Dynisco Polymer Test Systems



LMI 4000 Series Melt Indexer Manual

Version 4.1

CE

Manual Part Number: M0704 Original Language Version



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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.

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Introduction

About Dynisco Polymer Test

Dynisco Polymer Test 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 P.A., and is built on two principles: quality workmanship and outstanding service. Dynisco Polymer Test Systems' mission has grown to meeting the physical testing needs of the plastics, food, and rubber industries by providing high quality equipment and services at low cost. The company was purchased in 1988 by Dynisco, and is now a wholly owned subsidiary of Dynisco Inc. Dynisco, an ISO 9002 qualified supplier, is the world's largest manufacturer of pressure transducers and is located in Franklin, MA.

How to Use this Manual

This manual describes the setup procedure and basic operation of the Dynisco Polymer Test Systems Series 4000 class indexers. With the accompanying MIWORKS or Daisy software manual (if software was purchased) it provides the complete description of resources at your command. It is not necessary to read this manual in its entirety. However, even experienced rheologists and technicians can benefit from the SAFETY tips and cleaning suggestions learned over many years of operating these instruments in our applications laboratory.

The Getting Started Chapter explains the details of setting up the instrument and preparing it for use. Please take a few minutes to review the safety precautions that should be taken when operating Dynisco Polymer Test Systems Melt Index equipment.

Typographic Conventions

Italics: Rheological items that have defining equations presented in the manual are shown in italics. If you come across an italicized item that is unclear you can be sure it has a mathematical definition previously defined in the manual. *Bold Italics*: These are parameters that are set from the front panel on the

Bola nancs: These are parameters that are set from the front panel on the indexer (i.e. *Melt Time, Mtd A Time* etc.). These parameters are entered into the indexer's control programs via the indexer key pad.

BOLD ALL CAPITALS: This indicates an actual key found on the indexer key pad. Example: Press the **RESET** button to return to main screen. <u>Underlined Items</u>: Underlined items' head paragraphs or sections that pertain to the particular item or model underlined. If you do not have or are not interested in the underlined item skip the section that follows it. Underlining is also used to emphasize safety issues.

How to Contact Dynisco Polymer Test Inc.

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

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

Call Dynisco Polymer Test directly at (508) 541-9400 and ask for technical support. Should you wish to comment or query in writing, address to:

Dynisco Polymer Test Inc. : Customer Service 38 Forge Parkway Franklin, MA 02038

You can also reach us through the internet at: www.Dynisco.com.

Model Descriptions

The DYNISCO POLYMER TEST SYSTEMS Series 4000 Melt Flow Indexer incorporates the latest in micro-electronic technology. It is designed to make melt flow rate testing faster, more accurate and flexible, and generate more rheological information. The heart of the system is a 32 bit microprocessor developed by DYNISCO POLYMER TEST SYSTEMS. It controls temperature, responds to keyboard commands and implements testing programs. System capabilities:

- Communications with computers
- Direct output to printer
- Stores test conditions in program memory
- Shows instantaneous flow rate read-out
- Battery backed-up date & test conditions

- Download to MS-DOS compatible systems
- □ Methods A, B, or A/B conversion and flow rate ratio
- Automated purge or weight lowering
- Control temperature within 0.1° C.
 - Automatic check of RTD sensor probe and over temperature alarm.

On Overview of MI

A flow rate test is a measure of a polymer's mass flow rate (grams extruded in 10 minutes) using a particular orifice under specified conditions of temperature and load. Machines that determine flow rate are generally called Melt Indexers. Test methods by ISO, DIN, ASTM and others specify heat chamber and piston tip diameter such that the shear stress on the polymer is the same in all machines for a given load. In addition, material specification guidelines (by ISO, DIN, ASTM, GM etc) may exist which give further constraints on how a particular type of material may be run.

The tests we are interested in performing are those described by ASTM D1238 and ISO 1133. This manual in no way supersedes either of these documents. The precision and accuracy of the test has been determined by ASTM method D1238 and can be found at the end of this manual. Contributing to both precision and accuracy are operator variances such as; packing technique, cleaning, cutting, weighing etc. With moisture sensitive polymers, dryness can also play a major role, and time can be a factor with thermally unstable polymers, so procedures must be identical. *Dynisco Polymer Test Systems* has found that charging a consistent mass of material into the barrel (±0.1 grams) is the most critical factor in getting precise data.

ASTM Melt Index Tests

Two basic methods have been developed for running flow rate tests under D-1238, Methods A and B. Method A is simply the collection of extrudate over time, while Method B is the measurement of time for the flow of a fixed volume of polymer.

Method A	<u>Method A</u> the test is completely manual and is sometimes called the "cut-n-weigh" method.
Method A/B	<u>Method A/B</u> this employs the electronic eye or digital encoder. In order to run a method B test, an A/B run MUST be run first to determine the polymer's <u>melt</u> density
Method B	Method B can be conducted only after an A/B experiment is conducted. This is a "no-cuts" test that is the most convenient for busy laboratories.

Series 4000 Product Offerings:

Introduction The Series 4000 of melt indexers are actually 2 pieces of equipment in one. First, the instrument houses the hardware needed to conduct the measurement itself -- the heaters, temperature sensors, die/orifice assembly, and so on.

	Second, the instrument has a 32 bit micro-processor incorporated into its base to control the aforementioned hardware, but also to make calculations of the Melt Index, Viscosity, and in some cases (Model 4003, 4004) calculate rheological data about the sample as well.
Model 4001	This is the entry level machine. It will conduct method A tests only and cannot be upgraded to higher models. The 4001 has a 5 program memory.
Model 4002	This model performs Method A, Method B and flow rate ratio testing. Temperature and flow rate are displayed on the instrument front panel. It has a 20 program memory. The unit can be upgraded to higher models.
Model 4003	This model contains additional program capacity and can retain up to 60 test programs. In addition, it communicates with printers and can provide a series of more advanced technical information such as shear rate, shear stress and viscosity. Statistics on flow rate and viscosity can be averaged over replicated tests.
Model 4004	The 4004 is the most sophisticated of the Dynisco Polymer Test's models ever available. It has all of the capabilities of the 4001, 4002 and 4003, and will communicate with other computers, and can be multiplexed (8 machines to one PC). With the addition of the encoder option, 10 complete test programs (set ups) can be stored.
Method B Encoder Option	The Digital Encoder option replaces the optical flag and sensor with highly accurate digital flags and sensors. You can perform experiments using from one digital flag up to fifteen digital flags. The Dynisco Polymer Test Encoder for Method B tests employs a 1016 count optical encoder, coupled to high accuracy gears, to measure the piston travel. The optical encoder has dual outputs, enabling the signal to be processed in quadrature, yielding a resolution of 0.015 mm. The linear distance of the piston is transferred to rotary displacement via a precisely calibrated arm. The tip of this arm employs a hardened & ground tip for extra long life. Accuracy over the ASTM measurement range of 0.25" and 1.00" is +/-0.4%. The optical encoder, while being very accurate, also enables the test length signal to be processed and varied digitally. Thus, any test length up to 1"(ASTM) or 30 mm (ISO) may be selected. Up to 15 MFR results per barrel filling can be obtained. The unit is securely fastened to the rear of the Melt Indexer, and the arm latches down to facilitate cleaning of the unit after the test is completed.
	Encoder Advantages
	Completely Automatic Flag Length Selection on Single MFR Tests
	Up to 15 MFR readings per Barrel Filling
	Any test length ("Flag") up to 30 mm
	\Box User Defined test length & test spacing for single or multiple tests

 $\hfill\square$ Automatic Test Length & Test Spacing for multiple tests

Pneumatic Lift	The pneumatic lift accessory is a key piece of hardware if the flow ratio test needs to be conducted. The Flow Ratio test provides users with insight into a material's thinning ability. This test involves obtaining two flow rates at two different stress levels, of generally a 10:1 ratio. First, the lower mass weight is added to the piston. Next the higher mass weight is added to the piston. The high mass weight can be 21 Kilograms. The pneumatic lift automatically raises and lowers the weight safely.
Mini-Lift	This is a small Pneumatic Lift assembly that supports the piston rod itself. It is useful for high-flow materials and can be used in conjunction with a melt plug.
Intrinsic Viscosity Option	This is a firmware upgrade to the computer housed in the Series 4003 and 4004 melt indexers only. It enables a calculation of IV values, based on the correlation between melt-index and the "wet-chemistry" technique for determining the Intrinsic Viscosity of a polymer. The IV is used to determine the molecular weight of a polymer. This is described in greater detail in the Dynisco Polymer Test applications brief, "Correlating Melt Rheology of PET to Solution Intrinsic Viscosity" by J. Reilly and P. Limbach.

Getting Started

Unpacking the Indexer

	The DYNISCO POLYMER TEST'S Melt Flow Indexer comes in a heavy duty, double-walled cardboard container. First, open the main box and remove the instrument. Several boxes will also arrive by UPS; check that all boxes are received. They are coded 1 of 5 or 3 of 5, indicating the total number is five. It is recommended that the shipping carton be saved a few days until you are certain the machine works as expected.
Bench Requirements and Placement	Typical laboratory benches are too high for efficient use of the indexers. Cleaning can be difficult and requires awkward hand positions and forces that could lead to carpal tunnel syndrome or back discomfort. We strongly suggest a bench height of 29 inches (desk top height) for an average height operator. Place the front of the indexer 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 indexer and operator (total approx. 300 lbs.). DYNISCO POLYMER TEST recommends placing from left to right, if purchased, the melt indexer; printer; computer. Test shake the melt indexer for stability. The bench top should also be able to withstand hot dies and tools being dropped on them. Carpet will quickly burn spots in it.
	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.
	Most of the machine comes pre-assembled to your door, however, certain parts are prone to breakage if they were placed in their normal operating position during shipping. These items will need to be installed before safe operation of the machine is possible. Other issues important to getting accurate data must also be addressed before valid testing can begin.
Level the Melt Indexer	Using the small round bubble level, supplied, level the melt indexer. Place the level on top of the COLD barrel and using the adjustable screw feet, bring the machine into level. Tighten the locking nuts to keep the feet in level position Test shake the melt indexer for stability. Some companies bolt the machines

directly to their benches. <u>Be sure to remove the level before turning on the</u> <u>machine.</u> The level will be damaged if it gets hot.

*** Be sure to enable the following features in the SETUP Mode ***

Pneumatic lift setup

(Please refer to drawing below)

a.) Find the safety spike (1) taped to the guide post (2). The guide post is shipped in the down position. Remove the spike from the post and mount it into the aluminum weight bucket (3) as shown. The spike should be flush with the bottom of the weight bucket. Tighten set screw (4) with a 1/8" hex wrench.

b.) Loosen the guide block set screws (5) with a 3/32" hex wrench and slide the guide post (2) up until the bottom snap ring touches the bottom of the guide block. Tighten the guide block set screws (5).

c.) Connect a length of tubing (supplied) to the main solenoid valve (6). Push the tubing into the brass fitting and pull it to seal. Attach shop air supply (60 psi) to the other end of the tubing. Be sure to incorporate a water trap in the air line.



d.) (8) is the low voltage solenoid switch which controls the UP and DOWN functions via signals from the Indexer. The main solenoid valve (6) directs air pressure to the appropriate cylinder for UP and DOWN movement. Switch (7) is an extra safety "lock" feature that will lock the lift in place if closed.

Installing the Digital Encoder The following parts should be in the encoder upgrade kit or included with the encoder based melt indexer: (if ordered)

- Two 1.25" long #8-32 Socket Head Cap Screws (SHCS)
- Two 5/8" long #8-32 Socket Head Cap Screws
- Two 5/8" long #8-32 Reduced Head Socket Cap Screws
- Six #8 Lock Washers & Four #8 Washers
- One Encoder Housing
- One Long Plate, Plate #1

One Small Plate, Plate #2

Installation Steps (See Figure 1);

1. Using two 5/8 " Reduced Head Socket Cap Screws & two #8 Lock Washers, attach plates #1 & #2 to plate #3 (plate #3 is already attached to the MI). Tighten the screws.



2. Attach the Encoder Housing to the long plate (plate #1) using two 1.25" long SHCS, two 5/8" long SHCS, 4 washers & 4 lock washers (washers first then lock washers, i.e. lock washers under screw head).

Note for Pneumatic Lift Users: With the encoder arm in the down position, lower the pneumatic lift. Move the arm up and down to check for clearance with the pneumatic lift bucket. Clearance may be increased a small amount by rotating the encoder housing before tightening the screws. If no clearance exists and the arm hits the pneumatic lift bucket, contact Dynisco Polymer Test before proceeding.

Tighten the screws

3. Plug the phone jack cable into the encoder housing.

4. Move the encoder arm into the down position.

5. Turn the power on.

6. Enable the Encoder by pressing the **SETUP** key. Browse the options until you reach **B FLAG** and press **EDIT**. Press → Quick Key so that ENCODR is over the SELECT Quick Key. **SELECT** ENCODER and press **ENTER** to accept. Press **ESC** to back out to the main menu.

	7. Press the SYS key and select TEST. Browse the menu and select Digital Encoder Test. The display shows current position and should change when the arm is moved. Press QUIT ending the procedure. ESC out to main menu.
	8. See section entitled for detailed instructions on the variety of uses and programming available with the encoder option.
RTD Connection	The RTD, or thermocouple, is buried inside the instrument's barrel. The RTD and associated temperature control electronics are calibrated against NIST traceable temperature probes at Dynisco Polymer Test Systems. To achieve accuracy required by ASTM D1238 the RTD and electronics control should be kept together. Changing a RTD requires a complete temperature re-calibration. Please consult the factory when required to replace the RTD.
Power Cord	Connect the power cord to the melt indexer. There may be a number of standard power cords supplied; the heaviest is used for the melt indexer. The factory suggests using a noise filtering outlet strip to connect the instrument and associated components to the power source.
Printer Connection	Connect the printer (if you have one) to the melt indexer. A cable is provided with two distinctly different ends; one connects to the printer and the other to the melt indexer. The connections are on the back sides of both machines. Be sure the printer is OFF when connecting the indexer.
Computer Connection	The data processing system (4004 models only) consists of a PC and the MIWORKS software package. If your melt indexer is so configured, you will find the proper cable included with the equipment. The cable will connect to COM 1 on the computer side and to the smaller connector on the back of the indexer. Set COMM PORT to PC under the SYSTEM key on the indexer. Refer to the MIWORKS software manual for more details.
	If system purchased after February, 2012, the system will only have a USB connection. Please see "Setting-Up USB Connections" section of this manual.
Options:	The Series 4000 melt indexer has various options to make conducting specific measurements easier, or make running multiple samples faster and more convenient. As previously described, the Melt Indexer maybe shipped with a Digital Encoder, a Pneumatic Lift System or a Mini-Lift system. The installation of these options is described in the appropriate Appendix of this manual.

