

# PET Plus Viscometer

including Polymer Dryer and Polymer Grinder

## User Manual



## **WARRANTY**

This instrument is warranted against defects in workmanship, material and design for one (1) year from date of delivery to the extent that AMETEK will, at its sole option, repair or replace the instrument or any part thereof which is defective, provided, however, that this warranty shall not apply to instruments subjected to tampering or, abuse, or exposed to highly corrosive conditions.

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This warranty is voidable if the purchaser fails to follow any and all instructions, warnings or cautions in the instrument's Instruction Manual.

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## ICONS



### WARNING

The raised hand icon warns of a situation or condition that may lead to personal injury or death. Do not proceed until the warning is read and thoroughly understood. Warning messages are shown in bold type.



### DANGEROUS VOLTAGE

The lightning icon warns of the presence of an uninsulated dangerous voltage within the product enclosure that might be of sufficient magnitude to cause serious shocks or death. Never open the enclosures unless you are an authorized and qualified LLOYD INSTRUMENTS' service personnel. Never open any enclosure when power is connected to the system or its components.



### CAUTION

The exclamation point icon indicates a situation or condition that may lead to equipment malfunction or damage. Do not proceed until the caution message is read and thoroughly understood. Caution messages are shown in bold type.



### NOTE

The note icon indicates additional or supplementary information about the action, activity or concept. Notes are shown in bold type.

## CAUTION

**THIS EQUIPMENT IS ONLY FOR USE BY TRAINED PERSONNEL**

**IF NOT USED IN MANNER STATED MAY CAUSE HAZARD**

**ALL SPECIFIED SAFETY PROCEDURES SHOULD BE STRICTLY ADHERED TO**

**HIGH TEMPERATURES AND MOLTEN POLYMER ARE INVOLVED IN THE VISCOMETER TESTING PROCESSES - ALWAYS WEAR HEAT PROTECTING GLOVES WHEN HANDLING THE DIE, FOLLOWERS, PROBE, CLEANING TOOL AND AREAS AROUND THE HEATED BARREL**

**THE VISCOMETER AND SOME OF ITS ACCESSORIES ARE POWERED BY MAINS SUPPLY VOLTAGE**

**TO MAINTAIN ALL ASPECTS OF THE SPECIFICATION, ONLY DAVENPORT/LLOYD INSTRUMENTS APPROVED ACCESSORIES CONNECTIONS AND COMPONENTS SHOULD BE USED**

**NITROGEN GAS UNDER PRESSURE IS USED - WORKING PRESSURE 30 BAR - MAX PRESSURE 35 BAR**

**READ THIS MANUAL BEFORE USING THE VISCOMETER OR ANY OF ITS ACCESSORIES**

### General Safety

General safety precautions must be followed when using this LLOYD INSTRUMENTS product. Failure to observe precautions and warnings may result in damage to the equipment, or injury to personnel.

It is understood that safety rules within companies vary. If a conflict exists between the material contained in all LLOYD INSTRUMENTS' User's Guides and the rules of a company using a DAVENPORT product, the more stringent rules should take precedence.

### Safety Considerations

The PET Plus is completely enclosed and provides no potentially hazardous outputs. Safety considerations are related to the power connections and physical mountings.

Electronic and mechanical components housed within the PET Plus covers are to be serviced by authorized DAVENPORT representatives only.



MEASUREMENT & CALIBRATION TECHNOLOGIES DIVISION

## EUROPEAN DECLARATION OF CONFORMITY

### NAME, ADDRESS OF MANUFACTURER AND LOCATION OF TECHNICAL CONSTRUCTION FILE

Ametek, Inc.  
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Largo, FL 33773, USA, PHONE : (727) 538-6084, FAX : (727) 535-4391

### DESCRIPTION OF EQUIPMENT

Product: Ametek PET Polymer Measurement Instrument  
Models: LLOYD: PET-Plus 01/3440 and PET-Plus 01/3480



Effective Date: June 2014

### HARMONIZED STANDARDS USED

A sample of this product has been assessed against the essential health and safety requirements of the Low Voltage and the EMC Directives listed below. Based on conformity with the listed directives, Ametek, Inc. declares the products mentioned above are in compliance with the following:

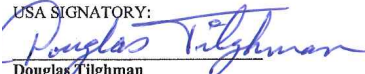
EMC Directive 2004/108/EC and Machinery Directive 2006/42/EC

BS EN 61010-1: 2010 Safety Requirement for Electrical Equipment

BS EN 61326-1: 2006 Electrical Equipment for Measurement, Control and Laboratory use:  
General Requirements

This declaration of conformity is the result of testing and evaluation performed by Ametek, Inc. and approved CE testing laboratories. LLOYD is a brand name owned by Ametek, Inc.

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**DESCRIPTION OF EQUIPMENT**

Product: Ametek Polymer Dryer Device  
Models: LLOYD: 01/2776 & 01/2777 Polymer Dryer



Effective Date: November 14, 2014

**HARMONIZED STANDARDS USED**

A sample of this product has been assessed against the essential health and safety requirements of the Low Voltage and the EMC Directives listed below. Based on conformity with the listed directives, Ametek, Inc. declares the products mentioned above are in compliance with the following:

BS EN 61010-1: 2010	Safety Requirement for Electrical Equipment
BS EN 61326-1: 2013	Electrical Equipment for Measurement, Control and Laboratory use: General Requirements

This declaration of conformity is the result of testing and evaluation performed by Ametek, Inc. and Product Safety Engineering. LLOYD is a brand name owned by Ametek, Inc.

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Certificate Number: 1518-01

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## 1.0 INTRODUCTION TO THE PET PLUS VISCOMETER

### 1.1 GENERAL

The PET Plus is a microprocessor controlled, capillary Melt Viscometer that is specifically designed to test the flow properties of PET. The system can be used to test other polymers and because it does not require solvents, it can also be used to test insoluble polymers.

It consists of a heated vertical barrel, a calibrated die, a small piston called a "follower", a displacement transducer called a "probe" and control systems for the nitrogen gas flow and extruder temperature.

The equipment is based on a system developed by ICI. It is designed for both research and quality control of polyethylene terephthalate (PET), which is hygroscopic and liable to degrade due to oxidation in the melt phase. The apparatus is recommended for use in the quality control of polyester polymers to measure the Intrinsic Viscosity and degradation rates.

The Viscometer extrudes molten PET similar to an MFI but is specially developed to prevent the polymer from degrading due to contact with the atmosphere. The polymer is isolated from moisture and oxygen because the barrel is sealed and the polymer is extruded through the die using pure nitrogen at high pressure.

Therefore, an external supply of Nitrogen gas of 30 Bar pressure is required. This nitrogen gas not only provides an oxygen and moisture free environment for the sample but also applies the extrusion force.

The quantity of polymer extruded is calculated similar to the MFI method B using an LVDT "probe" to measure the movement of the "follower".

The rate of extrusion is accurately measured by the "probe", which is designed to operate under the high-pressure conditions within the barrel during extrusion.

The system calculates the total thermal and hydrolytic degradation.

Therefore, a "Melt" test can show the degradation

due to the presence of moisture in the polymer so can be used to check the performance of the production drying system. A "Melt" test can also be used to measure the thermal degradation factor caused by the production extruder.

PET has historically been measured using the solution method and this indicates the POTENTIAL Intrinsic Viscosity (IV) of the polymer but does not show how the polymer will behave or degrade during extrusion.

The solution technique is being phased out due to the insoluble nature of highly crystalline polymer plus high environmental and disposal costs of solutions. It is also not so suitable for testing reground or recycled material because the quantity tested is very small and may not show the "Average IV" of the mixed granules.

The PET Plus is designed to replace the solution method so directly reports the Intrinsic Viscosity (IV) of the polymer and not the Melt Flow Rate.

PET is hygroscopic and any water molecules present at melt temperatures > 250C will give an irreversible break in the chain at the ester linkage, which gives a drop in the IV.

Therefore, the PET Plus can ONLY give the same IV as the solution method if ALL the moisture is removed from the polymer before it is tested (less than 20ppm moisture).

The PET Plus is a semi-automatic system so can be left unattended after the sample has been loaded and pre-conditioned. The test takes a total of 17 minutes after loading the sample.



## 1.2 EQUIPMENT

The equipment is fitted with a control console containing a blue 4 line by 40-character backlit LCD display and a membrane keypad. The LCD can display the test set-up, test procedure and test results in various languages as selected by the user.

The equipment can be used stand-alone or can be connected to a PC running the optional NEXYGEN Plus software.

When a test is performed in the stand-alone mode, the test results are stored using a Date-Time ID that is created from the internal clock inside the equipment.

This internal clock has been set during the production process at Davenport / Lloyd Instruments Ltd but should be checked during the initial installation. Note that if the equipment is used with the NEXYGEN Plus PET software, then the internal clock will be automatically synchronised to the PC clock when the software is connected to the equipment.

The internal clock always operates on UTC (GMT) time so the time displayed on the control console should be altered if the local time is not UTC, e.g. British Summer Time or if the equipment is used in a country other than the UK.

The equipment can store up to 600 test results internally and the statistics and results can be easily viewed on the control console display. If 600 tests are already stored, then when the next test is performed, the first test will be deleted to allow the new test to be stored. This feature allows the last 600 test results to be stored.

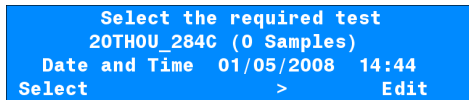
There are connection points on the cabinet for the nitrogen gas supply and the optional computer.

A fused switched socket is fitted for connection to the mains electricity supply.

Ancillary equipment developed for use with the PET Plus to handle the grinding, drying and transfer of samples is available as either a package or as individual items.

## 1.3 EQUIPMENT OPERATION

When the equipment is first switched on, the "Select Test" screen is displayed as shown below.



The screenshot shows a blue LCD screen with white text. The text is arranged as follows:  
Line 1: Select the required test  
Line 2: 20TH0U\_284C (0 Samples)  
Line 3: Date and Time 01/05/2008 14:44  
Line 4: Select > Edit

When this "Select Test" screen is initially displayed, the heaters and nitrogen gas valves are automatically switched off. The heaters will operate when any of the test set-ups are selected by pressing the key under SELECT.

However, note that there is a special test set-up called TRAINING and the heaters do NOT operate when this set-up is selected.

The nitrogen gas valves are can only be operated when the relevant screens are displayed and the gas valves are automatically switched OFF when any other screen is displayed.

## 1.4 EQUIPMENT USE

The “Incoming” or “Supplied” Resin can be checked before acceptance of delivery.

The “Dried” resin can be checked daily from the drying hopper to check the efficiency of the drying heaters and / or drying desiccant.

The preforms can be checked before and after storage to monitor the IV drop in the extruder and also to check the suitability of the storage area (humid storage conditions will reduce the IV of the preform and cause problems during the blow moulding stage).

