> 240 s
<b>Customer:</b> KONA Hot Runner	<b>Delay Time:</b> 180 s	<b>Die Diameter:</b> 0.030 mm
<b>Device:</b> No PT Device		<b>Die Length:</b> 1.200 mm
<b>Comments:</b>		<b>RCAL:</b> 0 N
		<b>PTS:</b> 9

Point	Force (N)	Pos (mm)	Ram (mm/min)	Time (min)	Stress (Pa)	Rate (1/s)	Visc. (Pa-s)	Avg Force (N)	Rms
1	284.5	4.3	.7	6.5	110412	499.9	220.9		
2	282.5	4.6	.7	9.9	109636	499.9	219.3		
3	288.9	4.9	.7	13.3	112119	499.9	224.3		
4	295.1	5.2	.7	16.7	114526	499.9	229.1		
5	302.6	5.5	.7	20.2	117436	499.9	234.9		
6	310	5.8	.7	23.6	120308	499.9	240.7		
7	313.5	6.1	.7	27	121666	499.9	243.4		
8	324.1	6.4	.7	30.4	125780	499.9	251.6		
9	329.2	6.7	.7	33.8	127759	499.9	255.6		

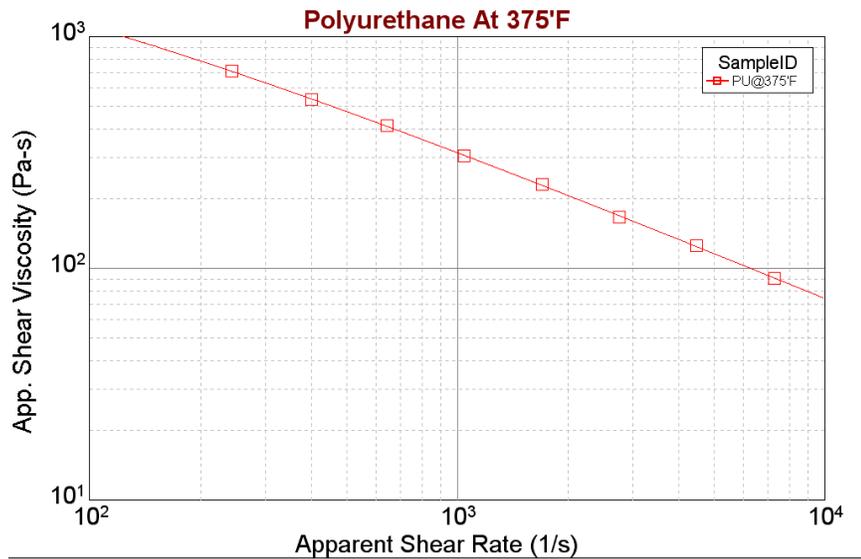


## 4. Glass filled Polyurethane

Charge #: 3322										
Date:	15-May-00 14:02	Sample ID:	PU@375F	Temperature:	190	C				
Operator:	Jeff	Workorder:	3322	Packing Force:	2653	N				
Material:	polyurethane	Lot#:	Unknown	Start Position:	90	mm				
Supplier:	Bill Rousseau	Die:	Z394-20	Melt Time:	360	s				
Customer:	KONA Hot Runner	Delay Time:	0	s	Die Diameter:	1.000	mm			
Device:	LC 9 kN, 2000 lb	Die Length:	20.000	mm	RCAL:	6147	N			
Comments:	PU Test				PTS:	8				

Point	Force (N)	Pos (mm)	Ram (mm/min)	Time (min)	Stress (Pa)	Rate (1/s)	Visc. (Pa-s)	X-D (mm)
1	3812.1	114.7	600	6	665186	7296.8	91.2	1
2	3245.8	140.5	369	6.1	566375	4487.5	126.2	1
3	2647.8	165.6	227	6.2	462014	2760.6	167.4	1
4	2259	190.5	140	6.4	394171	1702.6	231.5	1
5	1841.4	204.5	86	6.6	321316	1045.9	307.2	1
6	1535.7	209.1	53	6.7	267972	644.5	415.8	1
7	1237.2	213.1	33	6.8	215881	401.3	537.9	1
8	995.1	219.2	20	7.1	173636	243.2	713.9	1



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# 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  $\pm 0.0002$  of nominal, ASTM  
specs (D3835) are  $\pm 0.0003$  of  
nominal)

## 2. Cleaning

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

## 3. Bore Gages

nspex 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

For example: Standard Material 1476 is a branched polyethylene with  
(a MFR of  $1.19 \pm 0.01$ ) 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

## 6. Thermometers

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

### ***A. 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.

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# Running a Grey Polypropylene Standard Test

## PROCEDURE

**Purpose of Test:** To determine and confirm the accuracy and repeatability testing for the Dynisco 7000 series rheometers using Montell Grey (Profax 7523) polypropylene standard.