Safety

Use gloves, it's HOT!

To prevent burns, gloves and a long sleeve shirt (or lab coat) are essential. Dies and piston rods are extremely hot and are designed to quickly transfer heat 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 indexer barrel housing can also get fairly hot, however, at barrel temperatures lower than 350 °C these will not cause burns if touched for a brief period. Consider where dies may fall. If they are dropped on Nylon carpeting or similar materials they can quickly form holes. Protective mats may be needed.

DYNISCO POLYMER TEST recommends keeping a hot piston rod in the chamber; this precludes someone picking it up inadvertently. Be sure to hold the piston by the top insulator.

Electrical Hazard

Your Dynisco Polymer Test indexer contains high voltage inside the steel housing. DO NOT remove the housing unless you are instructed to do so by a DYNISCO POLYMER TEST representative or are experienced with high voltage devices. <u>Be sure</u> the outlet used to power the indexer is properly grounded.

Calibration Thermometers use Mercury

To calibrate the temperature on the indexer, 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 where it will not be crushed or otherwise broken. When using the thermometer be careful not to drop or bend the glass. Place a hot thermometer onto cotton patches to cool . Never put a hot thermometer in contact with cold metal or cold solvent because 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 hazardous waste disposal laws when disposing of any broken thermometers. 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.

Pinch Points

Do not place weights in precarious positions where they can be bumped and fall to the floor. For large weights (over 10 kg) the pneumatic lift system is recommended. In using a pneumatic lift system for weights up to 21,600 gram, the safety pin (spike) that goes through the weights must be used. When the machine is in operation the lift system moves the weight downward quickly creating an area where anything lying beneath could be crushed. Press the **UP** and **DN** keys only when the areas above and below the weight are clear.

Fumes from Materials

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

Pre-Run Check List

The following points should be addressed before running the instrument for the first time:

- 120V power outlet properly grounded? (230 V Europe/Asia)
- Indexer level and on a sturdy bench?
- Protective oil wiped out of barrel?
- Exhaust hood or snorkel working?
- Arm protection, long sleeves or lab coat
- Operator using high temp gloves?
- Operator using safety glasses?

Series 4000: A Guided Tour

Four Options from the main menu screen.

The Series 4000 Melt Indexer has been designed for ease of use. The VFD display prompts the user for input necessary to program, configure and run the **Overview** instrument. From the main menu screen, the user has the choice of four instrument routines. The user may: Configure the Instrument (by pressing the SETUP Key) Π Edit a Program (by pressing the EDIT Smartkey) \square Run a Program (by Pressing the RUN Smartkey) Enter Calibration and Test Routines (by pressing the SYS key) It is important that the instrument first be properly configured. This involves Setup pressing the SETUP key and checking instrument options and units for particular parameters Melt Index experiments (particularly method A tests) rely on the instrument to Edit reach the proper temperature and (in method B and A/B tests) collect information on the travel of the plunger. The user is first expected to input the appropriate parameters for the experiment and subsequent calculations in the EDIT routine. After this is accomplished, the user may RUN the program. The melt index unit relies on the operator to properly program the unit, load the Run sample, start the experiment, wait the appropriate length of time, and (in method A tests) collect the sample to weigh. All tests require that the instrument be properly cleaned between runs Many calibrations can be performed in the SYS, CALIBRATE mode. Although System the unit is not expected to change calibration values over years, it is always wise

to check the previous values. Maintenance should typically be performed on our instruments every 6 months. Your lab procedures may be different.

Each of these instrument routines is covered in greater detail in the following sections. Before the user attempts to work with the instrument, it is important to become familiar with the keyboard itself and the keystrokes necessary to perform particular functions.

Navigating the Keypad

The keypad provides all of the means necessary for programming the Dynisco Polymer Test Melt Indexer (Unless it is connected to a PC.) Other functions that can be performed on the keypad include calibration, instrument tests, parameter setups and pneumatic lift controls.



Introduction

Keypad Definitions

1 ESC (ESCAPE)

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

2 PROG (PROGRAM)

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

3 SYS (SYSTEM)

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

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

CALIB- Select either Digital Flag, RTD Electronics, RTD Sensor, Optical Flag or IV Offset calibrations by toggling prompt with UP or DOWN QUICK KEYS and using SELECT to start the calibrations. Please reference Configuring the Instrument sections (Page) or Appendix # for more detailed operations of calibrations.

TEST- Select either RTD Display, Digital Encoder, Printer or Display Brightness tests by toggling prompts with the UP or DOWN SMART KEYS and using SELECT to start the test. Please reference Configuring the Instrument sections (Page) for more detailed operations of tests.



4 <u>SHIFT</u>

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

5 **ID (IDENTIFICATION)**

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

6 SETUP

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



If your instrument is equipped with the Pneumatic lift option, these keys will manually operate them.



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

9 <u>BACKSP (BACKSPACE)</u>

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

10 <u>NUMERAL/ALPHA INPUT</u>

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

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

11 DISPLAY

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

12 <u>SMART KEYS</u>

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

Configuring the Instrument

Intro: The SYS (SYSTEM) Mode

Many functions can be performed on the instrument using its operator interface. Hardware calibrations, tests and option configurations will be discussed in this section.

The system mode lets the user perform calibrations or hardware tests. The system mode is entered by pressing the SYS keypad. For complete information on the keypad and its function's reference **Navigating the Keypad Sections** in the manual. Once you have entered the CALIBRATE and TEST Select mode you can select between calibrate and test.

Selecting CALIB (Calibration) in the SYS Mode

CALIB:Digital Flag Home	The Digital Encoder is calibrated at Dynisco Polymer Test prior to shipment. Because the arm is locked into place, calibration is rarely required. The instrument does allow you to reset the home position of the encoder. If calibration needs to be checked due to suspected encoder damage, then contact Dynisco Polymer Test for assistance and the necessary tool kit.
	Please reference the section entitled "Instrument Calibration" for further details on actual calibrations.
CALIB:RTD Electronics	The RTD electronics are calibrated at Dynisco Polymer Test prior to shipment. Please do not reconfigure this option.
	Please reference the section entitled "Instrument Calibration" for further details on actual calibrations.
	Calibration of the RTD Sensors requires one or two thermometers or equivalent.
CALIB:RTD Sensor	The RTD sensor is calibrated at Dynisco Polymer Test prior to shipment. There should be no need to re calibrate the RTD sensor.
	Please reference the section entitled "Instrument Calibration" for further details on actual calibrations.
CALIB:Optical Flag	The instrument allows for the adjustment of the Optical Flag sensitivity. This determines how sensitive the Optical Flag LED Receiver is in either of two states, blocked or open. Sensitivity directly affects the measured length of the tape flag.
	The Optical Sensor is calibrated at Dynisco Polymer Test prior to shipment. There should be no need to re-calibrate the Optical sensor. For this procedure you must have the <i>Flag Calibration Unit</i> (Factory Part # D2044).
	Please reference the section entitled "Instrument Calibration" for further details on actual calibrations.
CALIB:Intrinsic Viscosity Offset	The IV offset is the difference between the computed IV and the experimentally obtained IV. If option available, enter the difference here for correlation during IV runs. See the I.V. section.
	Please reference the section entitled "Instrument Calibration"

for further details on actual calibrations.

Selecting TEST in the SYS Mode

TEST:RTD Display	Press SELECT to view actual temperature reading accurate to 1/100 of a degree Celsius. Dynisco Polymer Test technical support may have you do this under certain conditions. RTD Display Test may also come in handy when equilibrating RTD's and thermometers.
TEST:Digital Encoder	Displays the current digital encoder arm position. Readings for home position should be 105.00 mm, +/- 1.5 mm.
TEST:Printer	Your model must support printer output to perform this test. A test pattern output will be produced on the connected printer. Press STOP to end printer output.
TEST:Display Brightness	The operator can control the VFD brightness level. Select a percentage of default brightness.

The SETUP mode

Introduction	This software routine is used to set user preferences for data collection. This routine also turns off instrument options such as the digital encoder or pneumatic lift. Please scroll through the settings prior to commissioning the instrument and make a note of the values particular to your instrument. If for some reason the settings are changed or erased they can be easily re-entered from this menu. By pressing the SETUP key the following series of inputs are requested of the user. Press the ENTER SMART Key to input the appropriate value. After entry is complete, press the ESC key to save modified information. After pressing SETUP, scroll through the list of settings using the UP or DOWN SMART KEYS . Select the EDIT SMART KEY to change the parameter accordingly. Please reference Configuring the Instrument Section for a detailed description of each parameter. Note the order the items appear can vary.
REJECTION	[15, OFF]] Exclude data points that are more than the operator entered number of standard deviations from the mean. Exclude these points from all statistical calculations. Points are not deleted but marked on the printouts with asterisks. The rejection option is used for systems configured with a printer. Data points are printed but not included in statistics.
AVERAGE	[AUTO, OFF, ON] ON: average is computed for one or more data collection runs. Generates statistics for printout. AUTO: compute the average of a single data collection run without operator interaction at the end of the run.
MFR / MVR	[MFR or MVR] The operator can choose the calculated result. The choices are Melt Flow Index or Melt Volume Index respectively.
B FLAG VISCOSI	[ENCODR or FLAG] Enable either the Digital Encoder or the Optical Flag devices, respectively.
TY	[POISE, PA-S, REYN] Select units of viscosity for displayed results.
PRINTE	[MINI, OFF, EPSON, IBM, LASER] Select output device. Mini printer, No device, Epson compatible, HP Laser Jet compatible, respectively. (Only 4002 models will have the mini printer option)
ĸ	
FORM FEED	[OFF or ON] Perform a Form Feed (A page is sent out of the printer) after the run data is printed.

COMM PORT	[PC, OFF, DIAG] Open up communications port for computer output, no output or diagnostics mode, respectively.
MINI LIFT	[OFF or ON] Enable the mini lift to function during material runs when installed.
PNEU LIFT	[OFF or ON] Enable pneumatic lift to function during material runs, when installed.
LOCKOUT	[OFF or ON] Enable password protection. When ON users can not edit test or system parameters only run tests. The default password is 1234, if the LOCKOUT is enabled this number must be put in to turn the LOCKOUT off. The NUMBER item allows a new password number to be entered.
DATE and TIME	Set the appropriate date and time here. Follow prompts
MACHINE ID	[##] Assign a unique machine number to the instrument. If using MIWORKS be sure this is set to one if you have only a single machine or to the Porter box port number for multiple machines. This allows you to tell what machine your sample was run on when you have more than one machine.
LANGUAGE	[CUSTOM, ENGLISH, FRENCH, ITALIAN, SPANISH, GERMAN] Switch
ORIFICE DIA	between desired languages.
ORIFICE LEN	[XXX] Store standard orifice diameter, 2.096 mm. Used in calculations.
	[X.XXX] Store standard orifice radius, 8.000 mm. Used in calculations Scroll
	through this menu and make appropriate changes. Press ESC twice and any
	changes that have been made will be implemented.
	Programming
Introduction	This section is intended for a quick reference of method parameters. Please refer to the appropriate sections in the manual for more specific information about a particular method.
	Before a measurement can be made, the RUN PARAMETERS must be entered. These parameters dictate how the experiment will be performed. Many of these parameters are set by particular ASTM test procedures. The SERIES 4000 software has many of these values pre-loaded into memory.
Create (New)	Press the EDIT SMART Key to start the EDIT mode. Enter the new program number into the upper left hand corner of the display and press ENTER. Proceed to EDIT.
Recall (Old)	Press the PROG button and enter the program number in the upper left hand corner of the display or BROWSE with the Smart Keys. Press ACTIVE to use the selected program number.
Save Program	From the EDIT mode, ESC out until prompted to SAVE PROGRAM. Select SAVE to save. In order to receive the SAVE PROGRAM prompt, a value must be changed while in the editing mode.
While EDITING	Browse the suggested values appearing on the 3rd line of the Edit Screen with the \Re SMART Key (Browse Key). Select the desired value with the

	SELECT SMART Key. Accept the value with the ENTER SMART Key. The user always has the option to enter values manually via the numeric keypad. Below is a listing of the run parameters that are required for each test method.
Method A Run Parameters	METHOD = [A, B, A/B or RATIO] SELECT and ENTER Method A. SET POINT = [Temp. Celsius] Dependent on the material, ASTM requirements. Enter or Select value using browse key \Im .
	MELT TIME : [Time, Seconds] ASTM D1238 equilibration time for most materials. Enter or Select value using browse key \mathfrak{D} .
	CUT TIME =[Time, Seconds] This is the sample collection time, the length of time an extrudate sample is squeezed out. Enter or Select value using browse key. Dependent on Flow Rate characteristics.
	NO OF CUTS =[Number] Dependent on material - up to 5 cuts allowed LOAD =[Weight, Grams]. Mass of weight plus piston assembly, ASTM requirements
	QC LIMITS [On or Off] Allows QC limiting of data to be used
	QC Limits High [Number] Highest MFR limit saved
	QC Limits Low [Number] Lowest MFR limit saved
	PROGRAM ID [Characters] Name of program up to 14 characters long
	METHOD = [A, B, A/B or RATIO] SELECT and ENTER Method A/B.
	SET POINT = [Temp., Celsius] Dependent on the material, ASTM
Method A/B Run Parameters	requirements. Enter or Select value using browse key \mathfrak{R} .
	MELT TIME : [Time, Seconds] ASTM D1238 equilibration time for most materials. Enter or Select value using browse key \mathfrak{R} .
	CUT TIME =[Time, Seconds] This is the sample collection time, the length of time an extrudate sample is squeezed out. Enter or Select value using browse key R . Dependent on Flow Rate characteristics.
	FLG LENGTH =[Length, millimeters] Flag length in millimeters. Dependent on Melt Flow Rate. Digital Encoder equipped instruments will be asked further questions. Reference Digital Encoder Section in manual.
	LOAD =[Weight, Grams] Mass of weight plus piston assembly, ASTM requirements
	PROGRAM ID [Characters] Name of program up to 14 characters long
	METHOD = [A, B, A/B or RATIO] SELECT and ENTER Method B.
	SET POINT = [Temp. Celsius] Dependent on the material, ASTM
	requirements. Enter or Select value using browse key.
Method B Run Parameters	MELT DENSITY =[X.XXX,g/cm ³] Numeric value, calculated from A/B Run. MELT TIME : [Time, Seconds] ASTM D1238 equilibration time for most materials. Enter or Select value using browse key.

