Dynisco LMI5000 Series Melt Indexer

Setting the Standard for Evaluating Polymer Melt Flow Characteristics

Operating Manual

Dynisco

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www.dynisco.com

SUPPORT CONTACT INFORMATION

Dynisco Polymer Test can be contacted for questions and support at:

By Phone and Fax:
Phone +1 508 541 9400
Fax +1 508 541 6206

To help us handle your questions as quickly as possible, have the following items ready before you call:

1. Instrument name and model number (on back panel)
2. Instrument serial number (on back panel)
3. Current version of instrument firmware (Power up unit to see, version shown on “About” screen under “Maintenance Tab”)
4. Computer system make and operating system and version (if applicable)
5. Current version of LaVA Suite software (if applicable)

By Internet:
For Technical and Service Support: http://www.dynisco.com/polytest-services
For all Other Inquiries: http://www.dynisco.com/

By Mail/Post:
Dynisco
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Document History

Document Number	Date	Comments
M0726 (0)	December 2012	Original edition; initial release
M0726 (1)	May 2103	Updated, Product Launch Release
M0726 (2)	Feb 2014	Updated for Versions 1.9 and 2.0 Internal Firmware, Improved Wording
M0726 (3)	Oct 2014	Added Lift, Packer and Encoder Appendices, Updated for 2.13 Internal Firmware
M0726	Jan 20, 2020	Updated ECO#, Rev., Footer

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SAFETY INSTRUCTIONS

All safety instructions must be understood and observed. Non-observance of safety instructions may cause damage to life and health of persons, environmental damage and/or extensive damage to property.

Observing the safety instructions included in the operating instructions will help to avoid dangers, to operate the product profitably and to secure the full use of the product.

Warnings, Danger and Informational Symbols

General safety instructions concerning the activities are given at the beginning of the relevant chapter. Special safety instructions concerning the individual steps of action will be given together with the corresponding step of action.

The following pictographs are used in the present operating instructions

 Danger	• This symbol indicates that death, **serious** bodily injuries or **considerable** damage to property **will** occur if the corresponding safety measures are not observed!
 Warning	• This symbol indicates that death, **serious** bodily injuries or **considerable** damage to property **may** occur if the corresponding safety measures are not observed!
 Caution	• This symbol indicates that **minor** bodily injuries or **minor** damage to property **may** occur if the corresponding safety measures are not observed!
	• This symbol indicates that special danger to the life and health of persons is present due to electric potential!
	• This symbol indicates that special danger to the life and health of persons is present due to hot surfaces!
	• This symbol indicates that sufficient personal protective equipment must be worn when working with the LMFI. The type of personal protective equipment will be defined in detail!
	• This symbol signalises user hints and other special information, which may be useful. This symbol does not signalize safety instructions!

Please also note that a safety symbol may never substitute the text of a safety instruction - therefore, the text of a safety instruction must always be read completely!

Safety summary

The following are recommended safety precautions unrelated to any specific procedures in this manual and therefore do not appear elsewhere. Personnel must understand and apply them as appropriate during all phases of operation and maintenance. IN ALL CASES, BE PRUDENT.

- **Keep away from live circuits**

	Do not replace components or make adjustments inside equipment with power turned on. To avoid injuries, always remove power source and discharge and ground a circuit before touching it. When making electrical connections, the services of a qualified electrician must be employed. Contact with live electrical circuits can cause serious personal injury or death. Be sure no circuits are energized during installation, connection or removal of any electrical cables or lines.

- **Wear protective clothing**

	Wear protective clothing (gloves, apron, goggles, etc.) approved for the materials and tools being used.

- **Provide adequate ventilation**

 Warning	Provide ventilation to remove heat and noxious odors and to prevent the accumulation of asphyxiates such as nitrogen gas.

- **Avoid hot surfaces**

	Keep hands away from hot surfaces and materials. Contact with hot surfaces or materials can cause blistering and third degree burns. Wear approved, clean, thermally insulated gloves when handling these components. Should injury occur; immerse injured area in cold water and get immediate medical attention.

Use gloves--it's very 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 any barrel temperature, these should not cause burns if touched for a very brief period except very near the barrel top and bottom. 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 hot piston rod and hot die in the chamber/barrel; this precludes someone picking it up inadvertently. Be sure to hold the piston by the top insulator.

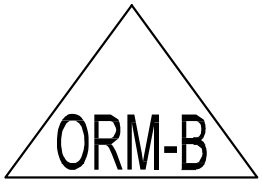
Electrical Hazard

Your Dynisco Lab Melt Flow Indexer contains high voltage inside the housing. DO NOT remove the housing or any part of its outer covers; there are no user serviceable parts inside. Service should only be done by a qualified DYNISCO Service Technician. Be sure the outlet used to power the indexer is properly grounded.

Calibration Thermometers may use Mercury

To calibrate the temperature on the indexer, a thermometer containing about 8 grams of mercury may be 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 on the Internet. 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 test weights (over 10 kg) the pneumatic lift system is recommended. The lift system has a mechanical capture rod which will not allow the weights to "fall" out of the machine. When the machine is in operation the lift system moves the weight downward somewhat quickly creating an area where anything lying beneath could be crushed. The lift system has a safety door that, when installed correctly and not tampered with, will prevent the lift from moving when open. There are redundant internal firmware and mechanical hardware safeties in place in the lift system.

Fumes from Materials

Please plan for unexpected issues to arise. 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.

Obligation of the operator to exercise due care

The LMFI was designed and constructed taking into consideration a hazard analysis having carefully adhering to the harmonised safety standards as well as further technical specifications. Thus, the LMFI conforms to the state of the art technology and ensures a maximum of safety.

In practical operation, this safety can, however, be achieved only if all necessary measures are taken. The obligation of the plant operator to exercise due care includes planning these measures and supervising their execution.

Especially, the operator has to ensure that:

1. The LMFI will be used only in accordance with the intended purpose.
2. The LMFI will be operated in a flawless, functionally efficient condition and that, in particular, the functional efficiency of the safety devices will be checked at regular intervals.
3. No modifications will be made to the component except by a Dynisco service person.
4. The necessary personal protective equipment for the operation, maintenance, and service will be available and used by any personnel performing these functions.

5. The operating instructions are always available completely and fully legible at the installation location of the LMFI. It must be guaranteed that all persons who work with the LMFI can consult the operating instructions at any time.
6. Only sufficiently qualified and authorized personnel will operate, maintain, and repair the LMFI.
7. All safety and warning labels, attached to the LMFI, must not be removed and must remain fully legible.
8. Additional instructions, in accordance with facility, local, state and federal industrial safety regulations and their corresponding, related ordinances for the use of work materials, will have to be made available as a supplement to the operating instructions.

INTRODUCTION

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 performed are those described by ASTM D1238 and ISO 1133-1. This manual in no way supersedes either of these documents. The precision and accuracy of the test has been determined by ASTM method D1238. 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.

Several methods have been developed for running flow rate tests under D1238, with Methods A (mass of material over time) and B (volume of material over time) being the basic of all methods. Method A is simply the collection of extrudate over time, while Methods B, C and D are the measurement of time for the flow of a fixed volume of polymer. All but Method A require an encoder to measure distance traveled to determine volume of material extruded during the test.

Method A	**Method A** -- the test is completely manual and is sometimes called the "cut-and-weigh" method. Measurement in g/10min.
Method A/B	**Method A/B** -- this employs a digital encoder. In order to run a method B test, an A/B test MUST be run first to determine the polymer's melt density. This test takes both A-type and B-type results to determine melt density. Measurement in g/cc.
Method B	**Method B** can be conducted only if material melt density is known as is determined by an A/B type test. This is a "no-cuts" test that is the most convenient for busy laboratories. Measurement in cc/10min.
Method C	**Method C** can be conducted only if material melt density is known as is determined by an A/B type test. This is also known as the "half die" method. A half-length die is used and is generally used for testing high flow rate polymers. This is a "no-cuts" test that is the most convenient for busy laboratories. Measurement in cc/10min.
Method D	**Method D** can be conducted only if material melt density is known as is determined by an A/B type test. This is also known as a FRR (Flow Rate Ratio) test. It uses two weights with the same charge of material to determine the materials FRR. The FRR is the ratio of the average flow rate of the material under higher load to the average flow rate of the material under lower load. This is a "no-cuts" test that is the most convenient for busy laboratories. Measurement is unit-less ratio.

SPECIFICATIONS

UTILITIES:
Electrical Requirements: 100-120 VAC / 220-230 VAC, 6A/4A-Peak at Power-up, 5A/2.5A, 500VA-normal operational power, 50 Hz / 60 Hz

PNEUMATICS:
Lift Option (PSI/Bar): MIN: 60/4.2 MAX: 80/5.5
Packer Option (PSI/Bar): MIN: 20/1.4 MAX: 50/3.5

DIMENSIONS:

	Base model	With Lift System (no weights installed)
Height (in/cm)	20/51	51/132
Width (in/cm)	13/33	13/33
Depth (in/cm)	25/64	25/64
Weight (lb/kg)	47/21.4	100/45.5

Instrument with weight system could have up to 70lbs/31.6Kg weights installed (shipped uninstalled with unit).

OTHER:

Complies with ISO ASTM D1238 and ISO 1133-1

INSTRUMENT MAINTENANCE RECOMMENDATIONS

Daily: Remove the orifice and clean thoroughly with brush and precision drill bit. Clean your indexer barrel with cotton patches 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 exposed metal 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 recommended that a repetitive maintenance schedule be created for your instrument.

Clean unit: Clean the dust and dirt out of the electronics module with clean air every six months to one year, more often in dirty environments. Perform this with power removed from the system. Remove the orifice and clean thoroughly. Clean your barrel. Use oven cleaner to clean the piston rod assembly and orifice.

