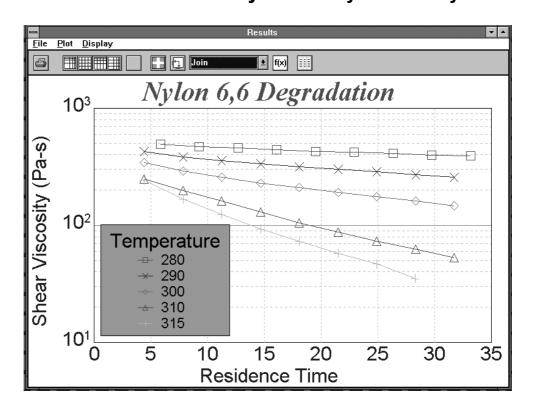
**Instrument and KARS for Windows Software** 

# LCR 5000 & 6000 Series Operation Manual

Version: 1.1 4/00 Dynisco Polymer Test Systems



#### **Notice**

This manual contains descriptions, drawings and specifications for a Dynisco Polymer Test Systems Product. Equipment or products made prior to or subsequent to the publication date of this manual may have parts, features, options or configurations that are not covered in this manual.

Specifications contained herein are subject to change by Dynisco Polymer Test Systems without prior notice. Dynisco Polymer Test Systems is not responsible for errors or omissions that may be contained herein or for incidental or consequential damages in connection with the furnishing or use of this information. The information contained in this manual is the property of Dynisco Polymer Test Systems, unless otherwise stated.

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:

Dynisco Polymer Test Systems **Attention:** Technical Publications Department PO BOX 709 Morgantown, PA. 19543-0709

www.dynisco.com



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## 1. Introduction

## 1.1 About Dynisco Polymer Test Systems

Dynisco Polymer Test Systems is a manufacturer of laboratory test equipment supplying capillary rheometers, melt indexers, a variety of impact testers, coefficient of friction testers, contact angle testers, film tensile testers and other small test devices. Through its innovative work with electronics and mechanical design, Dynisco Polymer Test Systems has carved a niche in the market place by providing the highest performance per cost ratio in the business.

The company was founded in the late '60's in Honey Brook PA, and is built on two principles: quality workmanship and outstanding service. Named Kayeness Polymer Test Systems at the time, The mission of the company has grown to meet the physical testing needs of the plastics industry by providing high quality equipment and services at a low cost. Kayeness Corp was purchased in 1988 by Dynisco Inc located in Sharon, MA. Dynisco, an ISO 9002 qualified business, is the world's largest supplier of pressure transducers for polymer processing and is now located at 38 Forge Parkway, Franklin, MA 02038.

## 1.2 How to Contact Dynisco Polymer Test Systems

Before calling Dynisco Polymer Test Systems be sure you have gone through the "Answers to common questions" section of this manual. In order to help us handle your questions as quickly as possible, have the following items ready when you call:

- Instrument name and model number
- Instrument serial number (on back panel)
- Current version of firmware (Power up the unit to see)
- Computer system make and model
- Current version of operating software (if applicable)

Call DYNISCO POLYMER TEST SYSTEMS directly at (610) 286-7555 and ask for technical support. Should you wish to comment or query in writing, address to:

Dynisco Polymer Test Systems: Customer Service 115 Thousand Oaks Blvd., PO Box 709

Morgantown, PA 19543-0709

Email any comments, suggestions, complaints or requests to Dynisco Polymer Test Systems via the INTERNET at:

www.dynisco.com

## 1.3 What's new with the LCR 5000 & 6000 Series

- Auto Scaling The use of potentiometers for force calibrations has been practically eliminated. You still must perform one initial force calibration. After that, you select the new device you are using in the software. The software then performs the necessary scaling to that measurement device
- Bi-directional Communications The instrument can now be controlled by a personal computer
- Steady State Algorithms The software can now determine when your material
  has met a steady testing state and proceed to acquire the next experiment point
- Windows Based Software- KARS is now a fully functioning Windows application featuring multitasking and OLE compliant cutting and pasting
- **45 Point Experiments** The instrument is now capable of acquiring up to forty-five data points in a single barrel charge.
- **BTP Temperature Probe** A new temperature probe, specifically designed for our thermal chamber (barrel), has been introduced. This probe eliminates mercury filled thermometers and provides a greater degree of accuracy

# 2. Getting Started

## 2.1 Unpacking the Instrument

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. You may also want to save the crate for future shipments back to the factory for upgrades, service or for your own relocation.

Remove the lid to the crate, labeled TOP, by removing the wood 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.

## 2.2 What you've Received

Depending on what you've ordered, you should receive five to seven boxes. A *typical* shipping list would include packages for:

Computer (Shipped separately)
Monitor (Shipped separately)
Printer (Shipped in instrument crate or separately)
Accessories Box (Shipped in instrument crate or separately)
Laser Micrometer (Shipped separately)
Chart Recorder (Shipped separately)
Plotter (Shipped separately)

The accessories box contains manuals, tool kits, safety guards, thermometers, power tree and small options. Please inventory against original order and confirm all pieces are present.

## 2.3 Bench Requirements and Placement

Typical laboratory benches are too high for efficient use of the instruments. Cleaning can be difficult and requires awkward hand positions and forces that could lead to repetitive motion or back discomfort. We strongly suggest a bench height of

29 inches (desktop height) for an average height operator. Place the front of the instrument flush with the edge of the table. This will prevent the operator from having to bend forward excessively when cleaning the barrel and allow easier access to the back of the machine. As a minimum, the lab bench should easily be able to support the instrument and operator (approximately 500 lb.). The bench top should also be able to withstand hot dies and tools being dropped on them. Carpet protection is necessary near the instrument 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 your material supplier to assess the magnitude of your ventilation needs. You may wish to consider these ventilation needs when positioning the instrument in the laboratory.

## 2.4 LCR5000 at a Glance

The LCR5000 is versatile and easy to use; yet offers a very sophisticated material characterization, data analysis and reporting capabilities. The instruments' wide range of standard features include: 45 Shear Stress or Shear Rate points, automatic melt stability determination, Bi-directional communications, PID based temperature control and precision servo-drive motor.

The LCR5000 uses a 175 mm (7.0") barrel, thermally controlled by dual zone heaters, Platinum RTD and adaptive PID algorithms to ensure temperature control to within +/- 0.2° C up to 430° C. Measurements are taken by a load cell driven by a highly efficient DC drive servo coupled through a low friction Ball Screw gear box obtaining measurement speeds from 0.1 to 600 mm/min. Maximum force is 9Kn.

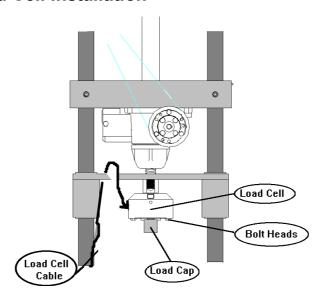
## 2.5 LCR6000 at a Glance

The LCR6000 is versatile and easy to use; yet offers the most sophisticated material characterization, data analysis and reporting capabilities. The instruments' wide range of standard features include: 45 Shear Stress or Shear Rate points, automatic melt stability determination, Bi-directional communications, PID based temperature control and precision servo-drive motor.

The LCR6000 uses a 200 mm (7.88") barrel, thermally controlled by triple zone heaters, Platinum RTD and adaptive PID algorithms to ensure temperature control to within +/- 0.2° C up to 430° C. Measurements are taken by a factory load cell or barrel pressure transducer. A highly efficient DC drive servo coupled through a low friction Ball Screw gearbox obtains measurement speeds from 0.1 to 600 mm/min. with a maximum force of 9Kn.

## 2.6 Load Cell Attachment

#### 2.6.1 LCR5000 Load Cell Installation



Find the load cell (blue) with connected cable in one of the boxes and be sure it is NOT labeled "Calibration Cell". The actual deflection (which measures the force) occurs on the side of the cell with 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 bottom side of the load cell with the exposed bolt heads (active end). The instrument plunger should fit into the load cap when a test is underway. Depending on your instrument, connect the load cell to the crosshead of the machine as follows:

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

#### 2.6.2 LCR6000 Load Cell Installation

Reference Figure 1 shown below

Tools Required: None

- Remove load cell from its labeled packing, and "plug in" the load cell to the load cell receptacle on the back panel of the instrument.
- Plug the Rheometer unit (or LMR) into the power source and turn ON the instrument. "Caution! Keep hands and fingers out of LMR at all times". Normally the load cell identifies obstacles in the path of the ram and will shut the instrument down if an obstacle is present. But with no load cell installed on the LMR at this time, this safety device is active. After the LMR has found its home position (i.e. motor has stopped moving), press the down button (the down arrow on the keypad) to move the crossbar down to an accessible location.
- When the lock nut (see Figure 1) is fully in view and past the back panel edge, depress the reset key.
- Turn the instrument OFF and unplug it from the power source and disconnect load cell.
- Install the load cell by screwing it fully on to the lock nut as shown in Figure 1. Hand tighten (snug tight) the load cell onto the lock nut. The cable as it exits the load cell, should be pointed at the 10:00 position as viewed from the top of the instrument. The strain relief of the load cell cable as it exits the load cell housing must clear the back panel of the LMR. If it does not contact Dynisco for assistance.
- Loop the load cell cable around the crossbar and route the load cell cable down the back left-hand side of the instrument to the load cell receptacle. Plug the load cell connector into the receptacle located on the back panel of the LMR
- Plug the instrument into the power source and turn the unit on.
- Press the "Test" button on the front of the LMR. The display will ask if you want
  to do a "Temp Test?" Press the "No" button. Next the display will ask if you
  want to do a "Force Test?", press the "Yes" button and verify that the load cell is
  working properly. The display should change as you push on the load cell. If
  the displayed values change you're done. If not, recheck all connections or call
  DYNISCO POLYMER TEST.

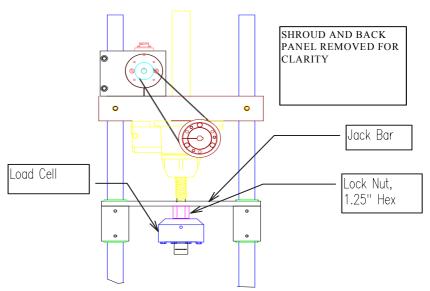


Figure 1

#### 2.6.3 Load Cell Shim Installation

Reference Figure 1 shown below

Tools Required: None

- Plug LMR into power source and turn instrument on. After the LMR has found home position (i.e. motor has stopped moving), depress the down button (the down arrow on the keypad)
- When the lock nut and load cell (see Figure 1) are fully in view and past the back panel edge, press the reset key.
- Turn the instrument OFF, unplug it from the power source and remove existing load cell.
- Install the second load cell by screwing it fully on to lock nut as shown in Figure 1. Hand tighten the load cell onto the lock nut. The cable, as it exits the load cell, should be pointed at the 10:00 position as viewed from the top of the instrument. The strain relief of the load cell cable as it exits the load cell housing must clear the back panel of the LMR.
- If the cable for the new load cell does not point in the correct position, then note the approximate number of degrees of rotation required to bring the load cell into the correct position. 0.010" thick shims are provided with the LMR 6000 and each shim represents 65° of counterclockwise rotation Install the required number shims to bring the load cell cable to the proper exit point.
- Loop the load cell cable around the cross bar and route the load cell cable down the back left hand side of the instrument to the load cell. Plug the load cell connector into the receptacle located on the back panel of the LMR
- Plug the instrument into the power source and turn the unit on.
- Press the "Test" button on the front of the LMR. The display will ask if you want to do a "Temp Test?", press the "No" button. Next the display will ask if you want to do a "Force Test?" press the "Yes" button and verify that the load cell is working properly. The display should change as you push on the load cell. If

the displayed values change you're done. If not, recheck all connections or call DYNISCO POLYMER TEST.

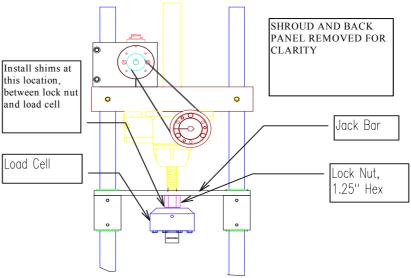
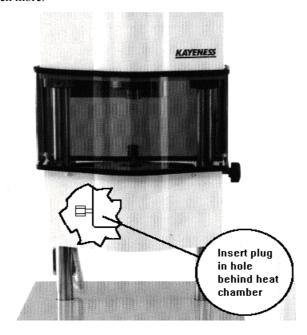


Figure 1

## 2.7 Installing the Barrel Transducer Plug (LCR6000)

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

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

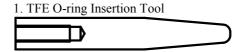


## 2.8 Installing an O-Ring on the Piston Tip

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

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

#### **Tools & Parts**

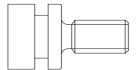


(KAYENESS Part No. 2052-152)



2. TFE O-ring (KAYENESS part No. 2052-66R)

For testing low viscosity materials at temperatures below 300 °C a PTFE O-ring can minimize leakage past the plunger tip.



3. O-Ring Grooved Tip

(KAYENESS Part No. 2052-66G)

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

With the tip free, screw the tip into the insertion tool, and then remove the old oring from the groove using a knife. Soften the new oring by heating it (the top of the barrel works well), and insert the new ring over the tool and into the groove in the tip.

The new o-ring must now be sized to fit the barrel. To do this, apply a small amount of anti-seize to the tip threads and reassemble the tip onto the rod. With the barrel at its normal operating temperature and the die removed, try to install the tip and o-ring into the barrel. While twisting the rod clockwise (counterclockwise will loosen the tip), slowly push the tip into the barrel, removing the extra o-ring material and conforming the new o-ring to the barrel size. This operation will actually shear a

small amount of the o-ring off. Once the tip and new o-ring can be inserted into the barrel, clean the barrel. The machine is now ready to be operated again.

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

## 2.9 RTD Connection

Current instruments do not require installation of the RTD probe (unlike earlier versions of the instruments). LCR Series instruments come with the RTD already installed under the top of the barrel insulator plate.

## 2.10 Safety Guard Attachment

#### 2.10.1 Series 5000

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

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

#### 2.10.2 Series 6000

Safety Shields are attached to the LCR6000 prior to shipment. There is no need for assembly

## 2.11 Power Connection

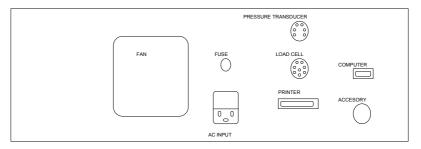
Make sure you have the correct voltage to run your instrument (i.e. 120V or 220 V). The voltage is clearly marked on the back of the instrument. First find the surge protector/power box (Power Tree). This is an extension cord of sorts. Plug it in and make sure the power is OFF (the ON switch should not be lit). There are a number of power cords supplied with your order, the thickest cord (heaviest) is used for the LMR instrument. Connect the thickest cord to the back of the instrument and into the isolated filter bank #3 in the power box. Power to the PC and its Monitor should connect to isolated filter bank #1. The printer and plotter can be connected to any remaining outlet.

## 2.12 Powering up the Instrument

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

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

## 2.13 Connecting Peripherals



#### **2.13.1 Printer**

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

Verify the printer in enabled. Press **RESET** then **SHIFT** then **PRINT** on the keypad to enter the options mode. Enter **YES** until the display reads **PRINTER ATTATCHED** =. Press **NO** until a YES is displayed after **PRINTER ATTATCHED** =. Now the display should read **PRINTER ATTATCHED** = **YES**. Press the **YES** key and then press **RESET**.

Test the printer by pressing **PRINT** on the keypad and enter **YES** to print all programs.

## 2.13.2 Computer

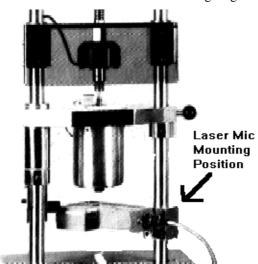
The data processing system (optional) consists of a personal computer and the WinKARS software package. The link between the computers COM1 port and the instrument is connected via a DB9 connector on the computer side and a round AMP connector on the instrument side. All connection ports are clearly labeled. Generally

there is only one round 9 pin connector on the back of the instrument to connect to; however, sometimes real time analog force output is also supplied which has the same type connector. Connect the PC COM1 line to the 9 pin round AMP connector on the back left side of the instrument which does not have a round zero adjust knob above it. This knob adjusts for a zero offset on the analog force signal (when the option is requested).

Verify the computer is enabled and that the printer is disabled. Press **RESET** then **SHIFT** then **PRINT** on the keypad to enter the options mode. Enter **YES** until the display reads **PRINTER ATTATCHED** = ?. Press **NO** until a NO is displayed after **PRINTER ATTATCHED** =. Now the display should read **PRINTER ATTATCHED** = **NO**. Press the **YES** key to save and continue to press **YES** until **PC ATTACHED** = ? Is displayed. Press **NO** until a NO is displayed after **PC ATTATCHED** =. Now the display should read **PC ATTATCHED** = **YES**. Press the **YES** key to save and the press **RESET**.

#### 2.13.3 Laser Micrometer

The Laser Micrometer Measurement head is mounted to the Instrument pole, just underneath the barrel chamber. Please reference the following diagram



The Laser Micrometer (Optional) can be connected directly to COM2 on the PC. Connect the Laser Display unit (connection labeled SCANNER 1) to the measurement head using the long 20' cable with round connectors at each end. Connect the Laser Display Unit (connection labeled SERIAL) to the computers COM 2 port with the supplied cable.