Note that the drier and preforms CANNOT be checked using the solution method because this checks the IV of the polymer and NOT the changes in IV due to the production process.

The PET Plus also reports the Thermal Degradation Factor, which is a combination of the Thermal Degradation Rate and the Hydrolytic Degradation Rate.

It is found in practice that the IV drop from polymer chip to preform is usually in the range of 0.02 to 0.03 IV.

## 1.5 MEASUREMENT RANGE

The IV measurement range is approximately 0.45 to 1.40 using dies from 0.015 in (0.0381cm) to 0.040 in (0.1916cm).

Every die is machined to be 0.5 in (1.27cm) long and has a very accurately defined bore. Each die is supplied with a calibration certificate, which states the exact size of the die, and these values are to be entered into the test parameters.

The software calculates Intrinsic Viscosity (IV) and the degradation rate according to the ICI algorithm for PET.

Although primarily designed to test hygroscopic polymers in an oxygen and water free environment, the flow properties, viscosity and degradation of most polymers may be tested.

The range of IVs that can be measured using the available dies is shown below:

Die Size	Die Internal Diameter (cm)	IV Range at 284°C	IV Range at 295°C
15 thou	0.0381	0.43 to 0.62	NA
20 thou	0.0508	0.53 to 0.70	0.70 to 0.83
30 thou	0.0762	NA	0.74 to 1.14
40 thou	1.1016	NA	0.93 to 1.43

## **1.6 PET PLUS ADVANTAGES**

1. Avoids handling toxic chemicals, which are not only expensive but are costly to dispose of.
2. Crystalline samples can easily be measured - as crystallinity INCREASES solubility DECREASES.
3. Samples can be taken directly from the plant to measure: -
  - IV of incoming raw material.
  - IV of regrind/virgin polymer mixture.
  - Drier Efficiency
  - Drop of IV in extruder.
  - Target value for moisture determination.
  - Melt stability/degradation rate.
  - IV in finished product / re-grind.
4. Relatively easy test to operate - does not need skilled laboratory staff, so shop floor staff can easily be trained to obtain reliable and consistent results.
5. Saves time - quicker than the solvent method - results can be obtained in twenty minutes.
6. Saves money - after initial cost of equipment and accessories, electricity and nitrogen gas are the only on-going costs.

## 2.0 EQUIPMENT AND ACCESSORIES

### 2.1 PET PLUS AND ACCESSORIES

The PET Plus is supplied with the following tools and accessories.



1. Top Blanking Nut and PTFE Sealing Washer (p/n 778/98 & 778/99)



Used during die cleaning and pressure testing.



2. Follower (p/n 750/45)



Follows the movement of the polymer down the barrel during extrusion.



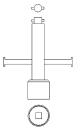
3. Contraction Taper Tool (part of p/n 750/41)

Used to fit a new "O" ring to the die.



4. Expansion Taper Tool (part of p/n 750/41)

Used to fit a new "O" ring to the die.



5. Tightening Tool (p/n 778/35)

Used to fit and remove the die retaining nut, top blanking nut and bottom blanking plug.



6. Barrel Cleaning Tool (p/n 778/55)

Used to charge the sample, push a cleaning cloth down the barrel and for die "O" ring fitting.



7. Die Retaining Nut and Fibre Sealing Washer (p/n 778/53 & 778/54)

Secures the die in the barrel.



8. Die and PTFE O Ring (p/n 778/82 & 750/1019)

Certified die used for extrusion testing.



Shown: PET PLUS with Probe attached



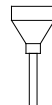
9. Bottom Blanking Plug and PTFE Sealing Washer (p/n 778/51 & 778/52)

Used during pressure testing.



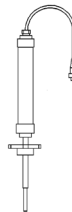
10. Standard Filling Funnel (p/n 778/50)

Used for loading dry granules into the barrel.



11. Long Filling Funnel (p/n FND/0114/00)

Used for loading dry powder into the barrel.



12. Probe, Probe Sealing Washer and Connecting Cable (p/n 750/048 & 750/76 & 09/0980).

Displacement transducer used to measure the movement of the follower to calculate the flow rate.

13. Barrel Cleaning Brush (p/n 750/1020)

Used for cleaning a badly crusted barrel.

14. Coiled Nitrogen Connecting Hose  
(p/n 778/31)

Used to connect the PET Plus to the nitrogen supply.

15. Thermometer 283°C - 287°C  
(p/n 338/104/16)

Used when calibrating to 284°C.

16. Thermometer 293°C - 297°C  
(p/n 338/104/1032)

Used when calibrating to 295°C.

17. Tweezers (p/n 92/0404)

Used to hold hot die and followers.

18. RS232 Cable (p/n 09/0639)

## 2.2 CONSUMABLE ITEMS

The PET Plus is supplied with the following consumable items.

QTY	Description	Part No.
4	Box of Cleaning Patches	338/132
50	Die "O" Ring	750/1019
5	Probe Sealing Washer	778/67
5	Plug Sealing Washer	778/52
5	Die Nut Washer	778/54
12	Follower	750/45
5	Top Cap Sealing Washer	778/99

## 2.3 POLYMER DRYER AND ACCESSORIES

The Polymer Drier is an optional item and is used to dry PET samples before testing to measure the POTENTIAL IV.

It MUST be used with a high quality vacuum pump that can achieve a very good vacuum of better than 0.1mm Hg and Davenport recommend the BOC Edwards RV rotary vane pump.

The Polymer Drier is supplied with the following accessories.

- 1 Sample Phials (p/n 757/14) Quantity 6.

Used to hold PET granules or powder when drying in the polymer drier.

- 2 Vacuum Taps and Hose Quantity 6.



This assembly is used to connect the phials to the polymer drier.

It is also used to isolate the phials from the vacuum line at the end of the drying time.

- 3 Hose for Vacuum Pump (p/n 468/27) Quantity 1

Used to connect the polymer Drier to the Vacuum Pump.

## 2.4 VACUUM PUMP

The Vacuum Pump is an optional item and is used together with the Polymer Drier to dry PET samples before testing to measure the POTENTIAL IV.

A high quality vacuum pump that can achieve a very good vacuum of better than 0.1mm Hg MUST be used.

AMETEK recommends the BOC Edwards RV rotary vane pump, which is shown above.



*Shown: Polymer Drier*



*Shown: Vacuum pump with oil mist filter attached.*

## 2.5 POLYMER CHIP TRANSFER VESSEL

The Polymer Chip Transfer Vessel is an optional item and is used to collect dried polymer from the production hopper to check the IV of the polymer before it is extruded.

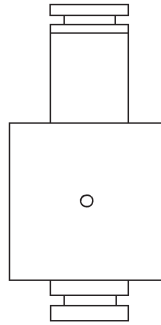


*Shown: Polymer chip transfer vessel*

## 2.6 SAMPLE MELT POT

The Sample Melt Pot is an optional item and is used to collect molten polymer from the production extruder to check the IV of the polymer during the extrusion process.

It is mainly used to collect a sample from a textile spinneret and is not usually used when making preforms etc.



*Shown: Sample melt pot illustration*

## 2.7 POLYMER CHIP GRINDER

The Polymer Chip Grinder is an optional item and is used to grind polymer chips to a powder to allow a much faster drying time.

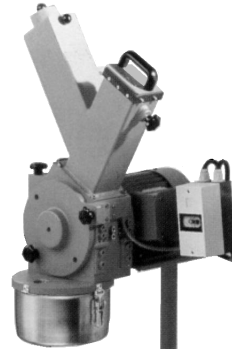


*Shown: Polymer chip grinder*

## 2.8 INDUSTRIAL GRINDER

The Industrial Grinder is an optional item and is used to grind pre-forms or other finished products to produce polymer chips.

AMETEK recommends the Fritsch Pulverisette 15.



Shown: Industrial grinder

## 2.9 EQUIPMENT AND MATERIAL REQUIRED (Not Supplied by AMETEK)

### 2.9.1 Nitrogen Supply

1. Nitrogen gas supply with a water content of less than 5ppm (parts per million), oxygen free, 'white spot' specification.
2. The supply is to be fitted with an on/off gas tap and a regulator that can be set to 30 bar (440 psi).

### 2.9.2 Safety Equipment

1. Cut resistant gloves made of DYNEEMA material. Useful when removing O Ring from die or scraping polymer off die or follower.
2. Heat resistant gloves.
3. Safety glasses.

### 2.9.3 Hot Plate

To aid cleaning of follower, put the dirty followers on the hot plate, PET will carbonate and be easier to remove.

### 2.9.4 PTFE Tape

To seal gas connection joints.

### 2.9.5 Razor Blade, Scalpel

To scrape molten polymer off dies and followers.

### 2.9.6 Scotchbrite Pad

To polish followers and dies until shiny.

## 2.10 OPTIONAL SPARE ITEMS

The following optional spares items are available.

QTY	Description	Part No.
1	Probe Rod	750/1042
1	Probe Calibration Tool	750/35
1	Probe Repair Kit	778/103
1	Chip Transfer Vessel Repair Kit	MT/CON/CTV

## 2.11 OPTIONAL EQUIPMENT (Not Supplied by AMETEK)

### COMPUTER

A computer with the minimum specification as stated below will be required if the optional NEXYGEN™ Plus software is to be used.

- 2GHz Pentium® III or better
- RAM 256MB or more
- Monitor resolution 1024 x 768 or higher
- CD ROM drive
- Unused COM Port with a 16550 UART
- Microsoft® Office 2000 or later for reports



### 3.0 INSTALLATION



**CAUTION:** The Viscometer and Polymer Grinder are heavy instruments. Great care must be taken when lifting the equipment, EMPLOY SAFE LIFTING PRACTICES. Use lifting equipment and/or two persons to lift the equipment into position. Do not position hands or fingers so that they may become trapped.

### 3.1 CONSIDERATIONS

Consideration should be given to the following before starting to set up the supplied equipment.

1. The location and strength of the bench where the equipment is to be used. The bench must be strong enough to support the equipment without deforming and be solid enough not to vibrate. Consider that:
  - The Viscometer weighs 34Kg
  - The Polymer Grinder weighs 41Kg.
  - The Polymer Dryer weighs 15Kg.
  - The weight of the optional computer system

Plus, when cleaning the barrel of the Viscometer a large proportion of the operator's weight may be applied to the top of the barrel via the cleaning tools.

2. The equipment should only be used in a well-ventilated environment, however the area needs to have a stable temperature and be draught free.
3. The Viscometer requires an Oxygen free Nitrogen supply with <5ppm of water (White Spot). The Polymer Grinder requires a compressed air supply capable of delivering 16cfm (0.45m<sup>3</sup>/min) at 80psi (5.5 Bar). The Polymer Dryer requires a vacuum line capable of maintaining a 0.1mm Hg vacuum.