Check Mechanical Tolerances: All dimensions and tolerances per ASTM D1238 and ISO 1133-1. Piston tip diameter = 0.3730”/9.474mm +/-0.0003”/0.0076mm; piston tip length = 0.2500”/6.35mm +/- 0.0050”/0.127mm. Assembled piston rod weighs 100 grams. GO/NO-GO gage works properly on the orifice. Orifice length = 0.3150”/8mm +/- 0.0010”/0.0254mm. Weights still weigh correctly. Digital Encoder calibration. Gages can be purchased from Dynisco Polymer Test.

Barrel Diameter: Once the barrel is extremely clean, all dimensional measurements are to be made at room temperature it can be checked using a bore gage. Gage can be purchased from Dynisco Polymer Test. At the time of manufacture, the barrel center bore measures 0.3760”/9.55mm +/- 0.0002”/0.00508mm. All dimensions and tolerances per ASTM D1238 and ISO 1133-1.

Pneumatic Lift maintenance: 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 air manifold through the manifold’s air intake to lubricate all internal parts.

Ask for help: Call Dynisco Polymer Test directly at (508) 541-9400 or visit http://www.dynisco.com/polytest-services and ask for technical support. To help us handle your questions as quickly as possible, have the following items ready before you call:

1. Instrument name and model number (on back panel)
2. Instrument serial number (on back panel)
3. Current version of instrument firmware (Power up unit to see, version shown on “About” screen under “Maintenance Tab”)
4. Computer system make and model (if applicable)
5. Current version of LaVA Suite software (if applicable)

Equipment Setup

Unpacking the Indexer

The Lab Melt Flow Indexer comes in a heavy duty, wood container. First, remove the container's lid—a bit tool should be within the crate packing documents on the outside of the container that can be inserted into an electric type drill to remove the lid and bracing screws. Several boxes may be packed inside along with the instrument; remove them and check that all boxes are received. For example, they are coded 1 of 5 or 3 of 5, indicating the total number is five.

For non-weight lift model instruments, the instrument is braced into the box with test weights and possibly accessories under the instrument in a foam pattern. Remove the instrument's bracing. Minding that a base unit weights 45lbs/21.4Kg, either lift the unit out from the top of the container with two people or carefully lay the container on its side and slide the instrument out of the container.

For weight lift models, the instrument is braced into the box with test weights and accessories in separate, internal boxes removed from above step. Noting which side is the bottom of the instrument and leaving the cross bracing in place, stand crate upward with the bottom of the instrument downward. Remove the cross bracing and slide the instrument carefully out of the container onto the floor or a lift. Use a minimum of 3 personnel for this operation for lifting and stabilizing the instrument as it is being moved. Note the shipping weight of the instrument with lift is 100lbs/45.45Kg.

It is recommended that the shipping container be saved a few days until you are certain the machine is functioning as expected.

Bench Requirements and Placement

Typical laboratory benches are too high for efficient use of the indexers. Cleaning and material packing 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 front edge of the table. This will prevent the operator from having to bend forward excessively when cleaning and packing 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. 300lbs/136.36Kg).

DYNISCO POLYMER TEST recommends placing from left to right, if using these options, the melt indexer; sample scale and computer. Shake test the melt indexer for stability. The bench top should also be able to withstand hot dies and tools being dropped on them. If carpet is present on the floor near the LMFI, carpet protection is necessary since a hot die, hot material or hot piston dropped on the 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 will want 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 obtaining accurate data must also be addressed before valid testing can begin.

Level the Melt Indexer

Using a small round bubble level, 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 may choose to bolt the machines directly to their benches.

This can be done by removing the levelling feet and using their mounting holes to mount the instrument to a bench or table. Use caution and remove power before doing so. Mounting this way will entail removing some covers for internal access to the internal, main instrumentplate.

Be sure to remove the level before turning on the machine. The level will be damaged if it gets hot.

Instrument Overview

Front View

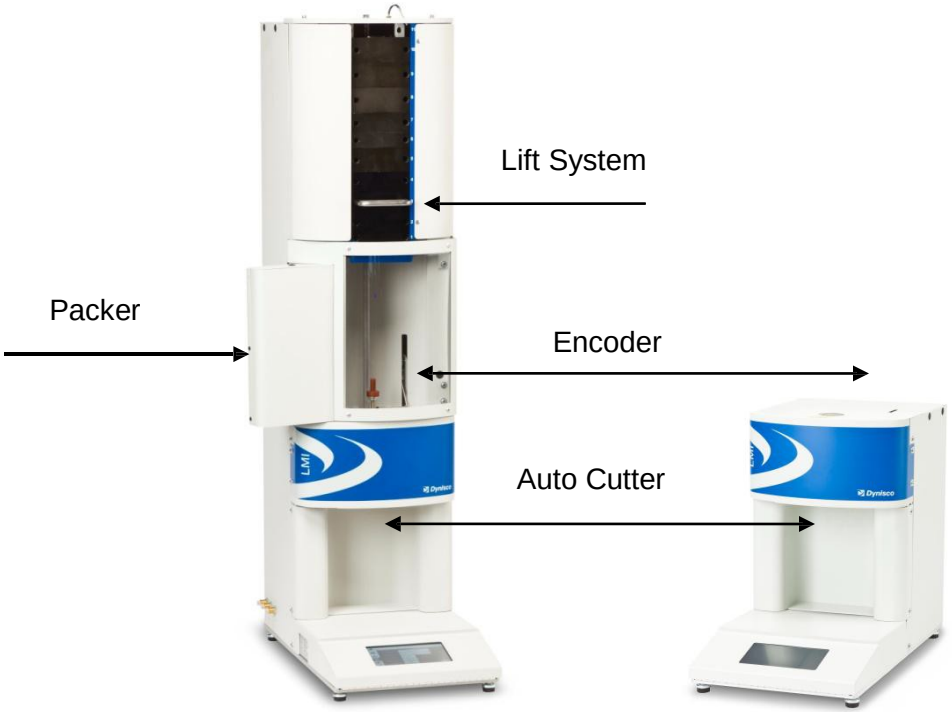


Figure 1: LMFI with lift, encoder and packer. (Cutter Location Shown)
Figure2: LMFI base unit (Encoder and Cutter Location Shown)

Rear View

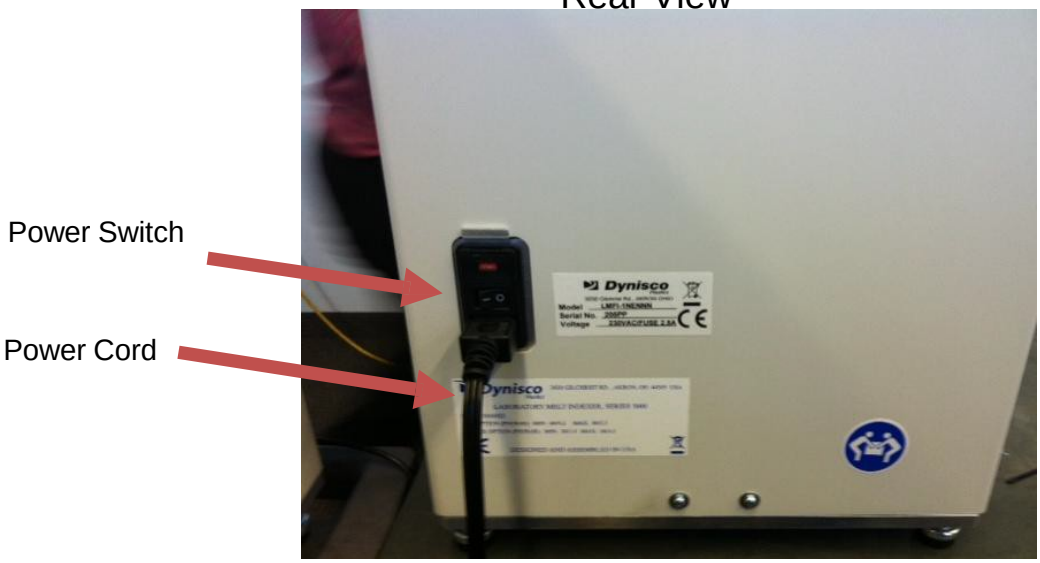


Figure 3: LMFI rear view Power connections

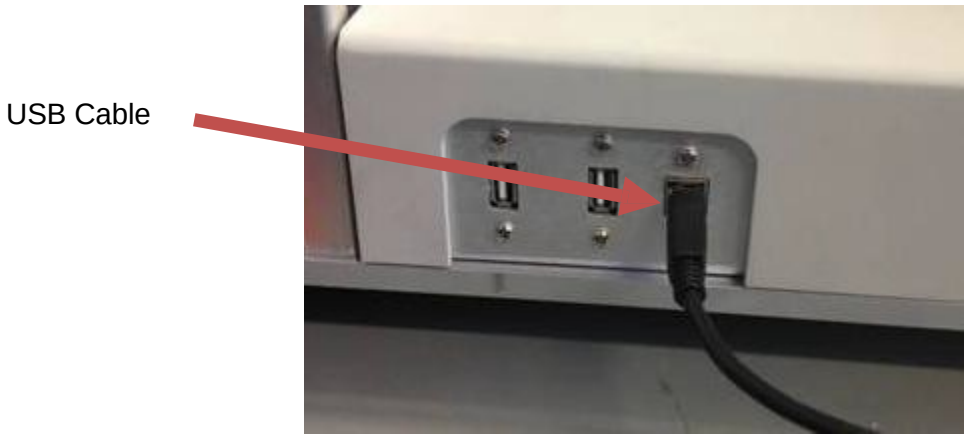


Figure 4: LMFI side view USB connections

Figure 3 shows the rear connections of the LMFI. Figure 4 shows the side USB connections with a PC cable connection to the USB, Type A connection. If using the LaVA Suite software, there is one cable connection that must be made from the instrument to the computer: a USB cable. The product sticker on the back of the LMFI will show the model, power requirements and the instrument serial number.