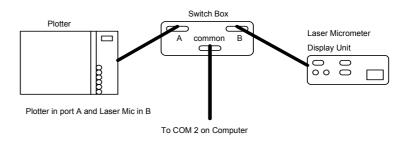
#### 2.13.4 Plotter

With the supplied cable, connect the plotter output port to COM 2 of the computer. Load the supplied pens and power up the components. KARS software should recognize a plotter is attached in the upper left-hand corner of the main screen.

## 2.13.5 Both Plotter and Laser Micrometer Together

The cable connects from the back of the Series 900 Laser scanner box to the 25-pin connector labeled SERIAL. The other end of the cable, a 9 pin (DB9) connector,

goes into Channel A of the switch box. Connect the end of the molded cable (DB25, 25 pin) to the plotter serial connector and the 9 pin (DB9) side to Channel B of the switch Box. Finally connect the output from the switch box (middle bottom) to the DB9 COM2 port on the back of the PC. Leave the switch box set to Channel A (LASER normally, this will allow KARS to initialize the laser when the KARS program is started. Switch to Channel B only when plotted output is desired. Laser data collection will not be possible when the plotter is being used.



#### 2.13.6 Chart Recorder

This connection is an option and is only available if specifically ordered. Connect the Chart Recorder to the instrument port labeled CHART RECORDER. Located above this connection is a ZERO set potentiometer. Power up the Chart Recorder and adjust the zero scale with this potentiometer.

## 2.14 Software Installation

KARS Software is available for DOS and Windows environments separately. Please follow the procedure for the operating environment that you purchased. If you purchased your PC from DYNISCO POLYMER TEST SYSTEMS, the software is already installed on your computer.

#### 2.14.1 DOS version installation

Insert the program diskette into the diskette drive. Change to that drive (i.e. A:\\_) and type INSTALL. Follow prompts on computer screen.

If you have an existing copy of KARS, either overwrite it or install KARS to a new directory. Installing over another KARS version will only delete the main KARS program, replacing it with the current version. It may add additional example files to those that exist. No data directories or data files in the directory will be deleted. It is a good idea to back up your old copy of KARS.EXE.

To back up your old .EXE file, either have your IS department rename the file, call Dynisco Polymer Test Systems technical support or use the following DOS command: C:\COPY C:\KARS\KARS.EXE KARS.OLD, press enter.

#### 2.14.2 Windows version installation

KARS for Windows uses one "distributible utility" for database management called Borland Database Engine (2 diskettes). WinKARS is approximately 7 diskettes.

1. Insert 1 of 2 Data Base Engine Installation Diskette

- 2. From Windows run SETUP off of the diskette.
- 3. Follow directions on screen. Accept all defaults
- 4. Insert 1 of 7 WinKARS Installation Diskettes
- **5.** From Windows run INSTALL off of the diskette.
- 6. Follow directions on screen. Accept all defaults
- 7. When installation complete, quit Windows and Soft Boot the computer
- 8. Launch Windows
- 9. Run DBECFG.EXE located in the Kars for Windows Group.
- 10. Click on "ALIASES" at bottom of screen, see figure below.
- **11.** Add the following aliases:

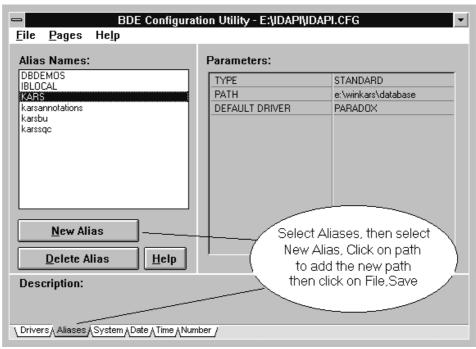
KARS PATH=C:\WINKARS\DATABASE

KARSBU PATH=C:\WINKARS\BACKUP

KARSSQC PATH=C:\WINKARS\SQC

KARSANNOTATIONS PATH=C:\WINKARS\SQC

12. Select File and Save.



13. Exit BDE Configuration and run WinKARS located in KARS for Windows group on desktop.

## 2.15 Upgrading to KARS 6.0

#### 2.15.1 Overview

This section is only for customers presently using Galaxy 5 equipment. The following section describes in detail the steps necessary to upgrade your system to KARS 6.0. Please read through the entire section before starting your upgrade.

The KARS upgrade package includes software and firmware for the system upgrade. The software is supplied on a 3.5 inch floppy disk or can be e-mailed and is added to your computer's hard drive via the installation program. This will be covered later in this section. The firmware upgrade involves replacing EPROM chips on the inside of the Galaxy V instrument itself. Care must be taken while performing the firmware upgrade or the instrument may be damaged. After reading all of the instructions and you have ANY questions about performing this upgrade, please call DYNISCO POLYMER TEST SYSTEMS technical support before proceeding.

#### 2.15.2 Tools Required for Upgrade

The following tools are needed for installation of the upgrade:

- 1 medium sized standard (slotted) screwdriver
- 1 small sized standard (slotted) screwdriver, jewelers size
- 1 medium sized Phillips (star) screwdriver
- 1 eighth inch (1/8") Allen (hex) wrench

#### **2.15.3 WARNING**

Follow the upgrade instructions in order. If you have any questions or concerns about the software/firmware upgrade please call DYNISCO POLYMER TEST SYSTEMS before beginning the upgrade process. If you are uncomfortable performing this upgrade, please make arrangements with the DYNISCO POLYMER TEST SYSTEMS service department for an on-site upgrade package.

#### 2.15.4 Firmware Installation

#### **Read-Me First!**

Please read through these instructions first before beginning the installation. If after reading the following directions you have any questions, please call **DYNISCO POLYMER TEST SYSTEMS** Technical Support:

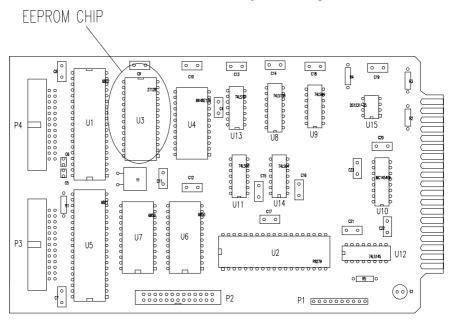
#### 2.15.4.1 The first EPROM

The first step of the upgrade involves writing down instrument parameters from the old software for comparison after the installation of the new software/firmware is complete.

**1.)** Make sure you have a hard copy of all your test parameters. Press **SHIFT** then **9** on the keypad. Record the number (machine configuration code) displayed. Press RESET to escape out.

Record R-CAL values that are stored in the currently running version of KARS. From the KARS main menu, press <F9> . Record the R-CAL values that are on the screen: "factory load cell R-CAL value" and "Pressure Transducer R-CAL Value".

- **2.)** Briefly touch the poles of the instrument to discharge any static electricity built up on your body. Turn off and unplug the machine
- **3.)** Detach the stainless steel cover on the machine by removing the screws located on the side of the machine. You only need to remove the front cover on Capillary Instruments.
- **4.)** Remove the front panel bolt with a one-eighths inch (1/8") Allen wrench and swing open the front panel.
- **5.)** The electronics card, known as the **Main Computer Board**, located in the front of the machine contains the EPROM. Remove the electronics safety shield and the board clamp mounted on the base of the machine. Remove all of the connectors leading to the MCB taking note of where they where originally connected, and remove the board.
- **6.)** Locate the EPROM on the MCB. It is clearly marked on the schematic below and is indicated physically by a white tag. Note the notch on top of the old IC chip. The new chip's notch should physically be in the same place as the original. Remove the chip by gently prying underneath it with a medium size screwdriver and replace it with the new chip. Take care not to bend any of the legs of the IC when replacing it.
- 7.) Replace the chip with the new chip labeled KWIN#.##, where #.## is a version such as 1.01. Reinsert the circuit card following the reverse procedure.

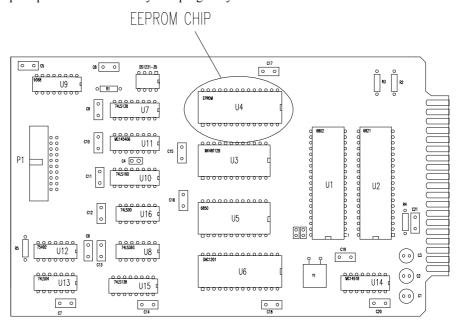


MAIN COMPUTER BOARD LAYOUT

#### 2.15.4.2 The second EPROM

If you have received only one EPROM, you may skip this section

- 1. The electronics card, known as the Computer Board One, located second from the front of the machine contains the EPROM. Remove the electronics safety shield and the board clamp mounted on the base of the machine. Remove all of the connectors leading to CB1 taking note of where they where originally connected, and remove the board.
- 2. Locate the EPROM on CB1. It is clearly marked on the schematic below and is indicated physically by a white tag. Note the notch on top of the old IC. The new chip's notch should physically be in the same place as the original. Remove the chip by gently prying underneath it with a medium size screwdriver and replace it with the new chip. Take care not to bend any of the legs of the IC when replacing it. Reinsert the circuit card following the reverse procedure.
- **3.** Reassemble the machine. When you power up the machine it is important to perform a memory clear. Press **SHIFT**, **NO** and answer **YES** to the clear all memory prompt. You are now ready to reprogram your unit.



#### 2.15.4.3 Re-Assembly

#### 2.15.4.3.1 Check the Configuration Code

Press **SHIFT> then <9>** to check the machine configuration code. This should be the same as what you recorded earlier (step 1).

- If it is the same, press **YES**> then **RESET**>.
- If not the same, press **<NO>** and enter the correct value.

Then press<YES> then <RESET>.

#### 2.15.4.3.2 Initialize PC Communications

Press **<SHIFT>** then **<PRINT>** to access options mode. Press **<NO>** eight times and then press **<RESET>**. Press **<RESET>** again and verify that the computer beeps. This verifies proper communications between the devices

This completes the firmware installation. The next section of this manual covers load cell and transducer calibration. This calibration is required for machines that use English units. It is strongly recommended for Metric users as well.

#### 2.15.4.4 Load Cell and Transducer Calibration

Firmware installation is now complete. The next step of the upgrade involves resetting the instrument parameters. The parameters must now be re-entered to ensure proper instrument operation. This involves calibrating the load cell and the pressure transducer (if you have one) and inputting the Load Cell RCAL value to the KARS 6.0

#### 2.15.4.4.1 Part 1: Load Cell

1. Press the <+> key on the instrument front panel to activate the load cell sensor. The display should show "Wait LC #00".

#### Verify the Options

**2.** At this point, you need to verify the options. This is done by pressing **SHIFT>** then **PRINT>**. If needed, toggle off with the **NO>** key and select **YES>**. Verify that  $AUTO \ FOffset = ON$ . Press **RESET>** when options have been properly set.

#### **Load Cell Offset Calibration**

**Note:** If you do not have a pressure transducer, R-CAL option is not present so the R-CAL value will be zero (0). Calibrate the load cell Offset and R-CAL by pressing the **<DEL>** key. NOTE: make sure there is no load on the load cell. Verify that the values recorded in step 1 (The first EPROM) are within 5% of the new values. At this point, if you have a metric unit, the R-CAL value is correct the load cell is calibrated.

#### 2.15.4.4.2 Part 2: Pressure Transducer

1. Install the pressure transducer by screwing it into the barrel until hand tight. Make sure the barrel is clean and the electrical cable connected. (If you have more than one pressure transducer RCAL with the largest range transducer unless told to do otherwise by Dynisco Polymer Test Systems. This will allow the *PT Autoscale ON* mode to work best). Give the transducer 5 minutes to come to temperature, then tighten snug with a wrench. Press the < - > key to activate the pressure transducer sensor. The display should show "Wait PT #00".

#### Verify the Options

**2.** Set *PT ATTACHED* = *ON* by pressing **<SHIFT>** then **<PRINT>**. Verify that *PT ATTACHED* = *ON*. Toggle off with the **NO** key and select **ON** if needed. Press **<RESET>** when options have been properly set. Also verify that *PT INSTALLED* = *YES* 

#### **PT Offset Calibration**

3. Make sure the barrel is clean and that the pressure transducer is installed and connected. At the set point temperature tighten it!. Calibrate the pressure transducer Offset and pressure R-CAL by pressing the <DEL> key. Tightening after pressing <DEL> will decrease the accuracy of the measurement. Verify that the values recorded in step 1 (The first EPROM) are within 5% of the new values. In the Set Options screen of KARS for DOS, verify the R-CAL value is correct when the pressure transducer is calibrated.

#### 2.15.4.4.3 R-CAL Values to use in KARS

Note RCAL values for Load cells are nominal, record RCAL after using calibration unit is best.

Device	Nominal	Nominal	Plunger	RCAL	RCAL	-	Max Terminal	Min "Good"	Min "Good"
	Range	Range	Range	English	Metric	Force	Force	Reading	Reading
	(psi)	(Bar)	(lb.)	(lb.)	(N)	(lb.)	(N)	(lb.)	(N)
PT	30000	2069.0	3331.1	2798.1	12446.0	3331.1	14816.7	333.1	1481.7
PT	20000	1379.3	2220.7	1865.4	8297.4	2220.7	9877.8	222.1	987.8
PT	10000	689.7	1110.4	932.7	4148.7	1110.4	4938.9	111.0	493.9
PT	29000	2000.0	3220.1	2704.8	12031.2	3220.1	14322.8	322.0	1432.3
PT	20300	1400.0	2254.0	1893.4	8421.8	2254.0	10026.0	225.4	1002.6
PT	14500	1000.0	1610.0	1352.4	6015.6	1610.0	7161.4	161.0	716.1
PT	10150	700.0	1127.0	946.7	4210.9	1127.0	5013.0	112.7	501.3
PT	2900	200.0	322.0	270.5	1203.1	322.0	1432.3	32.2	143.2
PT	725	50.0	80.5	67.6	300.8	80.5	358.1	8.1	35.8
LC*	45030	3105.5	5000.0	3500.0	15568.0	3000.0	13344.0	30.0	133.4
LC*	18012	1242.2	2000.0	1400.0	6227.2	2000.0	8896.0	20.0	89.0
LC*	9006	621.1	1000.0	700.0	3113.6	1000.0	4448.0	10.0	44.5
LC*	4503	310.6	500.0	350.0	1556.8	500.0	2224.0	5.0	22.2
LC*	2702	186.3	300.0	210.0	934.1	300.0	1334.4	3.0	13.3

Note RCAL values for Load cells are nominal, record RCAL after using calibration unit is best.

Conversion Factor Lb. to N  $\rightarrow$  4.448; Factor Psi to Lb. plunger  $\rightarrow$  0.111036 Factor Bar to Psi  $\rightarrow$  14.5; lb. to RCAL for PT's 0.84; nominal lb. to RCAL for LC  $\rightarrow$  0.7

# 3. Safety Precautions

## 3.1 Use Gloves - "Its HOT!"

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

## 3.2 Electrical Hazards

Your DYNISCO POLYMER TEST SYSTEMS instrument contains high voltage inside the steel base. When this steel cover is opened a plastic cover is revealed. This plastic shield aids in protecting you from these High Voltages. DO NOT remove this cover unless you are instructed to do so by a DYNISCO POLYMER TEST SYSTEMS representative or are experienced with high voltage devices. There are holes cut in the shield that allows access to the necessary items used in routine calibrations. Be sure the outlet used to power the instrument is properly grounded.

## 3.3 Calibration Thermometers use Mercury!

To calibrate the temperature on the instrument a thermometer containing about 8 grams of mercury is used. Every lab with mercury thermometers or equipment containing mercury must be prepared for breakage. Note that mercury exposed to air "evaporates" at room temperature, producing an invisible, tasteless, odorless and dangerous vapor. Thermometers have been used for decades in laboratory equipment and when used properly provide an accurate and effective means of calibration. Keep the thermometer in a safe place were it will not be crushed or otherwise broken. When using the thermometer be careful not to drop or bend the glass. Place hot thermometers onto cotton patches to cool when removed. Never put a hot thermometer in contact with cold metal or cold solvent, the thermal shock can crack or shatter the glass. Mercury is extremely toxic and should be handled accordingly.

A material safety data sheet (MSDS) for mercury (Hg) can be found in the appendix. Observe local, state and federal hazards waste disposal laws when disposing of any broken thermometers. You can find the names of mercury spill kit suppliers in the appendix under support vendors. If packaged in a sealed plastic container and labeled with the following symbol:



Broken thermometers and their spilled mercury can be sent back to the manufacturer. UPS will accept these packages provided they are labeled and the material is in a secure container.

See Princo support vendor for address information at end of manual. Mercury MSDS is also provided at the end of this manual.

## 3.4 Pinch Points

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

An optional front guard with power shut off interlocks can be provided. This shield is tied to a mechanism that prevents operation of the machine when the shield is not in place. The shield inhibits access to the area between the crosshead and the swing arm

## 3.5 Don't remove the RTD

Don't remove the platinum resistance thermometer (PRT or RTD) **UNLESS**, the set point temperature (Melt Temperature) has been set to zero (0.0). Check the temperature in the barrel using the separate hole drilled just for the thermometer that can be found by removing the small Allen head screw just in front of the RTD well.

The RTD is a 2-3 mm thin rectangular section of platinum wire grid used to measure the temperature. It is housed in a 4.75 mm (3/16") steel tube about 125 mm

(5 inches) long with a 90 degree bend on one end. On Galaxy V machine this tube is just to the left of the top of the instrument barrel under a protective metal shield.