### 3.2 SAFETY CONSIDERATIONS

- 1 Before connecting the equipment to the mains supply make sure that it is marked with the voltage of the power supply to be used. The Viscometer and its accessories are not adjustable for different supply voltages and must only be used on the supply that they were originally ordered for.
- 2 There are 2 standard versions, one for 230V ac  $\pm 10\%$  and the other for 115V ac  $\pm 10\%$ .
- 3 This equipment complies with electrical safety grade Class 1, which means that it is earthed apparatus and requires the mains plug to contain a protective earth terminal. The mains plug shall only be inserted in a socket outlet provided with a protective earth contact. The protective action must not be negated, e.g. by the use of an extension cord without a protective conductor.

### 3.3 SITTING THE EQUIPMENT

The Viscometer should be situated within two metres of both electrical and nitrogen gas supplies.

The polymer grinder must be situated as close as possible to the vacuum pump if this drying option is used. The polymer grinder is operated by compressed air so should be situated close to a suitable air line if this grinding option is used.

The industrial grinder (cutting mill) can be noisy in operation so this may need to be sited outside of the testing laboratory.

The polymer grinder and industrial grinder require liquid nitrogen and this should be handled with care and the user should wear protective gloves because of the danger of cold burns (this has a temperature of  $-196^{\circ}\text{C}$ ).

### 3.4 CONNECTING THE PET PLUS TO THE MAINS

Connect the mains supply lead to the switched and fused input connector on the rear of the vertical column. The mains switch is marked O and I. The switch is ON when the I end is pressed and OFF when the O end is pressed.

Only connect to the supply specified on the Serial Number nameplate situated under the mains input connector.



*Shown: PET Plus On/Off Switch*

### 3.5 CONNECTING THE NITROGEN GAS SUPPLY

Connect the viscometer to a 30 Bar nitrogen supply via a gas regulator and / or gas tap. Turn off the gas supply then connect the flexible inter-connection tube between the regulator / tap and the inlet connector on the left hand side of the Viscometer base.

The connections must be firmly tightened to ensure that they are gas tight.

Check for gas leaks around the inlet connection by:

- Obvious hissing
- Soap solution around the joints.

If hissing is heard or a leak is shown up by soap solution then gas will escape allowing a drop in pressure. A leak can usually be cured by using PTFE tape on the leaking connection and then re-connecting and tightening using a spanner.



*Shown: Nitrogen supply connection*



**WARNING:** Never subject the viscometer, the connecting hose or any of the accessories to pressures greater than 35 Bar.

Have your gas regulator checked at regular intervals.



**NOTE:** The Viscometer has an overpressure detector that disconnects the Nitrogen Supply if a pressure exceeding 35 Bar is inadvertently applied. Should this detector be activated; it can only be re-set by reducing the pressure then switching the viscometer off then on again.

### 3.6 CONNECTING THE PROBE

The probe is connected to the socket on the top rear of the vertical column.

Store the probe in the probe holder at the right hand side of the vertical column. Ensure that the body of the probe rests onto the probe holder so that the large knurled nut is below the holder.



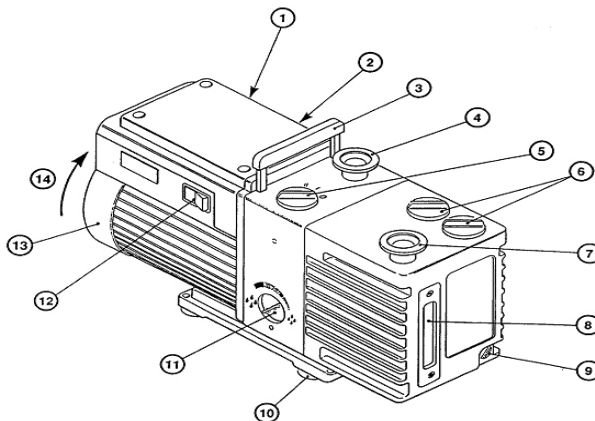
Shown: Probe connection

### 3.7 VACUUM PUMP

The recommended pump is a BOC Edwards RV rotary vane pump, which is a two-stage, oil-sealed, sliding-vane vacuum pump. The pump has a NW25 inlet port (item 4) and a NW25 outlet port (item 7), a gas ballast control (item 5) and a mode selector (item 11). When the pump is switched off, an inlet-valve seals the inlet and prevents the suck-back of air and oil into the vacuum system.

An internal oil pump delivers pressurised oil to the vacuum pumping mechanism in the RV pump. The level and condition of the oil can be viewed through a sight glass (item 8). Two oil filler-plugs (item 6) and an oil drain-plug (item 9) are provided on the oil-box.

The motor is fitted with an on/off switch (item 12) and an internal thermal overload device. When the motor is too hot, the thermal overload device switches off the pump. The thermal overload device has an automatic reset and when the motor cools down, the device resets.



- |                               |                          |                                   |
|-------------------------------|--------------------------|-----------------------------------|
| 1. Electrical inlet-connector | 4. NW25 inlet-port       | 10. Rubber feet                   |
| 2. Voltage indicator          | 5. Gas-ballast control   | 11. Mode selector                 |
| 3. Lifting handle             | 6. Oil filler-plug       | 12. On/off switch                 |
|                               | 7. NW25 outlet-port      |                                   |
|                               | 8. Oil-level sight-glass | 13. Motor fan-cover               |
|                               | 9. Oil drain-plug        | 14. Correct direction of rotation |

### **3.8 FILLING THE PUMP WITH OIL**

The recommended oil is Edwards Ultragrade 19 (hydrocarbon oil). The maximum volume is 0.7 litres and the minimum level is 0.42 litres.

Remove one of the oil filler plugs (item 6) then pour the oil into the pump until the oil level just reaches the MAX mark on the bezel at the top of the sight glass (item 8). If the oil level goes above the MAX mark, remove the drain plug (item 9) and drain the excess oil from the pump.

After a few minutes, recheck the oil level and top up if necessary. Refit the oil filler plug and tighten firmly by hand but do not over tighten.

### **3.9 FITTING THE OIL MIST FILTER TO THE PUMP**

Unscrew the red thumb nut from the bolt in the securing clamp then remove the through bolt.

Place the rubber seal washer over the top of the outlet port (item 7).

Remove the caps from both ends of the oil mist filter then place the oil mist filter on top of the washer.

Fit the clamp around the boss on the outlet port and the boss on the bottom of the oil mist filter.

Refit the through bolt and red thumb nut to the securing clamp. Hand tighten the red thumb nut to secure the filter.

### **3.10 CONNECTING THE VACUUM HOSE TO THE PUMP**

Unscrew the red thumb nut from the bolt in the securing clamp then remove the through bolt.

Place the special rubber seal filter washer over the top of the inlet port (item 4) noting that the conical filter must be positioned downwards into the port.

Place the hose connector on top of the filter washer.

Fit the clamp around the boss on the outlet port and the boss on the hose connector.

Refit the through bolt and red thumb nut to the securing clamp.

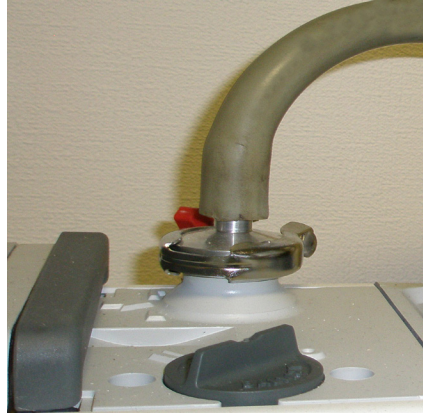
Hand tighten the red thumb nut to secure the connector.

### 3.11 CONNECTING THE PUMP TO THE DRIER BLOCK

Push the large diameter rubber hose over the hose connector on the inlet port of the pump.

Note that an additional clamp is not required because the vacuum will secure the hose to the hose connector.

Push the other end of the rubber hose over the hose connector on the rear of the drier block.



*Shown: Connection at the pump*



*Shown: Connection at the drier block*

### 3.12 CONNECTING THE DRIER BLOCK TO THE MAINS

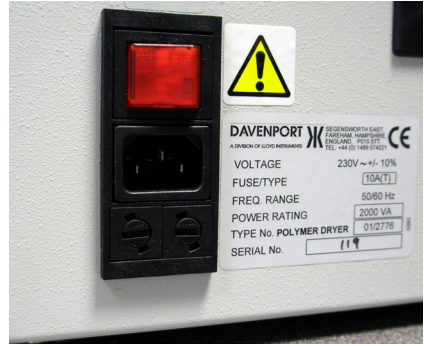
Connect the mains supply lead to the switched and fused input connector on the side of the drier block. The mains switch has an internal lamp and the equipment is powered when the lamp is lit.

Only connect to the supply specified on the Serial Number nameplate situated alongside the mains input connector.

The front panel of the drier block also has an indicator lamp to show when the equipment is powered.

The installer shall not position the equipment so that it is difficult to access the disconnect device.

The power cord shall be replaced only by an adequately rated power supply cord.



*Shown: Mains input connector*



*Shown: Front panel on drier block*



### 3.13 CONNECTING THE PUMP TO THE MAINS

The pump is powered from the drier block for ease of use. Connect the special lead between the mains input connector on the rear of the pump (item 1) to the mains outlet on the rear of the drier block.

The pump is switched on and off by the Vacuum Power switch on the front panel of the drier block.

This switch is marked O and I. The switch is ON when the I end is pressed and OFF when the O end is pressed.

The switch also has an indicator lamp to show when the pump is operating.



*Shown: Rear of pump*



*Shown: Rear of drier*



*Shown: Front panel on drier block*

### 3.14 DE-GASSING THE OIL

The pump must be run for at least 1 hour (preferably overnight) to remove any traces of gas and to thoroughly purge the oil of contaminants.

This procedure **MUST** be carried out to achieve maximum vacuum.

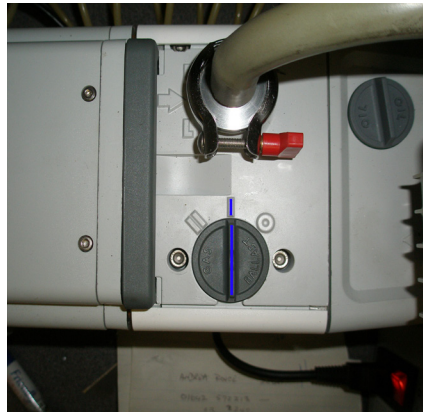
Rotate the mode selector (item 11) fully anti-clockwise to select the High Throughput mode (Large Pear Drop). Note that this control may be difficult to rotate.

Set the gas ballast control (item 5) to low flow (position '1').

Operate the pump for at least 1 hour (or overnight) to thoroughly purge the oil of contaminants.