Item	Part No.	Qty (Each)
LMFI 5000 Operator's Manual	M0726	1
Barrel (If wore, out of specification)	4051-25A	1
Piston Assembly (Tip, Piston, Weight Top)	7051-72	1
Piston Tip	0051-41	1
Die/Orifice (Standard 8mm Height)	0051-46	1
Die/Orifice (Half-Die 4mm Height Half Diameter)	0051-46S	1
Insulator, Top Mica (Die Entry Point)	4051-20MA	1
Barrel Cleaning Patches	GP0104	1 bag (100ea)
Barrel Cleaning Patches	GP0103	1 bag (1000ea)
Die Go/No-Go Gauge	0051-55	1
Die Cleaning Drill	0051-39	1
Barrel Cleaning Brush	B0555	1
Barrel Cleaning Tool (Used with Patches)	0051-40	1
Die Removal Tool	0051-35	1
Funnel, Polymer Charging	0051-80	1
Polymer Packing Hand Tool	0051-36	1
Knife, Palette (For Cutting—Method A)	0051-53	1

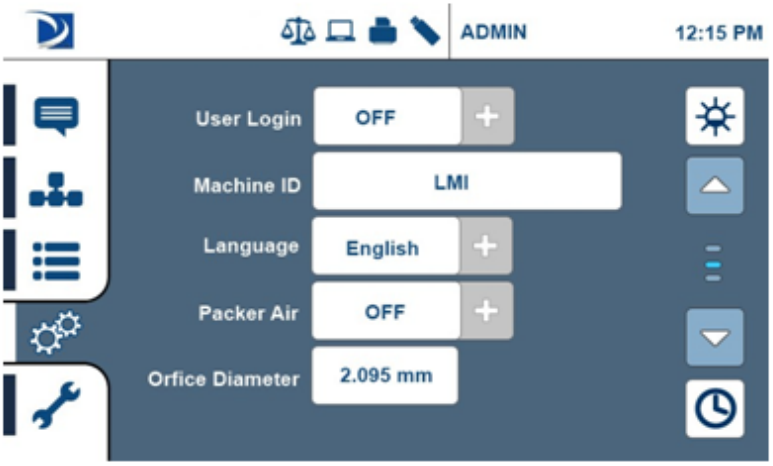
Instrument Operation/Log-In

When the system is powered-up, a log-in screen will appear. Below is a list of default user names and password and levels of access. The system has 5 levels of default user types. Admin and Maintenance user levels are defaulted with instruments at shipment. Users of Guest, Tester, and Manager levels will have to be added by the Admin level user if desired.

User Groups	Default User	Password	Access Rights
GUEST			Viewing only. No programs, system configuration (except volume and screen brightness), or calibration can be modified. Tests cannot be performed.
TESTER			Testing only. Cannot modify programs that are stored in memory. Can run tests and change test variables for the current test in which the values are not saved into memory. Can modify some of the system configuration, but cannot run calibration.
MANAGER			Program Editor. Same rights as "Tester" plus modification of programs that are stored in memory.
ADMIN	ADMIN	ADMIN	System Admin. All rights as "Manager" plus access to calibration data.
MAINTENANCE	MAINTENANCE		Maintenance. All rights as "Admin" plus access to special maintenance information.

Logging-in at power-up can be turned off if not desired. Simply log-in at the Admin level.

Go to System Configuration's second screen using the  and then  to get to the screen. Then simply set User Login to "OFF" as shown here.



Instrument Operation/Icons

Icons –An icon will be displayed when a device has been connected to either the master or host USB connections.

Remote computer is connected	Printer is connected
Error during initialization of the remote computer	Error during initialization of the printer
Mass storage device is connected	Scale is connected
Error during initialization of the mass storage	Error during initialization of the scale

Instrument Operation/Menus

Action	*Not Selected*	*Selected*	*Disabled*
Displays the Test Status Screen			
Displays the Programs Screen			
Displays the Test Setup Screen			
Displays the System Configuration Screen			
Displays the Maintenance Screen			

Instrument Operation/Buttons

Buttons *–A button is an active touch area that will perform a specific action on the release of the button. There are three states to a button: released, pressed, and disabled along with the option of a press and hold which will repeat the desired action as long as the button is pressed*

Action	*Released*	*Pressed*	*Disabled*
Displays the "About" window.	LMI	LMI	LMI
Sets the screen brightness to to the maximum level.			
Sets the screen brightness to to the medium high level.			
Sets the screen brightness to to the medium low level.			
Sets the screen brightness to to the minimum level.			
Closes the currently active window.			
Displays the weight configuration window.			
Continues/Moves Forward the currently running test.			
Copies the currently selected test conditions from the USB device into the system or from the system to the USB device.			
Deletes the currently selected test condition.			

Buttons Cont.

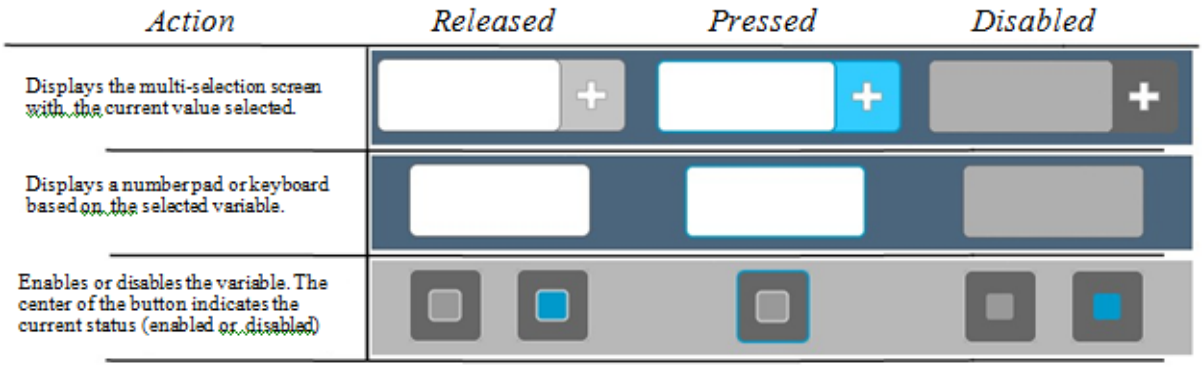
Action	*Released*	*Pressed*	*Disabled*
Displays the test conditions Edit screen with the currently selected test conditions.			
Stops the currently running test or stops the series if the system is running a series.			
Displays the lift override/ manual operation window.			
Creates new test conditions and changes to the test conditions Edit screen.			
Prints a test page to a connected printer.			
Displays the import test conditions window.			
Displays the export test conditions			
Displays the test results export			
Saves the current information.			
Selects all			
Deselects all			

Buttons Cont.

Action	*Released*	*Pressed*	*Disabled*
Selects the current test conditions and displays the			
Displays the edit date and time screen.			
Starts a test with the current test information and displays the test status screen.			
Sets the system volume to the the maximum level.			
Sets the system volume to the medium level.			
Sets the system volume to the minimum level.			
Sets the system volume to			
Select/ OK button that will accept the current condition.			
Displays the tool tip			
Logs the user in or displays the Edit user window.			
Displays the RTD calibration			
Displays the LCD/ Display Calibration			

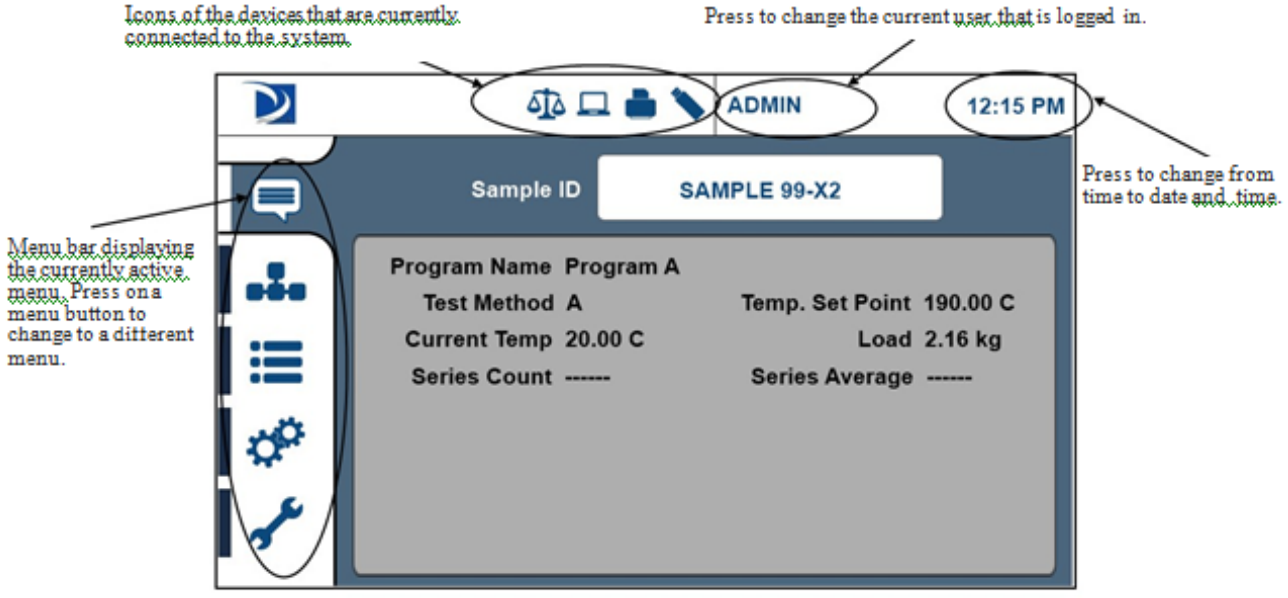
Instrument Operation/Miscellaneous Touch Areas

Miscellaneous Touch Areas—*The following items are touch areas that will perform the specified operation after the button has been released. There are three states to a button: released, pressed, and disabled.*



Instrument Operation/Panel Overview

Panel Overview



Test Calculations

Calculations: Method A

Calculations for the Method A test are:

$$MFR = \frac{M\ 600}{T}$$

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).