	NO FLAGS : [X] Stands for <u>Number of Flags</u> . Enter the number of stripes on your flag. This applies only to units with an optical eye for type B and A/B measurements. This step is different for users with a digital encoder. Please reference Digital Encoder Section.
	FLG LENGTH =[Length, mm] Flag length in mm for Optical Eye Instruments. Dependent on Melt Flow Rate. Digital Encoder equipped instruments will be asked further questions. Reference Digital Encoder Section in manual.
	LOAD =[Weight, Grams] Mass of weight plus piston assembly, ASTM requirements
	QC LIMITS [On or Off] Allows QC limiting of data to be used
	QC Limits High [Number] Highest MFR limit saved
	QC Limits Low [Number] Lowest MFR limit saved
	PROGRAM ID [Character string up to 14 characters long]
RATIO (Flow Ratio) run parameters	METHOD = [A, B, A/B or RATIO] SELECT and ENTER RATIO. SET POINT = [Temp. Celsius] Dependent on the material, ASTM requirements. Enter or Select value using browse key.
	MELT DENSITY =[X.XXX, g/cm ³] Numeric value, calculated from A/B Run. MELT TIME : [Time, Seconds] ASTM D1238 equilibration time for most materials. Enter or Select value using browse key.
	FLG LENGTH =[Length, mm] Flag length in millimeters for Optical Eye Instruments. Dependent on Melt Flow Rate. Digital Encoder equipped instruments will be asked further questions. Reference Digital Encoder Section in manual.
	FLG LENGTH HIGH =[Length, Inches] Flag length in millimeters for second flag strip.
	LOAD =[Weight, Grams] Mass of weight plus piston assembly, ASTM requirements
	LOAD HIGH =[Weight, Grams] Mass of weight plus piston assembly of second weight, ASTM requirements
	PROGRAM ID [Character string up to 14 characters long]

Run a Program

Runs are initiated from the SMART key panel after your run parameters have been properly set. Press RUN on the SMART keys. Each test method has a different set of test protocols. Please refer to the further manual sections for more specific information about a method.

Method "A" Run

General Description	Method A test involves collecting extrudate from the instrument over a fixed period of time, then converting the result to grams/10 minutes. After the melt time is over, a sample is collected by first cutting the extrudate across the orifice face, then waiting a predetermined amount of time and then making a second cut. (It is often very inconvenient or even impossible to wait 10 minutes). The sample is weighed and the flow rate is converted to grams of flow that would have occurred over 10 minutes. Test temperatures can be obtained from the manufacturer, from tables in ASTM D1238 or through experimentation. Materials with Melt Flow Rates below 50 g/10 min. are generally done using Method A.
Calculations: Method A	Calculations for the Method A test are straightforward $MFR = \frac{M600}{T}$
	<i>I</i> where M is the mass in grams of material collected over time T in seconds. The MFR value has units of g/10 min. (grams/10 minutes).
Power-Up	The instrument is turned on by throwing the switch located on the rear right top corner of the instrument (as one faces the instrument). Toggle the switch to the right and the unit will power-up. The large, friendly VFD display will power up and display system information as follows:
	Melt Indexer Serial NumberXXXXXXModel Number 400XVer 1.01(c) DYNISCO POLYMER TEST, 1999
	The instrument will beep twice after this screen. At that point, the screen will refresh and the main menu screen will be displayed.
	PROG # TEMPERATURE TIME

PROG #	TEMPERATURE	TIME	
METHOD	PROGRAM NAME		
	STATUS		
EDIT		RUN	

Programming a Method A

Introduction

The instrument is powered-on and the main menu screen is seen. If you plan to use an existing method A program skip to step 4 NOTE: The unit will load the last program run into memory and attempt to equilibrate the barrel temperature to that set-temperature.

1) Press the EDIT SMART Key. -

Machine SetupThe square cursor blinks in the upper left-hand corner of the screen prompting
the user to input the number of the program that will be created or edited.

2) Enter 05 (for example) and press ENTER on the numeric keypad. The method in program 05 will now be configured for a method A experiment. Press the center EDIT SMART Key to confirm. You can press ENTER to edit the existing program or input a new program number to create a new program.

3) Enter Run Parameters

The user will now be prompted to enter the run parameters for the method A experiment. The 2nd line of the VFD display displays the parameter to be edited. The 3rd line lists a group of suggested values. The bottom line of SMART Keys is used to **ENTER, SELECT or SCROLL** data input, respectively.

METHOD =

Press the \mathfrak{R} SMART Key (**browse**) until the 3rd line of the display has the A over the **SELECT** SMART Key.

Press the **SELECT** SMART Key to select the method A test. The second line should now read METHOD = A

Press SELECT/ENTER to accept and move to the SET POINT.

SETPOINT =

The user may enter a temperature on the numeric keypad and press enter or use the **ENTER**, **SELECT** and **SCROLL** SMART Key keys to choose a typical value.

Press SELECT/ENTER to accept the value and move to the MELT TIME

MELT TIME =

The user may enter a melt time from the numeric keypad and press enter or use the **Enter, Select and Scroll** SMART Key keys to choose a typical value Press SELECT/ENTER to accept the value and move to the NO OF CUTS

NO OF CUTS =

The user must enter the number of cuts from the numeric keypad and press ENTER to accept the value and move to the LOAD screen.

LOAD =

Enter the LOAD from the SMART Keys or from the numeric keypad. Pressing ENTER then enters the QC limits screen.

QC LIMITS =

If **YES** is entered, the user is prompted to enter the HI and LOW QC limits from the numeric keypad. Please reference "Edit a Program" section for descriptions of HI/LOW QC Limits. Pressing **ENTER** then prompts the user to enter the PROGRAM ID.

PROGRAM ID =

Enter the program ID and press ENTER when finished. Numeric and alphanumeric values may be input. See "Navigating the Keypad" for more information on alphanumeric input.

At this point, the screen will return to the EDIT Program METHOD = ? screen. All data has been input at this point. Press **ESC**. The program can be verified at this point by pressing the **UP** or **DOWN** SMART Keys. This ends the program edit mode.

To SAVE the program: Press **ESC** twice to back out to the SAVE Program mode and select **SAVE** to save the program under the OLD program name. Change the program name in the EDIT mode.

4) Choosing the Program

Press the PROGRAM button. A square block will appear in the upper left corner of the LED display. Enter the number of the program that was just created (05) or the number of the previously entered program of your choice. You can also press **UP** and **DOWN** to browse preprogrammed programs.

The unit will then attempt to latch into the specified temperature. MAKE SURE THE PISTON and DIE are in the unit. The temperature must equilibrate with the assemblies in place. The user must wait until the specified temperature has been achieved to continue with the experiment. When the instrument has latched-in, continue with sample loading as follows:

Loading the Sample

Loading the BarrelRemove the piston rod and lay it on a cotton cloth. Check to see if the die is at the bottom of the barrel. Fill the 10 ml beaker with an accurately weighed charge of material (see section on Charge Weight). Fill heat chamber with this appropriate charge. Use about 5 grams if nothing is known about the material. Add about two-thirds of the beaker into the barrel, using the brown fill funnel. The loading should be done in two increments, the first consisting of approximately 60% of the material. Tamp down the material with the packing tool using approximately 20 pounds of force. The packing tool can be inserted through the fill funnel. If bridging (clogging-up) of the material occurs in the fill funnel, slide the fill funnel off to one side (without lifting) and tamp down on the material in the barrel. Move the fill funnel back into place. The remaining material should drop down into the barrel directly. Repeat with the remainder of the material.
Bubbles are in your sample if snapping/popping noises are heard as the proceeding for the directly is for each the place of the place of the place of the directly is for each the place of the directly is for each the place.

Bubbles Bubbles are in your sample it snapping/popping hoises are heard as the extrudate is forced through the die orifice. If bubbles appear in the extrudate use less material between tamping down. If bubbles continue the sample may not be sufficiently dry.

RUN
 Place the plunger into the barrel. (Position the Optical Flag post to the right side). Seat the guide bushing down into the barrel if it as not already fallen into place. The guide bushing should move freely on the plunger. Place the weight on the plunger rod and immediately press RUN. RUN must be pressed immediately after loading the barrel to be compliant with the ASTM 1238 specification. Pressing RUN also starts the internal computer program running. This program will start an internal timer. Wait for the melt time (pre-heat) clock to count down from 360 seconds.



If the indexer has printing capability, flow rate will be printed along with other test details. When multiple tests have been completed an average, standard

deviation and coefficient of variance can be printed once, when the **END** key is pressed.

Cleaning Up Push down on the weight and purge any material remaining in the barrel. Remove the rod by twisting it clockwise to break the seal created by the molten plastic then pull straight up. *Warning: If you pull the rod out too quickly you may cause a suction that pulls the die out along with it.* Wipe the plunger rod with a cotton rag. Remove the die with the die removal tool and clean with the appropriate cleaning tools. 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 die and plunger back into the barrel. This allows the plunger and die 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 exact same material. For materials that degrade or are moisture sensitive, we recommend cleaning both the barrel and the die completely. To clean the die, use the die removal tool to push the die up from the bottom of the barrel and out the top. Wipe it with the cotton rag and clean it by running the drill bit through it several times. Remove material that collects in the grooves of the drill bit and repeat until the drill passes easily through the die. Scrape the die with the cutting knife if needed to clean the top and bottom faces. For materials that crystallize quickly, you can clean the die by first running a drill bit up into the die while it is hot and in the indexer. This will make it much easier to get the cleaning drill bit in when the die is removed and the material starts to solidify.

While the die is out, put on safety glasses and look down the barrel bore to be sure it is clean. A clean barrel will have a mirror-like shine to it. If it is not run a couple of patches up and down it before putting the die back into position.

User Tips:

User Tip #1 Drop the die into the barrel and listen for it to double bounce off of the bottom of the barrel. If the barrel is dirty, the die hangs up and will not bounce when it hits the bottom of the barrel.

User Tip #2 If you are using a PVC die (D3364 unstable materials) be sure to get the material out of the conical top section. (standard dies have a flat entrance and exit).

When the die is out of the barrel it cools down quickly. The longer it is out, the longer one must 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 0.2 C of the set point you can begin your next test. Loading material will cause a small temperature change even if the temperature steeping was locked in. The melt time (360 seconds) will allow ample time for the temperature to reach the steeping before the first data point is collected.

Always leave the barrel 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 materials that are extremely hard to clean (Polystyrene, EVA and elastomers, etc.) solvents may be required. Never use flammable solvents on a HOT barrel. In general however, DYNISCO POLYMER TEST does not recommend using solvents for health, safety, and environmental reasons.
Cleaning Up a really Big Mess	Oven cleaner (Easy-Off sprayed onto a cold plunger and left overnight will do an excellent job of cleaning degraded material off of the shaft, the outside of the die and the die retainer plate. Be careful not to inhale oven cleaner vapors.

Method "A/B" Run

General Description

In a Method A/B test both a Method A test and Method B test are performed on the same charge of material. The melt flow rate, derived from Method A, is equated to the Method B flow equation and solved for apparent melt density. The value in doing this test is obtaining a valid apparent melt density which can later be used in a sole Method B test (no manual cuts of extrudate nor weighing) to achieve results equivalent to Method A (operator must make manual cuts and weigh sample).

Calculations: Method A/B

Method B MFR = $\frac{R L}{2} \xrightarrow{600}{}^{2}$ Method A MFR = $\frac{T_{B}}{M600} \xrightarrow{T_{A}}{}^{2}$ $A = \frac{MT_{B}}{\Box LR} \xrightarrow{2}{}^{2}T$

Where R= radius piston (cm), T_B is time to traverse the Method B distance L (sec), L = Method A length of flag (cm), M is the mass in grams of material collected over the time T_A in seconds. MFR has units of g/10 min. (grams/10 minutes). \rangle is called the apparent melt density and is defined by equating the methods, the Method B flow rate must equal Method A.

This apparent melt density definition forces the two test methods to agree. DYNISCO POLYMER TEST recommends taking an average of apparent melt densities from at least five separate A/B tests on representative samples of polymer. This average apparent melt density can then be used for Method B tests to get Method A values without having to make cuts! It is called an "apparent melt density" because it is actually a correlation coefficient that forces Method A and Method B to agree. If there were no leakage past the plunger tip and the extrudate were bubble free and few other minor factors were taken into consideration then a true melt density could be assessed. All lengths are in mm.

Programming Method A/B

Introduction	The instrument is powered-on and the main menu screen is seen. If you plan to use an existing method B program skip to step 4 NOTE: The unit will load the last program run into memory and attempt to equilibrate the barrel temperature to that set-temperature.
Machine Setup	1) Start a new program by pressing the EDIT SMART Key The square cursor blinks in the upper left-hand corner of the screen prompting the user to input the number of the program that will be created or edited.
	2) Enter a program number "#" and ENTER on the numeric keypad. Now you are in the VIEW program mode of a new program. You can browse the program parameters for EDITING by pressing the EDIT SMART Key.
	3) Enter run parameters by pressing EDIT again. The instrument is now in the VIEW Program mode starting with METHOD. Press the EDIT SMART Key to set the run parameters to method A/B. The 2nd line of the VFD display displays the parameter to be edited. The 3rd line lists a group of suggested values. The bottom line is SMART Keys used to ENTER , SELECT of SCROLL data input, respectively.
	METHOD =
	Press the \bigotimes SMART Key (browse) until the 3rd line of the display has the A/B over the SELECT SMART Key
	Press the SELECT SMART Key. Press the SELECT SMART Key to select the method A/B test. The second line
	should now read METHOD = A/B Press SELECT/ENTER to accept the value and move to the SET POINT.
	SETDOINT -
	The user may enter a temperature on the numeric keypad and press enter or use the ENTER , SELECT and SCROLL SMART Key keys to choose a typical value
	Press SELECT/ENTER to accept the value and move to the MELT TIME
	MELT TIME = The user may enter a melt time from the numeric keypad and press enter or use
	Press SELECT/ENTER to accept the value and move to the CUT TIME
	CUT TIME = The Method A part of the run needs to know the amount of time between extrudate start and stop cuts to calculate the correct result. The user must enter the time manually from the keypad or browse preset values. Press SELECT/ENTER to accept the value and move to the FLAG LENGTH screen.
	DENSITY =

Enter the predetermined Melt Density of your material. Press ENTER.