### System Configuration

Plug the rheometer power cord (female connector) into the power connection located at the left rear of the rheometer and the male end into a 110 VAC grounded power outlet (230V for a 230VAC machine).

Plug the PC power cord into the rear of the computer unit and the other end into a 110 VAC volt (230V for a 230VAC PC) grounded outlet.

Repeat step 1.2 with the PC video monitor.

Use the computer connection cable (4051-34) being supplied with the rheometer to connect the computer port located in the rear of the rheometer to the Comm. port #1 (9 pin RS232) located at the rear of the PC CPU.

Verify that the rheometer is level by placing a bubble level on top of the rheometer barrel. Use a crescent wrench (5/8 inch for lock nut, 1/2 inch for foot nut) to thread the feet of the rheometer IN or OUT of the base plate to achieve a level machine.

Turn ON the rheometer by pushing UP on the black power toggle switch and pulling out the red mushroom emergency switch on the front of the rheometer control panel.

Turn ON the PC and video monitor by depressing the power buttons (1= on, 0=off) on the front of each unit.

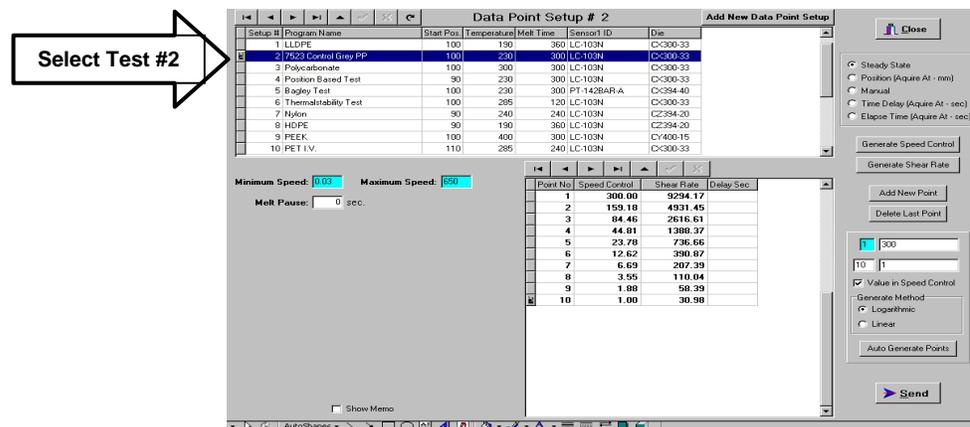
Test Initialization

Start the MS Windows operating software (LabKARS should already be installed on the PC).

Start the LabKARS software by double clicking on the LabKARS icon located on the Windows Desktop Page, the LabKARS Home Page will appear on the monitor.

Click on the "Set Up" Icon at the top of the page to set up your test. The LabKARS "Set Up" page will then appear on the screen.

Verify all testing parameters for "Set Up #2, Profax SB7523 (Grey)" is correct before sending the test setup.



Verify that the data points are setup to perform 10 Data Points from 300 mm/min to 1 mm/min in a logarithmic sequence. (See Figure 2.1 for actual settings)

Verify that Test temperature, Melt time and Start Position are set to proper settings (See Figure 2.1 for actual settings)

Click on the "Send" icon in the lower right hand corner of the page to send the test setup to the rheometer and return to the Rheometer Control Screen.

### Performing a Test Run

Click On the "RCAL" icon, to zero out sensors #1 and sensors #2 no the Rheometer Control screen.

Remove the piston from the rheometer barrel and charge the barrel with nine grams (9.0g) of Dynisco approved Montell Profax 7523 Grey Polypropylene Standard.

Place funnel on top of barrel.

Pour 1/3 of 9 grams into funnel. Use caution to prevent overflow.

Pack material in barrel by using charging tool.