Calculations: Method A/B

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 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 for the Method A/B test are:

$$\text{Method B MFR} = \frac{\pi R2L\rho\ 600}{TB}$$

$$\text{Method A MFR} = \frac{M\ 600}{TA}$$

$$\rho = \frac{MTB}{\pi LR2T}$$

Where R= radius piston (cm), TB (sec) is time to traverse the Method B distance L, L = Method B length of flag (mm), M is the mass in grams of material collected over the time TA in seconds. MFR has units of g/10 min. (grams/10 minutes). ρ (g/cc) 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 (MFR) 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, during the A/B test, there was no leakage past the plunger tip and the extrudate was bubble free and other minor factors did not occur, then a true melt density could be assessed. All lengths are in mm.

Calculations: Method B

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 test 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 laboratories use MVR directly without ever determining MFR.

The piston's downward travel time is determined from a counter initiated by a digital encoder. The encoder senses distance travelled by following the bottom of the test weight(s) which are at the top of the piston. With all Dynisco Polymer Test Systems flags, Method A and B start in the same place. Flags may be any length desired and a test can have any number as long as the total distance of flags is not longer than the distance from the start point and where the piston would land on the top of the die (≈25.4mm). Recently, Method B has become the more common test because it is simpler to run (hands free after material load) and more precise for routine analysis. In addition, the encoder system makes it possible to get more from one run.

Calculations for the Method B test are as follows:

$$MVR\rho = MFR = \frac{\pi R2L\rho\ 600}{T}$$

Where R= radius piston (cm), T (sec) is time to traverse the distance L, L= length of flag (mm), ρ = apparent melt density of polymer (g/cc).

You should determine the melt densities for your material using your 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, most likely, something is being done 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**.

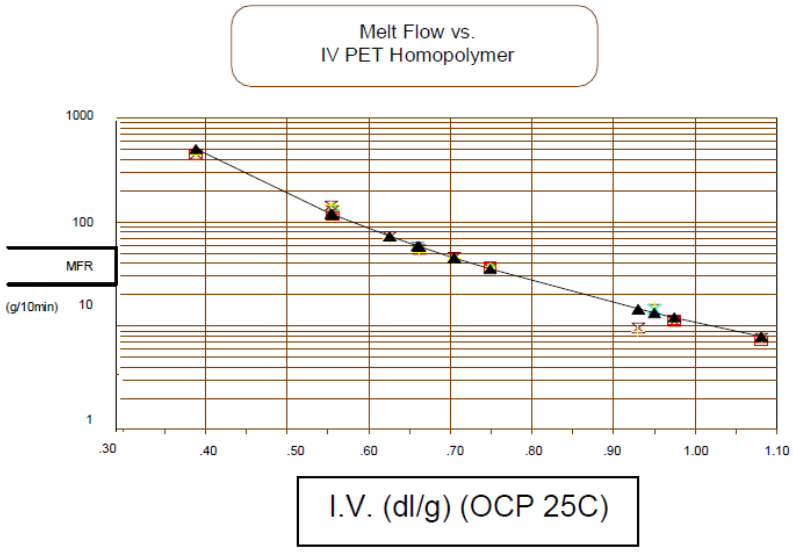
Some typical Melt Densities

Temperature(°C)	LDPE	HDPE	Polybutene-1	polypropylene
120	.797	-	.806	.880
130	.791	-	.800	.872
140	.785	-	.794	.864
150	.780	.780	.787	.852
160	.777	.777	.780	.840
170	.770	.770	.774	.819
180	.765	.765	.767	.758
190	.760	.760	.760	.754
200	.755	.755	.754	.750
210	.748	.748	.746	.746
220	.744	.738	.740	.742
230	.738	.738	.733	.738
240	.733	.733	.726	.734
250	.737	.727	.719	.730

Any inaccuracies in the melt density will be 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.

Calculating PET Intrinsic Viscosity (I.V.) from the Melt Indexer

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.



How to Do It and Why?
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.

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 ºC, use 6 grams material per test, clean machine thoroughly and run at a temperature of 285 ºC.

This option, if purchased, enables only at 285 ºC
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 ºC.
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

IV	MFI	Flag Length (cm)	Method A Time (seconds)	Pull Plug (seconds)
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)

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 piston's 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 recommended 6 minute melt time, some material flows out of the die so a larger charge is needed. The best situation would be to add 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 timed tests). 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:

$$Charge\ Mass = 3.6\rho + 0.6 * MFR$$

Where the charge mass is in grams, ρ is the melt density in g/cc and MFR is the anticipated 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 anticipated MFR of 3.5 at 230 C, using the melt density of 0.738 from the above table, the estimated charge mass would be:

$$3.6*0.738+0.60*3.5=4.76g$$

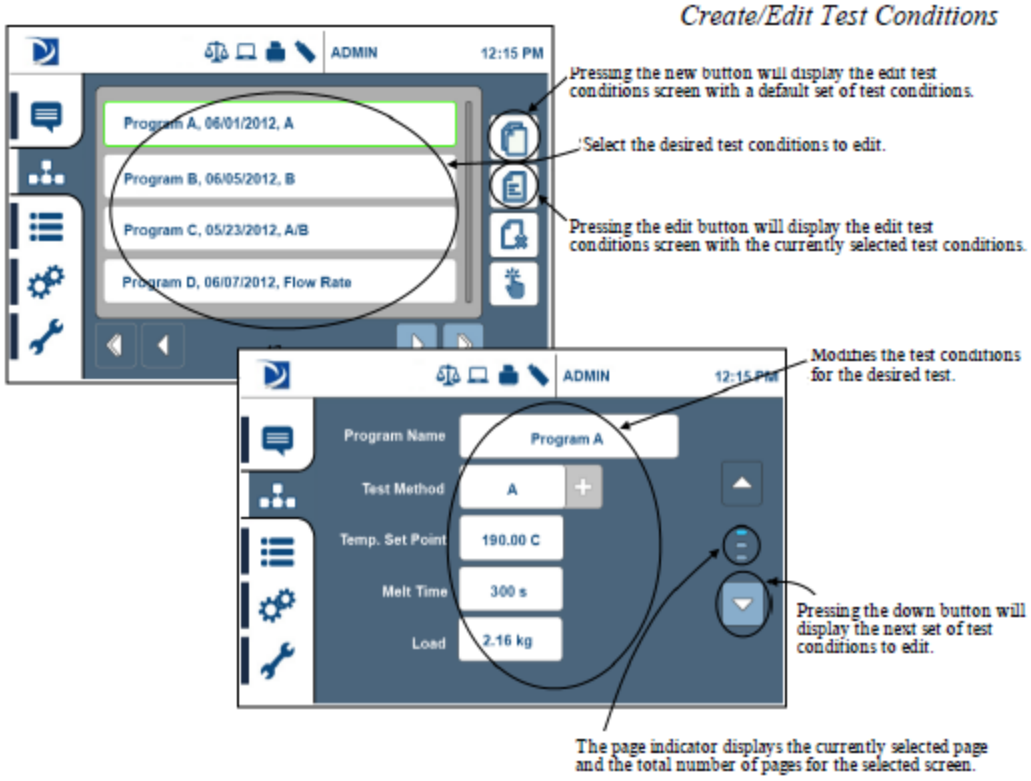
In our lab, we have found that 4.6 grams is a good charge for a 3.5MFR Polypropylene sample. The equation overestimates the charge since is assumes flow from the instant the material is packed into the barrel. The following table shows an estimate of charge weight in GRAMS based on anticipated MFR (g/10 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.