*Shown: Turn the mode selector fully counter clockwise*



*Shown: Set the gas ballast control to "1"*

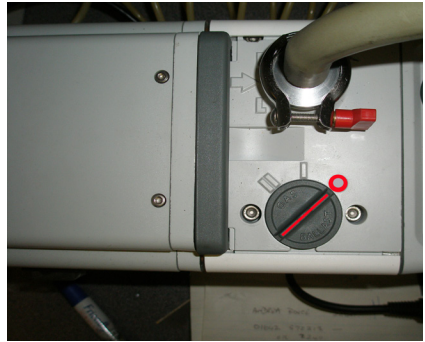
### 3.15 SETTING THE PUMP TO MAXIMUM VACUUM

This setting **MUST** be used to achieve maximum vacuum.

Rotate the mode selector fully clockwise to select the High Vacuum mode (Small Pear Drop).



*Shown: Rotate mode selector fully clockwise for High Vacuum mode*



*Shown: Set the gas ballast control to OFF (position "O")*

### 3.16 POLYMER CHIP GRINDER

Place the grinder within easy reach of a compressed air supply capable of providing 16cfm (0.45cmm) at 80psi (5.5 Bar).

Connect the air supply to the inlet connection at the rear of the grinder using the supplied plastic hose.

Unscrew the oil filler cap then fill the lubricator with the correct grade of oil, e.g. Shell Morlina 10 or Tellus R10.

The top of the oil must be visible anywhere inside the sight glass at the front of the lubricator.

Refit the oil filler cap after adding the oil.



Shown: Polymer Chip Grinder



Shown: Inlet connectin at rear of grinder



Shown: Oil filler location

Although the unit does not use mains power, it must be connected to a good earth point to prevent any build up of dangerous static charges. Therefore, an additional earth wire must be connected to the unit.

Note that an earth wire is not supplied with the equipment and this has to be provided by the user.

To operate the grinder, close the lid and fasten with the clip then push the start knob at the front of the unit.

When the grinder is running, check that the pressure gauge on the left hand side of the unit shows 80psi (5.5 Bar).

Note that the grinder stops automatically after 30 seconds so the grinder may need restarting during this procedure.

If the pressure gauge does not show the correct pressure when the unit is running, pull the orange knob downwards then rotate the knob to achieve the correct pressure.

Push the knob upwards again after adjustment to lock the regulator setting.

The grinder blade is driven by an air motor and this is lubricated by an oil mist carried by the compressed air.

A special oil lubricator is fitted to the air manifold assembly and this supplies oil drops into the air flow.

When the grinder is running, view the glass tube above the air manifold assembly and check the speed of the oil drops.

The required speed is approximately 1 drop per minute.

If the oil drop rate is not correct, adjust the oil drop regulator screw, which is on top of the glass tube.

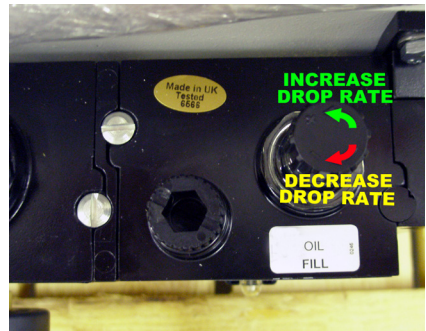
Note that the grinder stops automatically after 30 seconds so the grinder will need restarting several times during this procedure.



Shown: Pressure control gauge



Shown: Check oil drop rate

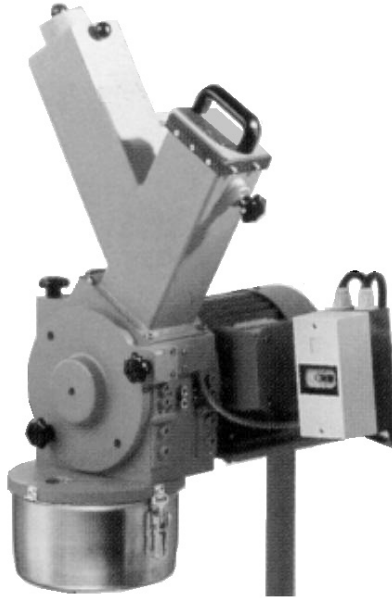


Shown: Adjustment for oil drop rate

### 3.17 INDUSTRIAL GRINDER

AMETEK recommends the Fritsch Pulverisette 15.

This equipment connects to the mains using a standard mains lead.



*Shown: Fritsch Pulverisette 15*

## 4.0 SETTING UP THE VISCOMETER

### 4.1 INITIAL CONFIGURATION

Switch the equipment on to display the “sign-on” screen. This screen indicates the embedded version number, the equipment serial number and the probe serial number.

```
Davenport
PET Plus S/No 105102
Version 1.0 Issue 7
Probe S/No 105102
```

The “Select Test” screen will be automatically displayed after 3 seconds. This screen is used to select one of the pre-defined test set-ups as shown later.

```
Select the required test
20THOU_284C (0 Samples)
Date and Time 01/05/2008 14:44
Select > Edit
```

Press the key under EDIT to set the date, time and test result format.

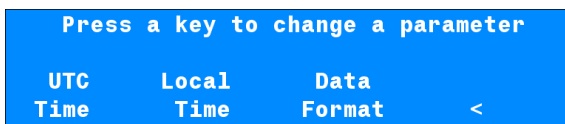
```
Press a key to select an option
20THOU_284C
Rename Global Delete Setup
```

Press the key under GLOBAL to display the first “Global Options” screen.

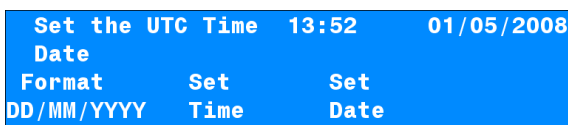
```
Press a key to change a parameter
Change Current
Password Language Calibrate
<None> English Mode >
```

The required language can be changed by repeatedly pressing the key under CURRENT LANGUAGE.

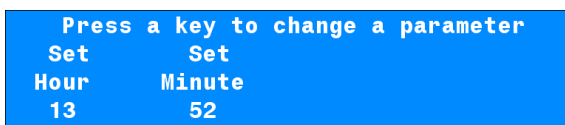
Press the key under > to display the second “Global Options” screen.



Press the key under UTC TIME.



To change the Time, press the key under SET TIME.



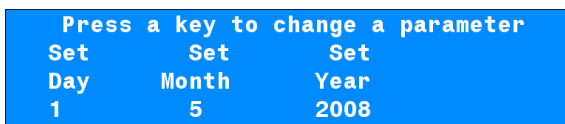
To change the Hour, press the key under SET HOUR.

To change the Minute, press the key under SET MINUTE.

Ensure that the time is set to UTC (GMT) and NOT to the LOCAL TIME.

Press the ENTER key to return to the UTC TIME screen.

To change the Date, press the key under SET DATE.



To change the Day, press the key under SET DAY.

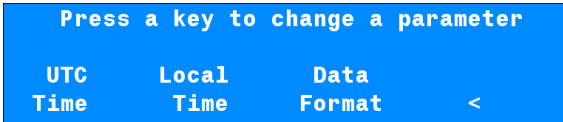
To change the Month, press the key under SET MONTH.

To change the Year, press the key under SET YEAR.

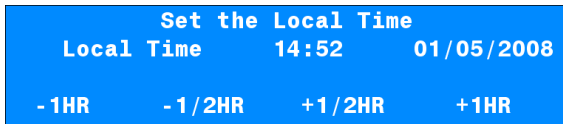
Press the ENTER key to return to the UTC TIME screen.



Press the ENTER key to return to the second “Global Options” screen.

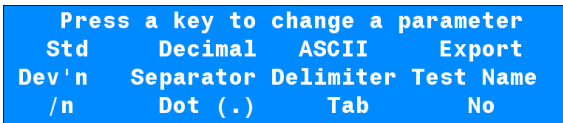


To set the difference between Local Time and UTC, press the key under LOCAL TIME.



Press the relevant keys to set the time offset then press the ENTER key.

Press the key under DATA FORMAT.



Each of the options toggle when the relevant key is pressed:

The STD DEV'N has options of /n and /(n-1).

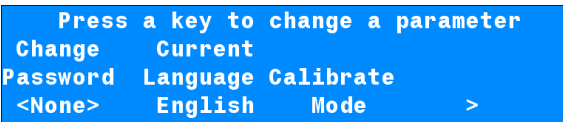
The Decimal Separator has options of Dot and Comma.

The ASCII Delimiter has options of Tab and Comma.

The Export Test Name has options of Yes and No.

Select the required options then press the ENTER key to return to the second “Global Options” screen.

Press the key under < to display the first “Global Options” screen.



#### 4.2 OVER TEMPERATURE DEVICE

A secondary temperature measuring device is fitted to protect the equipment should the main temperature measuring device fail for any reason. The temperature at which it is activated is pre-set at 400°C.

#### 4.3 SETTING THE TEST TEMPERATURE

The test temperature is selected using the relevant test set-up screen.

#### 4.4 WHICH TEST TEMPERATURE TO USE

Generally, samples of IV up to 0.70 should be tested at 284°C and IVs over 0.70 at 295°C.

##### Selecting Melt Temperature

Up to 0.7 IV	284°C
Above 0.7 IV	295°C

#### 4.5 WHICH DIE SIZE TO USE

Note that one die is supplied with the equipment (size specified at time or order) but additional dies may be purchased as required.

Die Size	cm	IV at 284°C	IV at 295°C
15 thous	0.0381	0.43 to 0.62	NA
20 thous	0.0508	0.53 to 0.70	0.70 to 0.83
30 thous	0.0762	NA	0.74 to 1.14
40 thous	0.1016	NA	0.93 to 1.43

## 5.0 CREATING A TEST SETUP

### 5.1 DEFAULT TEST SETUPS

The equipment is supplied with 4 pre-defined test set-ups called:

- 284\_20THOU
- 295\_20THOU
- 295\_30THOU
- TRAINING

The first test set-up is preset to give a barrel temperature of 284°C but the other 3 are preset to give a barrel temperature of 295°C.

The 295\_30THOU test set-up is preset for a 30 thou die (0.0762cm) but the other 3 are preset for a 20 thou die (0.0508cm).

The test set-up called TRAINING sets the equipment into a special training mode and does NOT perform a melt test.

Note that the special training mode is only available if the test set-up is called TRAINING.

Each test set-up contains separate results storage so that the statistics for each type of test can be easily viewed, so a different test set-up should be used for:

- A different polymer
- A different production drier or extruder

Therefore, a different test set-up should be used to store the test data for (say):

- Incoming resin
- Production Drier number 1
- Production Drier number 2 (etc)

## 5.2 RENAMING AN EXISTING TEST SETUP

Display the "Select Test" screen to select one of the pre-defined test set-ups.

```
Select the required test
20THOU_284C (0 Samples)
Date and Time 01/05/2008 14:44
Select      >      Edit
```

Press the key under > or < to select the test set-up that is to be renamed.