FLG LENGTH =

Enter or select the length of your flag. Remember, Method B is volume, using distance traveled to acquire results.
LOAD =

Enter the LOAD from the SMART Keys or from the numeric keypad. Pressing ENTER then enters the QC limits screen.

QC LIMITS =

If **YES** is entered, the user is prompted to enter the HI and LOW QC limits from the numeric keypad. Please reference "Edit a Program" section for descriptions of HI/LOW QC Limits. Pressing **ENTER** then prompts the user to enter the PROGRAM ID.

PROGRAM ID =

Enter the program ID and press ENTER when finished. Numeric and alphanumeric values may be input. See "Navigating the Keypad" for more information on alphanumeric input.

To SAVE the program: Press **ESC** twice to back out to the SAVE Program mode and select **SAVE** to save the program under the OLD program name. Change the program name in the EDIT mode.

4) Choosing the Program

Press the PROGRAM button. A square block will appear in the upper left corner of the LED display. Enter the number of the program that was just created (#) or the number of the previously entered program of your choice. You can also press **UP** and **DOWN** to browse preprogrammed programs.

The unit will then attempt to latch into the specified temperature. MAKE SURE THE PISTON and DIE are in the unit. The temperature must equilibrate with the assemblies in place. The user must wait until the specified temperature has been achieved to continue with the experiment. When the instrument has latched-in, continue with sample testing:

consisting of approximately 60% of the material. Tamp down the material with the packing tool using approximately 20 pounds of force. The packing tool can

The Actual Run

(Note that the standard Dynisco Polymer Test In-house test procedure section in this manual is an abbreviated version of what follows) How to do it: Step by Step Press EDIT and verify all entered program data values are correct. Make sure all equipment has been properly cleaned. Insert piston rod and let the system **Instrument Prep** come to temperature and wait for temperature lock-in. Lock-in has occurred when the display reads "READY". Heat up and lock-in must occur with orifice and piston rod in the heat chamber. During heat up the program may be adjusted and the Operator. and Sample ID can be entered by pressing the **ID** key. Remove the piston rod and lay on a cotton cloth. Check to see if the die is at the bottom of the barrel. Fill heat chamber with an appropriate charge or use Loading the Barrel about 5 grams if nothing is known about the material). Put the sample into the 10 ml beaker. Add about two-thirds of the material into the barrel, using the brown fill funnel. The loading should be done in two increments, the first

be driven through the fill funnel. If bridging (or clogging)of the material occurs in the fill funnel, slide the fill funnel off to one side (without lifting). Pack the material in the barrel directly with the packing rod. Move the fill funnel back into place. The remaining material should drop down into the barrel. Repeat with the remainder of the material.

Bubbles are in your sample charge if a snapping/popping sound is heard as the extrudate is forced through the orifice. If bubbles appear in the extrudate use less material between packing. If bubbles continue, the sample may not be sufficiently dry.

Place the plunger into the barrel and position the plunger arm directly over the slot on the indexer cover. Seat the guide bushing down into the barrel if it as not already fallen into place. The guide bushing should be moving freely on the plunger. Place the weight on the plunger rod.



Plunger for Method B and Method A/B Measurements (without digital encoder module)

Inserting the Timing Flag

Insert the plastic flag down into the slot on the top right side of the indexer then slide the flag onto the plunger arm through the flag hole. Make sure that the flag does not touch the sides of the flag slot. Twist the plunger as needed to get it to line up with the flag slot and slide the flag on the arm until it is in a vertical position. Immediately press **RUN. RUN** must be pressed as quickly after loading as possible to be compliant with the ASTM 1238 specification. Pressing **RUN** also starts the internal computer program running. This program will start an internal timer and will attempt to print initial test information to a printer or PC if the devices are available. The start of the flag time should begin between 6 and 8 minutes after the test starts or the test does not follow ASTM protocol. Wait for the melt time clock to count down from 360 seconds.

When melt time is over a flag timer appears set at 0.000. When the front edge of the black flag passes the optical eye the timer starts. If the sample extruded so quickly that it has already hit the front of the flag prior to the melt time passing one must either:

Add more material

	 Use a flow plug Reduce the test load (e.g. 2160 to 225 g) Reduce the test temperature (e.g. 230 to 190 C) Support the plunger and piston during melt time Change to a non-standard die (e.g., 1/2 diameter, 1/2 height) The first two are typically used. If, on the other hand, if the sample extrudate is so slow that you do not reach the flag before 8 minutes has expired you must
	take one of the following actions:
	 Add less material Increase the test load (e.g. 2160 to 21,600 g) Increase the test temperature (e.g. 190 to 230 C) Change to a non-standard die (typically larger diameter)
	Since Dynisco Polymer Test flags start at the same position as the first scribe mark, the amount of sample charge works for both Method A and Method B testing.
Making the Cut	When the lower scribe mark on the plunger reaches the top of the guide bushing simultaneously cut the extrudate and press RUN . Use the cutting knife in a wiping like action up against the bottom of the die. After pressing RUN , a countdown timer appears on the left of the front display. As it counts down it will give a warning beep when 10 seconds remain. Exactly when it reaches zero and beeps cut the extrudate up against the bottom of the die, as before. Weigh the sample and enter the weight in grams using the touch panel then press YES . You may have to wait for the flag to traverse the photo eye for slow flowing materials. A precision balance is needed in order to obtain correct flow rate measurements. The flow rate and melt density will be immediately calculated and displayed. A extrudate weight of about 1.0 gram or more will reduce weighing errors, adjust the cut time as needed to get at least 0.5 grams of material.
	If the indexer has printing capability, flow rate will be printed along with other test details. When multiple tests have been completed an average, standard deviation and coefficient of variance can be printed once, when the END key is pressed.
Cleaning Up	Clean up is the same as that for Method A.
Your data	Data will be displayed on the VFD or printed if you have the printer option. Follow the prompts on the VFD to END or SAVE the run.

Method "B" Run

General Description

Method B is an assessment of a material's flow characteristics based on the volumetric displacement rather than weight of extrudate with time as in Method A. Unlike Method A, no cutting and weighing of the extrudate is required to perform a Method B test. Results from Method B test can be expressed directly as Melt Volumetric Flow Rate (MVR) in ml/10 min. To relate the results of a Method B run back to Method A, the apparent melt density must be known. The determination of the apparent density is illustrated in the previous section using Method A/B. Without the apparent melt density MFR cannot be calculated using Method B. Some companies use MVR directly without ever determining MFR.

The piston's downward travel time is determined from a counter initiated by an optical sensor or digital encoder. The LED senses an opaque flag on a transparent tape hung off the top of the piston rod. With all Dynisco Polymer Test Systems flags, Method A and B <u>start</u> in the same place. Flags may be 1/8, 1/4, 1/2, or 1". Multiple flags are discussed in later. Recently, Method B has become the more common test because it is simpler to run and more precise for routine analysis. In addition, the encoder system makes it possible to get as many as 15 results from one run.

Calculations: Method B

Flow rate for method B is computed as follows:

$$\begin{array}{c} MVR \rangle & MFR & \underline{R \ \underline{L}} \rangle \\ = & = & \frac{R \ \underline{L}}{7} \end{array}$$

Where R = radius piston (cm), T is time to traverse the distance L (sec), L =

length of flag (mm), \geq apparent **melt** density of polymer (g/cc)

You should determine the melt densities for your material using your own melt indexer. Variations in technique and difference in material grades can cause differences from user to user. The following table of melt densities can be used as a general guide if you get values which differ by more than 10% from these chances are you are doing something incorrectly. Fillers, reinforcing agents etc. tend to increase the melt density of the material. Notice that apparent melt density is a function of temperature. In general the solid state density is a very poor estimate of the melt density and should not be used.

Some typical Melt Densities

Temperature (C)	LDPE	HDPE	Polybutene-1	polypropylene
120	0.797		0.806	0.880
130	0.791		0.800	0.872
140	0.785		0.794	0.864
150	0.780	0.780	0.787	0.852
160	0.777	0.777	0.780	0.840
170	0.770	0.770	0.774	0.819
180	0.765	0.765	0.767	0.758
190	0.760	0.760	0.760	0.754
200	0.755	0.755	0.754	0.750
210	0.748	0.748	0.746	0.746
220	0.744	0.744	0.740	0.742
230	0.738	0.738	0.733	0.738
240	0.733	0.733	0.726	0.734
250	0.737	0.727	0.719	0.730

Any inaccuracies in the melt density will propagated proportionally along to the MFR values. Thus a 1% error in the melt density means a 1% accuracy error in the MFR the value.

Programming Method B

This is a Method B setup for use with optical flags, (Small plastic strips with a black area), if you have and encoder based machine skip this section go to the "The Digital Encoder: Use and Calibration Chapter" Page 50.

Method B Flags Setup 1) Start a new program by pressing the EDIT SMART Key. -

The square cursor blinks in the upper left-hand corner of the screen prompting the user to input the number of the program that will be created or edited.

2) Enter a number and ENTER on the numeric keypad.

Now you are editing a new program of number #. You can press the **EDIT** SMART Key to edit the existing program and select a B Method test

3) After pressing EDIT SMART Key, enter run parameters by pressing EDIT again

The instrument is now in the VIEW Program mode starting with METHOD. Press the **EDIT** SMART Key to enter run parameters for the method B experiment. The 2nd line of the VFD display displays the parameter to be edited. The 3rd line lists a group of suggested values. The bottom line of SMART Keys is used to **ENTER**, **SELECT of SCROLL** data input, respectively.

METHOD =

Press the \mathfrak{R} SMART Key (**browse**) until the 3rd line of the display has the B over the **SELECT** SMART Key.

Press the **SELECT** SMART Key to select the method B test. The second line should now read METHOD = B

Press SELECT/ENTER to accept the value and move to the SET POINT.

SETPOINT =

The user may enter a temperature on the numeric keypad and press enter or use the **ENTER**, **SELECT** and **SCROLL** SMART Key keys to choose a typical value.

Press SELECT/ENTER to accept the and move to the DENSITY

DENSITY =

Enter the predetermined Melt Density of your material. Press ENTER.

MELT TIME =

The user may enter a melt time from the numeric keypad and press enter or use the **Enter, Select and Scroll** SMART Key keys to choose a typical value Press **SELECT/ENTER** to accept the and move to the NO OF FLAGS

OF FLAGS =

Method B runs can be made with multiple flags to observe the volumetric rates of materials over different distances. The user must enter the number of flags from the numeric keypad. Press **ENTER** to accept the value and move to the FLAG LENGTH screen.

FLG LENGTH =

Enter or select the length of your flag. Remember, method B is volume, using distance traveled to acquire results.

LOAD =

Enter the LOAD from the SMART Keys or from the numeric keypad. Pressing **ENTER** then enters the QC limits screen.

QC LIMITS =

If **YES** is entered, the user is prompted to enter the HI and LOW QC limits from the numeric keypad. Please reference "Edit a Program" section for descriptions of HI/LOW QC Limits. Pressing **ENTER** then prompts the user to enter the PROGRAM ID.

PROGRAM ID =

Enter the program ID and press **ENTER** when finished. Numeric and alphanumeric values may be input. See "Navigating the Keypad" for more information on alphanumeric input.

To SAVE the program: Press **ESC** twice to back out to the SAVE Program mode and select **SAVE** to save the program under the OLD program name. Change the program name in the EDIT mode.

4) Choosing the Program

Press the **PROGRAM** button. A square block will appear in the upper left corner of the LED display. Enter the number of the program that was just created (#) or the number of the previously entered program of your choice. You can also press **UP** and **DOWN** to browse preprogrammed programs.

The unit will then attempt to latch into the specified temperature. MAKE SURE THE PISTON and DIE are in the unit. The temperature must equilibrate with the assemblies in place. The user must wait until the specified temperature has been achieved to continue with the experiment. When the instrument has latched-in, continue with sample testing:

ASTM recommends two flags, a 1/4" and a 1" Flag. Use the longer 1" flag for higher flow rates (> 10 g/10 min.) and the 1/4" flag for all others. The aim is to have a flag long enough that the error in determining plunger speed is small resulting in precise flow rate measurements. Long flags for slowly flowing material can make for extraordinarily long tests and the material may actually degrade substantially during the test.

Dynisco Polymer Test provides 1/8" and 1/2" Non-ASTM flags in addition to the standard 1/4" and 1" flags to provide more flexibility in the time needed to traverse the flag (B time). Use the following table to aid your selection of the proper flag.

	FI	ag Selection	Table
MFR Min g/10m	MFR Max g/10m	Flag Length cm	Flag travel Time Range minutes
0.15	1.0	.635	29 - 4.
0.15	1.0	.3175	14 - 2
1.0	3.5	.635	4.3 - 1.2
1.0	3.5	1.27	8.7 - 2.5
3.5	10	0.635	1.2 - 0.4
3.5	10	1.27	2.5 - 0.9
10	25	1.27	0.9 - 0.4
10	25	2.54	1.8 - 0.8
25	50	2.54	0.8 - 0.4
50	300	2.54	0.4 - 0.06

From the first two lines 0.15 to -1 g/10 min. expected MFR we can see we have the option of running with a 0.635 cm (1/4") Flag or a 0.3175 cm (1/8") flag. If the flow rate is close to 0.15 the overall test time would be around 29 minutes! If we use the 1/4" flag instead of the 1/8" flag. The experimental run time would be halved.