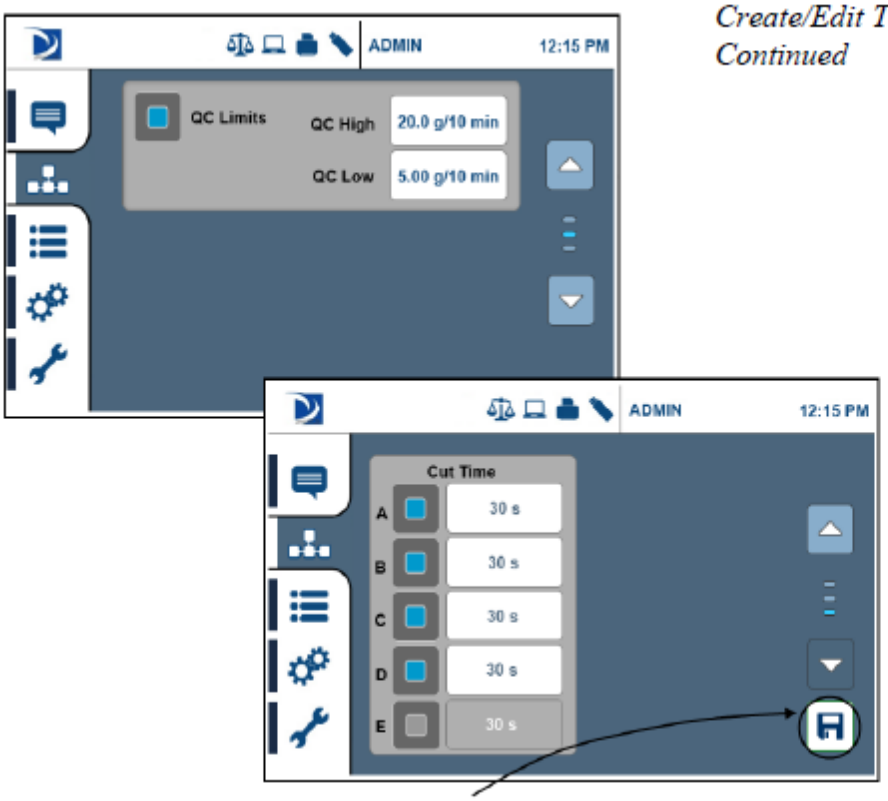
Barrel Charge Size in Grams

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

Create/Edit Test Conditions

Create/Edit Test Conditions

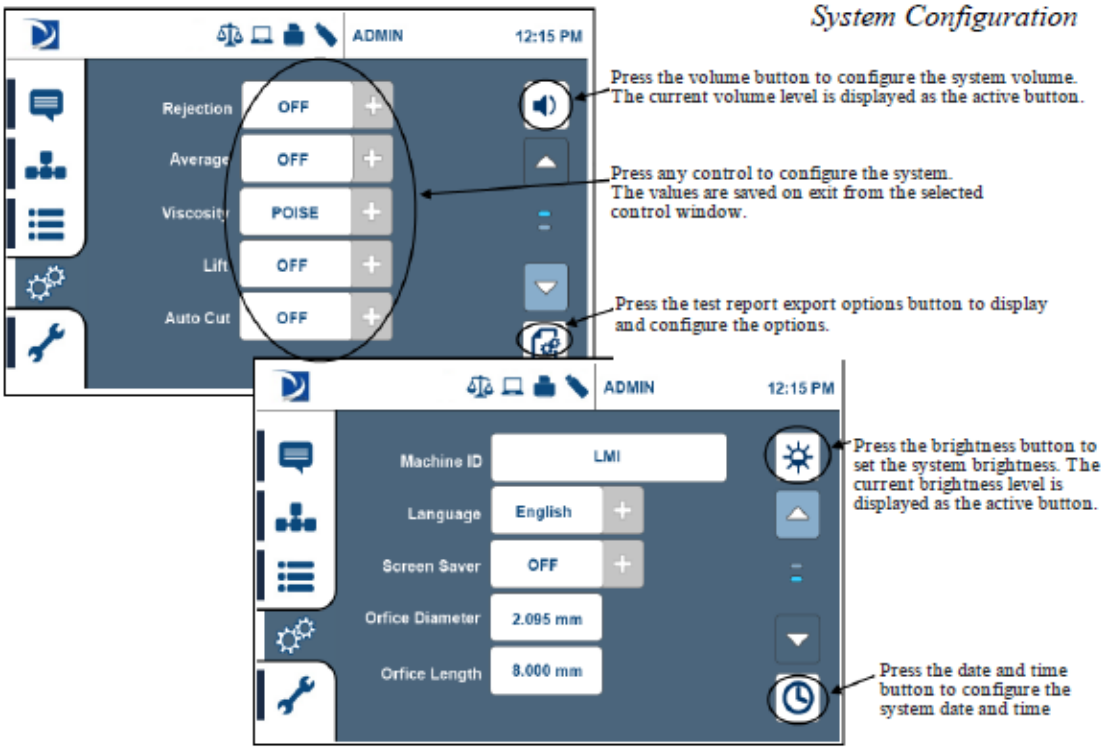




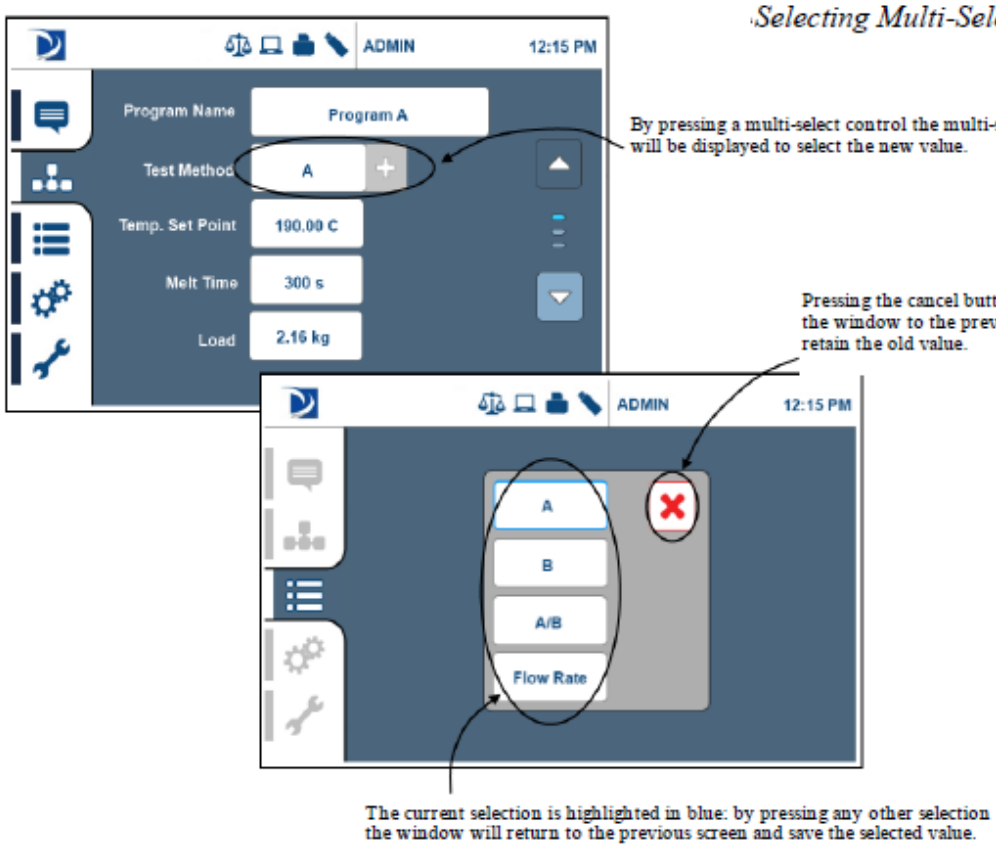
Create/Edit Test Conditions Continued

Press the save button to save the current test conditions. Test conditions are saved based on program ID so this is value must be unique for each program.

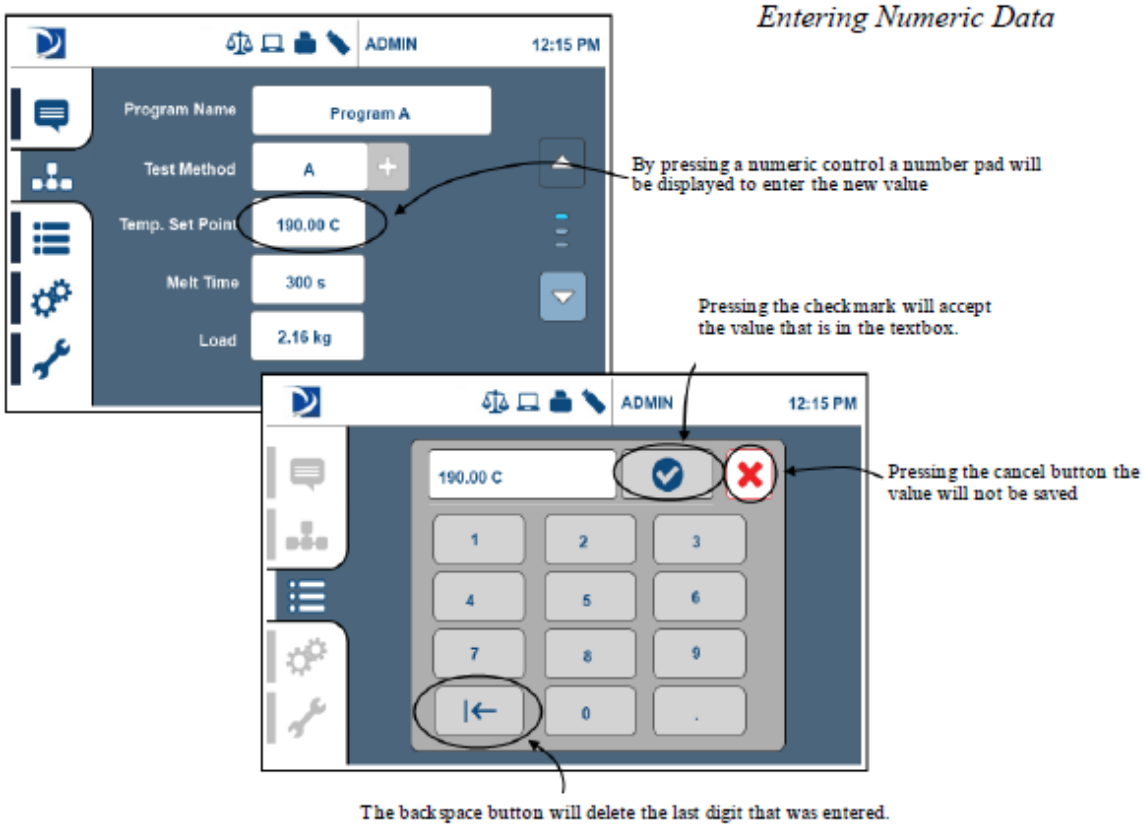
System Configuration



Selecting Multi-Select Data

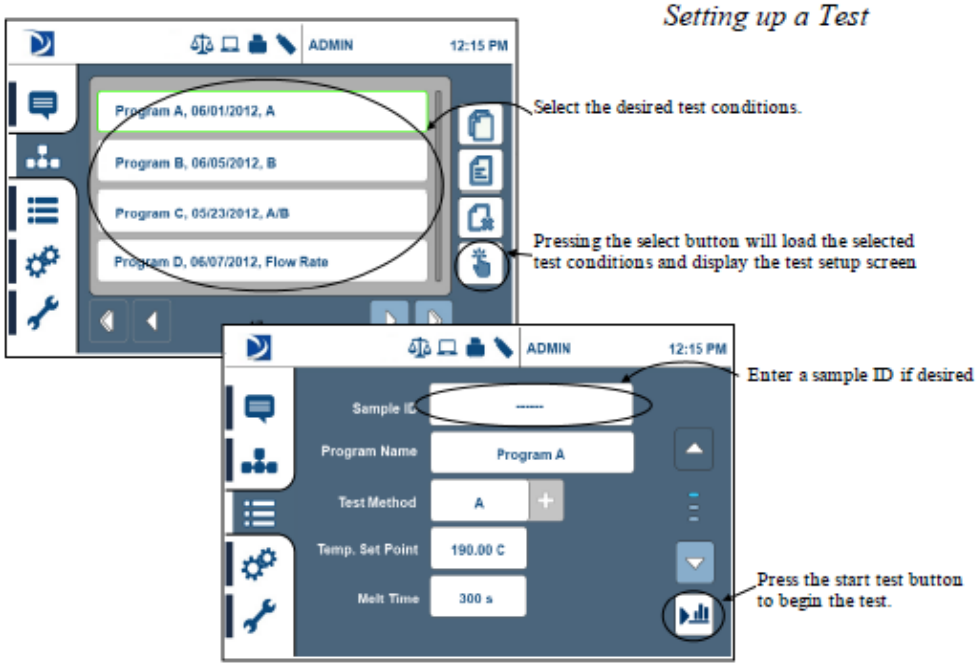


Entering Numeric Data



Setting-Up a Test

Setting up a Test



Loading and Packing Material into the Barrel

The piston rod should be inserted into the barrel during heating and temperature stabilization and between tests. 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/barrel with an appropriate charge or use about 5 grams if nothing is known about the material. Put the sample into a small beaker. Add about two-thirds of the material into the barrel, using a fill funnel. The loading should be done in two increments, the first consisting of approximately two-thirds of the material. Tamp down the material with the packing tool using approximately 20 pounds/9Kg 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.