When the required test set-up is displayed, press the key under EDIT to display the edit test screen.

```
Press a key to select an option
20THOU_284C
Rename      Global      Delete      Setup
```

Press the key under RENAME to display the rename test screen.

```
Rename Test 20THOU_284C
                20THOU_284C
BkSp                      Clear All
```

Press the key under CLEAR ALL to delete the existing name then enter the required name using the keypad.

This name could be a supplier name or a production ID such as drier identification.

Press the ENTER key to return to the edit test screen.

### 5.3 CREATING A NEW TEST SETUP

Display the "Select Test" screen.

```
      Select the required test
      20THOU_284C (0 Samples)
      Date and Time 01/05/2008 14:44
      Select      >      Edit
```

Press the key under > one or more times to select NEW\_TEST.

```
      Select the required test
      NEW_TEST (0 Samples)
      Date and Time 29/05/2008 13:09
      <      Create
```

When NEW\_TEST is displayed, press the key under CREATE to display the edit test screen.

```
      Press a key to select an option
      NEW_TEST
      Rename      Global      Delete      Setup
```

Press the key under RENAME to display the rename test screen.

```
      Rename Test NEW_TEST
      NEW_TEST
      BkSp      Clear All
```

Press the key under CLEAR ALL to delete NEW\_TEST then enter the required name using the keypad.

This name could be a supplier name or a production ID such as drier identification.

Press the ENTER key to return to the edit test screen.

## 5.4 DEFINING A TEST SETUP

Display the "Select Test" screen.

```
Select the required test
20THOU_284C (0 Samples)
Date and Time 01/05/2008 14:44
Select      >      Edit
```

Press the key under > or < to select the test set-up that is to be defined then press the key under EDIT to display the edit test screen.

```
Press a key to select an option
20THOU_284C
Rename      Global      Delete      Setup
```

Press the key under SETUP to display the first "Set-up" screen.

```
Press a key to change a parameter
Set      Die      Die
Temp     Length  Diameter
284      1.276   0.0508   >
```

The test temperature is generally 284°C for polymers having an IV below 0.7 and 295°C for polymers having an IV above 0.7 IV.

To change the test temperature, press the key under SET TEMP then enter the required temperature (in degrees C).

The die length (cm) and die diameter (cm) are shown on the die certificate and this should be referred to for this information.

To change a die dimension, press the key under DIE LENGTH or DIE DIAMETER then enter the required values (in cm).

```
Enter the required Pass/Fail limits
Upper      Lower
IV         IV
0.00      0.00      <
```

This screen is used to define the pass/fail feature. At the end of every test, the result screen will display the calculated IV and can also display the pass/fail status.

If a pass/fail indication is not required, then the Upper IV and Lower IV should be set to zero. If a pass/fail indication is required, then one or both of the IV values should be defined.

Note that if only the Lower IV is specified then the sample is considered to have passed if the calculated IV is above the Lower IV and no Upper IV is required.

Also note that if only the Upper IV is specified then the sample is considered to have passed if the calculated IV is below the Upper IV and no Lower IV is required.

Most polymers are specified with a tolerance of +/- 0.02 so a polymer with a nominal 0.8 IV could have an Upper IV specified as 0.82 and a Lower IV specified as 0.78.

```
Enter the required Pass/Fail limits
Upper      Lower
  IV       IV
  0.82     0.78      <
```

When the test parameters are correct, press the ENTER key to return to the edit test screen.

### 5.5 PASSWORD PROTECTION

The test parameters can be password protected to prevent unauthorised changes by non-technical personnel. This feature also prevents any stored test data from being erased.

Display the "Select Test" screen.

```
Select the required test
20THOU_284C (0 Samples)
Date and Time 01/05/2008 14:44
Select      >      Edit
```

Press the key under EDIT to display the edit test screen.

```
Press a key to select an option
20THOU_284C
Rename      Global      Delete      Setup
```

Press the key under GLOBAL to display the first "Global Options" screen.

```
Press a key to change a parameter
Change      Current
Password    Language Calibrate
<None>     English   Mode      >
```

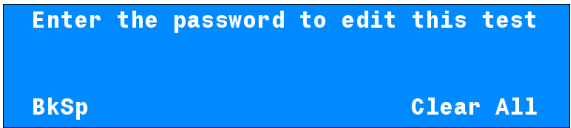
Press the key under CHANGE PASSWORD to display the password entry screen.

```
Change or create the password
BkSp      Clear All
```

Enter the required password then press the ENTER key.

Please make a note of the password because the test set-ups cannot be changed without entering the password.

If a password has been defined, then the following screen will be displayed if the key under EDIT is pressed when the "Select Test" screen is displayed.



If the correct password is entered then the first "Global Options" screen is displayed otherwise the "Select Test" screen is re-displayed.

Please contact your local agent / distributor or Lloyd Instruments Ltd if the password is forgotten and a test set-up is to be changed.



## 6.0 TRAINING MODE

### 6.1 SELECTING THE TRAINING MODE

The predefined test set-up called TRAINING is provided for operator training purposes. This is to familiarise the operator with the test procedure and the equipment controls without actually testing a sample.

Display the "Select Test" screen.

```
Select the required test
20THOU_284C (0 Samples)
Date and Time 01/05/2008 14:44
Select      >      Edit
```

Press the key under > or < to select TRAINING.

```
Select the required test
TRAINING (0 Samples)
Date and Time 29/05/2008 19:50
Select      <      >      Edit
```

Press the key under SELECT to display the "Analysis" screen.

```
Set Temp 295.0C   Barrel Temp 295.0C
Pressure 25.0Bar   Probe 1.23mm
                TRAINING
Test      Results  Stats    Suspend
```

This screen displays the requested barrel temperature, the actual barrel temperature, the nitrogen pressure within the barrel and the position of the probe rod.

Note that the training mode will automatically set the barrel temperature to 295°C, the nitrogen pressure to 25Bar and repeatedly increase the position of the probe rod to simulate a real test.

Note that the barrel heaters are not powered when the training mode is selected.

## 7.0 PERFORMING A TEST

### 7.1 SELECTING THE REQUIRED TEST SETUP

Display the "Select Test" screen to select one of the pre-defined test set-ups.

```

Select the required test
20TH0U_295C (0 Samples)
Date and Time 30/05/2008 07:48
Select      <          >          Edit
```

Note that the third line alternates between the Date and Time display and the Barrel Temperature.

```

Select the required test
20TH0U_284C (0 Samples)
Barrel Temperature 295.0C
Select      >          Edit
```

Also note that the barrel heaters are not switched on until either the key under SELECT is pressed or the PC software is started.

The heaters are then active and the barrel will heat and maintain the required test temperature until the equipment is switched off.

The required test temperature is specified either in the test set-up as shown earlier or in the PC Test set-up.

Press the key under > or < to select the test set-up that is to be used for the sample testing.

When the required test set-up is displayed, press the key under SELECT to display the "Analysis" screen.

```

Set Temp 295.0C   Barrel Temp 295.0C
Pressure 0.0Bar   Probe 15.85mm
                20TH0U_295C
Test      Results  Stats   Suspend
```

Press the key under TEST to display the "Pre-Test" screen.

```
Set Temp 295.0C   Barrel Temp 295.0C
Pressure 0.0Bar   Probe 15.85mm
Insert sample + follower then press GO
Gas Off   Purge   Full   Exit
```

Note that the "Pre-Test" screen will NOT be displayed if the probe is not electrically connected to the probe socket on the rear of the vertical column. If the probe is not connected, the warning screen below will be shown.

```
ERROR CONDITION
Probe NOT Connected

Press ENTER to Cancel
```

Also note that the intermediate screen shown below will be displayed if the barrel is not at the required temperature.

```
Set Temp 295.0C   Barrel Temp 292.0C
                20THOU_295C
Waiting for barrel temp to stabilise
                               Suspend
```

This screen will automatically change to the "Pre-Test" screen when the barrel has stabilised at the required temperature.

Note that the equipment MUST be set to the "Pre-Test" screen BEFORE trying to test a sample.

## 7.2 TEST PROCEDURE

The test procedure consists of the following actions:

- Check that the barrel is clean
- Prepare a clean follower and filling funnel
- Clean the die bore
- Purge the barrel with nitrogen gas
- Insert the polymer into the top of the barrel
- Insert the follower into the top of the barrel
- Press the GO key
- Apply pressure to the top of the follower using the charging tool for one minute
- Fit the probe to the top of the barrel
- Wait until the test has finished
- Clean the barrel, die and follower

## 7.3 CHECK THAT THE BARREL IS CLEAN

Fold a cleaning patch in two then place over the tip of the barrel cleaning tool. Push the cloth into the top of the barrel using the tool and push the tool down completely through the barrel.

Carefully remove the cloth from the barrel cleaning tool and withdraw the tool upwards.

Visually inspect the barrel and, if necessary, use another cleaning cloth until the barrel is completely free from polymer and the inner bore is shiny.



**CAUTION:** The cloth and barrel cleaning tool may become hot during the cleaning process. Wear heat protecting gloves when handling these items.

Polymer fumes may be emitted from the top of the barrel during the visual examination process. Wear protective glasses or goggles when viewing the interior of the barrel.

#### 7.4 PREPARE A CLEAN FOLLOWER AND FUNNEL

Ensure that a clean follower and the relevant filling funnel are ready for use.

Note that the large circular funnel is used for granules and the long funnel is used for powder. The long funnel prevents the powder sticking to the sides of the barrel.

Do NOT place the funnel onto the top of the barrel until the sample is being loaded otherwise it will become hot and the polymer may stick to it.

#### 7.5 CLEAN THE DIE BORE

The die bore can only be cleaned by blowing through with high-pressure nitrogen.

Do NOT try to clean the die bore by pushing wire etc through it. If the wire breaks inside the die, then a new die may be required.

Always clean the die immediately after use even if another test is not going to be performed afterwards.

Place the die into the recess of the die retaining nut with the end containing the O Ring facing upwards.

Screw the die retaining nut with the die to the bottom of the barrel and hand tighten using the tightening tool.

Loosen off by about 1/10th of a turn (about 30 degrees) otherwise it will be very difficult to remove once it has heated up and expanded.



*Shown: Retaining nut with die*

Fit the top cap to the top of the barrel and tighten securely using the tightening tool to seal the barrel.

Press the key under FULL to apply the nitrogen gas to the barrel. Wait until the gas can be heard flowing freely through the die, noting that the sound will change when the die is clean.

Press the key under GAS OFF to release the gas pressure, check that the display shows nominally zero then remove the top cap using the tightening tool.

#### 7.6 PURGE THE BARREL WITH NITROGEN GAS

Press the key under PURGE to allow a low volume of nitrogen gas to flow through the barrel to displace any air that may be present. Allow the gas to flow for a few seconds to ensure that the barrel is full of nitrogen.



## 7.8 APPLY PRESSURE TO THE FOLLOWER

Apply slight downwards pressure onto the top of the follower using the charging tool.