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

The LCR Series instruments have the RTD probe buried under the top insulator plate. The top insulator plate must be removed to remove the PRT.

## 3.6 Fumes from the material

Plan for the unexpected when it comes to materials giving off hazardous vapors. Many polymers (PVC etc.) are well known for giving off hazardous fumes at elevated temperatures. An exhaust system 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.

## 3.7 Pretest Safety check

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

## 3.8 Purging the Instrument

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

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

Press **EDIT** on the front panel of the instrument

Press **YES** until the word TEMP. = XXX will appear.

Press 2 3 0 then **YES**; the top display should show 230 for a second or two then the current temperature will be displayed. You should see it rising slowly to 230° C.

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

## 3.9 Before any Measurement

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

## 3.10 Cleaning Up

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

With materials that are thermally stable (less than 5% viscosity changes over 1/2 hour) we recommend cleaning only the barrel between runs of the same material. For materials that degrade we recommend cleaning both the barrel and the die completely. You can clean the die by first running a drill bit up into the die while it is hot and in the instrument. This will make it much easier to get the cleaning drill bit in when the die is removed and the material starts to solidify. Remove the die by extending the arm on the die nut removal wrench to its full length. Attach the socket completely over the die nut. A quick hit on the end of the socket arm is all it takes to remove even an over tightened die. Slide the arm back to its center position on the wrench and spin the wrench while pressing up with one finger below the socket. Be careful because the die is very hot when removed from the barrel. If you don't use gloves you will get burned eventually. Using a cotton rag wipe the top of the die clean. Be sure to get the material out of the conical top section, if your die has such a tapered entrance. Run the supplied drill bit into the die. Remove material that collects in the grooves and repeat until the drill passes easily through the die. On

long dies it may be necessary to clean from both ends to completely clean the entire length of the die. While the die is out look down the barrel bore to be sure it is clean. If it is not run a couple of patches up and down it before putting the die back into position. When the die is out of the barrel it cools down quickly. The longer it is out the longer the wait for the temperature to stabilize. Minimizing the time the die stays out of the machine will increase the number of tests you can run. When the temperature on the front display is within two degrees of the set point you can begin your next test. Loading material will cause a small temperature change even if the temperature set point was achieved. The melt time (360 seconds) will allow ample time to get the temperature to set point before the first data point is collected. Always leave the machine clean. If it is going to sit at room temperature for an extended period of time (More than one week) you may wish to coat the barrel with a light machine oil to prevent rusting. The oil will need to be purged from the machine before accurate data can be obtained.

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

# 4. Rheology

## 4.1 Scope of Manual

In depth discussions of rheology will not be included in this manual. Dynisco Polymer Test Systems will provide to you "The Kayeness Practical Rheology Handbook" either in a Windows Help format or in a hard copy format. There are also many other reference books recommended at the end of this instruction manual.

We will discuss some common rheological topics and ideology in this section, as they apply to the instrument and software.

## **Equations and Corrections**

Viscosity is Shear Stress over Shear Rate:

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

Where  $\eta$  is viscosity,  $\tau$  the shear stress and  $\gamma$  the shear rate.

Apparent Shear Stress:

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

Where  $\tau$  is the shear stress, **F** is force acting on surface area **A**. **F**<sub>p</sub> is the force on the plunger and  $\mathbf{r}_c$ ,  $\mathbf{D}_c$  is the inner radius and diameter of the capillary respectively,  $\mathbf{R}_b$  is the inner radius of the barrel and  $\mathbf{L}_c$  is length of the capillary and  $\Delta P$  is the pressure drop along the capillary

Apparent Shear Rate:

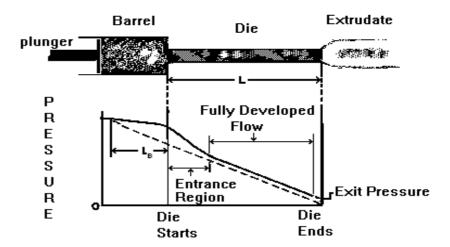
$$\dot{\gamma} = \frac{32Q}{\pi D_{c}^{3}} = \frac{4R_{b}^{2}S}{r_{c}^{3}}$$

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

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

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

#### 4.1.1 Pressure Profile



From the above diagram of the pressure vs. position it should be clear that the pressure falls as the die exit is approached. Even within the barrel there is, at least, some small pressure drop known as the barrel pressure drop or barrel drag effect. Close to the die entrance and just inside the die the pressure drop is fairly dramatic as we force the material to undergo a primarily extensional deformation to enter the die. Once past the entrance region the pressure falls in a linear fashion from the shearing action within the die. Note that the exit pressure is typically non-zero. The barrel pressure drop, the entrance pressure drop, frictional effects and the exit pressure all get measured in addition to the pressure drop in the die. In the previous equations we assumed we were just measuring the pressure drop in the die. If we use the data as collected, that is, the apparent values we over-estimate the viscosity because of the extra pressures in the system. By using several dies of the same diameter but differing lengths. And buy measuring the plunger force for a given shear rate at the same point in the barrel for each die. The extraneous pressures can be removed and the true viscosity calculated as described below under the section Corrected Shear Stress. Why use a Pressure Transducer and When?

#### 4.1.1.1.1 Advantages of a Pressure Transducer

- Frictional effects of plunger tip are removed.
- Barrel Pressure drop from plunger tip to the pressure sensor is removed.
- Some precision improvements can be gained.

#### 4.1.1.1.2 Disadvantages of a pressure transducer

- Reservoir needs flow into it for pressure sensors to stabilize.
- Increased thermal losses from pressure probe.
- Pressure reservoir needs cleaning for materials that degrade.
- Device itself is inherently not as accurate as a load cell
- Smaller dynamic range.

- Mercury used in transducers, care must be taken in handling and removal of device.
- More tedious to clean.
- Must be removed when hot to prevent damage of device. Polymer may adhere to the diaphragm and cause it to fail if removed cold.

#### 4.1.2 Common Assumptions

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

#### 4.1.3 Weissenberg-Rabinowitsch Correction

The Weissenberg-Rabinowitsch correction accounts for the fact that most polymeric materials when flowing through a capillary behave like a Newtonian fluid (shear stress is directly proportional to shear rate) only at very low shear rates. For a constant output flow rate, the velocity of the melt stream in the center of the die is at a maximum. The velocity then falls to zero at the capillary wall. The change in this velocity profile as we move from the center of the die to the wall is the shear rate for that position in the die. Polymeric materials at high flow rates show a much larger change in velocity near the wall of the die when compared to Newtonian materials. At higher shear rates then, the calculated apparent shear rate is an underestimate of the true shear rate and the viscosity is over estimated.

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

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

Where n is the tangent slope of the log true shear stress vs. log apparent shear rate curve at the apparent shear rate being corrected.  $\gamma$  is the true shear rate and  $\gamma$ , is the apparent shear rate described previously.

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

#### 4.1.4 Corrected Shear Stress

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

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

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

Where  $\tau$  is the true shear stress, P is melt pressure, Pc is the intercept obtained for a given shear rate from the above described plot. D is the die diameter, L is the die length. For plunger force measuring devices F is the force on the plunger, Fc is the intercept force on the Bagley plot described above and  $A_B$  is the cross sectional area of the barrel. Devices which measure plunger force must acquire data for a given shear rate (a given line on the graph) at the same position in the barrel for the various dies used. In this way barrel pressure drop effects will be removed along with the other stationary pressures in the system when the Bagley correction is performed.

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

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

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

# 4.1.5 Extradate Swell

Extradate swell is a measure of the increase in diameter of the extradate 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.

# 4.1.6 The Bagley Correction

K.A.R.S. will performs the Bagley correction when sufficient data exists to perform the calculation. By back calculating the applied force from stress values and the die geometry a linear straight-line relation is fitted to Force vs. L/D for each shear rate. The intercept calculated from this fit, if greater than zero, is subtracted from the applied force for all data points at the given shear rate before corrected viscosity's and stresses are calculated.

# 4.1.7 PVC

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

# 5. Test Drive

# 5.1 Purging the Instrument

Place the plunger into the barrel giving it a twist clockwise (looking down from above the plunger) be sure the guide bushing slides down into the top part of the barrel. This guide bushing forces the plunger top to go directly into the load cap attached to the load cell. Check to be sure the plunger is not moving (some materials can force the plunger upwards, especially if overfilling or degassing occurs) then close the swing arm. Tighten the ball-capped screw to fix the swing arm in position. Let the material heat up for about 3 minutes or longer. Time is not critical here; we merely want to give the material enough time to melt so we don't over force the machine trying to push it out.

After about 3 minutes press **SHIFT** then **DN** (down key). The front panel of the instrument will display *Purge?* press **YES** to empty the contents of the barrel through the die. The instrument will drive down until it sees 2 to 3 lb. of load. This occurs when coming into contact with the plunger. The instrument will then increase the plunger speed until either maximum speed is achieved or half the *Terminal Force* is felt by the load cell. The plunger is always driven to the bottom limit switch setting which is about 3 mm (1/8 ") above where physical contact between the plunger and die would be made.

# 5.2 Putting the Die into the Barrel

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

# 5.3 Loading your Sample

Turn the round ball handle on the right side of the swing arm counter clockwise and pull the swing arm open until it is in a comfortable position for you to load the sample. Don't be afraid of opening it too far; you can't. Use the 10 ml beaker to scoop a heaping beaker of pellets from the sample you wish to run. Get the filling

funnel and place it directly over the top of the barrel. The lip on the base of the funnel will fit onto the top of the barrel insulator. Holding the funnel in your left hand gently pour about 2/3 of the pellets from the beaker into the funnel (you may need to tap the beaker against the funnel lightly to eliminate bridging). After putting in only 2/3 of the beaker content use the packing tool to firmly press the material down into the barrel. Push down until you feel little or no movement of the packing tool. It is not necessary to use large amounts of force in packing.

Note: Try not to leave the packing tool in the barrel for too long. If the packing tool heats up material will melt around it and make packing difficult.

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

# 6. Using the Software for your own Experiments

# 6.1 Before any good Instrument Test

Purge the machine and clean everything thorwly. Place the plunger into the barrel giving it a twist clockwise (looking down from above the plunger) be sure the guide bushing slides down into the top part of the barrel. This guide bushing forces the plunger top to go directly into the load cap attached to the load cell. Check to be sure the plunger is not moving (some materials can force the plunger upwards, especially if overfilling or degassing occurs) then close the swing arm. Tighten the ball-capped screw to fix the swing arm in position. Keep packing the force set at 1500 lbs. or less so you don't over force the rheometer during packing do to packing unmelted material samples (don't over force the machine trying to push cold material through the die).

After about 3 minutes melt time, press **SHIFT** then **DN** (down key). The front panel of the instrument will display *Purge?* press **YES** to empty the contents of the barrel through the die. The instrument will drive down until it sees 2 to 3 lb. of load. This occurs when coming into contact with the plunger. The instrument will then increase the plunger speed until either maximum speed is achieved or half the *Terminal Force* is felt by the load cell. The plunger is always driven to the bottom limit switch setting which is about 3 mm (1/8 ") above where physical contact between the plunger and die would be made.

# 6.1.1.1 Notable Instrument Settings

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

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

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

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

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

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

# Other Instrument program parameters found under EDIT:

These parameters are values required to run a test and can be entered by pressing the **EDIT** key on the instruments front panel.

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

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

**Sample Length:** This is the distance over which the speed of the plunger is measured during stress 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 machine or when constant load (stress) runs are required. A typical sample length is about 1/3 of the distance between measurement positions.

During a run *Sample Length* works like this: You've set up a run with all positions 12.5 mm apart starting from a 115 mm start position. The load is constant at each point and set at 900N. The *Sample Length* is set to 5mm. After pressing RUN the test begins like any other test (described in the previous section). When melt time expires, the system then moves at what speed is necessary to keep 900 on the load cell (or pressure transducer). When position 127.5 mm is reached an internal timer is started. When position 132.5 mm is reached the time is stopped and the second point in the test starts (after any delay time pause).

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

#### LCR5000

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

**Terminal Force:** Terminal force is a software protection force for the machine. If the **Terminal Force** is observed by the instruments computer, the computer will abort the test. The plunger movement will stop; the instrument display will display "purge" and the machine will try to purge the barrel of its contents. If it can not the crosshead will return to start position. The terminal force also controls the maximum force allowed during the purging of material from the barrel. The speed of purging will be automatically adjusted so that the maximum amount of force applied is half the terminal force. Normally this value is set just below your load cell capacity (e.g. 1000lb cell set to 990 lb.).

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

S.I., Pascal

#### **ENGLISH**

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

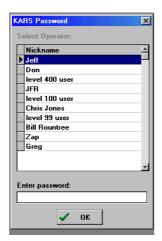
**RUN # START:** This is the run number the instrument includes with the data. Normally when a new **Sample ID** is entered this value should be cleared back to zero by pressing **NO** on the instrument keyboard. This value is automatically incremented every time the **RUN** button is pressed. The run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

# 6.2 Starting the Software

The WinKARS icon is located in the KARS for Windows group on your Windows desktop. Double click on the WinKARS icon on your desktop. Or simply Launch WinKARS by double clicking on its icon.



You will be prompted to chose a WinKARS operator, and enter the password for that operator. Click the OK button. If your name does NOT appears as an authorized operator of WinKARS, you will need to exit out of WinKARS and go into the Maintenance / Operator: DB and add yourself to the list.



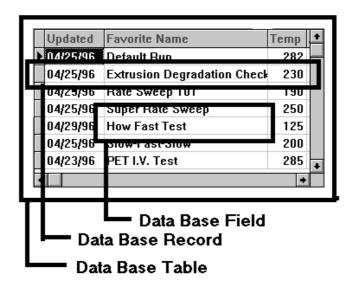
The following operator screen will appear:



Select Help to access on-line help, select Dynisco Polymer Test Systems Practical Rheology Handbook Icon to run rheology help or select Exit to quit WinKARS. RUN, ANALYZE, SETUP and MANTENANCE screens are discussed in their respective section in this manual.

# 6.2.1 Data Bases

WinKARS is extremely Database intensive. This provides the user with easy data manipulation such as sorting and extraction. Following are database definitions that this manual follows.

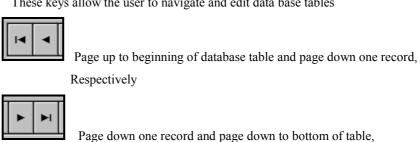


# 6.2.2 Data Base Record Manipulation Keys

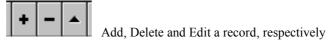
Common to most WinKARS screens are the record Manipulation Keys shown below.



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



Respectively





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

# 6.2.3 Password

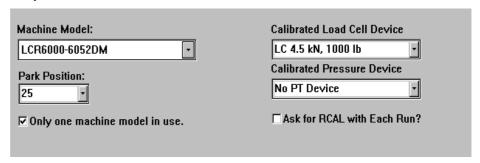
WinKARS protects critical instrument and experiment parameters with a password. The default password is KAYENESS.

To change the password, go into the *Setup* screen from the WinKARS main menu. Select the *Machine Defaults* tab. A radio button will be displayed labeled New Password. Click the button and you will be prompted for a new password. After entering the new password, you will be asked to reenter the new password for verification purposes.

# 6.3 First things First: Setting up the software for your machine

Prior to any experimentation it is extremely important that the software is configured for the type of instrument it is controlling. If it is not properly configured, damage may occur to the instrument or the operator may be injured.

- Enter the **Setup** screen from the main screen by clicking on Setup
- Select the **Machine Defaults** Tab from the upper four tab selections.
- Select the down arrow in the **Machine Model** and select the model that you purchased.
- Click once inside the Machine Nickname box and enter a new nickname for your instrument.



- Select a Load Cell Device and Pressure Device. These devices should be set to what the internal instrument electronics are calibrated to.
- Enter the desired Park Position.

You are now ready to control the instrument with the software. If you have any problems or need assistance, please call Dynisco Polymer Test Systems Technical Support.

# 6.4 Creating a new run, step by step

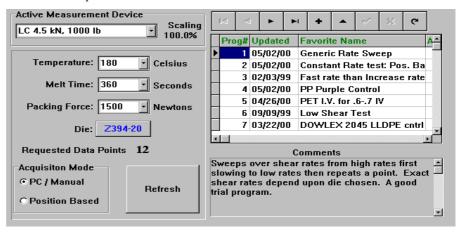
This procedure will familiarize you with creating a new experiment. Be sure that your instrument is connected to the computer and is properly configured as stated in previous sections of the manual.

If you are unfamiliar with any of the following directions or screen references, please reference their respective sections contained in this manual.

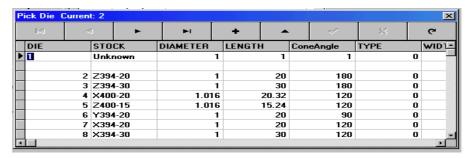
- Select the General Setup tab in the setup of screen of WinKARS
- Select Default Run in the list of programs under the **Favorite Name.** If you want to add a totally new program, click on the button labeled with a plus (+) and enter its new number.
- Select the Active Device that will be used during the experiment under Active Measurement Device.

Scaling is the compensation needed for the electronics present RCAL value. If the electronics have an RCAL for a 4.5 km. Load cell, and you select an 9 km. load cell, scaling will increase by approximately %100 or double in value. This scaling factor is taken into account when calculating results.

 Enter a Temperature, Melt Time and Packing Force. Values can be manually entered or picked from the list of values.



Click on the Die to select a die from the Die table. To add a new Die, click on
the button labeled with a plus (+) and enter its new number. Use the Tab to
advance to each field in the Die table to enter its characteristics.