The Packer Option, if purchased, can be used to tamp down the material. Packer air pressure is set, depending on material, to achieve the desired packing force. The packer must be enabled, through the System Configuration screen. Material is loaded, as stated above, with a funnel. The funnel is removed. The packer is pivoted around and held above the barrel. The packer's push button is pressed to extend the packer rod down into barrel. The packer's push button is released. The packer rod is released and the entire packer assembly will pivot back to its starting position. If a lift option is also installed, the lift will not lower if the packer is not back in its starting position.

Bubbles are present in the test 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 tamping down. If bubbles continue, the sample may not be sufficiently dry.

Place the piston rod into the barrel by placing the piston rod directly over the slot/opening on the top of the indexer barrel cover. Seat the guide bushing down into the barrel if it has not already fallen into place. The guide bushing should be moving freely on the piston plunger. Place the desired test weight on the piston plunger rod.

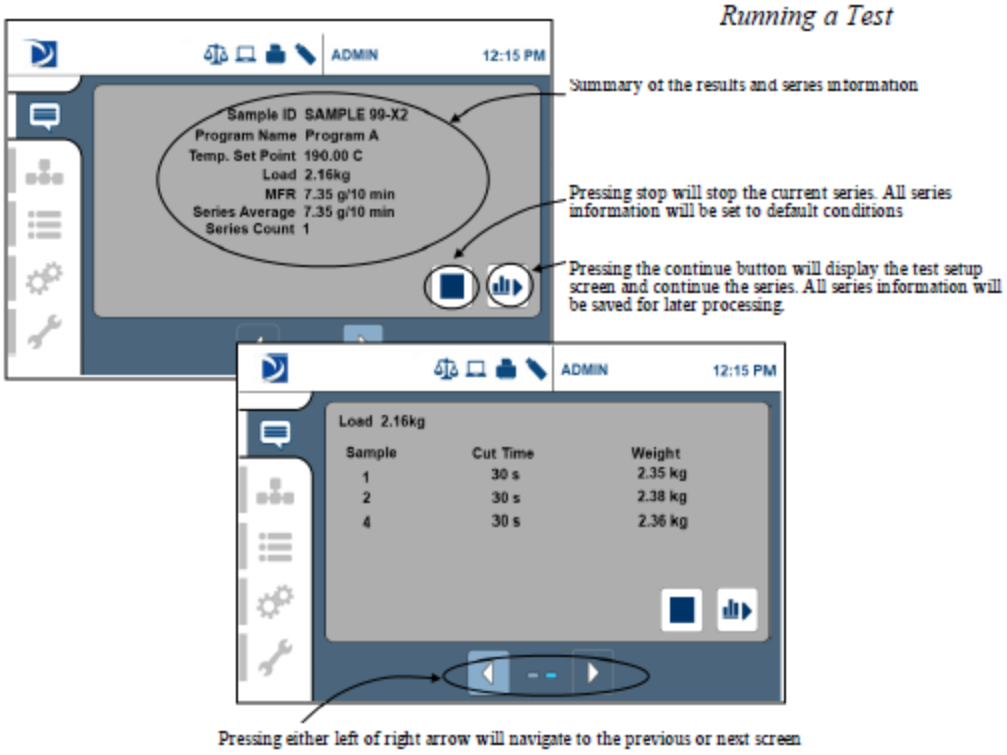
The lift option can be used to lower weight onto the piston. The lift must be enabled, through the System Configuration screen. On the test setup screen, Auto raise, Auto lower and Auto Hold can be enabled and used for weight sequencing.

Running a Test

To run a test, press the  icon from the Test Setup screen after either defining a new test or selecting a pre-defined test. Follow the screen prompts for temperature stabilization, packing/loading of material, weight selection and placement, encoder position (if used for test), and melt time for starting of test.

Once the test has begun, follow the screen prompts for cuts and input of cut weights. After the test is run, cut weight results can be rejected if desired.

Running a Test



Cleaning Up

If using hand weights without the lift system, push down slowly on the weight and purge any material remaining in the barrel through the die and out of the barrel. If using a weight lift system, you may want to turn off the “Auto Raise” feature to allow the test weights to dwell at the end of the test and purge any remaining material from the barrel. Remove the piston 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 the rod which may lead to a very hot die coming off and travelling through the air or onto the floor or some other undesired location.**

While wearing protective gloves, wipe the piston rod with a cotton rag. Remove the die with the die removal tool. Put two cleaning 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, and 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 piston plunger back into the barrel. This allows the piston plunger and the die to reheat before the next test.

With materials that are thermally stable (less than 5% viscosity change over 1/2 hour of heat exposure) we recommend only cleaning the barrel between every second run. For materials that degrade or are moisture sensitive it is recommended that both the barrel and the die be cleaned completely between each test. 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 a 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 sample 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 inserted when the die is removed and the material starts to solidify.

USER TIP: Experienced users often listen for a 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 of the barrel, look down the barrel bore to be sure it is clean (use safety glasses when looking down the barrel and beware of noxious fumes that may come from heated material inside!). Ensure 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 PVC die (D3364 for 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 will be.

Minimizing the time the die stays out of the machine will increase the number of tests you can run per time. 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 the temperature set point is locked in. A sufficient melt time, (360 seconds, for example) will allow ample time to get the temperature to the set point and stabilize 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 cleaned with cleaning patches from the machine before accurate data can be obtained.

Warning: **Do not heat the barrel back to elevated temperatures before cleaning the machine oil from the barrel. Noxious fumes and/or fire hazard exists if not cleaned.**

TROUBLESHOOTING

Actions

Getting incorrect or non-repeatable test values:
Was the instrument clean?
Check density value
Check tip diameter (> 0.3727)
Check Die (clean, diameter length OK?)
Check Machine is Level
Check Temperature
Check weights used +/- 0.5%
Check balance used to weigh extrudate
Piston Rod Bent?
Check die can get to bottom of barrel?

Generic Run Check List

Prior to loading Sample:
Is material properly prepared (dried, mixed, checked for contaminates)?
Machine on for 20 minutes to allow temperature to stabilize?
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:
Are die diameters within specification (passed G0-No Go gage, ASTM, ISO, DIN)?
Temperature calibration OK?
Piston Tip Diameter within specification?
Barrel Diameter within specification?

Support Vendors

1. NIST Standard Reference Materials (SRM)
   For example: Standard Material 1476 is a branched polyethylene with a MFR of 1.19 ± 0.01. As of 2013, cost was $870/US for 12 grams.

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

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

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

APPENDIX A—DIGITAL ENCODER OPTION

Introduction
A digital encoder module is available as an option with the instrument. It permits the collection of multiple data points from one charge of the barrel. The digital encoder makes operation of the instrument much faster and easier.

Encoder Option
Tests, using from one digital flag up to fifteen digital flags, can be performed. The Dynisco Encoder for Method B tests employs an encoder coupled to a precisely calibrated arm. The tip of this arm employs a hardened & ground tip for extra long life. Accuracy of the measurement range ±0.025mm over 25.4 mm. The digital encoder, while being very accurate, also enables the test length signal to be processed & varied digitally. Thus, any flag length up to 1"(ASTM) or 30 mm (ISO) may be selected. Up to 15 MFR or MVR results per barrel filling can be obtained.

The unit is securely fastened to the top, rear of the LMFI, and the arm latches down to facilitate cleaning of the unit after the test is completed.

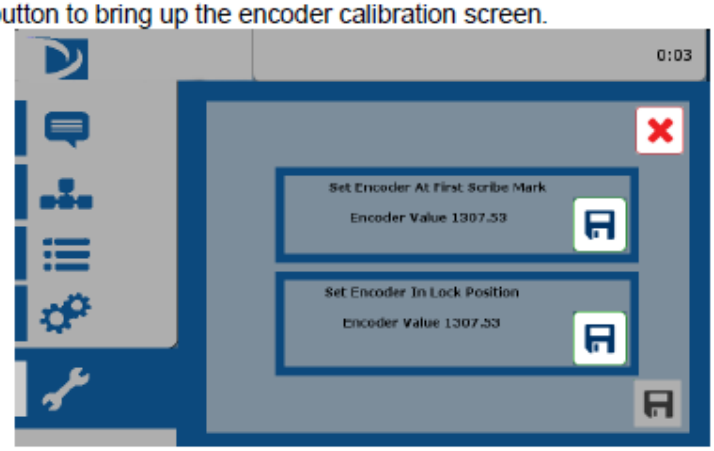
Encoder Advantages
- Completely Automatic Test Length Selection on Single MFR Tests.
- Up to 15 MFR readings per Barrel Filling can be obtained.
- Any test flag length up to 30 mm can be used.
- User Defined test length and test spacing for single or multiple tests can be used.
- Test conditions are stored internally.