This part of the test takes one minute and the screen indicates this time period by a series of >>> moving across the screen.

At the end of the time period, the equipment will beep and the "Fit Probe" screen is displayed.

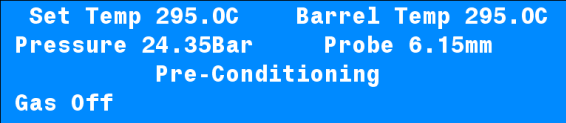


**Fit probe then press GO**

## 7.9 FIT THE PROBE TO THE BARREL

Using heat-protecting gloves, screw the probe onto the top of the barrel and secure the large nut hand tight.

Press the green GO key to indicate that the probe has been fitted. The "Pre-Conditioning" screen will then be displayed.



**Set Temp 295.0C    Barrel Temp 295.0C**  
**Pressure 24.35Bar    Probe 6.15mm**  
**Pre-Conditioning**  
**Gas Off**

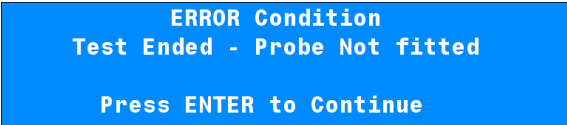
Note that the gas will be automatically switched to full pressure to perform the test. The gas pressure inside the barrel is displayed on this screen.

If the required gas pressure is known (testing a sample with a "known" IV), then the pressure can be checked and altered at this stage.

If the pressure is too low, increase it using the pressure regulator on the right hand side of the equipment.

If the pressure is too high, rotate the pressure regulator on the right hand side of the equipment anti-clockwise, press the key under GAS OFF to release the pressure then press the same key (now labelled GAS ON) to re-apply the pressure. Finally, increase the pressure, using the pressure regulator, to give the required value.

Note that if the GO key is not pressed within one minute after the "Fit probe then press GO" message is displayed, then the test will end and the warning screen below will be displayed.



**ERROR Condition**  
**Test Ended - Probe Not fitted**  
**Press ENTER to Continue**

## 7.10 WAIT UNTIL THE TEST HAS FINISHED

The test is now running.

The “Pre-Conditioning” part of the test allows the sample to heat up and become fluid and so start to flow through the die. The pre-conditioning ends 3 minutes after the sample was first inserted into the barrel then the “Speed Check” screen is displayed.

<b>Set Temp 295.0C</b>	<b>Barrel Temp 295.0C</b>
<b>Pressure 24.35Bar</b>	<b>Probe Speed 0.522</b>
<b>Gas Off</b>	

The Probe Speed should initially be between 0.5 and 1.0 (mm/min) and this is determined by the IV of the polymer and the applied gas pressure.

A high IV polymer will require a high gas pressure and a low IV polymer will require a low gas pressure.

<b>Set Temp 295.0C</b>	<b>Barrel Temp 295.0C</b>
<b>Pressure 18.75Bar</b>	<b>Probe Speed 0.445</b>
<b>Probe Speed Slow - Increase Pressure</b>	
<b>Gas Off</b>	

If the Probe Speed is below 0.5, the equipment will beep and display the screen below to request that the gas pressure is increased. Increase the pressure using the pressure regulator on the right hand side of the equipment until the probe speed is above 0.5.

<b>Set Temp 295.0C</b>	<b>Barrel Temp 295.0C</b>
<b>Pressure 18.75Bar</b>	<b>Probe Speed 0.445</b>
<b>Probe Speed Slow - Increase Pressure</b>	
<b>Gas Off</b>	

If the Probe Speed is above 1.0, the equipment will beep and display the screen below to request that the gas pressure is decreased.

If the pressure is too high, rotate the pressure regulator on the right hand side of the equipment anti-clockwise, press the key under GAS OFF to release the pressure then press the same key (now labelled GAS ON) to re-apply the pressure. Finally, increase the pressure, using the pressure regulator, to give a probe speed below 0.7.

<b>Set Temp 295.0C</b>	<b>Barrel Temp 295.0C</b>
<b>Pressure 28.58Bar</b>	<b>Probe Speed 1.005</b>
<b>Probe Speed High- Decrease Pressure</b>	
<b>Gas Off</b>	



Note that the probe speed MUST be set within the first 5 minutes after the sample was first poured into the barrel, i.e. the speed SHOULD only be altered when the bottom line of the display shows "Gas Off".

Note that the gas pressure must NOT be altered for the rest of the test duration.

After 5 minutes of total test time, the "IV" screen is displayed. This shows the current IV value and this changes during the test as the sample degrades.

```
Set Temp 295.0C   Barrel Temp 295.0C
Pressure 24.35Bar Probe Speed 0.581
IV Now 0.790
Time to end of test 12.0 minutes
```

After 6 minutes of total test time, the "Calculated IV" screen is displayed. This shows the "estimate" of the actual IV and this also changes during the test as the sample degrades.

```
Set Temp 295.0C   Barrel Temp 295.0C
Pressure 24.35Bar Probe Speed 0.592
IV Now 0.788     IV at Zero 0.807
Time to end of test 11.0 minutes
```

The screen is updated every second during the test until the test finishes.

```
Set Temp 295.0C   Barrel Temp 295.0C
Pressure 24.35Bar Probe Speed 0.680
IV Now 0.772     IV at Zero 0.799
Time to end of test 3.0 minutes
```

The test finishes 17 minutes after the sample was first inserted into the barrel. At the end of the test, the equipment will beep then automatically turn off the gas pressure to the barrel to stop the extrusion process.

The Calculated IV result and the degradation factor will be displayed together with the pass/fail indication if this was specified in the relevant test set-up.

```
Sample 1 Passed
IV      Degrad'n   Date      Time
0.798   0.010        01/05/2008 15:39
                          Save      Delete
```

The Degradation Factor is the reduction of IV per minute so a value of 0.010 means that the polymer has degraded by 0.010 IV per minute during the test. This value indicates how the polymer may degrade inside the production extruder.

Therefore, if the polymer stays within the heated zone of the production extruder for 3 minutes, the IV will drop by an estimated 0.03.

The test result should normally be saved into the internal memory so that the statistics can be calculated. If, for any reason the result is not to be stored, it can be deleted.

When either action is selected by pressing the relevant key, the "Pre-Test" screen is re-displayed as below.

```
Set Temp 295.0C   Barrel Temp 295.0C
Pressure 0.0Bar   Probe 15.85mm
Insert sample + follower then press GO
Gas Off   Purge   Full   Exit
```

The equipment is now ready for cleaning.

### 7.11 CLEANING THE BARREL

Ensure that the pressure displayed on the screen is nominally zero.

Using heat-protecting gloves, remove the probe from the barrel and, if necessary, wipe any polymer from the tip.

Store the probe in the probe holder at the right hand side of the vertical column.

Ensure that the body of the probe rests onto the probe holder so that the large knurled nut is below the holder.

Unscrew the die retaining nut from the bottom of the barrel with the tool provided. Note that the die will NOT be attached to the die retaining nut.

Insert the charging tool into the top of the barrel until it touches the top of the follower.

Gently push downwards on the barrel cleaning tool until the die is pushed out of the bottom of the barrel.



*Shown: Top of PET Plus with probe holder*

Remove the charging tool from the top of the barrel.

Hold the die with tweezers then remove any polymer from the sides and faces using a sharp blade.



**WARNING:** Wear cut-resistant gloves when cleaning.

Insert the charging tool into the top of the barrel until it touches the top of the follower.

Push the charging tool completely down through the barrel to remove the remaining polymer and the follower.

Hold the follower with tweezers then remove any polymer from the sides and faces using a sharp blade.

Fold a cleaning patch in two then place over the tip of the barrel cleaning tool. Push the cloth into the top of the barrel using the tool and push the tool down completely through the barrel.

Carefully remove the cloth from the barrel cleaning tool and withdraw the tool upwards. Visually inspect the barrel and, if necessary, use another cleaning cloth until the barrel is completely free from polymer and the inner bore is shiny.

A final cloth coated with a small amount of silicone oil may be used to coat the barrel and make future cleaning easier.



**WARNING:** The cloth and barrel cleaning tool may become hot during the cleaning process. Wear heat protecting gloves when handling these items.

Polymer fumes may be emitted from the top of the barrel during the visual examination process. Wear protective glasses or goggles when viewing the interior of the barrel.

If traces of polymer are still present in the barrel bore, these can be removed using the wire brush.

Note that this procedure should not be required if the barrel is cleaned immediately after each test.

Put the cleaning tool through the loop in the wire brush handle then move the brush up and down the length of the barrel to remove the traces of polymer.

Use another cleaning cloth until the barrel is completely free from polymer and the inner bore is shiny.

Do NOT use the brush until after the barrel has been cleaned using one or more cleaning clothes otherwise the brush will become clogged with polymer.

### **7.12 CLEAN THE DIE BORE**

The die bore can only be cleaned by blowing through with high-pressure nitrogen.

Do NOT try to clean the die bore by pushing wire etc through it. If the wire breaks inside the die, then a new die may be required.

Always clean the die immediately after use even if another test is not going to be performed afterwards.

Place the die into the recess of the die retaining nut with the end containing the O Ring facing upwards.

Screw the die retaining nut with the die to the bottom of the barrel and hand tighten using the tightening tool.

Loosen off by about 1/10th of a turn (about 30 degrees) otherwise it will be very difficult to remove once it has heated up and expanded.

Fit the top cap to the top of the barrel and tighten securely using the tightening tool to seal the barrel.

Press the key under FULL to apply the nitrogen gas to the barrel. Wait until the gas can be heard flowing freely through the die, noting that the sound will change when the die is clean.

Press the key under GAS OFF to release the gas pressure, check that the display shows nominally zero then remove the top cap using the tightening tool.

### **7.13 STORING THE DIE**

If another test is not going to be performed, remove the die from the barrel using the barrel cleaning tool and store safely.

Note that the die will be hot after cleaning!

Do NOT leave the die in the barrel for long periods when not in use.

### **7.14 CLEANING THE FOLLOWERS**

The followers are cleaned by heating to burn off the excess polymer.

Note that polymer fumes may be emitted from the followers during the heating / burning process so this should be carried out in a fume cupboard or an enclosed laboratory oven.

There are 3 ways that they can be heated.

- 1 Heated on a hot plate.
- 2 Placed on a wire gauze on a tripod stand then heated using a bunsen burner under the gauze
- 3 Placed in a muffle furnace using a maximum temperature of 450°C.

Do not handle the hot followers with tweezers or tongs as this might damage them by causing distortion or indentation, which would render them useless.

After the followers have cooled, brush off the film of ashed polymer using a brass wire brush.

Finally, the followers should be polished until shiny with a Scotchbrite pad, available from any super-market.

Do NOT use a file or similar tool to clean the followers because any scratches or similar marks will affect the IV values during the test.