Pour 2/3 of the 9 grams into funnel.

Repeat 3.3.3.

Remove funnel and charging tool.

Separate funnel and charging tool to prevent fusion due to molten polymer.

If testing the LCR 7002 rheometer repeat steps 3.2.1 through 3.2.7 for the second barrel.

Replace the piston (or pistons) back into the barrel.

Move crosshead down to piston bob using the down arrow (↓) located on the keypad. Pressing ↓ and Enter on the keypad simultaneously causes the crosshead to move at a higher velocity. Be sure only to lower the cross to come in contact with piston bob. The pre-loading requirement is a system safety and is necessary to begin the run.

Press the RUN button at the top of the Rheometer Control page to start the test. For Sample ID type "Grey PP Control", for Lot # type "Machine Accuracy Test".

Press the RUN icon.

## Data Analysis

After LabKARS is finished running the test and has purged the excess material from the barrel, you will need to analyze the data for accuracy.

Click on the "Analyze" bar at the top of the Rheometer Control screen to enter the LabKARS Database.



Choosing the Charge number of the test you just ran or would like to view (The last test run should be the last charge number in the LabKARS Database).

Click on the PLOT icon at the top of the page to graph the data for analysis.

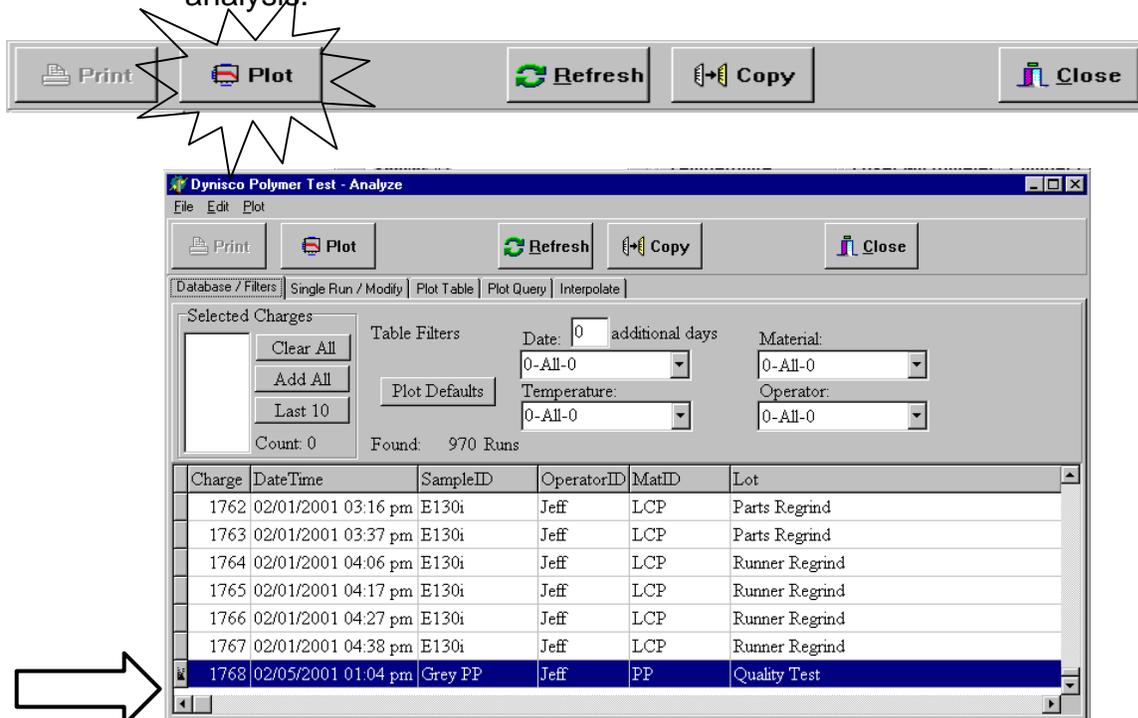
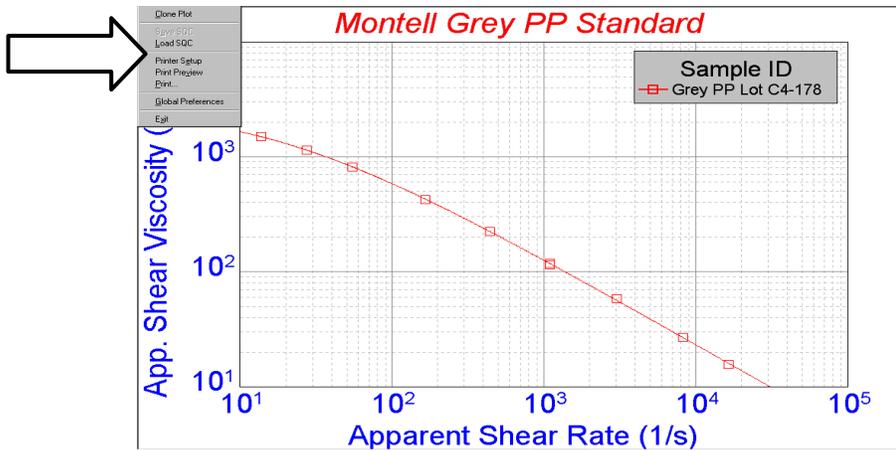