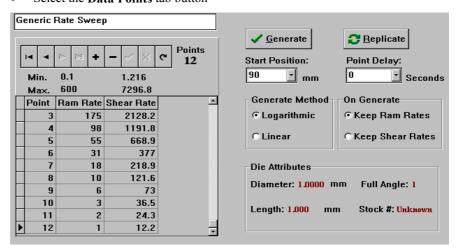


• Choose an Acquisition Mode of operation:

<u>PC/Manual</u> - In the PC 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 run. In the Manual mode, the operator presses the **END** key on the instrument when they feel the measurement is producing stable data.

<u>Position</u> - The instrument is purely positioned based. When a certain position is reached, data is acquired and the instrument proceeds to the next rate.

• Select the **Data Points** tab button



• On the left of the screen you'll see a table listing Data Points. You can modify each point individually or delete the points in between the first and last points using the minus (-) key. Modify the beginning ram rate and the ending ram rate to reflect the ranges that you want to experiment in. Modify the last point's number to increase or decrease the number of points (i.e. change to thirty for thirty points). The ending ram rate, or last point, determines how many points will be acquired (1-45).

# **EXAMPLE**

If you want to do a 30 point test. The data points view shows only 5 points covering a ram rate that you do not desire.

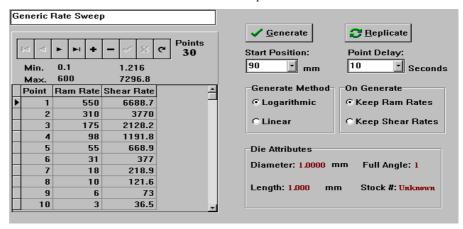
- Click on the fifth point
- ♦ Click on the add record button
- A new point is added and numbered sequentially after the last point.
- ◆ Type in 30 for 30 points (1-30)
- Modify the starting (point 1) and stopping (point 30) rates or positions
- Click on the Generate button to create the rest of the points
- Press the generate button and the software will automatically generate points for you. Do not exceed the min./max. Ram rate!
- Enter a **Start Position** and a **Point Delay**.

Point Delay is a delay before the next point is taken, or a residence time. It is used for thermal degradation tests and other residence time analysis.

 Select a Generate Method. <u>Rates</u> are generated on a logarithmic scale or using a linear scale.

Generate method determines how the data is generated for testing purposes. If logarithmic is selected, the testing procedure will physically follow a logarithmic process throughout the test. That is, the rates or stresses will increment logarithmically during the test. Linear will produce linear runs throughout the testing process.

• Select an **On Generate** method. Either keep the Ram Rate or Shear Rate.



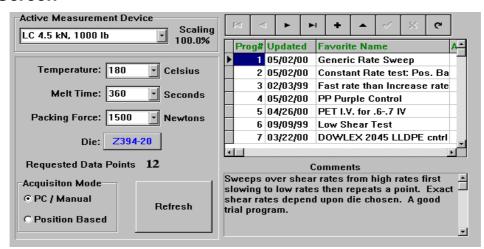
When you select a new die, Ram Rates and Shear Rates are automatically updated for use with the new die characteristics. Therefore, you want to select which current rates you want to maintain, and which rates will be modified to work with the new die dimensions.

- Remember to press the **REFRESH** button after making any changes to the test setup.
- Click the **SEND** button at the top of the page and the program is transferred to the instrument. A graphical display will show download progress.
- Allow the instrument to reach the set temperature, an audible beep will sound
  when temperature is locked. When the instrument has locked in at the desired
  temperature and your sample is loaded, Click on the CLOSE button to exit the
  Setup page and click on the RUN button located in the RUN screen.

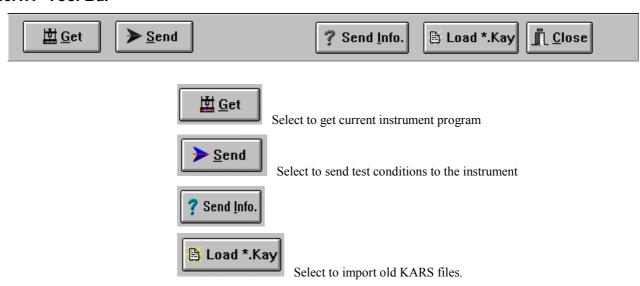
# 6.5 WinKARS Setup Screen

The SETUP Screen allows the user to create new experiments and edit pre-existing experiments. Instrument configuration is also performed here. This section will describe, in detail, the operation of each function in the Setup Screen.

# 6.5.1 The Screen



# 6.5.1.1 Tool Bar

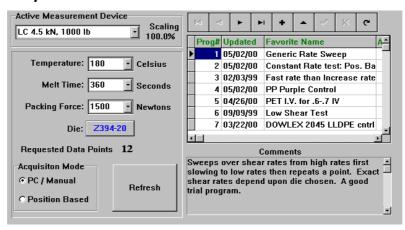


When you select Load \*.KAY a file load dialog box appears. Search your directories for the file you want to import. When data has been imported, verify correct Die, Machine Defaults and Sample Defaults.



Exit the Setup screen.

# 6.5.1.2 General Setup



# **Active Measurement Device**

This setting selects the device used for acquiring data during the experiment. It can be a load cell or pressure transducer. The measurement device can be changed with out re-calibrating. The scaling factor will change according to the scale of the device and the current electronics R-Cal value. This scaling factor is applied to the final results obtained during the experiments.

# **Temperature**

Click on this box to manually enter a new temperature or select the down arrow to browse a list of temperature to select. The instrument temperature is updated when the user proceeds to the next variable box. Any temperature above 430° C will produce the following visible warning:



**Machine damage will occur** if the instrument exceeds the maximum temperature recommended by the factory.

The instrument does allow temperatures to be entered that are above the mechanical limitations, but the factory does not recommend doing so.

# **Melt Time**

The *Melt Time* is the minimum amount of time the material will sit in the barrel before the first rate begins. If the *Melt Force* is insufficient to get the material to the *Start Position* then the *Packing Force* will be exceeded. The front panel of the instrument 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*. If the instrument is not at the *Start Position* when *Melt Time* is zero, then the front display changes to showing the Run Time or the amount of time passed (including the *Melt Time*), in seconds, since the RUN button was pressed (e.g. RT=412).

# **Packing Force**

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

# Die

Select the die used in the experiment. Click on the **Die** button to select a die from the Die table. Double click on the desired die to accept. To add a new Die, click on the button labeled with a plus (+) and enter its new number. Use the Tab to advance to each field in the Die table to enter its characteristics.

To determine a stock number, let's take a look at an X400-15 die.

- The X stands for the entrance angle. Where as,  $X = 120^{\circ}$ ,  $Y = 90^{\circ}$  and  $Z = 180^{\circ}$
- 400 stands for the diameter, in inches. 400 = .004" or 1.016 mm, D=.004
- 15 is the L/D ratio. D = .004"
- The length of the die is then  $(15)(.004)^{\circ}=.06$  or 15.24 mm

# **Requested Data Points**

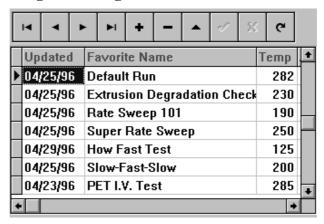
Displays the amount of data points to be taken during the experiment. The number of points is modified in the Data Points section of Setup.

# **Acquisition Mode**

<u>PC/Manual</u> - In the PC 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 run. In the Manual mode, the operator presses the **END** key on the instrument when they feel the measurement is producing stable data.

<u>Position</u> - The instrument is purely positioned based. When a certain position is reached, data is acquired and the instrument proceeds to the next rate.

# **Data Base Program Listing**



This screen contains a list, or table, of all "favorite" runs that are stored in the program database. This screen is where the user can add new programs, edit program parameters or delete programs.

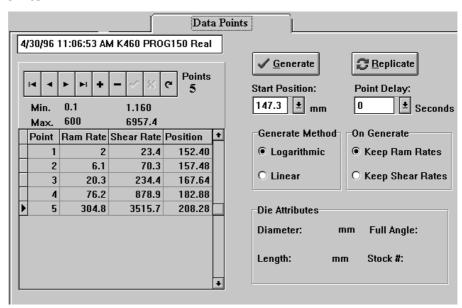
The tool bar on top enables the user to browse, add, insert and refresh data in the database. Please reference the section on Data Base Record Manipulation Keys.

To create a new run, click on the button labeled with a plus (+) and enter its new "Favorite Name". Fill in the parameters previously mentioned (i.e. Temp. Melt Time, Force, Die and so fourth)

# **Comments**

The comment block contains any text message that the user wishes to enter about the program. Click inside the text section and begin to type. Arrow up and down to scroll through the text.

# 6.5.1.3 Data Points



The Data Points Tab allows the user to directly edit or view the parameters for each data point taken in the experiment. The program you are editing is displayed in the upper left-hand corner.

Click on any data point field to edit that value. Use the Database Manipulation Keys in order to add or delete new points.

# **EXAMPLE**

Let's say you want to do a 30-point test. The data points view shows only 5 points covering a ram rate that you do not desire.

- Click on the fifth point
- Click on the add record button
- A new point is added and numbered sequentially after the last point.
- Type in 30 for 30 points (1-30)
- Modify the starting (point 1) and stopping (point 30) rates or positions
- Click on the Generate button to create the rest of the points

# Generate

When WinKARS is given a range of points, rates or positions the Generate key is used to automatically generate points in between. Please reference the previous example.

# Replicate

For quality assurance, use the replicate button to append the exact same data points to the end of your run.

#### **Start Position**

Enter where you want to the testing process to begin in the testing chamber. If you change this position, remember to select the **regenerate** button to modify the point's data.

#### **Point Delay**

Point delay is the minimum time between points. After the first rate finishes, there is a point delay and then the next point is taken. This option is helpful in thermal degradation studies.

# **Generate Method**

Generate method determines how the data is generated for testing purposes. If logarithmic is selected, the testing procedure will physically follow a logarithmic process throughout the test. That is, the rates or stresses will increment logarithmically during the test. Linear will produce linear runs throughout the testing process.

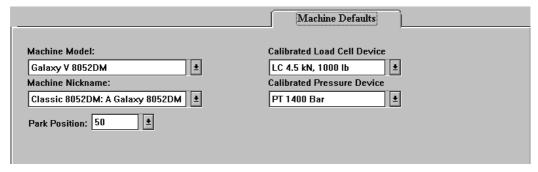
#### On Generate

When you select a new die, Ram Rates and Shear Rates are automatically updated for use with the new die characteristics. Therefore, you want to select which current rates you want to maintain, and which rates will be modified to work with the new die dimensions.

# **Die Attributes**

Die attributes are shown for your reference. To change an attribute, just click on the attribute value and select or add a new one from the database table that appears.

# 6.5.1.4 Machine Defaults



The machine defaults screen allows the user to configure the software to match their instruments factory settings.

# **Machine Model**

To select the instrument model you are using with the software. A list of factory instruments is provided. Click the down arrow to browse and select the instrument model you have.

# **Calibrated Load Cell Device**

Select the device measurement device that your instrument is using **and** is calibrated to. The instrument should be factory calibrated to the measurement device you ordered.

# **Calibrated Pressure Device**

Select the device measurement device that your instrument is using **and** is calibrated to. The instrument should be factory calibrated to the measurement device you ordered.

# **Park Position**

The park position is the position that the instrument rests in before and after runs. When the unit is power up, it will assume the park position. Select the desired park position.

#### **Important**

Prior to any experimentation, it is extremely important that the software is configured for the type of instrument it is controlling. If it is not properly configure, damage may occur to the instrument or the operator may be injured.

- Select the arrow in the **Machine Model** and select the model that you purchased.
- Select a Load Cell Device and Pressure Device. These devices should be set to
  what the internal instrument electronics are calibrated to.
- Enter your desired Park Position.

You are now ready to control the instrument with the software

# 6.6 WinKARS Run Screen

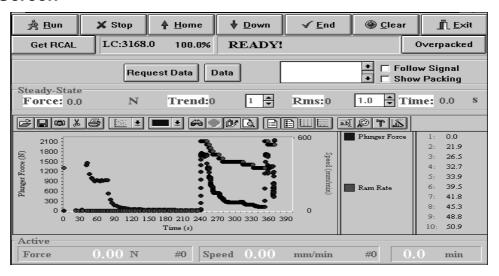
The RUN screen provides instrument control via the computer.

Many of the instruments keyboard functions can be performed from the RUN screen

The run screen is displayed while an experiment is being performed. Real-time data charting is performed on the bottom half of this screen.

\*\*\* Important - Next to the Get RCAL button on the RUN screen, verify that your measurement device is selected (e.g. PT or LC).

# 6.6.1 The Screen

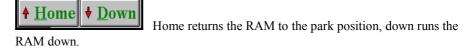


# 6.6.1.1 Instrument Controls



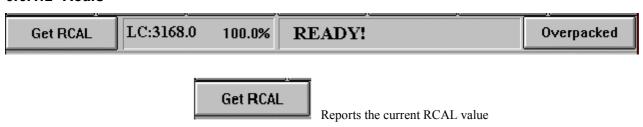
This begins the experiment process. When the experiment parameters have been entered into WinKARS, click on the RUN button to start the run.

The STOP button allows the user to stop an experiment in progress, or stop any type of motor/ram movement.



Press END to record the force in manual mode when the material has reached steady state. The steady state algorithm can also be over ridden with this button. Clear resets or clears the real time graph and data acquired.

# 6.6.1.2 Rcals



LC:3168.0 100.0%

Reports current RCAL number and scaling factor of

load cell (LC) or pressure transducer (PT)

Overpacked

If you overfill the sample chamber, you cannot close the swing arm. The over packed button raises the RAM one-inch to allow for piston clearance so the operator can close the swing arm.



# 6.6.1.2.1 Follow Signal and Show Packing Check boxes

If the *Follow Signal* checkbox is checked the last force data point sent by the instrument will be used to scale the Force axis. This may cause earlier data to disappear but is useful in determining if the forces are getting stable. If the *Follow Signal* checked box is not checked the Force axis (left side) will be set to a minimum Force value of Zero and the maximum Force sent from the machine.

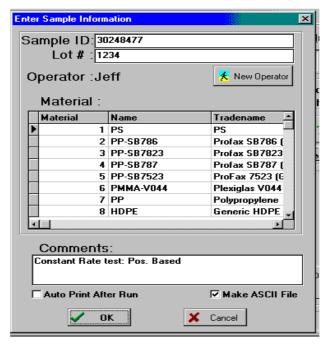
The *Show Packing* check box enables the viewing of pre-run force data. That is, force data as the material is getting packed down to the starting position for the test. It is easy to see if the melt force or packing force has been achieved or if the barrel was under packed with material. Viewing the packing stage has no impact on the final test data.

# 6.6.1.3 Starting the Run

Click on the Run button at the top of the run page.

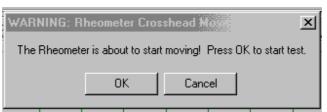


You will enter the WinKARS Sample Information page. On this page you can enter all your test information such as Sample ID, Material Name, Lot Number and any other comments you wish to include in your test.



If you need to enter a new Material to the WinKARS Material Database, You need to enter the Material Database through the Maintenance software and add a new line and material (See Maintenance \ Material.DB section below). When your sample is loaded, Temperature is locked and you're ready to start the run. Click the **OK** button.

The instrument will beep and WinKARS will warn you that a test is about to start. Click **OK** to proceed.



# 6.6.1.4 Steady State Data

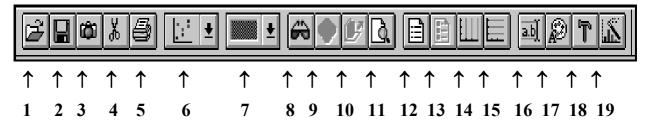
The steady state data field displays information about the steady state force, RMS values, and elapsed time of the test and trending functions.



The force is displayed so when you are in the manual mode, you can verify steady state has been reached and press the END key to proceed to next rate.

# 6.6.1.5 Plot Functions

WinKARS provides powerful plotting and OLE functions.



- 1- File Open: This icon lets you load previously saved chart (.chf) files
- 2- File Save: This icon lets you save the current chart as a chart file
- **3-** Copy Graph: This icon copies the graph displayed to the Windows clipboard. From any OLE compliant software package, click EDIT then PASTE to insert the graph
- **4-** Copy Data: This icon copies the raw data to the Windows clipboard. From any OLE compliant software package, click EDIT then PASTE to insert the data points.
- 5- Print Click printer icon to print the graph
- **6-** Chart Click arrow icon to change the plot characteristics
- **8-** 3D Click 3D icon to change graph to three dimensional
- 9- Rotate Click rotate icon to rotate data points in 3D view
- **10-** Z Clustered Click Z Cluster icon to separate the data points on a 3D graph
- 11- Zoom Click the zoom icon, position the cursor above the data to zoom into. Drag the cursor over the data to zoom into. Click the zoom icon to restore the normal view
- **12-** Show/Hide Legend Click icon to Show or Hide legend
- 13- Show/Hide Series Click icon to show or hide the series legend
- 14-15 Show/Hide Grids Click icon to show or hide grid lines, vertical and horizontal
- 16- Titles Click icon to edit titles
- **17-** Fonts Click icon to change fonts
- **18-** Chart Options Click icon to change chart options
- **19-** Properties Click icon to modify chart properties

# 6.6.1.6 Real Time Data

This screen provides the user with real time data results. Force, speed and time are displayed as they happen.