### 7.15 VIEWING PREVIOUS TEST DATA

When a test is performed in the stand-alone mode, the test results are saved into the internal memory. This test result is stored together with the test name ID and a Date-Time ID that is created from the internal clock inside the equipment.

Therefore, statistics can be calculated and displayed for each of the defined test set-ups and the test data can be easily viewed and exported.

Display the “Select Test” screen to select one of the pre-defined test set-ups.

```
      Select the required test
      20THOU_295C (0 Samples)
      Date and Time 30/05/2008 07:48
      Select      <          >          Edit
```

Press the key under > or < to select the test set-up for the data that is to be viewed.

When the required test set-up is displayed, press the key under SELECT to display the “Analysis” screen.

```
      Set Temp 295.0C      Barrel Temp 295.0C
      Pressure 0.0Bar      Probe 15.85mm
                        20THOU_295C
      Test      Results  Stats      Suspend
```

Press the key under RESULTS to view the individual test results.

Note that a warning message will be displayed if there is no data present for this test set-up.

```
      There is no data for this test setup

      Press ENTER to continue
```

The "Results" screen shows the test number, IV, Degradation Factor, Date and Time at the end of test.

The screen below shows the results for test number 12.

Test No 12 of 12 (Select using keys)			
IV	Degrad'n	Date	Time
0.798	0.010	25/04/2008	11:51
No 2	No 11		

To view the data for any other test, select the test using the keys.

Note that the inner 2 keys move through the data one test at a time and the outer 2 keys move through the data 10 tests at a time.

The screens below show tests number 11 and 2.

Test No 11 of 12 (Select using keys)			
IV	Degrad'n	Date	Time
0.801	0.011	25/04/2008	10:13
No 1	No 10	No 12	

Test No 2 of 12 (Select using keys)			
IV	Degrad'n	Date	Time
0.799	0.011	20/04/2008	13:52
	No 1	No 3	No 12

Press the key under STATS to view the test statistics.

Note that a warning message will be displayed if there is no data present for this test set-up.

**There is no data for this test setup**

**Press ENTER to continue**

The "Statistics" screen shows the Mean and Standard Deviation values for the IV and the Degradation Factor together with the number of samples used to calculate the statistics.

Note that the Standard Deviation uses either the /n or the /(n-1) formula as selected in the GLOBAL options.

The data can be exported, using the supplied RS232 cable, to HyperTerminal etc. The RS232 protocol is 9600 Baud Rate, 8 Data Bits, 1 Stop Bit and No Parity.

To export the data, press the key under EXPORT.

	<b>IV</b>	<b>Degrad 'n</b>	<b>Samples</b>
<b>Mean</b>	<b>0.0798</b>	<b>0.011</b>	<b>12</b>
<b>Dev 'n</b>	<b>0.015</b>	<b>0.003</b>	
<b>Export</b>	<b>Reset</b>		

The test results and statistics can be erased from the internal memory by pressing the key under RESET. A confirmatory screen will be displayed to ensure that data is not erased by accident.

Note that if the system is password protected, then this erasing action can only be performed after the password has been entered.

## 8.0 SAMPLE PREPARATION

The PET Plus system can be used to test both the PET resin and the finished product at various stages throughout the production process.

- 1 Check the IV of the supplied PET granules
- 2 Check the IV of the PET granules after being dried in the production drier
- 3 Check the IV of the molten PET after being heated in the production extruder
- 4 Check the IV of the finished product

Test number 1 is to measure the POTENTIAL IV of the PET and is equivalent to the solution method. This test will ONLY give the same IV as the solution method if ALL moisture is removed from the polymer before testing (less than 20ppm moisture).

The supplied granules must be dried for 16 to 24 hours before testing but the granules can be ground to a powder in which case the drying time is reduced to 50-55 minutes.

Test number 2 is to measure the check the efficiency of the heaters and / or drying desiccant used in the drying hopper.

Test number 3 is mainly for use by fibre producers to measure the IV of the extrudate from a spinneret.

Test number 4 is to measure the IV of a finished pre-form or other moulded item.

The equipment required and the sample preparation is shown in the following sections.



## 9.0 SUPPLIED PET (GRANULES)

### 9.1 TESTING PRINCIPLES

The polymer must be dried using the polymer drier and vacuum pump, both of which are optional extras.

When the polymer is dry, it **MUST** be transferred to the PET Plus barrel using a procedure that ensures that the polymer does not come into contact with any external air.



**NOTE:** Drying time is a function of both temperature and vacuum.

A <0.1mm Hg vacuum is essential.

### 9.2 POLYMER DRYER



*Shown: Polymer drier*

The Polymer Drier has been designed for drying PET and other hygroscopic materials and incorporates the following features:

1. A temperature controlled heated block that has been machined to hold and heat six sample phials. The temperature controller can control the temperature from ambient to 200°C ( $\pm 2^\circ\text{C}$ ).
2. Six quick release valves at the top of the unit to connect each sample phial to the vacuum supply.
3. Two "Storage" or "Parking" areas to hold six sample phials. These areas are at the side of the heated block but are not heated. They are provided to allow the dried samples to be stored under vacuum until required.
4. An On/Off Switch for the Vacuum Pump.
5. A timer, settable from seconds to hours, with an audible alarm. This is mainly for use when drying polymer powder.

### 9.3 CHIP SIZE

For optimum drying the chip size should be approximately 1mm diameter, i.e. an average chip weight of 20mg.

Larger granules, i.e. 40 mg and above, should be ground to powder in the polymer chip grinder then the SUPPLIED PET (POWDER) procedure followed.

### 9.4 DRYING PRINCIPLE

Weigh out 3.8g of polymer chip and transfer to a sample drying tube (glass phial).

Dry the chip samples under vacuum at 150°C overnight, (optimum drying time is 16 - 24 hours).

The above values are guidelines based on the experience of many users. Adjustment of temperature and drying time may be required to get the best and most repeatable results.

### 9.5 DRYING PROCEDURE

Using a suitable container and laboratory balance, weigh out 3.8g of polymer chip. Pour the sample into a sample drying tube (glass phial) then screw the cap onto the top of the tube. Tighten the cap sufficiently to give a good seal between the silicone rubber gland and the glass tubing but do not over tighten.

Repeat for the other phials as required noting that a maximum of 6 samples can be dried at a time.

Set the temperature controller on the Polymer Drier to 150°C, switch the drier on and wait for 15 minutes for the drier to reach this temperature and stabilise.

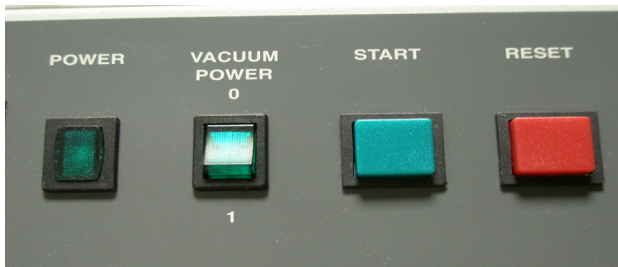


**WARNING: CAUTION HOT SURFACE:** Metallic Top Plate Surface can reach Temperatures up to 200°C from Dryer Block heat transfer. Wear Heat Protective Gloves.

Start the vacuum pump by pressing the Vacuum Power switch on the front of the polymer drier.



Shown: Temperature control



Shown: Vacuum power switch

Place each phial into the storage area at the sides of the polymer drier then push each of the metal tubes into a quick release connector along the top of the drier. Note that the tube clicks into place in the connector and cannot be pulled out again until the release lever is pressed.

Check that each of the vacuum valves is open (lever is in line with the vacuum pipe).

Wait for 5 minutes to ensure that a good vacuum is applied to the samples then place each of the phials into the heated block. This is to ensure that the polymer is not initially heated when air and / or moisture is present in the tube.

The samples **MUST** be dried for a time period between 16 and 24 hours. A typical procedure is to place the samples in the drier at (say) 4PM so that they are ready for testing the next morning from 8AM onwards.

At the end of the drying time, remove the phials from the heated block and place in the storage areas at the sides of the polymer drier.



**WARNING:** Hot tubes! Wear Heat Protective Gloves.

Note that the plastic tubes **MUST** still be attached to the quick release connectors and the vacuum pump **MUST** be left running.

Dried samples of polymer can remain in the parking area for up to 24 hours before testing with the vacuum pump running. Therefore, a number of samples, from 1 to 6, can be dried at the same time.

## 9.6 TESTING THE POLYMER



**NOTE:** It is very important that the Die, the Barrel and the Follower are scrupulously clean and have been assembled correctly after cleaning.

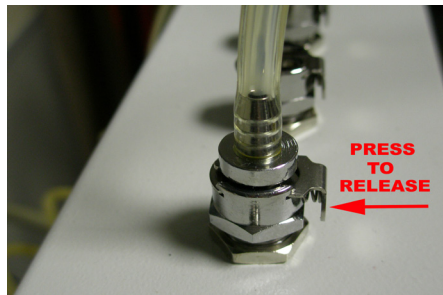
It is also important that the PET Plus is set to the “Pre-Test” screen ready for testing.

THIS PROCEDURE IS PART OF THE MAIN PROCEDURE SHOWN IN SECTION 7.7

Select a sample phial and turn the vacuum valve in the plastic vacuum tube to closed (lever is at 90 degrees to the vacuum pipe).

Press the release lever on the front of the quick connector so that the metal tube springs up out of the connector.

Note that the quick release connector has a sealing valve so that the vacuum is not released from the other sample phials.

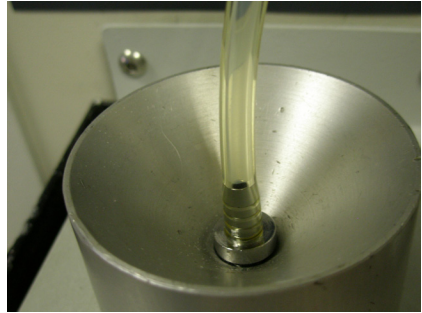


Press the key under PURGE to allow a low volume of nitrogen gas to flow through the barrel to displace any air that may be present. Allow the gas to flow for a few seconds to ensure that the barrel is full of nitrogen.

Place the standard filling funnel onto the top of the barrel.

Place the open metal end from the sample phial into the top of the filling funnel.

Turn the vacuum valve in the plastic vacuum tube so that the lever is in line with the vacuum pipe, to release the vacuum. A slight hissing sound will be heard as the vacuum draws hot dry nitrogen into the sample phial.



Press the key under GAS OFF to stop the flow of nitrogen gas.

Unscrew the top cap from the sample phial then carefully pour the polymer from the phial into the top of the filling funnel.

Remove the filling funnel from the top of the barrel.

Insert a clean follower into the top of the barrel.

Insert the charging tool into the top of the barrel.

Immediately press the green GO key to start the test. The “Charging” screen will be displayed.

## 10.0 SUPPLIED PET (POWDER)

### 10.1 TESTING PRINCIPLE

The polymer must be ground to a powder using the polymer grinder then dried using the drier and vacuum pump, all of which are optional extras.