Why use it?
- No manual cuts are required.
- A sample can be checked as homogeneous within a charge.
- Get better precision (averages are less variable than single observations) for tests.
- Verification as to whether barrel pressure drop is significant for sample.
- Gain ability to reject data point if bubble or air pocket existed in sample.

Verifying or Calibrating the Digital Encoder

1. Press  button to bring up calibration screen.

2. Press  button to bring up the encoder calibration screen.



3. Mark sure the encoder arm is locked into its locking holder.
4. Press  next to "Set Encoder in Lock Position" section to save current position of encoder arm.
5. Insert a 48mm long spacer into the barrel.
6. Insert piston into barrel so that it will sit on the spacer. The piston's first scribe mark should be aligned with the top of its sliding bushing.
7. Place a weight onto the top of the piston. Weight's bottom must be flush, without any recess.
8. Raise the encoder arm to pivot against the bottom of the weight.

9. Press  button next to "Set Encoder at First Scribe Position" section to save current position of encoder arm.
10. Press  button in the lower right hand corner of screen to save both calibration locations.
11. Screen reverts back to the calibration screen.
12. Lower arm back into its locking holder.
13. Remove weight from piston.
14. Carefully remove piston and spacer as they may be very hot.

Maintenance

1. Always place the arm into the lowered and locked position before cleaning and the end of the day. This prevents damage and ensures that the encoder arm will be in the proper position upon power up.
2. Never place any severe force against the side of the arm.

APPENDIX B—PNEUMATIC LIFT

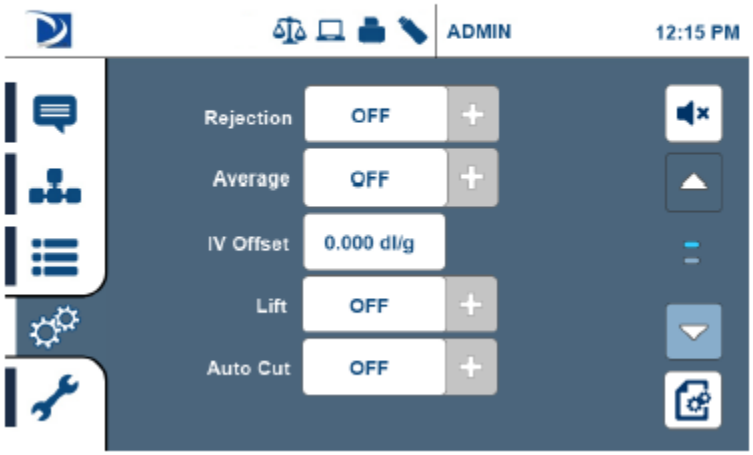
Introduction

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

Flow Ratio provides the user with insight into a materials shear thinning ability and can 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.

Activation and Operation

Press  button to bring up the System Configuration screen.

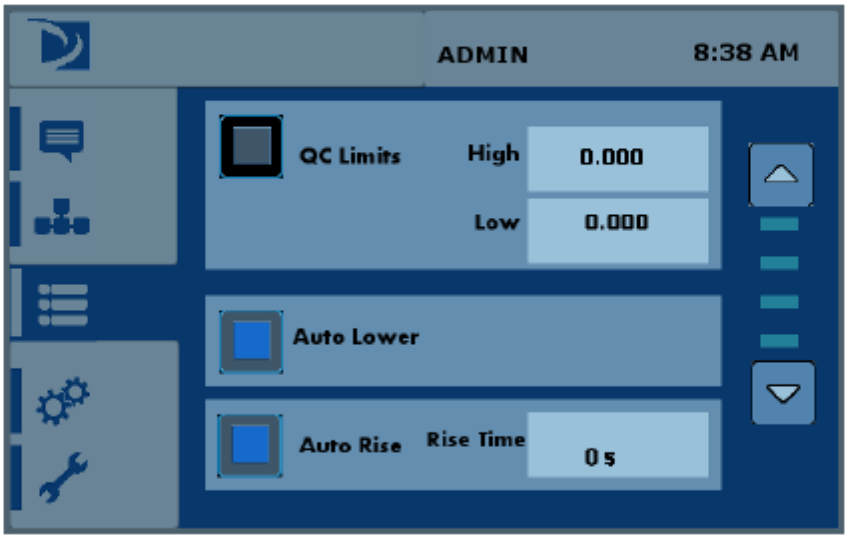


Lift

Change to use lift; chose on or off from drop down menu. Option is only visible when the control cable for the lift is assembled onto the main board.

The lift will be used during test if set to “ON” in the configuration screen above. The screen will prompt the user during test steps when the lift is going to be moved and where to key or place the weight fork during the test procedure.

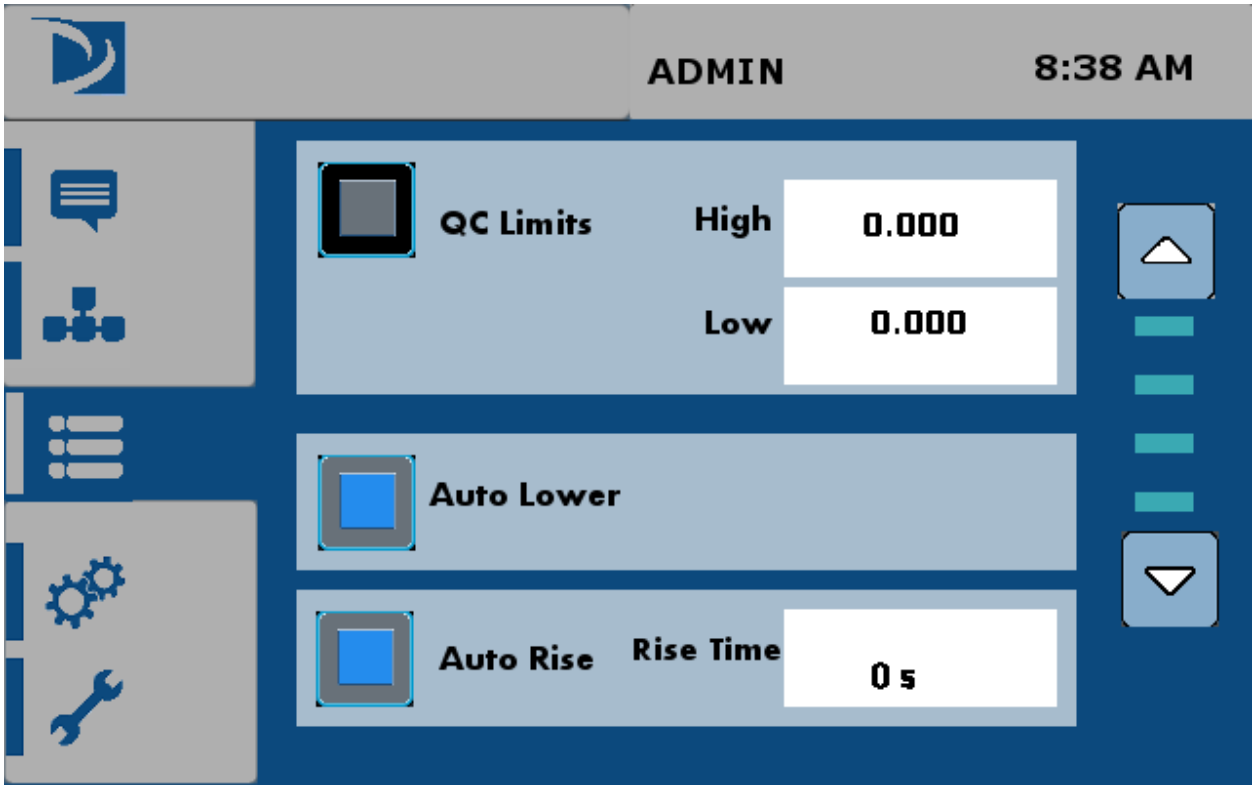
When setting up the test procedures or recipes, press  or  buttons to page up and down as required to see lift options.



Press  buttons next to each lift option to enable it.

Auto Lower
Enable to let the lift bucket lower the weight onto piston after barrel is packed with sample. If Auto Lower is not enabled, the operator will have to press a screen prompt to lower the weights and weight bucket during a test.

Auto Raise
Enable to let the lift bucket raise the weight off piston after the test has ended automatically. If Auto Raise is not enabled, the operator will have to press a screen prompt to raise the weights and weight bucket.
Enter any delay time as required in the box next to “Rise Time”. The “Rise Time” delay will expire before the lift is raised. A raise delay can be used to “Purge” the barrel of remaining material after a test