# 6.6.2 What's Happening During a RUN?

What happens during a run depends upon the acquisition mode. All tests start out the same way, either the RUN button is pressed on the machine or the RUN button is pressed from the Real-time Control window. On the PC, an internal test timer starts as soon has RUN is pressed. The instrument starts moving down looking to engage the plunger. When it sees about 10 N of force, it assumes it has engaged the plunger and starts applying the packing force. If the *Show Packing* check box is on, the force data associated with this packing stage starts scrolling across the plot at the bottom of the Real-time Control Window. During the packing stage, the speed varies to keep a constant melt force on the material and is not plotted (forced to zero). The top status area shows the test is **RUNNING.** In position mode testing, (can also be standalone operation for the instrument) this continues through the entire test. Only force information is sent and steady-state analysis is not performed.

In PC based tests, the force and speed data are transferred to the PC, when the PC decides steadystate has been achieved it commands the instrument to go to the next speed. The operator can also press the END button on the instrument or on the software screen to change to the next force before steadystate is achieved. The TREND and RMS values can be adjusted to meet specific steady-state needs, though the defaults should work with most materials. Setting the trend to zero will cause the PC never to change speeds, allowing the operator to change speeds using the END key only.

The instrument will automatically collect and display data.

# 6.6.3 Pre RUN

Cleanliness of the barrel and components is a critical factor in the behavior of materials. The barrel of the unit should be cleaned as well as possible. All components associated with the instrument (Piston tip and Orifice) should be cleaned of all debris and material build-up.

The precision and accuracy of the test and instrument has been standardized by ASTM D3835. DYNISCO POLYMER TEST has found that the contributing factors to the accuracy of tests includes sample weights (the amount of material tested), timing (how long the material is in the heated barrel), packing technique, dryness (has the material been properly dried?) and cleaning technique. It is recommended to follow ASTM Specification D3835 for your material or control sample. Also, pay attention to the recommendations of the manufacturer or suppliers.

# 6.6.3.1 RCAL

Please be sure that the calibration is up to data. Make sure your measurement devices, pressure transducer or load cell, are functioning properly and have been calibrated. Please reference RCAL sections in this manual for further instructions.

# 6.6.3.2 Cleaning

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

clean. When finished, put the plunger back into the barrel. This allows the plunger to heat up before the next test.

With materials that are thermally stable (less than 5% viscosity change over 1/2 hour) we recommend only cleaning the barrel between runs of the same material. For materials that degrade we recommend cleaning both the barrel and the die completely. You can clean the die by first running a drill bit up into the die while it is hot and in the instrument. This will make it much easier to get the cleaning drill bit in when the die is removed and the material starts to solidify. Remove the die by extending the arm on the die nut removal wrench to its full length. Attach the socket completely over the die nut. A quick hit on the end of the socket arm is all it takes to remove 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, the die is very HOT when it comes out. If you don't use gloves you will get burned eventually. Using a cotton rag, wipe the top of the die clean. If your die has a tapered entrance, be sure to clean any material out of the conical top section. Run the supplied drill bit into the die. Remove material that collects in the orifice and repeat until the drill passes easily through the die. On long dies it may be necessary to clean from both ends to completely clean the entire length of the die. While the die is out look down the barrel bore to be sure it is clean. If it is not run a couple of patches up and down it before putting the die back into position. When the die is out of the barrel it cools down quickly. The longer it is out the longer the wait for the temperature to stabilize. Minimizing the time the die stays out of the machine will increase the number of tests you can run. When the temperature on the front display is within two degrees of the set point you can begin your next test. Loading material will cause a small temperature change even if the temperature set point was achieved. The melt time (360 seconds) will allow ample time to get the temperature to set point before the first data point is collected. Always leave the machine clean. If it is going to sit at room temperature for an extended period of time you may wish to coat the barrel with a light machine oil to prevent rusting. The oil will need to be purged from the machine before accurate data can be obtained.

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

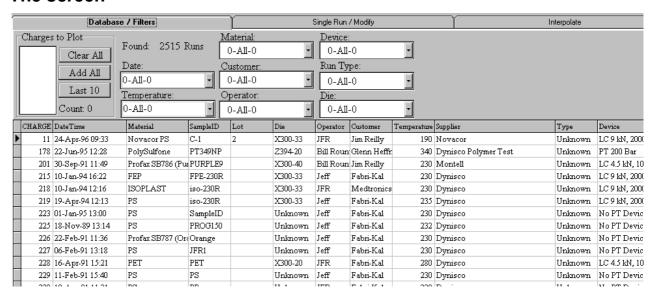
# 6.6.4 When RUN is done

After a run has been completed, the instrument will purge any left over sample material and retract the ram to the park position. At this point, WinKARS will automatically go to the Analyze screen. Analyze will allow you to enter comments, check the acquired data and verify its validity.

# 6.7 WinKARS Analyze Screen

The ANALYZE screen is where the user can view and modify the data they created during the experiment. It will allow you to enter comments, check the acquired data and verify its validity after a completed experiment. It also provides tools for viewing and sorting previous experiment data. Plotting and merging functions are also performed here.

# 6.7.1 The screen



# 6.7.2 Function Bar



### Print

Select Print to print the current data report. Reports will be printed to the default printer selected in Windows Print Manager.

# Plot

Plot provides an on screen plot of the current data. From the **Raw Data** tab (screen), plot will chart all the data runs displayed. You can double click a charge in the Raw Data screen to plot just that charge. From the Modify/Preview screen, plot only charts the current experiment displayed. To print the plot, click the printer icon in the upper left-hand corner of the screen. Please reference further sections about plotting.

# Append

Use append to merge plot files. If you select Append from the Raw Data tab (screen), all of the charges will be appended. Please reference further sections on appending.

# Refresh

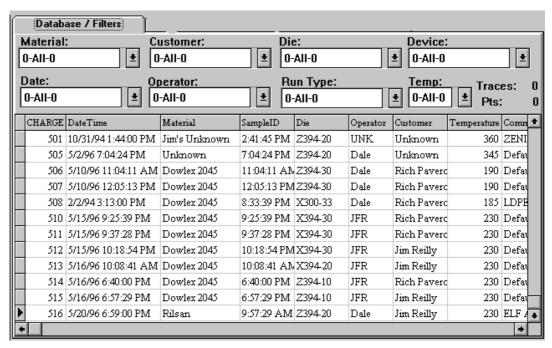
When a data filter is used, refresh will filter all the data and redisplay the sorted charges.

### Copy

Copy allows the user to copy data and data points.

# **Old Files**

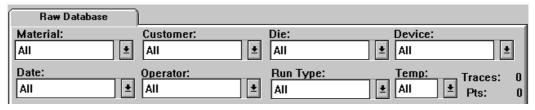
# 6.7.3 Database / Filters



The Database / Filters screen allows the user to view all of the previously saved experiments that were performed. The user can filter charges by material, customer and so forth. The user can also plot and merge charge data.

You cannot modify any data here. This screen is intended for browsing only. To modify data points or any characteristic about the charge, go to the Modify/Preview screen in Analyze. Make sure you select or highlight the run you want to analyze before doing so.

# 6.7.3.1 Data Filtering



WinKARS allows you to filter the charges database to display only the files pertaining to certain characteristics. These characteristics include material, customer, die, device, date, operator, run type and temperature.

- Select the down arrow of the characteristic you want to filter by
- You will see a list of available titles to filter by
- Scroll up or down to select the characteristic
- Click on the characteristic to update the field
- Select refresh to display only experiments that include the selected data filter

You can use multiple characteristics for data filtering.

# 6.7.3.2 A Word about Plotting and Merging

Plotting and plot merging can be performed from the Database / Filters screen.

# 6.7.3.2.1 Plotting

- Browse the list of experiments to find the data you want to plot
- Double click on the charge you wish to plot. This will plot it on screen for you
- Select the printer icon to print the plot to your printer

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 experiments, and then select the plot button to merge them all into one plot.

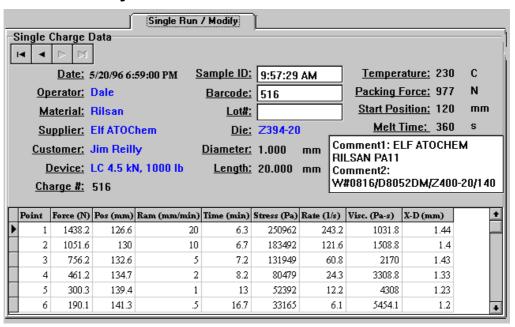
# 6.7.3.2.2 Merging

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 button to merge them all together.

Another merging technique is as follows:

- Double click on a single run file that you want to merge into. This will bring up a plot window of the data.
- Switch back to Analyze/Database / Filters screen
- 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

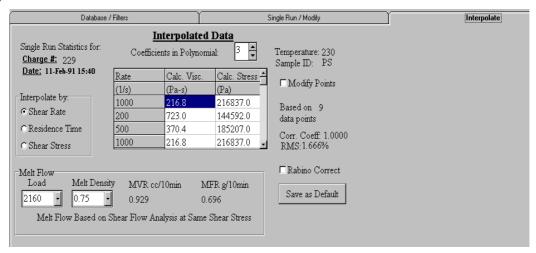
# 6.7.4 Single Run / Modify



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 buttons just above the date display.

# 6.7.5 Interpolate Table



Interpolate table is the run statistics information for that particular run. You can collect Melt flow, Melt density, MVR, Shear rate and RMS information as well as statistical averages on your run.

This data can be copied and pasted directly into other Windows applications.

- From the Plot Data screen click the copy button on the function bar. This copies the data directly into the clipboard
- Enter your Windows application
- Select Edit-Paste and the data will be transferred from the clipboard directly into your application.

# 6.7.6 SQL

Standard SQL language scripts written for the data filter on the Raw Database screen. These scripts are dynamically generated when the user performs data filtering. These scripts affect the plot outputs produce by WinKARS. This module allows the user to write his own SQL query of the database for use in custom plotting.

The following script is stating: For the plot output, select from the CAPDATA.DB table the data field "Sample ID" and display in plot. From the RDATA.DB table or file, select the data field "Time" and rename it as "Rtime" and display it in the plot, and so on and so on.

```
SELECT
CAPDATA."SampleID",
RDDATA."Time" as RDDATA."RTime",
RDDATA."VISCOSITY" as RDDATA."SViscosity",
RDDATA."STRESS" as RDDATA."SStress",
RDDATA."Fate" as RDDATA."SRate",
RDDATA."Force",
RDDATA."RamRate",
RDDATA."Position",
CAPDATA."SetPointTemp" as CAPDATA."Temperature",
CAPDATA."Charge"
FROM CAPDATA, RDDATA
WHERE CAPDATA."Charge" = RDDATA."Charge" and
CAPDATA."Charge" =:SelectedCharge and
RDDATA."Viscosity">0 and
```

RDDATA."Force">0

# 6.7.6.1.1 Custom SQL

If the user is familiar with Standard Query Language, they can add their own SQL reference in the plot query screen to produce their own custom plots.

- Check the Use as Default box to allow the new query to work on current data.
- Enter the SQL reference in the editor box
- Plot a run on screen to verify the changes affected the plot
- Uncheck Use as Default to use the WinKARS generated SQL
- Save your SQL by selecting the Save SQL button and save accordingly

# **Example**

A user may want to see viscosity squared on previously acquired data. They should check the **Use as Default** box and modify the following line:

RDDATA."VISCOSITY" as RDDATA."SViscosity",

To read:

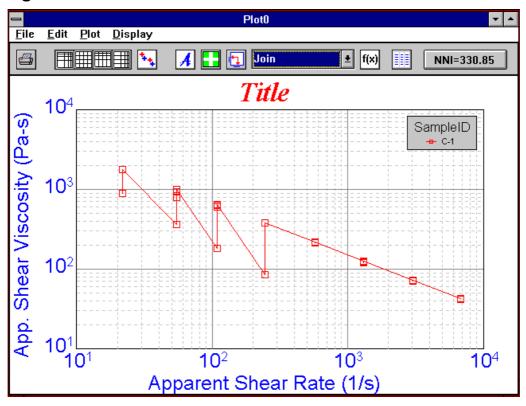
RDDATA."VISCOSITY\*2" as RDDATA."SViscosity",

Or, if the user only wants to plot data with forces over 100, enter the following SQL script:

RDDATA."Force">100

Other examples may include adding and O-ring, such as Operator "Jdoe" and "Bsmith" or material "PET" and "HDPE"

# 6.7.7 Plotting



Plotting is simply accomplished by clicking the plot button on the function bar. If the Plot button is clicked from Raw Database screen, the entire set of displayed experiments is merged and plotted. If the Plot button is clicked from Modify/Preview screen, only the current experiment is plotted.

From the plot screen, select either the printer icon or File-Print to output to your printing device.

# From Database/Filters Screen

- Browse the list of experiments to find the data you want to plot
- **Double click** on the charge you wish to plot. This will plot it on screen for you
- Select the printer icon from the plot screen 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.

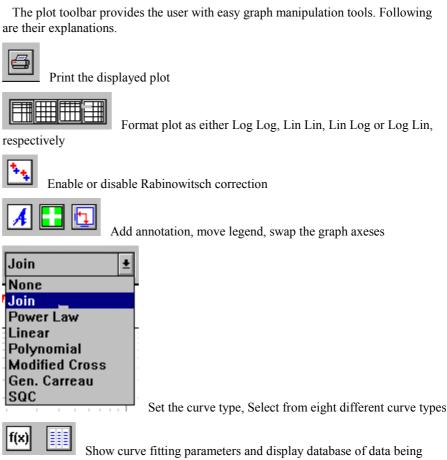
# From Single Run/Modify Screen

- 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

# 6.7.7.1 Modifying Plots

#### 6.7.7.1.1 Plot Toolbar











# 6.7.7.1.2 Modifying Plots

- 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.

# 6.7.7.2 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 button to merge them all together.

Another merging technique is as follows:

- Double click on a single run file that you want to merge into. This will bring up a plot window of the data.
- Switch back to Analyze/Raw Database screen
- Filter the data so that there is only one data run showing in the database list

# 6.7.7.3 Copying and Saving Plots

# 6.7.7.3.1 Copying

Plots can be copies 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.

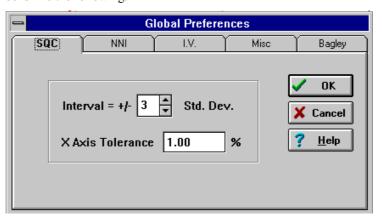
Plots can also be cloned. From the plot windows, select File-Clone. This will open up a new plot display window and create a copy of the original plot in it.

# 6.7.7.3.2 Saving Plots

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

# 6.7.7.4 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:



# 6.7.7.4.1 SQC

SQC allows the user to reject data not within predetermined limits. An interval of standard deviation acts 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.

#### 6.7.7.4.2 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

#### 6.7.7.4.3 I.V.

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

#### 6.7.7.4.4 Misc.

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

# 6.7.7.4.5 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.

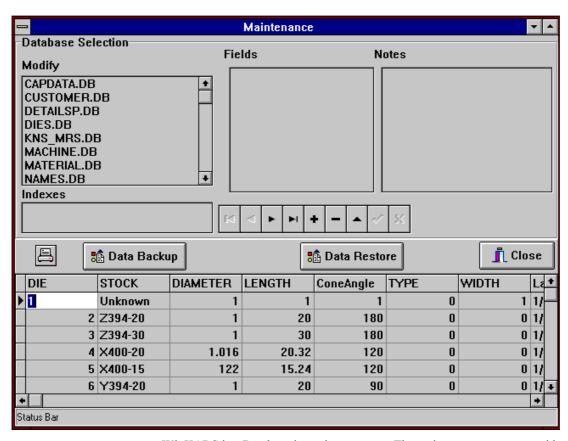
# 6.8 WinKARS Maintenance Screen

# 6.8.1 A Note of Caution

The screens contained in the Maintenance Menu provide direct manipulations of data and software/hardware parameters. The Maintenance Screen is provided for advanced users and Dynisco Polymer Test Systems Technical Support. Before manipulating any data or parameters in the following screen, please make sure that you have a backup of your C:\WINKARS\DATABASE directories.

# 6.8.2 The Screen

Access the WinKARS Maintenance screen from the main menu by selecting Maintenance. The following screen will appear.



WinKARS is a Database intensive program. The maintenance screen provides direct manipulation of data in the database files; it should be used with caution. Each database selection, on the left side of the screen, represents a database file in the C:\WINKARS\DATABASE directory.

Select a Database file to modify by clicking on it in the MODIFY section. Edit data in the field table below in the data edit section.

# **Modify**

These are the interactive database files. Click on a file to select it, then proceed to edit the data below. Some fields in these files automatically interact with WinKARS and other database files. We cannot begin to give a detailed explanation of these interactions, but we can say that as you become more familiar with the software, you will notice them. You may see references to some of their fields in other screen likes ANALYSIS.

# **Fields**

These are the data holders in the database files. They have labels like machine or customer. When we manipulate the files, store new programs, enter new sample identifications or perform new runs, these fields are updated with the new data.

Some fields can be modified and some cannot be modified.

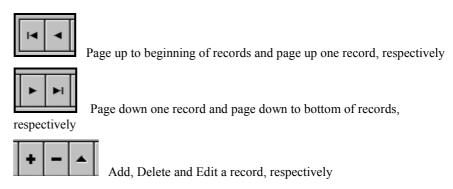
# **Notes**

Here is where the operator can modify, add or delete notes for a table in a database file. For instance, under the MATERIAL.DB file, one can add a note to material #3 stating detailed information about that material.