Figure 4.1: LabKARS database and Analyze screen

Connect the data points on the graph using the MODIFIED CROSS option (if testing the LCR 7002 you will have to plot sensor #1 and Sensor #2 together by pressing the "1/2" button at the top of the Plot page).

Click on FILE in the upper left-hand corner of the screen.

Choose the LOAD SQC option to overlay the SQC upper and lower control limits.



Choose the SQC ID that applies to the test and the material sample used to test the rheometer. In this case Profax 7523 @ 230C with the X300-33 Die.

Click on your selection to highlight the ID.

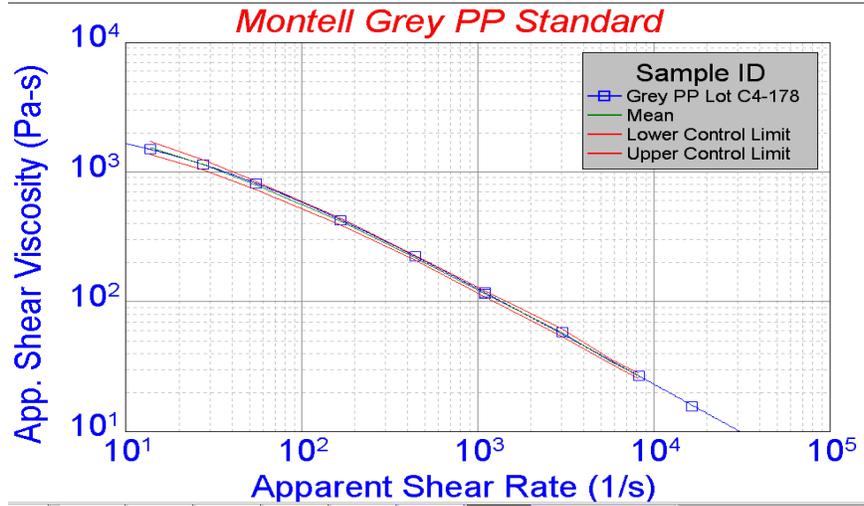
Click on the green LOAD bar to insert the data onto the plot.

ID	Comments
7523 LCR5000 6-29-98	Pos.B. test X300-33 charges 1854-1866
Dowlex 2045 lot:MG0301E153	
Profax 7523 PP@230C	Worn die test:X300-20 charge#s2177-2187&2241-2250
Profax 7523 @230C,X300-33	#s2158,59,62,63,2212,22-26,28-29,51-52,78,2310,11
Profax 7523 @230C	lot#C400017800; X300-33;#s2506-2516;prod. setup
sqc dowlex 19050	
jim's gray	training

Index	X Value	Y-Mean	Y-Std.Dev.	SQCCOV
40	13.7	1560.3	59.64	3.8224
40	27.5	1154.2	35.27	3.0561
40	55.0	804.5	19.91	2.4746
40	164.9	422.1	7.93	1.8785
40	439.8	220.8	3.41	1.5422
40	1099.4	116.9	2.15	1.8383
40	3023.5	58.0	1.31	2.2504
40	5772.1	34.9	0.49	1.3951

If data curve does not fit within control limits, recheck rheometer calibrations, verify material Lot Numbers and/or recheck the rheometer for proper assembly and configuration. If your data curve fits within the upper and lower control limits of the plot, the machine is operating correctly.