The Actual Run

How to do it: Step by Step	<u>The standard Dynisco Polymer Test In-house test procedure section in this</u> manual is an abbreviated version of what follows). Press EDIT and verify all entered program data values are correct. Make sure all equipment has
Instrument Prep	 been properly cleaned. Insert piston rod and let the system come to temperature and wait for temperature lock-in. Lock-in has occurred when the display reads "READY". Heat up and lock-in must occur with orifice and piston rod in the heat chamber. During heat up the program may be adjusted and the <i>Operator</i>. and <i>Sample ID</i> can be entered by pressing the ID key.
Loading the Barrel	Remove the piston rod and lay on a cotton cloth. Check to see if the die is at the bottom of the barrel. Fill heat chamber with an appropriate charge or use about 5 grams if nothing is known about the material). Put the sample into the 10 ml beaker. Add about two-thirds of the material into the barrel, using the brown fill funnel. The loading should be done in two increments, the first consisting of approximately 60% of the material. Tamp down the material with

the packing tool using approximately 20 pounds of force. The packing tool can be driven through the fill funnel. If bridging (clogging) of the material occurs in the fill funnel, slide the fill funnel off to one side (without lifting) and use the fill tool to pack the material in the barrel directly. Move the fill funnel back into place. The remaining material should drop down into the barrel. Repeat with the remainder of the material.

Bubbles are in your sample charge if a snapping/popping sound is heard at the extrudate is forced through the dir./orifice. If bubbles appear in the extrudate use less material between tamping down. If bubbles continue the sample may not be sufficiently dry. Place the plunger into the barrel and position the plunger arm directly over the slot on the indexer cover. Seat the guide bushing down into the barrel if it as not already fallen into place. The guide bushing should be moving freely on the plunger. Place the weight on the plunger rod.



Plunger for Method B and Method A/B Measurements (without digital encoder module)

Insert the plastic flag down into the slot on the top right side of the indexer. Next, slide the plastic flag onto the plunger arm "flag post" through the flag hole. *The flag should not touch the sides of the slot*! Twist the plunger as needed to get it to line up with the flag slot and slide the flag on the arm until it is in a vertical position. Immediately press **RUN. RUN** must be pressed as quickly after loading as possible to be compliant with the ASTM 1238 specification. Pressing **RUN** also starts the internal computer program running. This program will start the internal timer and will attempt to print initial test information to a printer or PC if the devices are available. The start of the flag time should begin between 6 and 8 minutes after the test starts or the test does not follow ASTM protocol. Wait for the melt time clock to count down from 360 seconds.

When melt time is over a flag timer appears set at 0.000. When the front edge of the black flag passes the optical eye the timer starts. If the sample extrudes so quickly that it has already hit the front of the flag before the melt time is completed one must take one of the follow actions:

- Add more material
- □ Use a flow plug

Timing Flag and Starting the Run

Method "B" Run 36

- \square Reduce the test load (e.g. 2160 to 225 g)
- Reduce the test temperature (e.g. 230 to 190 C)
- Support the plunger and piston during melt time
- Change to a non-standard die (e.g., 1/2 diameter, 1/2 height)

The first two are typically used. If, on the other hand, the sample extrudate is so slow that one does not reach the flag before 8 minutes has expired one must either:

- Add less material
- Increase the test load (e.g. 2160 to 21,600 g)
- □ Increase the test temperature (e.g. 190 to 230 C)
- Change to a non-standard die (typically larger diameter)

Since Dynisco Polymer Test flags start at the same position as the first scribe mark the amount of sample charge for Method A can also be used here for Method B testing.

NOTE: There is a 1/4" calibration flag that is higher (closer to the hole) on the flag and used only for calibration purposes. Do not use this flag in normal testing.

Cleaning Up Push down on the weight and purge any material remaining in the barrel. Remove the rod by twisting it clockwise to break the seal created by the molten plastic then pull straight up. *Warning: If you pull the rod out too quickly you may cause a suction the pulls the die out along with it.* Wipe the plunger rod with a cotton rag. Remove the die with the die removal tool. Put two patches directly over the barrel about 1/2 way overlapped and, 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. A minimum of two sets of patches are generally needed to clean the barrel properly, however, some materials are harder to clean that others. Repeat the process until used patches come out clean. When the system is clean, put the die and plunger back into the barrel. This allows the plunger and die 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 exact same material. For materials that degrade or are moisture sensitive it is recommend that both the barrel and the die be cleaned completely. To clean the die, use the die removal rod and push the die up from the bottom of the barrel and out the top. Wipe it with the cotton rag and clean it by running the drill bit through it several times. Remove material that collects in the grooves of the drill bit and repeat until the drill passes easily through the die. Scrape the die with the cutting knife if needed to clean the top and bottom faces. For materials that crystallize quickly you can clean the die by first running a drill bit up into the die while it is hot and in the indexer. This will make it much easier to get the cleaning drill bit in when the die is removed and the material starts to solidify.

USER TIP: Experienced users often listen for the double bounce of the die when they drop it back into the barrel to know that the barrel is clean.

Also, while the die is out look down the barrel bore to be sure it is clean (use safety glasses when looking down the barrel!). The inside surface of the barrel is smooth and shiny when completely clean.

If you don't use gloves you will eventually get burned. If you are using a PVC die (D3364 unstable materials) be sure to get the material out of the conical top

section. Standard dies have a flat entrance and exit. 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 0.2 C of the set point you can begin the next test. Loading material will cause a small temperature change even if temperature set point was locked in. 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.

"Flow Ratio" Runs

Flow Rate Ratio test and the
Pneumatic Lift Option

What is it?

Flow rate ratio (FR) is a tests used to obtain two flow rates at two different stresses or loads, of generally a 10:1 ratio. For example, the first might be a 2,160gram weight with the second being a 2,160gram weight. If you divide the high load flow rate by the first, you have flow rate ratio or one number relating the slope of the flow curve.

One example of this test is as follows, tapes are available with a ¹/₄" flag and a 1" flag right after it. The test is started using a low weight. Once the ¹/₄" flag passes the eye, the low weight is removed and the high load is applied or a high load is added. Immediately after the second flag passes, the two flow rates are calculated and the flow rate ratio is computed and printed.

It is strongly suggested that this test be conducted if you have purchased the Pneumatic lift option from DYNISCO POLYMER TEST, Inc. The weights involved are quite heavy and dangerous for an operator to be routinely lifting. The pneumatic lift automatically and safely lifts the weights as needed.

Assess shear thinning behavior of materialWhy do it?Correlation to MWD

You must have a Flow Ratio capable instrument. (Note: Flow Ratio can only be run in one pass in Method B).

In a Flow Ratio test, when the melt time is over, the sample data collection time starts. Flow Ratio programs execute in a Method B mode using **two** optical flags or **two** Digital Encoder flags. Between the first and second flag, the second weight will automatically be lowered by the pneumatic lift system onto the plunger. After the flags have been processed, the instrument will compute and display the two MFR's, two MVI's if selected and the Flow Ratio before proceeding to the next run.

1.) In the **EDIT** mode select RATIO for method. This triggers the computer to perform a new test.

2.) The addition of FLG LENGTH HI and LOAD HI appear in the edit mode. Enter the appropriate values for these parameters.

Flow Rate Ratio - How to Do It

3.) After the LOAD HI prompt, AUTO LOWER appears. If set to YES, the lift system will lower the weight onto the piston after the melt time expires. If set to NO, the lift must be manually told to lower the weight via the keypad.

4.) After the AUTO LOWER prompt, AUTO RAISE appears. If set to YES, the lift system will raise the weight after the run is complete. If set to NO, the lift must be manually told to raise the weight via the keypad.

5.) Proceed with run normally.

"Intrinsic Viscosity" Runs with PET

Calculating PET I.V. from the Melt Indexer

What is it?

This feature allows the Melt Indexer to correlate Melt Flow Rate (g/10 min.) to Intrinsic Viscosity (dl/g). Intrinsic Viscosity (IV, ASTM D3835) is a "wet chemistry" technique used to assess the specific volume of an isolated polymer chain, when dissolved in a good solvent. This value is directly related to the molecular weight of the polymer. Melt viscosity or MFI measurement is another, empirical (non-absolute like I.V.), way to assess molecular weight. Melt Viscosity and Intrinsic Viscosity are related in such a way that IV can be directly calculated from Melt Index values. This is described in greater detail in the applications brief, "Correlating Melt Rheology of PET to Solution Intrinsic Viscosity" by J. Reilly and P. Limbach, available from DYNISCO POLYMER TEST on request.



Why do it?	I.V. has been used to determine molecular weight relying on the key assumptions of isolated flexible linear chains. You can eliminate the hindrances of the expertise, laboratory and solvents required for solution measurements. Improvements in process control are achievable using capillary rheometry methods over solution measurements due to their simplicity and quick turnaround.
How to do it	IV correlation calculations are only enabled when IV is set to ACTIVE in the SETUP mode. IV results are based on an empirical study that included various PET resins. A reference material of known IV is needed to calibrate the melt indexer. You must get an IV value for your material prior to testing.
	Dry PET for 5 hours at 150 °C, use 6 grams material per test, clean machine thoroughly and run at a temperature of 285 °C.
	This option, if purchased (Models 4003/4004 only), enables only at 285 $^{\rm o}{\rm C}$
	1.) Clean the orifice, piston rod and barrel thoroughly. This is very important! Put piston rod back into barrel until temperature has locked.

2.) Enable IV calculations under the Setup on the indexer. Note that a typical Pet test temperature is to 285 $^{\circ}$ C.

3.) Set the following parameters accordingly in EDIT mode:

Acceptable Methods: A, A/B, B Melt time: 360 sec Temperature: 285 C Flag: 2.54 cm Weight: 2160 kg Note: Always perform a purge run of a new sample

Example Settings/Procedures for known MFI's

		Flag Length	Method A Time	Pull Plug
		(cm)	(secs.)	
1.081	8	1.27	60	NA
0.975	10	2.54	40	270
0.950	16	1.27	40	220
0.749	35	2.54	20	30
0.705	43	2.54	20	30
0.662	57	2.54	20	15
0.660	53	2.54	10	40
0.626	73	2.54	10	25
0.556	11	2.54	10	15
0.554	143	2.54	10	0
0.389	180+	2.54	5	0

4.) Perform a sample purge of material. Once done, clean thoroughly.

5.) Loads 6 grams of sample and perform run. Perform the test 3 times to obtain a repeatable MFI and record the value.

6.) Perform a run using parameters in step 3.

7.) After the run, an Intrinsic Viscosity will be displayed and printed.

After each run: Clean Barrel Clean Die (remove first)

Charge Weight

The Amount of Sample

ASTM gives a recommendation of how much material to put in the barrel to perform a test. However, by determining the proper charge and controlling it from run to run. Testing can be made easier and more reproducible. The distance from the top of the die to the first scribe mark is about 5 cm. Filling the barrel up to the first scribe mark is the minimum charge needed to run a test. During the 6 minute melt time some material flows out of the die so a larger charge is needed. The best situation would be if one added just enough material, so that, during the melt time, the plunger slowly falls and is just above the first scribe mark when the 6 minute melt time has expired. In this way the required 6 minute melt time is satisfied and there is no excessive waiting before the first cut is made (or timing flag starts in method B). A conservative estimate for charge weight can be calculated if an approximate melt flow rate and melt density for the material are known by using the following formula:

Charge Mass = 36 0 6 . * MFR

Where the charge mass is in grams, \rangle is the melt density in g/cc and MFR is the melt flow rate in the typical units of g/10 min. This equation will tend to slightly overestimate the charge needed. For a Polypropylene with an expected MFR of 3.5 at 230 C getting the melt density of 0.738 from the above table the estimated charge mass would be:

360*06 35476 = 738

In our lab we found 4.6 grams is a good charge for a 3.5 Polypropylene sample. The equation overestimates the charge since is assumes flow from the instant the material is packed into the barrel. The following table shows an estimate of charge weight in GRAMS based on anticipated MFR (g/10 min.) and melt density (g/cc). Where the word PLUG appears indicates you cannot put enough material into the barrel such that after 6 minutes there would be enough left to test, the barrel must have a flow plug inserted at the base of the die to keep the material from escaping.

Melt Density-> MFR g/10 min.	0.75 g/cc	1.0 g/cc	1.2 g/cc
0.1	2.7	3.6	4.4
0.2	2.8	3.7	4.4
0.5	3.0	3.9	4.6
1	3.3	4.2	4.9
2	3.9	4.8	5.5
3	4.5	5.4	6.1
4	5.1	6.0	6.7
5	5.7	6.6	7.3
6	6.3	7.2	7.9
7	6.9	7.8	8.5
8	Plug	8.4	9.1
9	Plug	9.0	9.7
10	Plug	9.6	10.3
11	Plug	Plug	10.9
12	Plug	Plug	11.5
13	Plug	Plug	Plug

Barrel Charge Size in grams

A-Standard Operating Procedure

Introduction:	Indexer Testing of GRAY polypropylene Control Resin This the procedure Dynisco Polymer Test uses to run a control material to check if the machine is operating properly. It is also used to qualify operators once trained. It can be used as a starting point for a SOP (standard operating procedure) to use within your company if one does not already exists.
	(MFR about 3.6 g/10 minutes, HIMONT PP, use only LOT # 13891) This standard operating procedure is to be used to qualify Dynisco Polymer Test employees in the use of melt indexers. It sets forth the proper operation of the equipment and specific test procedures and methodology to be used by the operator. In an effort to make the results as consistent as possible we have found it necessary to go beyond the standard ASTM D1238 method. Control limits $\pm 3 \int$ of 3.0% have been obtained using this method. NOTE the A/B method should always be used for operator training. Only the A/B method assesses cutting and flag methodology together.
	The Test Procedure for Indexer SQC runs:
	Use 2060 weight.
Instrument Preparation	Use the standard die. (D=0.0825", L=0.315")
•	Use the 1/4" flag
	Set MTD A Time to 180 sec
	All calibrations completed.
	All machine oils removed from barrel and machine is LEVEL.
	Die Dimension Check. (Go, No-Go)
	Die Length Check. (Micrometer, = 8.0 mm or 0.315")
	Die Clean Check, Clean if Dirty.
	Machine should be on for 20 minutes set to 230C, Temp locked.
	Barrel Clean Check, Clean II Dirly while HOT.
	Plunger Clean Check, Guide busining must move neery.
	Tools Poody (Pookor, Die romover, bross brush cleaner)
	Flag Nearby
	Fill Funnel Nearby
	Connect mini-printer or standard printer to indever
	Machine program set as shown on attached sheet. (1/4" Flag Density 0.735)
	Set Operator ID to operator initials and Material ID to GRAY

	At least one purge of gray material performed. (To purge: About 5 grams, wait 2 minutes hand purge) Clean MI barrel after purge (2 patches, double pass, two times). Reinstall plunger and let machine re-lock the temperature. Weigh out 4.6 grams ± 0.1 gram of Gray Polypropylene Lot # 13891 material
Conducting the Test	 Weigh out 4.8 grains ± 0.1 grain of Gray, Polypropytele, Lot # 13891 material. DO THESE AS QUICKLY AS POSSIBLE Pour 2/3 material through funnel into barrel, pack until little movement occurs when pushing on packer. Pour in remainder of material, Pack as before. Wipe plunger before placing into barrel. Seat plunger guide bushing. Place flag on arm & in flag hole. Place weight on plunger. Press RUN button. Make cut and Press RUN button when first scribe reached. Make second cut when MTD A time expired (beep)
	The test should run to completion by itself, enter the mass of extrudate collected over the cut time then press END then YES to print to the mini printer or Okidata 320. Push down on weight hard to purge remaining material out.
Clean Up	Always use two patches (must have enough frictional forces against barrel to clean). Eight or more strokes completely up and down the barrel. Toss patches & repeat. Or use the power cleaning tool and make two cleaning passes with fresh patches each time.
	If the last run of control: Push the die up and out using the orifice removal tool. Hold the die in a glove and run a drill bit through the die. Run MI die brush through the orifice. Clean top and bottom surfaces. Re-clean the barrel to get area blocked by orifice. Leave die out so it can be inspected before next runs. Leave die on cleaning drill bit.
	TURN MACHINE OFF ONLY IF IT IS CLEAN.
Your data	Data will be displayed on the VFD or printed if you have the printer option. Follow the prompts on the VFD to END or SAVE the run.