### **Record Manipulation Keys**



These keys provide editing functions and data browsing.



#### **Editor**

The editor is where the operator can change data and parameter settings stored by WinKARS. Caution should be exercised here.

### 6.8.3 Database Files

# All Values are in METRIC even if you have a STANDARD machine

There are approximately ten database (.dB) files in the \winKARS\database directory. The amount of files and names may vary depending when you purchased the software and how custom your installation is. Brief descriptions are included for some of the unclear fields in the databases. Following is a general description of each file and its tables.

### **CAPDATA.DB**

The CAPDATA.DB file is a master database file that incorporates most of the information from the other .dB files and from previous runs. Some fields are uneditable, in that the data cannot be changed and some are editable. Performing maintenance on the file CAPDATA.DB allows the operator to view and change the many parameters involved with the experiment.

Data Registration Field - Stores information

Rate Device - Device that controls the rate of the instrument. Usually the motor encoder value

**CTRL Device** - Numeric value of the device which is being controlled by the instrument for the particular experiment: Rate = Motor, Stress = Pressure Transducer.

**OPTION Device - TBD** 

Signature - Numerical value of older KARS version used in importing old data.

Units - Numerical value of units of data imported into WinKARS.

Version - Numerical value of firmware in instrument.

### **CUSTOMER.DB**

The CUSTOMER.DB file stores all the information about customers or vendors. Each customer is assigned a customer number. This database is provided for reference to the operator. Supplier, customer or supervisor information can be stored her for their reference.

### **DETAILSP.DB**

DETAILSP.DB contains information about a program. It can be used to set or browse data points in a program. There is no raw run data stored here. Each program is assigned a number in the PROGRAM field, and the data for each particular is displayed in the POINT field.

**Hold Time** - Test delay or pause between points of an experiment.

#### **DIES.DB**

The DIES.DB file holds all the information about the dies that you have. Each die is assigned a number for later use in program setups. When creating programs and its applicable, simple enter the die number from this table for instant programming.

### KNS\_MRS.DB (caution)

The KNS\_MRS file stores all the information about your instrument options and setting. It also contains information about every model instrument that the factory provides. Each model is given a machine number. This number is used in various parts of WinKARS.

An instrument **cannot** be simply changed to a new model in this file. Options cannot be added through this file. Models and options are determined by the instruments physical characteristics and embedded firmware. Changing this file may result in operator injury and instrumentation damage.

This file will be used covertly by the software and instrument. Factory technical support may also suggest changes to this file.

### **MACHINE.DB**

MACHINE.DB holds information about various instruments located at the testing facility. It is mainly used when multiple machines are in use. When creating programs and its applicable, simple select the machine number from this table for instant programming.

KNS - Machine number from KNS\_MRS.DB file.

### **MATERIAL.DB**

Each material used in testing is given a unique material number. Details about the material are also stored. When creating programs and its applicable, simple select the material number from this table for instant programming.

#### NAMES.DB

NAMES.DB holds relevant information about labels on plots that are produced. Symbols, units, formats and languages can be changed here. These changes will be reflected on plots.

### **OPERATOR.DB**

Information about the various operators at your facilities is entered here. When creating programs and its applicable, simple select the operator number from this table for instant programming.

### PLOT.DB

When the ANALYSIS screen request data for a plot, PLOT.DB files are created to hold this data. Plotting routines then access this file.

### PROGRAM.DB

The PROGRAM.DB file holds information regarding programs, the program number and notes defining the program. The associated die is also placed here. When creating programs and its applicable, simple select the program number from this table for instant programming.

**Protected** - Whether field is protected (TRUE = locked) or unprotected (FALSE).

### RDDATA.DB

RDDATA holds the raw information or data from a program that was run. Data can be manipulated here to produce various results in plots and reports.

### **SUPPLIER.DB**

The supplier database provides information about the supplier of material. When creating programs and its applicable, simple select the program number from this table for instant programming.

### TDUCER.DB

TDUCER.DB is the file that holds information about the various pressure measurement devices in use. Past and present RCAL values are stored here. This information provides easier calibrations and device swaps. When creating programs and its applicable, simple select the program number from this table for instant programming.

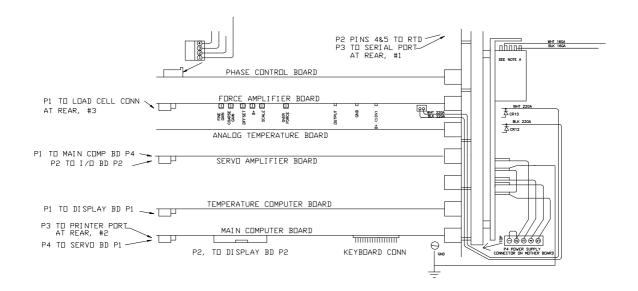
# 7. Maintenance

## 7.1 NIST Traceability

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

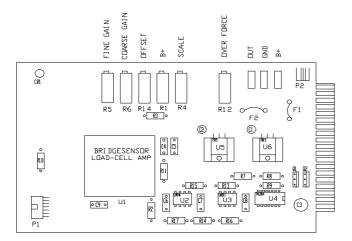
### 7.2 Circuit Card Location

Following is an internal layout of the instrument so that you can locate the various circuit cards reference in the following sections.



### 7.3 Force Calibration

The instrument, which can be easily calibrated with dead weights, must be calibrated with a calibration unit to assure proper adherence to ASTM methods. All adjustments are made to the potentiometers located on the Force Amplifier Board shown below.



### 7.3.1 A word about "AutoScale"

The use of potentiometers for force calibrations has been practically eliminated. You still must perform one initial force calibration. After that, you select the new device you are using in the software. The software then performs the necessary scaling to that measurement device eliminated recalibration to different measurement devices. This option works with Load Cells as well as Pressure Transducers.

Scaling factors are displayed in WinKARS software. Please reference those sections of the manual for more about scaling.

### 7.3.2 Calibrating the Calibration Unit

Obtain a weight or set of weights which are at least 10% (100 lbs. for 1000 lb. load cell) of the calibration load cells total capacity. Know the force exerted by these weights to  $\pm$  0.5%.

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

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

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

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

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

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

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

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

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

Read the Model: AD-4321A/B instruction manual that came with the calibration display unit for more details. Note that the 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 brought over about 2640 N (600 lb.) or the display will go blank.

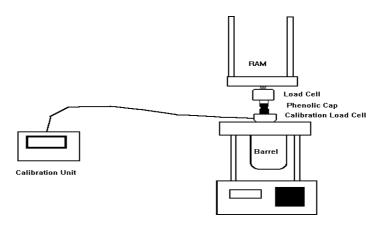
### 7.3.3 Calibrating the instrument load cell with the calibration unit

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

- Make the load cell the active device by pressing + on the instrument keypad
- Zero the instrument force reading.
- Put attachment bobs on calibration unit.
- Zero calibration unit.
- Move the crosshead down until it is about 6" from the swing arm.
- Insert the calibration cell up into load cap and press DN.
- Press **RESET** when calibration cell is 1/4" from hitting swing arm.
- Press the **EMERGENCY STOP** button.
- Turn the top large pulley counterclockwise until 1800 N is read on the calibration unit.
- Adjust the fine gain until both units match.
- Turn pulley clockwise and remove CAL cell.
- Press RESET then pull EMERGENCY STOP button out.
- Press UP

### Details:

• Zero the instrument force reading:



### 7.3.3.1 Machines equipped with barrel pressure transducers

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

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

### 7.3.3.2 Machines equipped with load cell only

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

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

#### Put attachment bobs on calibration unit.

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

### • Zero the calibration unit.

With the calibration cell in a vertical position (the same position it will be in when placed in the load cap where the plunger normally goes) on the table, bolt side up,

and no external loads applied press **ZERO** on the calibration display units front panel. Remember the calibration unit is the reference; use its reading to judge where you are in terms of load on the system. If you instrument force calibration is grossly in error you could overload your equipment if you trust the instrument front panel force display before it is calibrated.

• Insert the calibration load cell in series with the instrument load cell

Press DN on the instruments front panel and allow the crosshead to come down. Run until there is about 150 mm clearance between the crosshead and the swing arm. Press RESET to stop the crosshead motion. You need enough room to allow you to slip the calibration load cell into the upper load cap and hold it there. If the crosshead has come down too far press **UP** and the crosshead will move quickly upward. Press **RESET** when it has moved up enough to stop the ram and insert the calibration load cell.

# **DO NOT LET THE MACHINE DRIVE THE TWO LOAD CELLS TOGETHER!** Press **RESET** at anytime to stop the machines movement.

- Insert the calibration load cell up into load cap
- Press in the EMERGENCY STOP button.
- Turn the top large pulley counterclockwise to engage the two load cells. Proceed until 1800N of force is read on the calibration unit.

Position the calibration cell so that it is sitting flat and centered over the top of the barrel hole. Watch the calibration display unit and keep turning until you see about 1800N on the calibration display unit. **DO NOT EXCEED 2400 N** 

 Adjust the *fine force* potentiometer (screw on far left, found on the Force Amplifier board) until the calibration unit and the instrument force match.

The instrument display should read 1.0-1.2N higher than the calibration display unit to account for the plunger weight. (E.g. If the calibration display unit reads 1832.6 the instrument front display should read 1833.8)

• Turn the pulley clockwise by hand until you can remove the calibration cell.

Hold the calibration cell so it does not fall off the instrument. **Important step** Press **RESET** then and only then. Pull the EMERGENCY STOP button back out. Press **UP** on the instrument to return it to park position and you are ready for your next test.

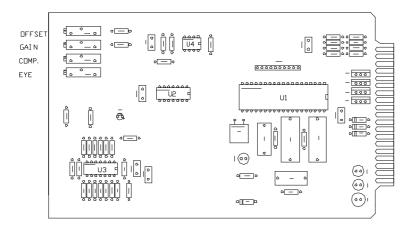
### 7.3.4 Calibrating without the calibration unit

Dynisco Polymer Test Systems has developed a platform to calibrate the load cells mechanically while they are attached to the instrument. We suggest placing the load on the floor and being sure the platform is on a level surface before attempting the calibration. Adjust the zero offset (see above for location) with no weights on the platform then adjust the fine gain to achieve the calibration weight. We suggest using a minimum of 10% of the load cells full-scale capacity to achieve a calibration.

## 7.4 Temperature Calibration

CAUTION: Do not adjust the COMP Potentiometer located three pots down on the Analog Temperature Board. The unit will have to be returned to the factory for calibration.

The instrument must be calibrated with a calibration unit to assure proper adherence to ASTM methods, the instrument must be able to accurately control and display temperature. All adjustments are made to the potentiometers located on the Analog Temperature Board shown below.



### 7.4.1 One Temperature Reference

To perform a temperature calibration go into EDIT and set the temperature of the machine to the temperature of the calibration thermometer. Allow the machine to heat up to within a few degrees of the thermometer's nominal value **then** remove the set screw which covers the thermometer well and carefully insert the thermometer into the thermometer well.

### Perform the calibration with the die and Piston Rod assembly in the barrel.

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

Remove the front top cover of the instrument and locate the slot in the clear plastic sheet on the LEFT side of the machine (not the slot on the top that is for force calibration). There are four adjustment screws from the top down they are labeled OFFSET, GAIN, COMP and EYE. See above picture of the Analog Temperature Board.

To perform the calibration we must first disconnect the temperature control system so it does not change the temperature of the machine as we are trying to calibrate it! To "lock" the temperature in place simply press down both the RED and BLACK buttons together on each side of the instruments front LED display. Hold the buttons down until you see the current temperature reading change to some new number. After letting go of the buttons you should see a dot flashing on the left side of the LED display (a dot on the right side may also be flashing). If you do not see any flashing dot press down both the RED and BLACK and release only after you see the number on the front panel change. Only when the flashing dot appears should you turn the OFFSET screw (TOP SCREW) on the left side of the instrument until the FRONT display matches the true temperature as determined from your thermometer and its calibration certificate. Once set correctly press either the BLACK or RED button down to start the system controlling again. The flashing dot should disappear once the RED or BLACK button is pressed.

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

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

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

### 7.4.2 Two Temperature References

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

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

### **Using an ICE BATH**

If using an ice bath, go into **EDIT** and set the **TEMP.** to 0.0 only then can the PRT be removed from the instrument. Unscrew the two fasteners that hold the top barrel insulator in position and remove the PRT. Place the PRT temperature probe from the instrument into a distilled ice / distilled water mixture which has been well stirred. Allow the system to equilibrate for 10 minutes then without performing the temperature "lock" (as is needed when the instrument is controlling the temperature) turn the OFFSET screw (THE TOP SCREW) until the front display reads 0.0. Wipe the PRT dry and insert it back into the machine. Set the machine temperature to match the calibration thermometer's nominal value (e.g. 230 C).

The second part of the calibration is exactly the same as the single point calibrations described above **except** now the second temperature is adjusted by

turning the **GAIN** screw (SECOND SCREW FROM TOP) NOT the offset screw. Perform the following actions and refer back to the single point calibration where needed but don't adjust the offset screw.

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

### 7.4.3 Using two reference thermometers

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

**DO NOT ADJUST THE COMP SCREW (Third pot down from the top)!** This corrects for the non-linearity of the voltage vs. temperature curves for the PRT KAYENESS POLYMER TEST SYSTEMS sets this at the factory. If you do change this setting, the instrument will need to be readjusted at the factory.

### 7.5 Pressure Transducer Calibration

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.

Therefore, when calibrating, set a minimum offset of 10 lbs. (40N) at 190C. This should compensate for the full range of temperatures that the instrument can sustain.

### 7.5.1 RCAL

The pressure transducer system provides an automatic check of the electronics system. A precision resistor is placed into the circuit and acts like a known pressure on the system. From the systems response to this simulated measurement, we can assess the current calibration of the machine. The value of the 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 instruments front key pad and then pressing **NO** until the words RCAL TEST appear in the display. Pressing **YES** will cause the current RCAL value to be displayed for the active device.

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

When calibrating, set a minimum offset of 10 lbs. (40N) at **190C**. This should compensate for the full range of temperatures that the instrument can sustain.

Calibrate the Pressure Transducer by pressing **DELETE** on instrument key pad <u>ONLY</u> when:

- Pressure reservoir cleaned
- Pressure transducer in barrel is snug (100 in-LB torque)
- Cross head is at the Park position (Up and away from plunger)
- Machine is "locked in" at test temperature (preferable 190 C)
- Piston Rod is in the barrel
- PT is active sensing device (Press on front panel so "PT" is displayed)

## 7.6 R-CAL Values

These values are supplied for DOS software and are typically not used in the Windows based software WinKARS.

Device	Nominal	Nominal	Plunger	RCAL	RCAL	Max Termina	Max Terminal	Min "Good"	Min "Good"
	Range	Range	Range	English	Metric	Force	Force	Reading	Reading
	(psi)	(Bar)	(lb.)	(lb.)	(N)	(lb.)	(N)	(lb.)	(N)
PT	30000	2069.0	3331.1	2798.1	12446.0	3331.1	14816.7	333.1	1481.7
PT	20000	1379.3	2220.7	1865.4	8297.4	2220.7	9877.8	222.1	987.8
PT	10000	689.7	1110.4	932.7	4148.7	1110.4	4938.9	111.0	493.9
PT	29000	2000.0	3220.1	2704.8	12031.2	3220.1	14322.8	322.0	1432.3
PT	20300	1400.0	2254.0	1893.4	8421.8	2254.0	10026.0	225.4	1002.6
PT	14500	1000.0	1610.0	1352.4	6015.6	1610.0	7161.4	161.0	716.1
PT	10150	700.0	1127.0	946.7	4210.9	1127.0	5013.0	112.7	501.3
PT	2900	200.0	322.0	270.5	1203.1	322.0	1432.3	32.2	143.2
PT	725	50.0	80.5	67.6	300.8	80.5	358.1	8.1	35.8
LC*	45030	3105.5	5000.0	3500.0	15568.0	3000.0	13344.0	50.0	220.0
LC*	18012	1242.2	2000.0	1400.0	6227.2	2000.0	8896.0	20.0	89.0
LC*	9006	621.1	1000.0	700.0	3113.6	1000.0	4448.0	10.0	44.5
LC*	4503	310.6	500.0	350.0	1556.8	500.0	2224.0	5.0	22.2
LC*	2702	186.3	300.0	210.0	934.1	300.0	1334.4	3.0	13.3

<sup>\*</sup> Note RCAL values for Load cells are nominal, record RCAL after using calibration unit is best.

Conversion Factor Lb. to N  $\rightarrow$  4.448; Factor Psi to Lb. plunger  $\rightarrow$  0.111036 Factor Bar to Psi  $\rightarrow$  14.5; lb. to RCAL for PT's 0.84; nominal lb. to RCAL for LC  $\rightarrow$  0.7

### 7.7 Dimensional Verifications

### 7.7.1 Die and/or Orifice

Perform a quick visual inspection of the die: The die bore and its finish is critical, there should be no visible tool marks. Top and bottom surfaces should be flat and smooth.

The die should be extremely clean! Clean a **cold** die with drills bits. Solvents are not recommended because of safety hazards. For Indexer dies use the small cleaning brush supplied. Do not clean the die with the Go No-Go pins!