Repeat steps 3 – 4.12 three times. Data will be logged for both sensors. All 3 curves should lie within the SQC tube.

**Clean the barrel and prepare the rheometer for the next test.**

## RAM Rate / Shear Rate Table

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									
DYNISCO POLYMER TEST Galaxy V Based on rheometer 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 in./min,  $\dot{\gamma}$  is the desired shear rate, D is the die diameter.

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

---

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# Glossary of Terms

<b>Cross head or RAM</b>	Mechanical device onto which the Load Cell is mounted.
<b>Laser Micrometer</b>	Device used to measure extrudate swell.
<b>Piston rod</b>	Plunger used to extrude material through die.
<b>Barrel Pressure Transducer</b>	A device used to measure pressure at the die in the barrel of the rheometer.
<b>Load cell</b>	Device used to measure piston rod pressure. Usually rated in pounds or newtons
<b>L.C. CAPACITY</b>	Maximum pressure the load cell will read with out becoming damaged.
<b>L.C. SCALING</b>	The percentage of the L.C. capacity that will be required to run a test.. Example: If you are using a 10kN L.C. or P.T., and only 5kN is needed to test, then the L.C. SCALING should be set at 50%.
<b>L.C. GAIN</b>	Rate of voltage to force scale. Example: 2.5 volts =200n. (V=F).
<b>L.C. RCAL</b>	Recalbrates the L.C. or P.T. back to a zero reading.
<b>RTD</b>	(Resistive Thermal Detector) A device used to measure temperature.
<b>Sample Length</b>	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 that control plunger speed. It should be set to values other than zero when mimicking a MCR rheometer 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 <b>Sample Length</b> 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 <b>Sample Length</b> 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 whatever speed is necessary to keep 900N 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).

## **Terminal Force**

Terminal force is a software protection force for the rheometer. If the ***Terminal Force*** is observed by the rheometers on board computer it will abort the test. The plunger movement will stop; the rheometer will try to purge the barrel of its contents. If it cannot 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. (if you are having trouble purging the rheometer) 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.

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# Index

## A

A RATE SWEEP 24  
A Time Sweep Step-by Step 20  
ABS 92  
Accuracy and Reproducibility 46  
Apparent Shear Stress 61  
Arrhenius expression 60  
ASTM D3835 75

## B

Bulk Viscosity 53

## C

CaCO<sub>3</sub>, 60  
carbon black 60  
Cleaning Up a really Big Mess 26  
Clearing program memory 13  
Computer connection 9  
Control material 88  
Conversion Factors 90

## D

Die nut removal wrench. 8  
Dilatant 59

## E

**EMERGENCY STOP** 12  
**ENGLISH** 30

Index

Example Programs (English Units) 98  
Extension 52

## F

Force Calibration 65

## G

Grooved Tip 11

## H

Hand Tools 8  
How to Order 82

## L

Laser Micrometer Connection 9  
Load cell 10  
Load cell assembly 10  
Loading the Barrel 17

## M

Rheometer Configuration Codes 13  
Maintenance 65  
**Melt Force** 28  
**Melt Time** 28  
mercury thermometers 14  
**Metric, Poise** 30

## N

NIST Traceability 65  
NYLON 6 92

## O

O-ring  
  installation 10  
  part number 10  
Oven cleaner 26

## P

***Park Position*** 28  
Parts List 82  
Pa-sec 90  
PET 60  
PMMA 91  
Poise 90  
***Positions*** 29  
Pressure Transducer  
  Activating 34  
  calibrating 72  
  Plug 10  
  Why Use? 62  
Printer connection 11  
PRT  
  what is it? 8  
Pseudoplastic 59  
Purging the rheometer 16  
Putting the Die in the Barrel 17  
PVC 16

## R

Rate Sweep  
  Example English 99  
RCAL 72  
Rheometer Keypad Functions 89  
Rheopectic 60  
***RUN # START*** 30

## S

***S.I., Pascal*** 30  
Setting the SAMPLE ID name 22  
**SHIFT DN** 89  
**SHIFT PRNT** 89  
**SHIFT RUN** 89  
Simple Shear 52  
SPARE PARTS LIST 85  
***Start Position*** 28

## T

Temperature Calibration 70  
***Terminal Force*** 30  
Test Delay 30  
Thixotropic 60  
Time Sweep  
  example english 98  
TiO<sub>2</sub> 60