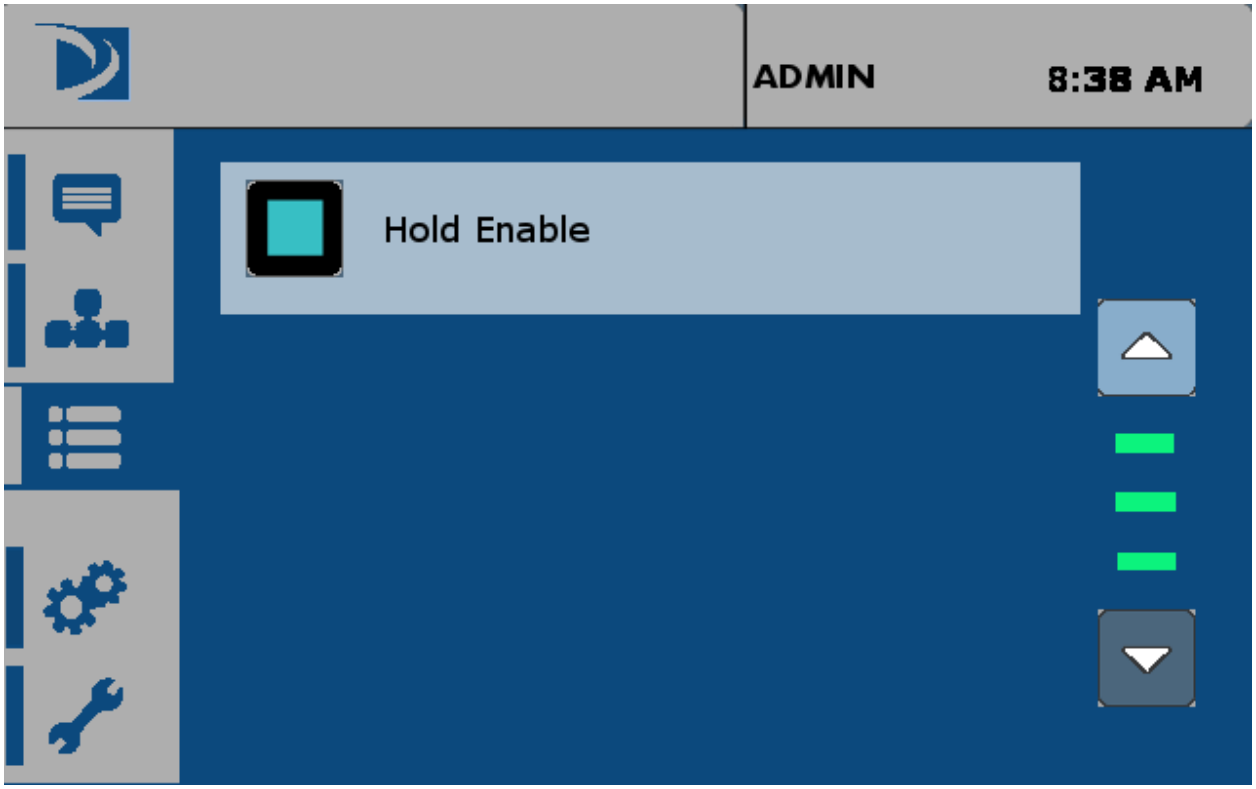


Press  buttons next to each lift option to enable it.

Auto Lower
Enable to let the lift bucket lower the weight onto piston after barrel is packed with sample. If Auto Lower is not enabled, the operator will have to press a screen prompt to lower the weights and weight bucket during a test.

Auto Raise
Enable to let the lift bucket raise the weight off piston after the test has ended automatically. If Auto Raise is not enabled, the operator will have to press a screen prompt to raise the weights and weight bucket.

Enter any delay time as required in the box next to “Rise Time”. The “Rise Time” delay will expire before the lift is raised. A raise delay can be used to “Purge” the barrel of remaining material after a test.



Auto Hold
Enable to let lift bucket lower until its weight-holding edge is 3.88” off top of barrel. This allows the weights to push the piston to the “First Scribe” mark and no further during the melt time of the test.

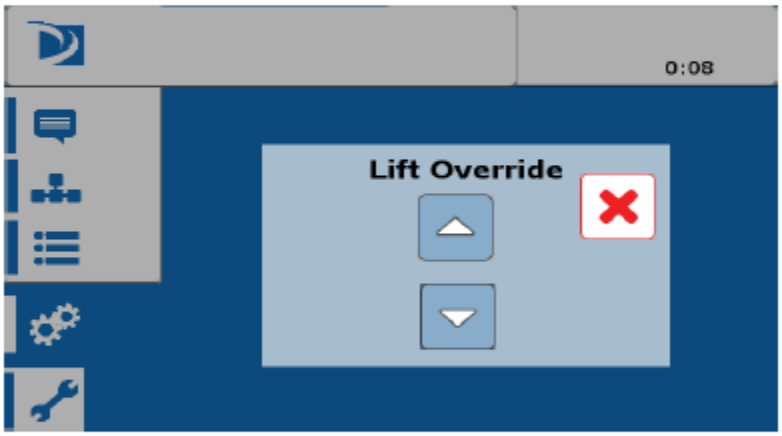
- Bucket may be lower than weights during hold if there is a lot of material in barrel causing the pistons and weights to sit higher.
- Hold is useful for high-flow materials and can be used in conjunction with a melt plug. The hold keeps the weights from extruding the melted sample before the melt time has expired.

After setting lift options, press  to get to  button and save in test procedures or recipes.

Lift Bypass

The “Lift Bypass” option is simply a manual way to test the lift without having to use a test procedure to do so. It is useful if one wanted to check movement or positions of the lift for accuracy or workability. Lift safety measures such as safety shield and weight fork must be employed for “Lift Bypass” to function.

1. Press  button to bring up the System Configuration screen.
2. Press  button until  button.  button to display lift bypass screen.

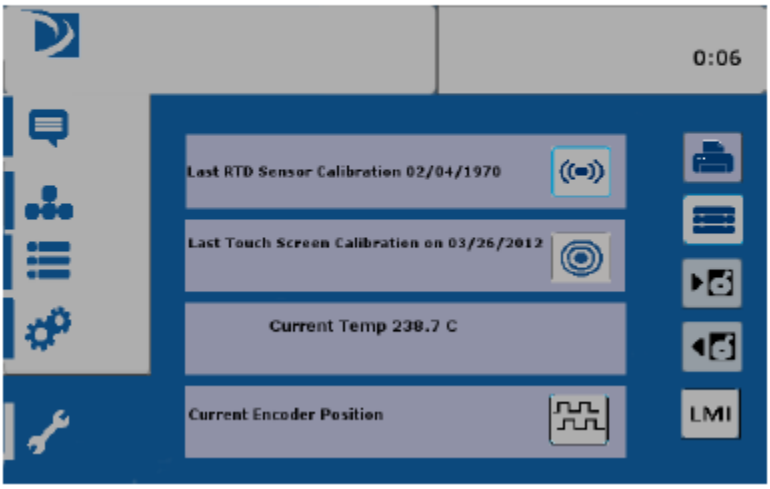


3. Press and hold  or  button to raise or lower the lift.
4. Press  button to leave screen. Screen reverts to previous screen.

Weight Configuration

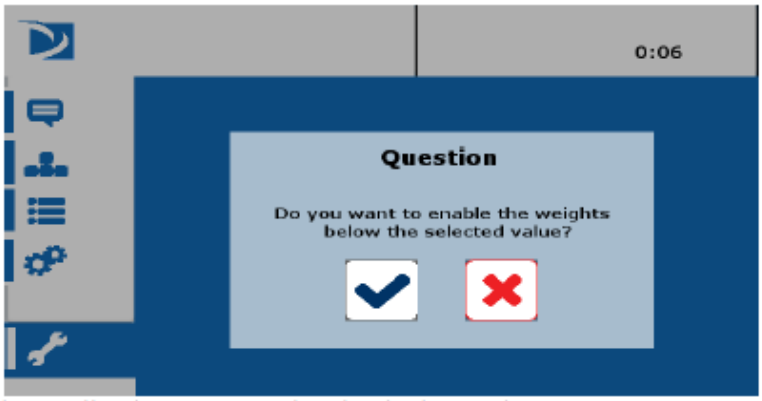
The weight configuration screen sets the weight values and positions within the LMI operation. When setup correctly, the system will alert the operator where to place the weight fork depending on the test procedures' defined test weight. An installation guide accompanies all weight system shipments showing how to install the physical weights in the LMFI.

1. Press  button to bring up calibration screen.



2. Press  button to configure weights. List of weights are displayed. The weight values listed are default. If special or non-default weights have been installed, simply press the value of the weight in the desired position to change, then type the weight value that is physically in that position. There are numbers along the weight stack showing position numbers.

3. Press  next to all weights required to enable them for the stack. Enable the highest position number weight first and the screen will prompt to allow it to automatically populate the lower weights.



4. Press either button to finish selecting the test weights.

Press  button to save weight configuration.

APPENDIX C—PNEUMATIC PACKER

Introduction

The packer accessory removes user variations on packing force. It applies a force that is controlled by an air regulator. The packer is pivoted around to the barrel. It must be held in place or it will return to its starting position. While the packer is held over the barrel, press its switch to extend the packer end into the barrel. When the switch is released, the packer tip will retract out of the barrel.

Activation and Operation

Press  button to bring up the System Configuration screen.

Packer Air

Turn the Packer option from “OFF” to “ON” by pressing the white area around the word “OFF” and then selecting the “ON” option that displays.

When activated or turned “ON” the Packer is available during test material loading times. Option is only visible when the control cable for the packer is assembled onto the main board.

If the packer is used with the lift option, the lift will not lower unless the packer is back in its starting position, outside of the unit’s working area.

APPENDIX D—AUTO CUTTER

Introduction

The Auto Cutter option is for cutting the sample during either Method A or Method A/B tests. The cutter is engaged at the end of each, defined cut time within the test. For example, if a Method A test is defined to have 3 sample cuts, at 60 second intervals, then the auto cutter will cut the extrudate at the end of each 60 second interval. Also, the auto cutter will cut the extrudate at the end of the defined Melt Time, before the test sample time is to begin.

The Auto Cutter option is triggered from the LMI main PCB via an 8 pin connection to the PCB. The Auto Cutter option cuts in a single, 360 degree rotation and drops the cut sample down below the barrel in the work surface above and to the rear of the display. The cutting action is accomplished by using a thin, flexible blade against the rigid, bottom die holding plate. The cutting blade is rotated via a stepper motor. Homing and Positioning are controlled within the motor's own, on-board electronics and a position sensor tied to the motor control.

The Auto Cutter comes with a clear, safety door. This door must be closed for the Auto Cutter to cycle. The door may be opened during a test to remove cut sample(s) and then closed to allow the cutter to continue to operate during a test. For example, if 60 second sample cut intervals are being used for a test, the operator may open the safety door, remove the sample, and then close the safety door in the 60 second interval between tests without affecting the Auto Cutter operation during a test.

At anytime during a test, the sample extrudate can be cut by the operator by simply pressing the  cut button.

Activation and Operation

Press  button to bring up the System Configuration screen.

Turn the Auto Cut option from "OFF" to "ON" by pressing the white area around the word "OFF" and then selecting the "ON" option that displays.

When activated or turned "ON" the Auto Cutter will automatically cycle during Method A tests at both End of Melt and Cut times.