Out of Control Action List

	Was the instrument clean?
Actions	Check density value
	Check tip diameter (> 0.3727)
	Check Die (clean, diameter length OK?)
	Check Machine is Level
	Check Temp
	Check weights used $+/-0.5\%$
	Check balance used to weigh extrudate
	Piston Rod Bent?
	Check insulator can hitting bottom of barrel?
	Prior to loading Sample
Generic Run Check List	Is material properly prepared (dried, mixed, check for contaminates)
Scherie Run Check List	Machine on for 20 minutes?
	Correct Die in machine?
	Correct program being run?
	Hand tools in position for run(packing funnel and tool, cleaning drill bit etc.)?
	Encoder Arm in place?
	Long term items (in order of importance)
	Are die diameters within spec. (passed G0-No Go gage. ASTM. ISO. DIN?)
	Temperature calibration OK?
	Piston Tip Diameter within spec.?
	Barrel Diameter OK?

Model 4004 and Computer Control

Introduction

The model 4004 Melt Indexer can communicate directly with a personal computer running MIWORKS Software. Features include:

- Direct programming of the instrument via personal computer
- D Point & click interface for program setup and data analysis
- Comprehensive data base type features
- Graphical display of program

Talk to your Dynisco sales person for more information. USA 508-541-9400

The Digital Encoder: Use and Calibration

Introduction	A digital encoder module is available from DYNISCO POLYMER TEST. It permits the collection of multiple data points from one charge of the barrel. It eliminates the need to use the clear plastic "flags" and replaces the electronic eye assembly in the instrument. The digital encoder makes operation of the instrument much faster and easier.
Method B Encoder Option	The Digital Encoder option replaces the optical flag and sensor with highly accurate digital flags and sensors. <i>You can perform experiments using from one digital flag up to fifteen digital flags</i> . The Dynisco Polymer Test Encoder for Method B tests employs a 1016 count optical encoder coupled to high accuracy gears to measure the piston travel. The optical encoder has dual outputs, enabling the signal to be processed in quadrature, yielding a resolution of 0.015 mm. The linear distance of the piston is transferred to rotary displacement via a precisely calibrated arm. The tip of this arm employs a hardened & ground tip for extra long life. Accuracy over the ASTM measurement range of 0.25" and 1.00" is +/-0.4%. The optical encoder, while being very accurate, also enables the test length signal to be processed & varied digitally. Thus, any test length up to 1"(ASTM) or 30 mm (ISO) may be selected. Up to 15 MFR results per barrel filling can be obtained.
Encoder Advantages	 Completely Automatic Test Length Selection on Single MFR Tests Up to 15 MFR readings per Barrel Filling Any test length ("Flag") up to 30 mm User Defined test length & test spacing for single or multiple tests Automatic Test Length & Test Spacing for multiple tests Ten test conditions stored internally

W/h-r	No manual cuts
why use it?	Check how homogeneous a sample is within a charge.
	\Box Get better precision (averages are less variable than a single observations)
	See if barrel pressure drop is significant
	Gain ability to reject data point if bubble or air pocket existed
Digital Encoder Installation	The following parts should be in the encoder upgrade kit or included with the encoder based melt indexer: (if ordered)
	Two 1.25" long #8-32 Socket Head Cap Screws (SHCS)
	Two 5/8" long #8-32 Socket Head Cap Screws
	Two 5/8" long #8-32 Reduced Head Socket Cap Screws
	Six #8 Lock Washers & Four #8 Washers
	One Encoder Housing
	One Long Plate, Plate #1
	One Small Plate, Plate #2

Installation Steps (See Figure 1);

1. Using two 5/8 " Reduced Head Socket Cap Screws & two #8 Lock Washers, attach plates #1 & #2 to plate #3 (plate #3 is already attached to the MI). Tighten the screws.



2. Attach the Encoder Housing to the long plate (plate #1) using two 1.25" long SHCS, two 5/8" long SHCS, 4 washers & 4 lock washers (washers first then lock washers, i.e. lock washers under screw head).

Note for Pneumatic Lift Users: With the encoder arm in the down position, lower the pneumatic lift. Move the arm up and down to check for clearance with the pneumatic lift bucket. Clearance may be increased a small amount by rotating the encoder housing before tightening the screws. If no clearance exists and the arm hits the pneumatic lift bucket, contact Dynisco Polymer Test Systems before proceeding.

Tighten the screws 3. Plug the phone jack cable into the encoder housing.

4. Move the encoder arm into the down position

5. Turn power on

6. Enable the Encoder by pressing the **SETUP** key. Browse the options until you reach **B FLAG** and press **EDIT**. Press → Quick Key so that ENCODR is over the SELECT Quick Key. **SELECT** encoder and press **ENTER** to accept. Press **ESC**. To back out to main menu.

7. Press the **SYS** key and select **TEST**. Browse the menu and select **Digital Encoder Test.** The display shows current position and should change when the arm is moved. Press **QUIT** to end procedure. **ESC** out to main menu.

8. See section entitled for detailed instructions on the variety of uses and programming available with the encoder option.

Programming for use with the Digital Encoder

Method B: Setup	This procedure assumes you are already set up to perform a Method B test as explained in the section entitled " Method B Run ". Make sure Method B is selected in the EDIT mode.
Enable the Digital Encoder	Press the SETUP key and browse the options until you reach B FLAG and press EDIT . Press —> Quick Key so that ENCODR is over the SELECT Quick Key. SELECT encoder and press ENTER to accept. Press ESC . twice to back out to main menu. Enter the EDIT mode.
Auto Flag ON/OFF	Press \longrightarrow Quick Key until AUTO FLAG appears. Select ON to enable the instrument to automatically determine one flag length based on real time flow rates during the actual run. ASTM standard flag lengths will be used. No data entry will be required. Auto flag is the easiest mode, it handles all the flag details, unless you have a detailed test method use this option. Select NO then you must enter more information in the options below about the number of flags and their length. Press ENTER for next prompt.

Flag Calculation MANUAL or AUTO?	Select AUTO here unless you are an MFR wizard. This lets you pick flag length which automatic adjusts the spacing between flags and start positions. In MANUAL MODE the operator enters the start position of each flag. The instrument will not perform any flag boundary or cross over checking. Dynisco Polymer Test recommends a START position of 0.5 mm and a STOP position not to exceed 30.0 mm. In AUTO MODE, the operator enters the data collection START and STOP plunger positions (105 mm - 230 mm). The instrument will then calculate all positions, flag lengths and flag separation lengths (equal to 25% of the flag length). The instrument will perform flag boundary crossover checking. Flag length will be decreased automatically until all the requested numbers of flags fit between the START and STOP positions. Minimum flag length is 1 mm.
AUTO flag selected	Now that AUTO flag has been selected, enter the number of flags (Data points collect for this loading of the barrel) and the flag length, press ENTER. Edit the next parameter or quit edit mode and save program.
So you picked MANUAL	Enter the number of flags in the next prompt, 1 to 15, and press ENTER . Select the length of the flags and press ENTER . The next prompt will say "# ENCODER FLAGS NOT INITIALIZED". This means that you must enter start positions for each flag. Press EDIT an enter start position for flag 1, press NEXT for flag 2 and so on. The instrument will suggest positions for you, but you don't have to accept them. EXIT when finished editing. Dynisco Polymer Test recommends 1 mm minimum flag spaces.
Digital Flag Home	<section-header><section-header><text><text><text><list-item><list-item><list-item></list-item></list-item></list-item></text></text></text></section-header></section-header>
Testing and Maintenance	TEST: Digital Flag Position The instrument allows you to test the position of the encoder arm. This procedure verifies the circuitry is working and can be used to test calibration.
	Test Steps:

	 Press the SYS key and select TEST. Browse the menu and select Digital Encoder Test. The display shows current position and should change when the arm is moved. Press QUIT to end procedure. ESC out to main menu.
	 Maintenance 1) Always place the arm into the lowered and locked position before cleaning and at the end of the day. This prevents damage and ensures that the encoder arm will be in the proper position upon power up. 2). If position gets "lost" during calibration procedure, at the start of the test., go back and perform a "re-home". 3) Never place any severe force against the side of the arm. 4) The real time flow during the test is an approximate value and should not be used as an absolute value. Fractional flows below 1.0 MFI will appear to change by larger steps due to resolution of sample time. 5) The encoder absolute resolution is .015 mm overall accuracy +/-0.0254 mm. 6) Results on multiple flag runs are printed at the end of the test. This differs from our standard Galaxy Series melt indexer.
Complete Digital Encoder Calibration	The Digital Encoder is calibrated at Dynisco prior to shipment. Because the arm is locked into place, calibration is rarely required. If calibration needs to be checked due to suspected encoder damage, then the following steps should be followed <i>if</i> an Encoder Calibrator is available. If an encoder calibrator is not available, and damage to the arm is suspected, then contact Dynisco for assistance.
	Tools Required:
Deferenced Stree D1229 05	 Encoder Calibrator, Dynisco Part Number 7051-59, See Figure 3 Allen wrenches: 1/8", 9/64", 3/32", 5/64" The calibration table (below) applies to ASTM D1238-95.
Referenced Spec D1238-95	Changes made, if any, to ASTM 1238 after 95 are not reflected in the table or drawings.
	Calibration Steps: 1. Turn the instrument on and press <test> and answer <yes> to encoder test. The encoder position is now displayed in millimeters.</yes></test>
	2. Remove piston rod and rotate the stainless steel RTD cover out of the way. Install Encoder Calibrator onto Melt Indexer as shown in Figure 3. Turn the micrometer head

on the calibrator counterclockwise until the MI displays a negative value (example -1.0mm). Slowly turn the micrometer head in the opposite direction while watching the front display. Stop turning the micrometer head when the MI display reads <+0.00>. Record the value of the micrometer head position to the nearest 0.0000". Continue rotating the micrometer head in the same direction until the MI displays 25.40. Again read the micrometer head to the nearest 0.0000".

Caution!: Once the first position is recorded, always turn the micrometer head in the same direction (i.e. clockwise) until the second measurement is recorded. Rotating the micrometer head counterclockwise during a measurement after passing through 0.00mm voids that test! Note!: If the distance used during testing is exclusively 6.35mm, then the micrometer head may be rotated until the MI displays 6.35mm. The difference in the micrometer head readings should be 6.35 +/-0.03mm. If calibration is required, then the method outlined in this procedure for 25.4mm should be followed.

3. Subtract the first micrometer head reading from the second reading in step 2 and multiply the result by 25.4 (example 25.45). Go to Table 1 and determine if calibration is required. The specified limits are +/-0.4% ASTM D1238-95 and +/-0.10mm for DIN 53735. If calibration is required, go to step 4, otherwise stop. *Note!: Repeating this measurement before proceeding with the calibration procedure is recommended*

4. If calibration is required, then the right hand cover on the encoder housing must be removed. Remove the cover from the Encoder Housing using a 3/32" Allen wrench. Loosening the arm set screw shown in Figure 1 (5/64" Allen Wrench). Be sure to loosen the proper set screw, otherwise the entire arm will come loose from the shaft. If the first set screw is accidentally loosened then the left cover must also be removed and the first set screw retightened when the arm is in the full down position when the gear is oriented as shown in Figure 2. The second set screw has been locked with removable Loctite and may be stiff. After loosening the second arm set screw, determine how far the adjustment screw on the arm (see Figure 1) must be turned by using Table 1 & the results from step 3. For example if the difference in the micrometer head readings from step 3 yielded 25.45mm,

then the adjustment screw must be turned -87.6 degrees (negative = ccw rotation as viewed from the rear of the instrument) or about ccw 1/4 of a full turn to bring it back into calibration per ASTM requirements. Use a 1/8" Allen Wrench to turn the adjustment screw.

5. Recheck the encoder calibration by repeating the micrometer measurements in steps 2 & 3. If further adjustment is required then again use Table 1 to determine the appropriate degrees of movement. Repeat this process until the arm is calibrated. Once the arm is calibrated, secure the arm in place by re tightening the second arm set screw (see Figure 1). Apply removable thread locker (Loctite type 290 or equivalent). Reinstall the cover and tighten all hardware.



		Number o	of Degrees	
Micrometer		of Rotatio	n Required	
	Reading When MI		Arm Into	
	Displays 25.4m	nm Calibratio	n	
	dial 25.25	degrees	_turn _n	
	25.26	243		
	25.27	226		
	25.28	208		
25.3:	25.29	191		
ASTM &	25.3	174		
DIN Min	25.31	156		
	25.32	139	1	Positive turn = cw
	25.33	122	5	screw rotation (push
	25.34	104	ć	arm out to make
	25.35	87	I	longer)
	25.36	69		
	25.37	52		
	25.38	35		
	25.39	17		
	25.4	0		
	25.41	- 17		Negative turn = ccw
	25.42	- 35		screw rotation (pull
	25.43	- 52	ć	arm in to make
	25.44	- 69	2	snorter)
	25.45	- 87		
	25.46	- 104		
	25.47	- 122		
	25.48	- 139		
	25.49	- 156		
25.5:	25.5	- 174		
ASTM & DIN Max	25.51	- 191		
	25.52	- 208		
	25.52	- 226		
	25.55	- 243		
	25.54	- 261		
	20.00	201		



ENCODER CALIBRATOR INSTALLATION

Figure 3

Pneumatic Lift Options

Pneumatic Lift

The pneumatic lift accessory is a key piece of hardware to invest in if the flow ratio test needs to be conducted. Also, if your material has a low flow rate and a heavy weight is used, the pneumatic lift system lowers and raises the weight for you.