#### Die Diameter

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

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

Dynisco Polymer Test Systems Lab policy is as follows: If the green side of the gage goes through both ends of the die then attempt to put the red side pin into the die, be sure to check both ends. If the RED pin enters more than 1 mm into the die on either end the die is rejected. Verify you have the correct nominal dimension and reject the die (DO NOT USE THIS DIE).

ASTM Policy is the same except for the 1mm minimum rule.

The die may also be good enough to meet the ISO specifications. This check can be made by selecting the ISO equivalent Go No-Go pin (for a nominal die dimension of 0.030" the ISO 0.03" gage would be used). Check to be sure the green side goes through both ends as before and the red side goes in less than 1 mm on each end. If it passes the die is a more valuable commodity and should be marked as meeting ISO specs. If it has not passed the ISO pins but passed the ASTM pins it should be marked as meeting ASTM specs. This is Dynisco Polymer Test System's incoming QC procedure for all new dies.

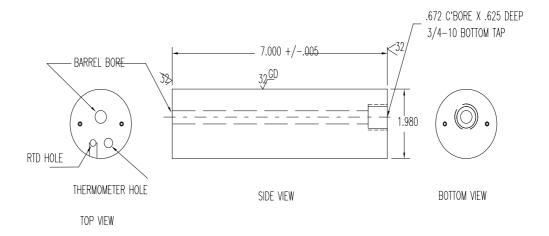
#### **Die Length**

If the die has a flat entrance angle a hand held micrometer can be used to measure the die length. For dies with an entrance angle other than flat a small precision ball can be mounted to a dial indicator which is only slightly larger in diameter than the die capillary diameter. Small errors in the cone angle will affect the die length measurement very little if the ball is close to the capillary size. Be sure the entrance cone and faces of the die are extremely clean before measuring.

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

### 7.7.2 Barrel Diameter

The support vendor list shows the address for a bore gage manufacturer. Once the barrel is extremely clean, all dimensional measurements are to be made at room temperature, it can be checked using a bore gage. Be sure to get or make a long enough extension so the entire barrel can be checked. The barrels are  $9.550\pm0.005$  mm leaving the factory.



### 7.7.3 Plunger Tip Diameter

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

### 7.8 Maintenance Schedule

Dynisco Polymer Test Systems offers a flexible on site service agreement, negotiable with your salesperson.

The Dynisco Polymer Test Systems capillary instrument service contract provides users with assurance of quality test results and adherence to the current testing standards. Capillary instruments are serviced to meet or exceed the latest ASTM D3835 specification values for the following:

- Temperature: set point, control and gradient in testing chamber
- Force Cell
- · Orifice diameter and length
- · Barrel diameter
- Piston tip diameter

The lineage of each calibrated item can be traced to NIST.

Instrument firmware and software will be upgraded to the latest version for items that were initially purchased with the instrument.

### 7.8.1 Lubrication

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

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

### 7.8.2 Daily

The Dynisco Polymer Test Systems Instrument was designed to meet low maintenance criteria. Therefore, there is no major maintenance schedule except for keeping the instrument clean.

Remove the die nut and die and clean thoroughly with brush and drill bit. Clean the threads of the orifice holder nut thoroughly. Clean the barrel with a cotton patch when hot. Piston should spin easily when placed into a clean barrel.

### 7.8.3 Weekly

Clean the barrel with a brass brush. Use oven cleaner to clean the piston rod assembly and orifice.

### 7.8.4 Monthly or Long Term

Depending on your company policy, a calibration or calibration check may be needed on a monthly, quarterly or yearly basis. It is a good idea to create a repetitive maintenance schedule for your instrument. We cannot do this for you, however we can suggest what to do:

#### Clean, Clean, Clean

Clean the dust and dirt out of the electronics module with clean air every six months to one year, more often in dirty environments. Remove the die nut and die and clean thoroughly. Clean your barrel. Use oven cleaner to clean the piston rod assembly and orifice.

#### Check mechanical tolerances

- Is your piston tip in spec?
- Does your GO/NO GO gage work properly on the die?
- Operating temperature within spec? +/- 0.2 degree Celsius
- Force Cell/Pressure Transducer calibrated and reading properly?

# 8. Trouble Shooting

## 8.1 FAQ's (Frequently Asked Questions)

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

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

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

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

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

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

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

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

NO. Shear rates calculated from plunger speed and forces measured depend greatly on the assumption that all material exits through the die. Check to see the tip is within ASTM specs that are between 0.3751 - .3745, high temperature machines will tend to be on the low side of this specification (bigger gaps when measured cold). If the tip is worn replace it, if it is OK and you still see significant material going past

the tip you may wish to try using an O-ring tip or one of other special designs available for low viscosity (or high pressure) applications.

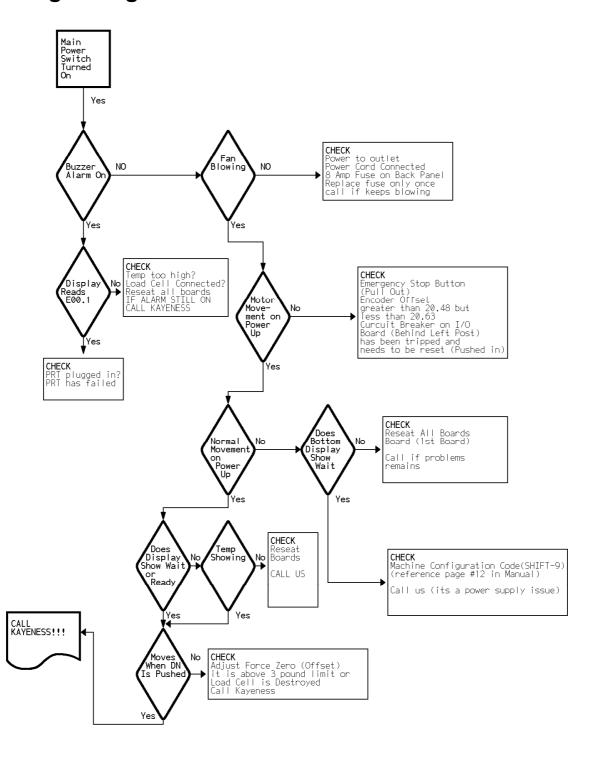
• How do I check the temperature?

You received with the machine a mercury thermometer. This thermometer comes with a calibration certificate from DYNISCO POLYMER TEST SYSTEMS comparing it to our NIST traceable reference box. Any deviation from our NIST traceable box will be noted and supplied with the thermometer. Set the machine to the temperature of the reference thermometer (see thermometer markings). Let the machine come to temperature. Only after the machine is stable insert the thermometer gently down the thermometer well, which lies under an Allen screw cap, directly adjacent to the PRT probe, on the top of the rheometer barrel.

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

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

## 8.2 Logic Diagram



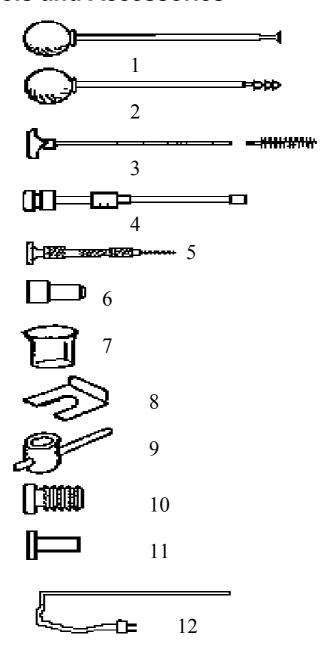
# 9. Parts List

# 9.1 Dies and Orifice

Order Name	Capillary Diameter	L/D	Entr.	Capillary Length	'apillary Length	Cleaninş Drill #	Fotal ength	apillaı Diam.	Jo-No Pin
	(in)		deg.	(in)	(mm)	# Eng.)	mm)	mm)	Note 1
Z295-1C	0.02953	1	180	0.02953	0.75000	70	0.7500	0.75	P295
Z295-16	0.02953	16	180	0.47244	12.00000	70	12.0000	0.75	P295
Z295-33	0.02953	33.3	180	0.98327	24.97500	70	24.9750	0.75	P295
Z295-40	0.02953	40	180	1.18110	30.00000	70	30.0000	0.75	P295
X300-1C	0.03000	1	120	0.03000	0.76200	69	3.2917	0.762	P30
X300-16	0.03000	16	120	0.48000	12.19200	69	14.7217	0.762	P30
X300-20	0.03000	20	120	0.60000	15.24000	69	17.7697	0.762	P30
X300-33	0.03000	33.3	120	1.00000	25.40000	69	27.9297	0.762	P30
X300-40	0.03000	40	120	1.20000	30.48000	69	33.0097	0.762	P30
Z394-16	0.03937	16	180	0.62992	16.00000	61	16.0000	1.0	P394
Z394-20	0.03937	20	180	0.78740	20.00000	61	20.0000	1.0	P394
Z394-30	0.03937	30	180	1.18110	30.00000	61	30.0000	1.0	P394
Y400-01	0.04000	1	90	0.04000	1.01600	61	5.2705	AMP	P40
Y400-15	0.04000	15	90	0.60000	15.2400	61	19.4945	AMP	P40
X400-1C	0.04000	1	120	0.04000	1.01600	61	3.4723	1.016	P40
X400-16	0.04000	16	120	0.64000	16.25600	61	18.7123	1.016	P40
X400-20	0.04000	20	120	0.80000	20.32000	61	22.7763	1.016	P40
X400-30	0.04000	30	120	1.20000	30.48000	61	32.9363	1.016	P40
Z492-16	0.04921	16	180	0.78740	20.00000	3/64	20.0000	1.25	P492
Z492-20	0.04921	20	180	0.98425	25.00000	3/64	25.0000	1.25	P492
Z492-25	0.04921	25	180	1.23031	31.25000	3/64	31.2500	1.25	P492

X500-20	0.05000	20	120	1.00000	25.40000	56	27.7830	1.27	P50
X600-20	0.06000	20	120	1.20000	30.48000	53	32.7897	1.524	P60

# 9.2 Hand Tools and Accessories



**Supplied Hand Tools** 

- 1. Charging tool for compacting pellets into barrel. Relief area to allow air to escape during packing. Part # 0051-36
- 2. Cleaning tool for barrel. Use with two patches for proper friction. Part # 0051-40
- **3.** Orange handled cleaning rod with bronze scouring brush. Use after primary cleaning tool to get the barrel nicely polished. Handle Part # 0051-47, Bronze brushes 0051-48
- **4.** Plunger for main rheometer barrel. Guide bushing should move freely up and down. Seat guide bushing in place prior to pressing RUN on rheometer. Complete assembly Part # 2052-300, Tip is removable, Tip Part # 2052-66; Sliding guide bushing Part # 0051-44
- **5.** Clean out drill bit and drill bit vise for cleaning capillary dies. Bit Vise Part # 0051-38, specify die size.
- 6. Sample filling funnel. Fits into counter sink on top of barrel. Part # 0051-45
- 7. Pyrex beaker used for sample filling of pellets or powder. Part # GP0300.
- **8.** Pull clip to automatically pull out plunger (use optional). When plunger is in barrel with no ram movement place pull clip into slots in load cap connected to the load cell. Press UP to remove the plunger. Part #2052-151
- 9. Die nut removal wrench. 7/8" T handle. Tighten only until snug. Part # 2052-156.
- **10**. Die nut. The capillary die slides into this. It is then screwed into the base of the barrel and allowed to heat up before tightening. Part # 2052-73
- **11.** Rheometer Die. Tungsten Carbide. Don't drop hot die into solvent! See table in appendix for ordering information.
- **12.** PRT platinum resistance thermometer. Temperature sensor for measurement and control of temperature. Has red banana clip connector. Part # GP1752

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

## 9.3 Spare Part Recommendations

Depending on how mission critical your operation is there are several recommendations. For operations limited to casual R&D use, you should have spare fuses, cotton cleaning patches and some bronze orifice brushes.

For more demanding environments, you should supply an abundance of brushes and cleaning patches. An extra piston tip and die will also come in handy, especially when testing corrosive or abrasive materials. Many customers prefer to keep and extra RTD (thermal detector) in stock. Although all parts are warranted from defects in craftsmanship for 1 year after the purchase, RTD's take some of the most severe thermal punishment of all components. Therefore, a spare RTD is a good investment. Go-No Go gauges may sometimes have a long lead-time. You may want to purchase extras for your site. A spare parts list should be supplied with the quote.

### 9.4 Detailed Parts List

# Part # and Description for common items on Galaxy Series and LCR5000 & 6000 Series Instruments only.

```
0051-80 LARGE POWDER FILL FUNNEL
2052-46 PISTON TOP INSULATOR
2052-66 PISTON TIPS
2052-66G PISTON TIPS W/GROOVE
2052-66R TFE PISTON TIP O-RINGS (DOZ)
2052-152 O-RING INSTALLATION TOOL
2052-72 POLYIMIDE ORIFICE INSULATOR (BOTTOM)
2052-66LL 1/2" Long piston tip needs short rod
2052-66LLS 1/2" Long piston tip for corrosion resist. barrels
0051-43LL Short piston rods for Long tips
2052-73 BRONZE ORIFICE HOLDER NUT
GP-1752 PLATINUM RTD
2052-42 LOAD CELL-500 KG.
2052-45I LOAD CAP
0051-36 S.S. CHARGING TOOL
0051-40 S.S. PATCH CLEANING ROD
0051-43 PISTON ROD - ONLY
0051-44 PISTON GUIDE BUSHING
0051-47 BORE BRUSH HANDLE
0051-48 BRONZE BORE BRUSHES
2052-100 CAPACITOR - SPRAGUE
2052-106 RELAY-MIDLAND ROSS
2052-107 RESISTOR .5 OHM 1%
2052-108 MAIN POWER TRANSISTOR - MJ11032
2052-111 MAIN POWER TRANSISTOR - MJ11033
2051-151 PULL CLIP (PISTON REMOVAL)
2051-156 T-WRENCH HANDLE
2051-157 SOCKET
2052-160 CERAMIC INSULATION BLANKET
2052-200 COMPLETE 4 TRANSISTOR PWR BLOCK ASSY
2052-300 COMPLETE PISTON ASSEMBLY: 0051-43,44 2052-
46,66
2052-22 MOTOR W/GLASS ENCODER (GAL V)
9052-22 MOTOR W/GLASS ENCODER (GAL III)
2052-32 WORM JACK W/ MICRO SWITCH BOX
2052-42 LOAD CELL-500 KG.
9052-42 LOAD CELL 1000 LBS. W/B101 BASE
9052-42 LOAD CELL 1000 LBS. W/ADAPTERS
9052-43 LOAD CELL 2000 LBS. W/B101 BASE
2052-43 FLANGE BEARING - FAF
8052-75 HEAT CHAMBER - TOP INSULATOR (BROWN)
2052-64A INSULATOR SLEEVE (BARREL MOUNT)
2052-70 BARREL HEATER (SINGLE ZONE)
2052-7DZ BARREL HEATER (DUAL ZONE)
2052-77 THERMISTOR COVER
7051-45 POLYIMIDE FILL TUBE (LARGE WHITE)
8052-62 HEAT CHAMBER COVER G7 (HI TEMP)
8052-65 HEAT CHAMBER (BARREL - STD)
8052-141 HEAT CHAMBER (BARREL) NON LINED HIGH
8052-141H HEAT CHAMBER (BARREL) LINED HIGH FORCE
2052-65H HEAT CHAMER (BARREL) LINED
8052-79 TOOL RACK
GP0300 LOADING BEAKER (PYREX)
GP-1111 PLUG-MIDGET-HUBBEL
GP-1122 POMONA PLUG -RED
GP-1400 FUSE HOLDER
```

 GP-1428
 CIRCUIT BREAKER - 8 AMP

 GP-1510
 TRANSFORMER 120/24 V.

 GP-3004
 LEVELING FEET - 3/8-16

 GP-7820
 RECTIFIER - 35 A.-BRIDGE

GP-1410 FUSE - BUSS - 8 AMP (MAIN)

# 10. Notes

# 11. HELP Utilities

## 11.1 KARS for Windows On-Line Help

The complete LCR manual is accessible in WinKARS by selecting Help from the WinKARS main menu. Help is also available by running the help icon located in the WinKARS group on your Windows desktop.

Windows help provides many powerful tools to aid in your search for help topics and references. Topics can be printed out for future reference. We suggest exploring the many options in Help for WinKARS.

## 11.2 Practical Rheology Handbook

Also available in on line help is the "Kayeness Polymer Test Systems Practical Rheology Handbook". This help topic provides useful information concerning rheometry and its processes.

# 12. Example Ram Rates

	Achievable Ram Rates (inches/min.) for Die diameters (in inches) of:								
Apparent Shear Rate	0.0820	0.0600	0.0500	0.0400	0.0300	0.0250	0.020	0.015	0.0120
0.1	0.003								
0.2	0.006	0.002							
0.5	0.015	0.006	0.003						
1.0	0.029	0.011	0.007	0.003					
2	0.059	0.023	0.013	0.007	0.003				
5	0.146	0.057	0.033	0.017	0.007	0.004	0.002		
10	0.293	0.115	0.066	0.034	0.014	0.008	0.004		
20	0.585	0.229	0.133	0.068	0.029	0.017	0.008	0.004	
50	1.463	0.573	0.332	0.170	0.072	0.041	0.021	0.009	0.005
100	2.925	1.146	0.663	0.340	0.143	0.083	0.042	0.018	0.009
200	5.850	2.292	1.326	0.679	0.286	0.166	0.085	0.036	0.018
500	14.625	5.729	3.316	1.698	0.716	0.414	0.212	0.090	0.046
1,000	29.250	11.459	6.631	3.395	1.432	0.829	0.424	0.179	0.092
2,000		22.918	13.263	6.790	2.865	1.658	0.849	0.358	0.183
5,000				16.976	7.162	4.145	2.122	0.895	0.458
10,000					14.324	8.289	4.244	1.790	0.917
20,000					28.647	16.578	8.488	3.581	1.833
50,000							21.220	8.952	4.584
100,000								17.904	9.167
200,000									18.334
500,000									
1,000,000									

	KAYENESS Galaxy V Based on machine speeds min. and max shear rates obtainable (0.005 - 24 "/min.)								
Diameter	0.0820	0.0600	0.0500	0.0400	0.0300	0.0250	0.0200	0.0150	0.0120
Min SR	0.17	0.44	0.75	1.47	3.49	6.03	11.78	27.93	54.54
Max SR	820	2094	3619	7068	16755	28953	56550	134045	261807

# 13. Conversion Factors

## 13.1 Conversions

The following conversion formulas are here for your reference and convenience.