Flow Ratio provides the user with insight into a materials shear thinning ability and may correlate to molecular weight distribution. This test involves obtaining flow rates at two different shear stresses, of generally a 10:1 ratio. First, the lower mass weight is added to the piston. Next the higher mass weight is added to the piston. The high mass weight may be well over 50 lbs. The pneumatic lift automatically raises and lowers the weight safely.

You must enable the lift system in the SETUP mode, PNEU = ON. When instrument is idle, the lift system is controlled by pressing the lift UP/ DOWN arrows on the keypad.

There are also references to lift operation in the EDIT and SETUP modes. Some notable settings are described:

Auto Purge = At end of test, last flag or cut, drop weight to purge material. **Auto Raise** = If enabled, this option requests that the lift system raise the

weight after the experiment has ended

Auto Raise time = If Auto Raise enabled, wait this long to raise the weight. See following graph

Mini-Lift

This is a small Pneumatic Lift assembly that supports the piston rod itself. It is useful for high-flow materials and can be used in conjunction with a melt plug.

You must enable the Mini Lift system in the SETUP mode, MINI LIFT = ON. When instrument is idle, the lift system is controlled by pressing SHIFT then the lift UP / DOWN arrows on the keypad.

There are references to Mini Lift operation in the EDIT and SETUP modes. Some notable settings are described:

MINI Lift Hold = If enabled, this option requests that the lift system holds the mini lift until the melt time has expired.

HOLD TIME = If MINI Lift Hold enabled, wait this long to lower the MINI Lift after raise time. Intrinsic Viscosity Option

Introduction

Activation and Operation

Activation and Operation

Introduction

Instrument Calibration

RTD Electronics

The instrument must compensate for any offsets occurred in the internal electronic circuitry. A known high precision resistor is inserted, through software, into the RTD conversion electronic circuitry and a converted temperature is displayed. If the temperature deviates from the expected value an offset is computed. Accepting the offset applies it to all future temperature conversions

The RTD electronics are calibrated at Dynisco Polymer Test prior to shipment and really should not be changed.

RTD Sensor

Calibration of the RTD Sensors requires one or two thermometers or equivalent.

The RTD sensor is calibrated at Dynisco Polymer Test prior to shipment. There should be no need to re calibrate the RTD sensor. However, if there is a need, please follow the following procedures.

Calibration Steps:	NOTE: You must have one (SINGLE CAL) or two thermometers or equivalent to perform this calibration.
	1. Press SYS Key and CAL Smart key to enter calibration mode. Select RTD Sensor in the Calibrate Select mode.
SINGLE	2. The instrument prompts you to select a SINGLE or DOUBLE procedure. Each procedure requires a thermometer, or two, rated at the desired calibration temperature. SINGLE uses one temperature reference to perform a calibration accurate to one temperature setting. DOUBLE uses two temperature standards to create a linear curve between the two temperatures. Temperatures falling between the two standards will be accurate.
	1. In the EDIT mode set the temperature on the instrument to the rated temperature of your thermometer and wait for it to lock in. Enter the SYS, CALIB mode and select RTD Sensor SINGLE. Insert the thermometer into the thermometer well. Let the RTD and the thermometer stabilize in the barrel for 15 minutes. Once the temperature has stabilized, press STABLE to proceed.
	2. Enter the thermometers temperature reading accurate to 1/10 degree Celsius.

3. The temperature offset is displayed. The offset is the difference
between the RTD temperature reading and the thermometer's
temperature reading. Select SAVE to use the new temperature offset or
manually enter an offset correction factor.

DOUBLE In the EDIT mode set the temperature on the instrument to the rated temperature of your thermometer and wait for it to lock in. Enter the SYS, CALIB mode and select RTD Sensor DOUBLE. Insert the first thermometer into the thermometer well. Let the RTD and the first thermometer stabilize in the barrel for 15 minutes. Once the temperature has stabilized, press STABLE to proceed.

2. Enter the first thermometers temperature accurate to 1/10 degree Celsius.

3. Set the temperature on the instrument to the rated temperature of the second thermometer and wait for it to lock in. Insert the second thermometer into the thermometer well. Let the RTD and the second thermometer stabilized in the barrel for 15 minutes. Once the temperature has stabilized, press STABLE to proceed.

4. Enter the second thermometers temperature accurate to 1/10 degree.

5. The temperature offset is displayed along with gain. The offset is the difference between the stabilized temperature reading and the thermometer's temperature readings. Select SAVE to use the new temperature offset and gain values or manually enter an offset correction factor and gain value.

Optical Flag

The instrument allows for the adjustment of the Optical Flag sensitivity. The sensitivity determines how sensitive the Optical Flag LED Receiver is to being in either two states, blocked or open. Sensitivity directly affects the measured length of the tape flag.

The Optical sensor is calibrated at Dynisco Polymer Test prior to shipment. There should be no need to re calibrate the RTD sensor. For this procedure you must have the *Flag Calibration Unit*.

 Select Optical Flag in the Calibrate Select mode.
 Make sure FLAG STATUS = OPEN when there is no flag blocking Optical Sensor. Typical sensitivity readings are 50.

3. Place the electronic eye calibrator on top of the instrument and hang a tape with a $\frac{1}{4}$ " flag on its pin. The tape should hang such that it extends down through the slot on the top right hand side of the instrument.

3. Adjust the calibrator to lower the flag down until it just blocks the optical sensor and the instrument display reads FLAG STATUS = CLOSED. Set the calibrator dial to zero. Continue to lower the flag until the flag leaves the sight line of the eye and the instrument display reads FLAG STATUS = OPEN. Note the dial travel of the tape, it should be within +/- 0.002 inches of 0.250 inches.

Calibration Steps:

4. If an adjustment is required, increase SENSITIVITY to lengthen the flag reading or decrease SENSITIVITY to shorten flag length.

5. Press SAVE to permanently save the new value to memory.

Digital Encoder Calibration

The Digital Encoder is calibrated at Dynisco Polymer Test prior to shipment. Because the arm is locked into place, calibration is rarely required.

The instrument does allow you to reset the home position of the encoder. If calibration needs to be checked due to suspected encoder damage, then contact Dynisco Polymer Test for assistance and the necessary tool kit.

Calibration Steps:

1. Press the **SYS** key and select **CAL**. Browse the menu and select **Digital Flag Home.** The instrument prompts you to place the encoder arm in the home position. This is the up most position that the flag arm can go into.

2. Place the encoder arm in the up most position and press **RESET** to set the encoder count to 105.00 mm at the top home position. Press **QUIT** to end procedure. **ESC** out to main menu.

The encoder should now be set to the proper encoder settings. A digital micrometer mounted in a special fixture is available from the factory to check and verify accuracy of the encoder unit. Please contact your sales representative.

Intrinsic Viscosity

Offset The IV offset is the difference between the computed IV and the experimentally obtained IV. If option available, enter the difference here for correlation during IV runs. Please reference previous IV sections in manual

Calibration: Digital Flag Home

Instrument Maintenance and Troubleshooting

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Maintenance

Daily	Remove the orifice and clean thoroughly with brush and drill bit. Clean your indexer barrel with cotton patch when hot. Piston should spin easily when placed into a clean barrel.
Weekly	Give good cleaning with brass brush. Use oven cleaner to clean the piston rod assembly and orifice.
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 orifice and clean thoroughly. Clean your barrel. Use oven cleaner to clean the piston rod assembly and orifice. Clean dust from optical flag sensor.
	Check mechanical tolerances Is your piston tip in spec? diameter = 0.3730 +/0003 length = 0.2500 +/0050 Does assembled piston rod weigh 100 grams? Does your GO/NO GO gage work properly on the orifice? Orifice length O.K.? length = 0.3150 +/0010 Operating temperature within spec? +/- 0.2 degree Celsius Weights still weigh correctly? Sample scale calibrated? Digital Encoder calibration OK?

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 measure 0.3760 +/- .0002 leaving the factory.

Reference the section "Series 4000: A Guided Tour"; Selecting TEST in the SYS Mode" to perform any hardware tests.

Support Vendors1. Pin Gages
Calibration or Certification of Cylindrical Pins
Zero Check
P.O. Box 903
Thomaston, Connecticut 06787
Tel: 203-283-5629
FAX: 203-283-4113

 $\label{eq:metric} \begin{array}{l} \underline{\text{Meyer Gage Company}}\\ 230 \ \text{Burnham St.}\\ \text{South Windsor, CT 06074}\\ (203)\ 528\text{-}6526\\ \text{Ask for Class X pins (ISO specs are \pm 0.0002 of nominal, ASTM specs}\\ (D3835)\ \text{are \pm 0.0003 of nominal)} \end{array}$

2. Cleaning Patches

<u>Skyline Center Inc.</u> P.O. BOX 3064 Clinton, IA 52732 (319) 243-4065 (800) 747-4065 Extension-4065 FAX (319) 243-9901

3. Bore Gages

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

4. NIST Standard Reference Materials (SRM)

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

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

5. Hg spill kits

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

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

Temperature

Conversion Factors

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

Viscosity

To Convert From		ltiply By
Poise	Pa-sec	0.10
centi-Poise	Pa-sec	0.001
centi-Stokes	m ² /sec	1e-6
lbsec/ft2	Pa-sec	4.788026E+01

Pressure or Stress

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

Troubleshooting

Flow Rates are Changing

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. **Remove** the orifice to clean it. Next, the units' component tolerances and operational components should be checked:

- □ The barrel **temperature** should be checked with the supplied thermometer. $(+/-0.2^{\circ} \text{ C})$
- □ The **orifice** should be checked for wear with the supplied GO-NO GO gauge. (0.0825 +/-0.0002 in.)

	□ The piston tip should be checked for correct tolerances. (MI 0.3730 +/- 0.003 in.)
	□ The guide bushing should be clean of material and easily slide up and down the piston rod.
	□ If running method B tests, are the factory supplied flags in good condition.
	The machine should be on a level surface. (Vertical alignment of the
	The precision and accuracy of the test and instrument has been standardized by ASTM D1238. Dynisco Polymer Test has found that the contributing factors to the accuracy of the test 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), cleaning and cutting technique. It is recommended to follow ASTM Specification D1238 for your material or control sample. Also, pay attention to the recommendations of the manufacturer or suppliers.
	After a good cleaning and purge, your control sample should be run a minimum of 5 times to verify that the problem is repeatable or even exists. If you feel it is necessary to call the factory, please have your model number, serial number and a complete program listing available (it may be helpful to fax the program data) when you call.
Temperature is Unstable	Unplug the unit and remove the front cover. Verify all cables and circuit cards are properly connected.
	While the cover is off and the unit is unplugged, clean out all debris from the unit's internal components with compressed air. Clean all debris from the units cooling fan. Air must flow freely to properly cool the unit.
Mercury Separation in Thermometer	Before using any thermometer it should be examined very carefully for mercury separation in the main mercury column, expansion chamber, contraction chamber and bulb. Mercury separation in the bulb will usually show as small bubbles. All the mercury must be united. If a thermometer does not read zero at the ice point mercury separation is typically the cause. There is no known method to completely insure that the mercury will not separate in a thermometer when the thermometer is subjected to shock. This can occur either in transit or by improper storage and handling. On the following procedure describes a procedure for reuniting a separated mercury column. Remember that the thermometer contains only two fluids, mercury and gas. The object is to get all the liquid below the gas or conversely all the gas above the liquid
Reuniting Mercury Thermometers	This method is the easiest to use and is the method PRINCO recommends. In a small Dewar flask or thermos bottle mix powdered dry ice with Methanol or Acetone. Holding the thermometer vertical, immerse about 3/4 of the lower section of the bulb into the mixture. <u>DO NOT</u> immerse the capillary or funnel section that is above the bulb into the mixture. The main portion of the mercury will retreat into the bulb, and the separated portion will follow.
	Occasionally the separated portion may cling to the walls of the funnel portion of the bulb. When all the mercury, including the separated portion, has retreated into the bulb, remove the thermometer from the dry ice mixture. (See Figure 1). The mercury should go together. Stand the thermometer in a vertical

position and allow the mercury to rise into the capillary of its own accord. <u>DO</u> <u>NOT TOUCH THE BULB WITH YOUR HANDS!</u>

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

<u>NOTE:</u> Where possible, thermometers should be stored in a vertical position.

Pneumatic Lift Troubleshooting

1.) The pneumatic lift system requires 60 to 80 psi for proper

operation. Please verify that your shop air meets these requirements.

2.) Clean out the in-line water filter.

3.) Verify that the unit is level and that the guide rod is not bent, see fig. C.

4.) Confirm that the hold valve switch is in the HOLD OFF position. See figure A.

5.) If there is still no response please follow these procedures:

a.) Verify that the solenoid is getting the proper voltage. See fig. B. Remove the solenoid electrical connector with a regular screwdriver. Remove housing. Connect one lead of the DC voltmeter to chassis ground and the other lead to either solenoid connectors. Enter the solenoid cycle in the SYS, TEST mode. Measure the DC voltage to each connection. Voltages should be a constant +4 to +5 volts or greater on one connector and should switch from +4 to +5 volts (UP) to less

than +1 volt (DOWN) on the other connector. If these conditions are not met, call Dynisco Polymer Test Systems tech support. If voltages are OK, proceed.

b.) Reassemble the solenoid and reenter the solenoid cycle. While the lift is cycling, listen for the manifold to open and close; it's usually pretty noisy. If it does not open and close, then listen if the solenoid clicks. If the Solenoid clicks then the manifold is probably bad, if the solenoid does not click, then the solenoid is probably bad. Call Dynisco Polymer Test tech support to order replacement parts.

The guide rod and the pneumatic cylinder rod can be lubricated. Dynisco Polymer Test suggests that WD-40 or machining oil be used as a lubricant. You can also spray a small amount of WD-40 into the manifold to lubricate all internal parts.



Pneumatic Lift maintenance



Questions and Answers

Typical Problems	PROBLEM : Unit will not power up SOLUTION : Check the fuse in the input power module, where the power cable connects to the instrument. The fuse is rated at 5 Amperes 120 Volts. Verify the supply voltage is correct (e.g. 110 VAC vs. 220 VAC). If you have any difficulty, please call Dynisco Polymer Test Technical Support.
	PROBLEM : The display works but the instruments keyboard does not respond. SOLUTION : The keyboards internal ribbon cable has become detached due to rough handling during shipment. Remove the top cover, swing open front display/keyboard panel mount. Locate keyboard ribbon cable coming through bottom center of the display/keyboard panel mount. Reconnect to the right side connector (J2) on the mother board. If you have any difficulty, please call Dynisco Polymer Test Technical Support.
	 PROBLEM: The digital temperature readout fluctuates, blinks or jumps around: SOLUTION: The temperature of the barrel cannot physically change temperature as fast as 1 degree C per second. If it does, usually a bad electrical connection or failing electrical component can be blamed. Make sure the die and piston rod are in the unit when heating. If you have any difficulty, please call Dynisco Polymer Test Technical Support.
	PROBLEM : The VFD on the instrument displays a negative temperature. SOLUTION : The RTD is in a shorting condition. Perform an RTD Display test. If the reading is abnormal call Dynisco Polymer Test Technical Support.
	PROBLEM : Temperature is stable but not reaching set point. SOLUTION : Heat the instrument with the die and piston rod assembly in the sample chamber. If you have any difficulty, please call Dynisco Polymer Test Technical Support.
	PROBLEM : The keyboard works but the instruments display does not work. SOLUTION : The VFD's internal ribbon cable has become detached due to rough handling during shipment. Remove the top cover, swing open front display/keyboard panel mount. Locate VFD ribbon cable. Reconnect to the left side connector (J1) on the mother board. Check connection to the VFD itself. If you have any difficulty, please call Dynisco Polymer Test Technical Support.

PROBLEM: How do I check the temperature?

SOLUTION: Enter a temperature value in the edit mode, preferable one that matches your thermometer. **Make sure the orifice and piston rod are in the barrel.** Allow the instrument to lock into temperature and equilibrate for 15 minutes. Remove the thermometer chimney set screw located on the top of the barrel. Insert thermometer and let stabilize for 10 min.. If display does not agree with thermometer perform temperature calibration.

PROBLEM: My digital Encoder is not responding! What's going on? **SOLUTION:** Make sure the encoder is enabled in the SETUP mode. Verify all cables are plugged in at base of Encoder housing and inside the Indexer.

PROBLEM: My method B numbers are wrong, even after inputting my manufacturer's values for apparent melt density! Why? **SOLUTION:** The value reported by the manufacturer is probably a solid state density, not the melt density. Run an A/B method experiment to acquire the proper melt density.

PROBLEM: My MFI is changing!

SOLUTION: Your MFI may vary as much as 2% due to operator inconsistencies, even the smallest things may affect MFI. The most common cause is not keeping the barrel and orifice properly cleaned. We recommend the power cleaning kit (PN 8052-97K) to help guarantee and promote a clean system. Also, make sure sample weight is consistent throughout tests.

PROBLEM: What flag do I use?

SOLUTION: Please refer to the section "Choosing a Timing Flag" in the manual.

PROBLEM: How long should my cut time be for a Method A run? **SOLUTION:** Dynisco Polymer Test suggests that you collect about 1 gram of sample extrudate, or more, before making your 2nd cut.

PROBLEM: My printer does not work! What gives? **SOLUTION:** Enable the printer in the SETUP mode. Make sure it's an online **PROBLEM:** My materials Melt Flow Rate is much higher than normal? **SOLUTION:** If the material is moisture sensitive, dry it. Moisture can make Flow Rates increase greatly, especially on Polyesters (PET, PBT), Nylons, Polysulfones and material with filler content (glass, calcium carbonate, and talc).

Typical Questions ANS

QUESTION: How often should I test my material?

ANSWER: This depends on how much material your plant receives and the method that it is received, a suggested guideline follows:

- Material supplied by tankers (3 tests)
- Material supplied in bags on pallets (3 tests per pallet, check lot numbers)
- Material supplied in individual bags (3 tests per bag)

Also conduct a test if there is a production problem

QUESTION: My material has a Certificate of Conformity and my results don't agree.

ANSWER: A Certificate of Conformity is based on only one test, usually. As a polymer may vary throughout the process, you cannot be sure that the polymer is the same throughout your delivery. It has been known for processors to have problems during production and for them to think that they are machine problems, when in fact it is their material that is out of specification.

QUESTION: How long should a test take from start to finish?

ANSWER: This depends, to a large extent, on the MFI of the material, but roughly the times are as follows:

- Loading/Weighing sample (2 minutes)
- Melting time (6 minutes)
- Testing time MFI > 8 (1-2 minutes)
- Testing time MFI 3 8 (3-6 minutes)
- Testing time MFI < 3 (6-10 minutes)
- Cleanup (2 minutes)
- Heat Stabilization (2 minutes)

QUESTION: What form should my sample be in?

ANSWER: For ease of loading, granules are best. Regrinds, plastic films, powders and liquids have all been successfully tested. Watch for air entrapment with some forms.

QUESTION: What type of sample preparation should I perform? **ANSWER:** Some type of drying conditions may need to be carried out before testing. Preferably, this should be done at the same conditions your production material goes through. LDPE, HDPE and PP therefore will not need any significant drying.

QUESTION: Should I check the MFI on my finished product? **ANSWER:** Sure! You want to know if what you made yesterday is the same as what you made today. If it's the proper quality and did it change? You also want to make sure that the performance of your process and end product are consistent day to day.

QUESTION: What are the lowest MFI and highest MFI we can test? **ANSWER:** Typically down to 0.1 gm/ 10 mins. Any values lower increase your weight or temperature.

Typically the highest would be 500 gm/ 10 mins. however it is possible to go up to 1500 gm/ 10 mins. Tests above 50 gm/ 10 mins. are uncommon and do require skilled operators.

QUESTION: How long does the die last?

ANSWER: The die is made of tungsten carbide which is extremely wear resistant. The die should last for several years if cleaned and maintained properly. Some manufacturers use stainless steel dies which last 2 years.

QUESTION: What should the barrel be cleaned with?

ANSWER: With the tools and cotton patches provided. There should be no oils on the patches. You can heat the machine to its highest temperature to burn off any residue. You really shouldn't use any solvents to clean the barrel due to fire and health hazards. Check with your company safety manager what the policy is.

QUESTION: Can we leave the machine on 24 hours a day?

ANSWER: Yes, in fact many of our instruments have been on for several years. Some customers use a timer to turn their instrument on before they arrive for work, and off after they have left.

Sometimes lightning storms can damage the instrument. As with all accessories that are plugged into wall outlets, a surge protector is a great idea.

QUESTION: What spare parts should I keep?

ANSWER: This depends on how mission critical your operation is. You should have spare fuses and maybe a piston tip and die. A spare parts list should be supplied with the quote.

Glossary of Terms

Digital Flag, Digital Encoder	A device used to measure piston travel		
Firmware	Software embedded into the instrument's hardware. Sometimes you may receive firmware upgrades		
Flow rate	A measure of polymer's mass flow rate (grams extruded in 10 minutes) using a particular orifice under specified conditions of temperature and load		
Flow Rate Ratio	A test used to obtain two different flow rates at two different shear stresses. This may provide insight to the slope of a flow curve.		
Melt Flow Indexer	A measure of the capacity of a polymer molecule to enhance viscosity.		
Method A	An instrument used to perform ASTM D-1238 testing of molten plastics and other polymers		
Method A/B	Measurement of the amount of extrudate accumulated for a given amount of time. The test is completely manual and is sometimes called the "cut-n-weigh" method		
Method B	This test measures the amount of material extruded over time and a volume extruded over a distance. A means of measuring travel is employed. In order to run a method B test, an A/B run MUST be run first to determine the polymer's melt density.		
MVI	Measurement of the volume of material that extrudes over time. Can be conducted only after an A/B experiment is conducted. This is a "no-cuts," automated test that is the most convenient for busy laboratories.		
Optical Flag or Optical Eye Sensor	Material is forced through an orifice at a specific temperature using a specific weight. The volume extruded in a 10minute period is an MVI, in cm/10 min. A component used to measure the amount of time it takes the piston rod to travel a certain distance.		
Pneumatic Lift			
RTD	An optional system allowing the user to automatically raise and lower weights onto the piston rod. Requires 70psi shop air.		
Timing Flag	(Resistance Temperature Detector) A component that senses temperature and changes in temperature. A device that the optical eye uses to measure distance and time		

USB Setup

PC and Daisy Instructions

LMI's with USB ports will need the COM Port that is automatically assigned by a PC, when an USB device is plugged in to it, set to match a COM Port in the Daisy configuration file. This can be done through the PC as follows:

Daisy Software Start-Up

Verify USB port assigned to LMI:

- 1) Power-up LMI with USB connected to PC
- 2) On PC, go to Control Panel/System/Hardware/Device Manager/Ports(Com and LPT)



- 3) Note Comm port of "ATEN USB to Serial Bridge"
- 4) Go to PC and open (with WordPad or NotePad) C:\ "Device1.cfg"

5) Scroll down to line, "RS232='X', 19200,N,8,1" and if necessary, change parameter 'X' to match the USB port assignment noted from Device Manager in step 3.

🖡 DEVICE1.CFG - Notepad 📃 🗖 🔰	
File Edit Format View Help	
$\backslash\!\!\backslash$ A Command is sent with the Unlock keyboard with a passcode of ' $\!$	
MENULINE=Dynisco Melt Indexer MENUCAT=MeltIndexer MENUCOM=RS232 MACHINEID=DynisMI	
********** The next items may be modified to overrule default settings. RS232 is in the format: ComPort, BaudRate, Parity, DataBits, StopBits RESPONSETIME is the maximum time for the device to respond to a command. DELAYTIME is the maximum time between two characters sent by the	111
// If the limits module detects an out of range situation at the er	
<mark>R5232=4</mark> ,19200,N,8,1 LIMITSALARM=2000 \\ Herz (0 = No alarm) PRINTDEVICE=EPSONPRN PLOTDEVICE=EPSONPLT	
PortNumber=1	
ShowEndSeriesMessage=TRUE	
TemplatePath=C:\LMI\Template\ DebugPath=C:\LMI\Debug\	
TESTDUMP=FALSE RESULTDUMP=FALSE NOTESDUMP=FALSE	-

6) Save Device1.CFG if COM Port was changed.

To Verify Communications:

7) Start LMI software by opening "Daisy for Melt Indexer" from desktop or through C:\Program Files\

Username: alpha Password: alpha



8) Either of the two windows below may open. If this first window does not open, go to "Daisy 8.50 Eclipse" window (the second below) and press upper, left button. If the first window fails to open, check communications to instrument.



PC and MI Works Instructions

LMI's with USB ports will need the COM Port that is automatically assigned by a PC, when an USB device is plugged in to it, set to match a COM Port choice within MI Works. This can be done through the PC as follows:

Verify USB port assigned to LMI

- 1) Power-up LMI and PC with USB connected from LMI to PC
- 2) On PC, go to Control Panel/System/Hardware/Device Manager/Ports(Com and LPT)



- 3) Note COM port of "ATEN USB to Serial Bridge"
- 4) If it COM1 or COM2, then skip ahead to the MI Works communications settings in step 8. If it is not COM1 or COM2, then you will need to change it to either COM1 or COM2 but COM2 would be preferred. COM2 most likely will not conflict with a hardware, 9-Pin, serial port on the PC. If the "ATEN USB to Serial Bridge" does not show in the "Ports" section or has errors, a driver may need to be installed. The driver for the PC's operating system can be found at:

http://www.aten.com/products/productItem.php?pcid=20050107104554001&psid=20050117102915002&pid=2005022316346005

At this webpage, select "Resources" and then "Software & Driver" to download. Install driver and then repeat from step 1.

5) Right click on "ATEN USB to Serial Bridge" and select Properties.

Communie	ations Port (CO	12) Properties	? ×
General	Port Settings Dri	ver Details Resources	
Į	Communications F	Port (COM2)	
	Device type:	Ports (COM & LPT)	
	Manufacturer:	(Standard port types)	
	Location:	on Intel(R) 82801DB LPC Interface Control	er -
Devic This If you start	e status device is working p u are having probler the troubleshooter.	roperly. ns with this device, click Troubleshoot to Troubleshoot]
	usage:		_
Use th	is device (enable)		
		OK Car	ncel

6) Select "Port Settings"

Communications Port (COM2) Properties	? ×
General Port Settings Driver Details Resources	
	- 1
Bits per second: 9600	
Data bits: 8	
Parity: None	
Stop bits: 1	
Flow control: None	
Advanced Restore Default	s

7) Select "Advanced". In "Advanced Settings", select "COM2" (or COM1), then "OK" to "Advanced Settings, then "OK" to "Communications Port Properties". Then, close Device Manager.

Advanced Settings for COM2							<u>?</u> ×
Use FIFO buffers (requ Select lower settings to Select higher settings fo	ires 16550 co correct conn or faster perfo	ompatible UAR1 ection problem rmance.	r) s.				OK Cancel
Receive Buffer: Low (1)				—Į	High (14)	(14)	Defaults
Transmit Buffer: Low (1)	1			—Ţ	High (16)	(16)	
COM Port Number: COM2	•						

8) Start MI Works and the following window should appear:



9) Select "Communications" and the following window should appear. Select the COM port that was assigned the USB port in step 7 in the "Select PC Port" section of the window. MI Works should now function with the LMI.

Active Charge #: 31 lbl_update		<u>I</u> . <u>C</u> lose
bl_Incoming		
— Line Memo Opened — Anyone there? Msg Sent: 153 3:05:43 PM	 Message to Melt Indexer #153 #13 0#13 Edit1 Show Raw Input Log to Disk Active Msgs 0 Sent/Acked 0 Channel 1 Set Comm IDs Purge all Msg 	Clear Clear Toggle Status Select PC Port © Comm 1 © Comm 2 Handshaking © ON (Best) © OFF Connection © Direct © Porter Box
4	▼ ▶	

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