### 13.1.1 Temperature

To Convert From	То	Use Formula
<b>°</b> C	°K	$T_k = T_c + 273.15$
°F	°C	$T_c = (T_{f}-32)/1.8$

### 13.1.2 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		

### 13.1.3 Pressure and Stress

To convert From	То	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/cm2)	Pa	9.80665E+4
bar	Pa	1.0E+5
dyne/cm2	Pa	0.10
ksi (kip/in2)	Pa	6.894757E+6

# 14. Support Vendors

### 14.1.1 Calibration/Certification of Pin Gauges

#### **Zero Check**

P.O. Box 903 Thomaston, CT. 06787 Tel: 203-283-5629 Fax: 203-283-4113

### **Meyer Gage Company**

230 Burnham St. South Windsor, CT. 06074 Tel: 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)

### 14.1.2 Cleaning Patches

### **Skyline Center Inc.**

P.O. Box 3064 Clinton, IA 52732 Tel.: 319-243-4065 800-747-4065 Fax: 319-243-9901

### 14.1.3 Bore Gauges

# Inspex Corp. 664 Bussee Hwy

Park Ridge, IL 60068
Tel: 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

### 14.1.4 Mercury (Hg) Spill Kits

Mercon Products: distributed by <u>Fisher Scientific</u> Unit 8, 7551 Vantage Way
Delta, B.C.
Canada V4G 1C9
Tech Assistance (800)926-8999
Tel: 604-940-0975
Or call Fisher Scientific

### 14.1.5 Thermometers

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

### 14.1.6 NIST Standard Reference Material (SRM)

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

Tel: 301-975-6776 Fax: 301-948-3730

# 15. Reference Books

## 15.1 Processes and Theory

### **15.1.1 Process**

The Dynisco Injection Molders Handbook, Tony Whelan and John Goff 1991
The KAYENESS Practical Rheology Handbook, Tony Whelan and John Bryson
1991

The Dynisco Extrusion Processors Handbook, Tony Whelan and David Dunning 1991

#### Melt Rheology and it's role in Plastics Processing,

Dealy & Wissburn, Van Nostrand Reinhold, 1990

### Modern Plastic Encyclopedia

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

### **Polymer Melt Rheology**

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

### **Plastics Engineering**

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

#### **Polymer Extrusion**

Chris Rauwendaal Hanser, 1986

### **Principles and Applications of Rheology**

A. G. Frederikson Prentice Hall, N.Y. 1969

### **Rheological Techniques**

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

### **Polymer Structure, Properties and Applications**

R.D. Deanin, Boston, Cahners Books, a division of Cahners Publishing Co. 1972

### 15.1.2 General Introduction to Polymers

### **Textbook of Polymer Science**

F.W. Billmeyer, Jr. Wiley-Interscience, New York 1962

### 15.1.3 Theoretical

### **Computer Programs for Rheologists**

G. Gordon, M. Shaw, SPE, Hanser 1994

### **Properties of Polymers**

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

### **Viscoelastic Properties of Polymers**

J. D. Ferry, Wiley & Sons, 1980

# 16. Appendix

## **16.1 Options Program (<Shift-Print>)**

Certain instrument options and features are enabled via pressing **<SHIFT>** then **<PRINT>** on the instrument keypad. Following is a brief discussion on their definitions and effects on the instrument.

**Auto F Zero** - This options cancels any forces present on the load cell before the test. Caution, press Run only when there is no force on the load cell. **Acquisition Mode** - The acquisition mode determine how data is collected during the experiment.

<u>PC/Manual</u> - In the PC 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 run. In the Manual mode, the operator presses the **END** key on the instrument when they feel the measurement is producing stable data.

<u>Position</u> - The instrument is purely positioned based. When a certain position is reached, data is acquired and the instrument proceeds to the next rate.

**PT Install -** If you have an instrument with a barrel pressure transducer installed, enable its operation by setting this option equal to yes.

**Machine ID** - For multiple machines, this can be set to any positive number to distinguish between different instruments.

To check the options program, press **<RESET>** then **<SHIFT>** then **<PRINT>** on the instrument keypad.

## 16.2 Firmware Settings (<Shift-9>)

Firmware settings tell the embedded software in the instrument what physical specifications the instrument has. The "config code" is accessed by pressing <RESET> then <SHIFT> then <9> on the instrument keypad. These "config code" numbers can sometimes be lost or corrupted by power outages, component failure or operator interaction. Therefore, usually the first thing technical support may suggest is check the "config code". This table is supplied for your reference. Entering the wrong number may result in operator and instrument damage. These numbers represent the physical options of the instrument from the factory. Therefore, you

cannot change model numbers, min/max speeds, max temperature or max load via these settings without encountering serious damage and risk to operators.

To check the "config code", press **<RESET>** then **<SHIFT>** then **<9>** on the instrument keypad. Display will read "MACH. CONFIG = 00XX" where XX is a number. Press **<YES>** to accept.

To change the "config code", press <RESET> then <SHIFT> then <9> on the instrument keypad to enter config mode. Press <NO> to clear the present value. Enter correct value from table and press <YES> to accept. Power off machine and then restart.

Instrument Model	"Config Code"
Galaxy 9052	34
Galaxy 9052M	36
Galaxy 0052	98
Galaxy 0052S	227
Galaxy 4052EM	
Galaxy 8052A	18
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## 16.3 Mercury Thermometer MSDS

PRINCO INSTRUMENTS >> METALLIC MERCURY THERMOMETER MATERIAL SAFETY DATA SHEET

FSC: 6685 NIIN: 008944513

Manufacturer's CAGE: 48620 Part No. Indicator: A

Part Number/Trade Name: METALLIC MERCURY THERMOMETER

#### General Information

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Item Name: THERMOMETER, SELF-INDICATING, LIQUID IN GLASS Manufacturer's Name: PRINCO INSTRUMENTS INC

Manufacturer's Street: SOUTHAMPTON INDUSTRIAL PARK

Manufacturer's City: SOUTHAMPTON

Manufacturer's State: PA Manufacturer's Country: US

Manufacturer's Zip Code: 18966-4095 Manufacturer's Emerg Ph #: 518-785-1703 Manufacturer's Info Ph #: 215-355-1500

Distributor/Vendor # 1: DEL-CO ENTERPRISES INC (215-284-5880)

Distributor/Vendor # 1 Cage: 0WYA8 Record No. For Safety Entry: 001 Tot Safety Entries This Stk#: 001 Status: SE

Date MSDS Prepared: 07MAY91 Safety Data Review Date: 03NOV93

Supply Item Manager: CX

MSDS Preparer's Name: MERCURY REFINING COMPANY

Preparer's St Or P. O. Box: 26 RAILROAD AVE

Preparer's City: ALBANY Preparer's State: NY Preparer's Zip Code: 12205 MSDS Serial Number: BRNPT Specification Number: NONE Spec Type, Grade, Class: NONE Hazard Characteristic Code: J6

Unit Of Issue: EA

Unit Of Issue Container Qty: NOT KNOWN

Type Of Container: NOT KNOWN Net Unit Weight: NOT KNOWN Report for NIIN: 008944513

### Ingredients/Identity Information

Proprietary: NO

Ingredient: MERCURY (SARA III) Ingredient Sequence Number: 01 NIOSH (RTECS) Number: OV4550000

CAS Number: 7439-97-6 OSHA PEL: S, C, 0.1 MG/M3 ACGIH TLV: S, 0.05 MG/M3; 9293

Other Recommended Limit: NONE RECOMMENDED

#### Physical/Chemical Characteristics

Appearance And Odor: ODORLESS, SILVERY LIQUID WITH A METALLIC

LUSTER Boiling Point: 674F,357C Melting Point: -38F,-39C

Vapor Pressure (MM Hg/70 F): 0.002

Vapor Density (Air=1): 7.0 Specific Gravity: 13.59

Decomposition Temperature: NOT KNOWN Evaporation Rate And Ref: NOT GIVEN Solubility In Water: INSOLUBLE Corrosion Rate (IPY): UNKNOWN

#### Fire and Explosion Hazard Data

Flash Point: NONFLAMMABLE Lower Explosive Limit: NOT GIVEN Upper Explosive Limit: NOT GIVEN

Extinguishing Media: DRY CHEMICAL, CARBON DIOXIDE, HALON, WATER SPRAY OR FOAM Special Fire Fighting Proc: MOVE CONTAINERS FROM AREA IF POSSIBLE. COOL CONTAINERS EXPOSED TO FLAMES WITH WATER FROM THE SIDES UNTIL FIRE IS OUT. STAY UPWIND. Unusual Fire And Expl Hazrds: NEGLIGIBLE FIRE HAZARD. MAY RELEASE INORGANIC

MERCURY VAPOR

Report for NIIN: 008944513

### Reactivity Data

Stability: YES

Cond To Avoid (Stability): AVOID ELEVATED TEMPERATURES AND OPEN FLAMES. Materials To Avoid: ACETYLIPIC COMPOUNDS, ALUMINUM, AMINES, AMMONIA, BORON,

BROMINE, METHYL AZIDE, TETRACARBONYLNICKEL, COPPER AND ALLOYS

Hazardous Decomp Products: TOXIC VAPORS

Hazardous Poly Occur: NO

Conditions To Avoid (Poly): NONE. WILL NOT OCCUR.

/M3 (SKIN)

Route Of Entry - Inhalation: YES Route Of Entry - Skin: YES Route Of Entry - Ingestion: YES

Health Haz Acute And Chronic: RESPIRATORY DISTURBANCES, GASTROINTESTINAL DISTURBANCES, MERCURIALISM, SKIN IRRITATION, SENSITIZATION DERMATITIS, MAY BE ABSORBED THROUGH THE SKIN, ASPIRATION HAZARD, VISUAL DISTURBANCES.

Carcinogenicity - NTP: NO Carcinogenicity - IARC: NO Carcinogenicity - OSHA: NO

Explanation Carcinogenicity: MERCURY IS NOT CURRENTLY CLASSIFIED AS CARCINOGENIC BY NTP, IARC OR OSHA.

Signs/Symptoms Of Overexp: DYSPNEA, COUGH, FEVER, NAUSEA, VOMITING, DIARRHEA, STOMATITIS, SALIVATION, METALLIC TASTE, NECROTIZING BRONCHIOLITIS, PNEUMONITIS, PULMONARY EDEMA, ABDOMINAL PAIN, DIZZINESS, CLUMSINESS, SLURRED SPEECH, HEADACHE, ALBUMINURIA, TREMORS, LOOSENING OF TEETH, BLUE NEPHRITIS, ANXIETY, WEIGHT LOSS, MENTAL DEPRESSION.

Med Cond Aggravated By Exp: NONE SPECIFIED BY MANUFACTURER.
Emergency/First Aid Proc: INHALATION: REMOVE FROM EXPOSURE. IF BREATHING REMOVE
CONTAMINATED CLOTHING. WASH WITH MILD SOAP AND WATER. GET MEDICAL
ATTENTION. INGESTION: IMMEDIATELY GIVE 4-5 GLASSES OF WATER AND INDUCE
VOMITING. KEEP HEAD LOWER THAN HIPS. GET MEDICAL ATTENTION. EYES: WASH
IMMEDIATELY WITH WATER FOR 15 MINUTES, GET MEDICAL ATTENTION.

### Precautions for Safe Handling and Use

Steps If Matl Released/Spill: ISOLATE AREA OF LEAK OR SPILL. WEAR APPROPRIATE PROTECTIVE EQUIPMENT ATTEMPT TO STOP LEAK. TAKE UP SPILLS WITH SAND OR

OTHER ABSORBENT MATERIAL AND PLACE INTO CONTAINERS. Neutralizing Agent: NONE SPECIFIED BY MANUFACTURER.

Waste Disposal Method: PREVENT WASTE FROM CONTAMINATING SURROUNDING ENVIRONMENT. DISCARD ANY PRODUCT, RESIDUE, DISPOSAL CONTAINER OR LINER IN ACCORDANCE WITH ALL FEDERAL, STATE AND LOCAL REGULATIONS.

Precautions-Handling/Storing: STORE AWAY FROM INCOMPATIBLE MATERIALS. STORE IN PLASTIC, GLASS OR STEEL CONTAINERS WITH TIGHT FITTING SEALS.

Other Precautions: STORE IN AN ISOLATED, COOL, WELL VENTILATED AREA. Report for NIIN: 008944513

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#### Control Measures

Respiratory Protection: IF VENTILATION DOES NOT MAINTAIN INHALATION EXPOSURES BELOW TLV, USE NIOSH APPROVED FULL FACE POSITIVE PRESSURE SELF-CONTAINED BREATHING APPARATUS.

Ventilation: PROVIDE LOCAL EXHAUST, PROCESS ENCLOSURE VENTILATION OR GENERAL DILUTION TO MEET PERMISSIBLE EXPOSURE LIMITS.

Protective Gloves: RUBBER IF NEEDED

Eye Protection: CHEMICAL SAFETY GOGGLES & FULL FACESHIED Other Protective Equipment: APPROPRIATE IMPERVIOUS CLOTHING & EQUIPMENT TO PREVENT SKIN CONTACT. MAINTAIN EYEWASH AND QUICK DRENCH FACILITY. Work Hygienic Practices: NO SMOKING, EATING, DRINKING.

Suppl. Safety & Health Data: THE MERCURY IS CONTAINED IN A GLASS TUBE.

#### Transportation Data

Trans Data Review Date: 93258

DOT PSN Code: IWD

DOT Symbol: A,W

DOT Proper Shipping Name: MERCURY

DOT Class: 8

DOT ID Number: UN2809 DOT Pack Group: III DOT Label: CORROSIVE

IMO PSN Code: JKJ

IMO Proper Shipping Name: MERCURY IMO Regulations Page Number: 8191

IMO UN Number: 2809 IMO UN Class: 8 IMO Subsidiary Risk Label: -IATA PSN Code: PYF IATA UN ID Number: 2809

IATA Proper Shipping Name: MERCURY

IATA UN Class: 8
IATA Label: CORROSIVE

AFI PSN

Code======

Disposal Data

Label Data

#### Luoei De

Label Required: YES

Technical Review Date: 15SEP93

Label Date: UNDATED

MFR Label Number: NOT KNOWN

Label Status: D

Common Name: METALLIC MERCURY,77-0

Chronic Hazard: X Signal Word: WARNING! Acute Health Hazard-Moderate: X Contact Hazard-Slight: X Fire Hazard-None: X

Reactivity Hazard-None: X
Special Hazard Precautions: ACUTE- INHALING VAPORS CAN CAUSE CNS DEPRESSION,
PULMONARY EDEMA, KIDNEY DAMAGE. SKIN CONTACT CAN CAUSE DERMATITIS. SKIN
ABSORPTION CAN OCCUR. CAN CAUSE EYE IRRITATION. INGESTION CAUSES CORROSION
OF MOUTH, THROAT & GI TRACT. CHRONIC- KIDNEY & LIVER

Report for NIIN: 008944513

DAMAGE, CNS DEPRESSION. STORE IN SECURE AREA. PROTECT FROM PHYSICAL DAMAGE. CLEAN UP SPILLS PROMPTLY. FIRST AID- CALL A DOCTOR IF SYMPTOMS PERSIST. EYE:IMMEDIATELY FLUSH WITH WATER FOR 15 MINUTES, HOLDING EYELIDS OPEN. SKIN:WASH WITH SOAP & WATER. INHALED:REMOVE TO FRESH AIR. PROVIDE CPR/OXYGEN IF NEEDED. ORAL:IF CONSCIOUS, INDUCE VOMITING. CALL A PHYSICIAN/POISON CONTROL CENTER IMMEDIATELY.

Protect Eye: X

Label Name: PRINCO INSTRUMENTS INC Label Street: 1020 INDUSTRIAL HWY

Label City: SOUTHAMPTON

Label State: PA

Label Zip Code: 18966-4095

Label Country: US

Label Emergency Number: 215-355-1500/800-424-9300(CHEMTREC)

# 17. Glossary of Terms

### 17.1.1.1.1 Cross head or RAM

Mechanical device used to mount the Load Cell onto

### 17.1.1.1.2 Laser Micrometer

A device used to measure die swell

### 17.1.1.1.3 Piston rod

Plunger used to extrude material through die

### 17.1.1.4 Barrel pressure transducer

A device used to measure pressure at the die in the barrel of the instrument

### 17.1.1.1.5 Load cell

Device used to measure piston rod pressure. Usually rated in pounds or newtons

### 17.1.1.1.6 RTD

(Resistive Thermal Detector) A device used to measure temperature

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