Note that a quantity of liquid nitrogen is required to assist with the grinding process, so suitable storage and handling procedures are required.

When the polymer is dry, it MUST be transferred to the PET Plus barrel using a procedure that ensures that the polymer does not come into contact with any external air.



**NOTE:** Drying time is a function of both temperature and vacuum.

A <0.1mm Hg vacuum is essential.

### 10.2 POLYMER DRYER



*Shown: Polymer drier*

The Polymer Drier has been designed for drying PET and other hygroscopic materials and incorporates the following features:

1. A temperature controlled heated block that has been machined to hold and heat six sample phials. The temperature controller can control the temperature from ambient to 200°C ( $\pm 2^\circ\text{C}$ ).
2. Six quick release valves at the top of the unit to connect each sample phial to the vacuum supply.
3. Two "Storage" or "Parking" areas to hold six sample phials. These areas are at the side of the heated block but are not heated. They are provided to allow the dried samples to be stored under vacuum until required.
4. An On/Off Switch for the Vacuum Pump.
5. A timer, settable from seconds to hours, with an audible alarm. This is mainly for use when drying polymer powder.

### 10.3 CHIP SIZE

The polymer chips must be ground to fine powder (<0.5mm) using the polymer chip grinder.

### 10.4 DRYING PRINCIPLE

Weigh out 3.6g of polymer powder and transfer to a sample drying tube (glass phial).

High IV polymers (>0.7) are dried under vacuum at 170°C for 55 minutes.

Low IV amorphous polymers (<0.7) are dried under vacuum at 170°C for 50 minutes.

Too short a drying time will result in the sample still being wet and so degrade in the test giving low results.

Too long a drying time will result in the sample starting to polymerise.



**NOTE:** The above values are guidelines based on the experience of many users. Adjustment to temperature and drying time may be required to get the best and most repeatable results.

### 10.5 USING THE POLYMER CHIP GRINDER

The Polymer Chip Grinder is designed to grind polymer chips down to particles of either 1mm or 0.5mm using the supplied grinder bowls.

Davenport recommends only using the 0.5mm bowl for the purposes of grinding polymer chips to powder.

The polymer grinder should only be used for the purpose for which it was designed, as an accessory to the Davenport PET Plus, as described in this operating manual.

Compressed air is used to drive the Polymer Grinder. Setting up, maintenance and service should only be carried out by trained technicians or service personnel.



**WARNING:** The blades of the Polymer Grinder rotate at very high speeds. Therefore they continue to rotate for a few seconds after the timer has operated or the Timer Valve knob is pulled.

**WAIT FOR 5 SECONDS BEFORE OPENING THE GRINDER AFTER IT HAS BEEN SWITCHED OFF.**

**NOTE THAT PULLING THE TIMER VALVE KNOB FORWARDS STOPS THE GRINDER AT ANY TIME.**

Release the clip at the front of the grinder and lift the lid.

Note that for safety the blades CANNOT rotate when the lid is lifted.

Ensure that the grinder blade, grinding bowl (with slits), collecting funnel and collecting bowl are thoroughly clean.

The recommended way to clean these items is by using a vacuum cleaner, and / or by hand using a soft brush.

Weigh out 20 to 30g of polymer, which can be in the form of cube cut, masson cut or lace cut material and place into the grinding bowl with 0.5mm wide slits.

Place a clean receiving bowl under the funnel at the front of the grinder to collect the ground sample.

Place the filled grinding bowl centrally in the top of the cleaned collecting funnel of the grinder.

Using a suitable container, pour liquid nitrogen into the grinding bowl until the level of the liquid is about 1cm or 1/2" above the polymer chip level.



Shown: Poymer grinder



Collecting Funnel



Collecting Bowl



Shown: 0.5mm Grinding Bowl



**WARNING:** ALWAYS WEAR SAFETY GLASSES AND HEAT RESISTANT GLOVES WHEN HANDLING LIQUID NITROGEN WHICH IS AT  $-196^{\circ}\text{C}$

Allow the liquid nitrogen to just evaporate to dryness then lower the grinder lid and lock with the clip.

Start the grinder by pushing the pre-set timer valve fully inwards to give approximately 30-45 seconds grinding.

The grinder stops automatically to prevent over grinding and heat build up which could degrade the sample.

Wait until the grinding period has finished and the grinding blade has ceased to rotate.

Release the clip at the front of the grinder and lift the lid.

Lift the grinding bowl and shake it gently by hand until all the powder has been sieved out into the collecting bowl.

Pour more liquid nitrogen into the grinding bowl until the level of the liquid is about 1cm or 1/2" above the remaining polymer chip level.

Allow the liquid nitrogen to just evaporate to dryness then lower the grinder lid and lock with the clip.

Start the grinder by pushing the pre-set timer valve fully inwards to give approximately 30-45 seconds grinding.

Wait until the grinding period has finished and the grinding blade has ceased to rotate.

Release the clip at the front of the grinder and lift the lid.

Lift the grinding bowl and shake it gently by hand until all the powder has been sieved out into the collecting bowl.

Continue the above procedure until most of the sample has been reduced to powder.

Remove the receiving bowl and transfer the powder to the sample phials as shown later.

Clean the grinder blade, grinding bowl, collecting funnel and collecting bowl.



*Shown: Preset timer valve*



## 10.6 SETTING THE TIMER

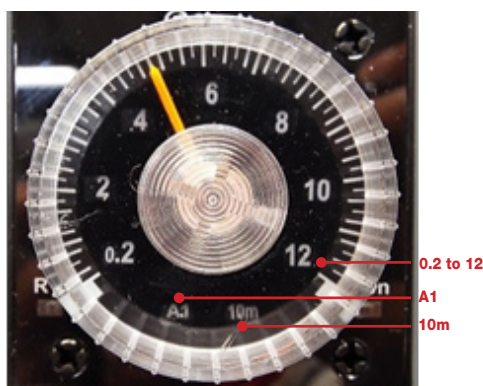
The timer can be set to a wide range of time periods but its main use is provide a 50 minutes or 55 minutes timing for drying polymer powder.

Check that the 3 controls are set as below:

- Function = Bottom Left Screw = A1
- Time Units = Bottom Right Screw = 10m
- Time Base = Top Right Screw = 0.2 to 12

A small terminal screwdriver is required to adjust.

Rotate the relevant screw to set the unit as shown below:



Set the dial to 0.82 to select 50 minutes.

Set the dial to 0.92 to select 55 minutes.

Using a suitable container and laboratory balance, weigh out 3.6g of polymer powder. Pour the sample into a sample drying tube (glass phial) then screw the cap onto the top of the tube. Tighten the cap sufficiently to give a good seal between the silicone rubber gland and the glass tubing but do not over tighten.

Turn the vacuum valve in the plastic vacuum tube to closed (lever is at 90 degrees to the vacuum pipe).

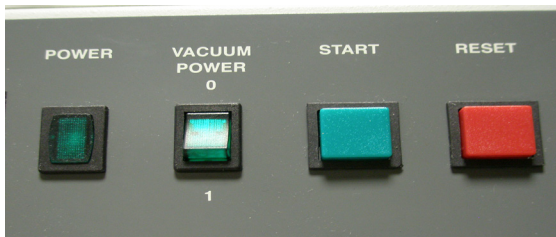


Repeat for the other phials as required noting that a maximum of 6 samples can be dried at a time.

Set the temperature controller on the Polymer Drier to 170°C, switch the drier on and wait for 15 minutes for the drier to reach this temperature and stabilise.



Start the vacuum pump by pressing the Vacuum Power switch on the front of the polymer drier.



Place each phial into the storage area at the sides of the polymer drier then push each of the metal tubes into a quick release connector along the top of the drier. Note that the tube clicks into place in the connector and cannot be pulled out again until the release lever is pressed.

Open each of the vacuum valves SLOWLY, by turning the lever until it is in line with the vacuum pipe, so that powder is NOT drawn up the plastic tube by the initial vacuum.

Wait for 5 minutes to ensure that a good vacuum is applied to the samples then place each of the phials into the heated block. This is to ensure that the polymer is not initially heated when air and / or moisture is present in the tube.

The samples MUST be dried for a time period of 50 minutes (IV < 0.7) or 55 minutes (IV > 0.7).

If the timer is to be used, press the START button on the front of the polymer drier. The light in the top left hand corner of the timer will light.

A buzzer will sound at the end of the timer period (if the timer was used). Press the RESET button to stop the buzzer.

At the end of the drying time, remove the phials from the heated block and place in the storage areas at the sides of the polymer drier.



**WARNING:** Hot tubes! Wear heat protective gloves when handling phials.

Note that the plastic tubes MUST still be attached to the quick release connectors and the vacuum pump MUST be left running.

Dried samples of polymer can remain in the parking area for up to 24 hours before testing with the vacuum pump running. Therefore, a number of samples, from 1 to 6, can be dried at the same time.

## 10.7 TESTING THE POLYMER



**NOTE:** It is very important that the Die, the Barrel and the Follower are scrupulously clean and have been assembled correctly after cleaning.

It is also important that the PET Plus is set to the “Pre-Test” screen ready for testing.

THIS PROCEDURE IS PART OF THE MAIN PROCEDURE SHOWN IN SECTION 7.7.

Select a sample phial and turn the vacuum valve in the plastic vacuum tube to closed (lever is at 90 degrees to the vacuum pipe).

Press the release lever on the front of the quick connector so that the metal tube springs up out of the connector.

Note that the quick release connector has a sealing valve so that the vacuum is not released from the other sample phials.

Press the key under PURGE to allow a low volume of nitrogen gas to flow through the barrel to displace any air that may be present. Allow the gas to flow for a few seconds to ensure that the barrel is full of nitrogen.

Place the long filling funnel into the top of the barrel. Note that the long funnel prevents the powder sticking to the sides of the barrel.

Also note that the funnel must be cold or the powder may stick to the funnel.

Place the open metal end from the sample phial into the top of the filling funnel.

Open the vacuum valve in the plastic vacuum tube (lever is in line with the vacuum pipe) to release the vacuum. A slight hissing sound will be heard as the vacuum draws hot dry nitrogen into the sample phial.



Press the key under GAS OFF to stop the flow of nitrogen gas.

Unscrew the top cap from the sample phial then carefully pour the powder from the phial into the top of the filling funnel.

Remove the filling funnel from the top of the barrel.

Insert a clean follower into the top of the barrel.

Insert the charging tool into the top of the barrel.

Immediately press the green GO key to start the test. The "Charging" screen will be displayed.

## 11.0 PRODUCTION DRIER

### 11.1 TESTING PRINCIPLE

The production drier / hopper is checked by collecting dried polymer from the bottom of the hopper then testing it WITHOUT FURTHER DRYING to check the drier efficiency. This can be used to check the drying temperature, drying time and desiccant.

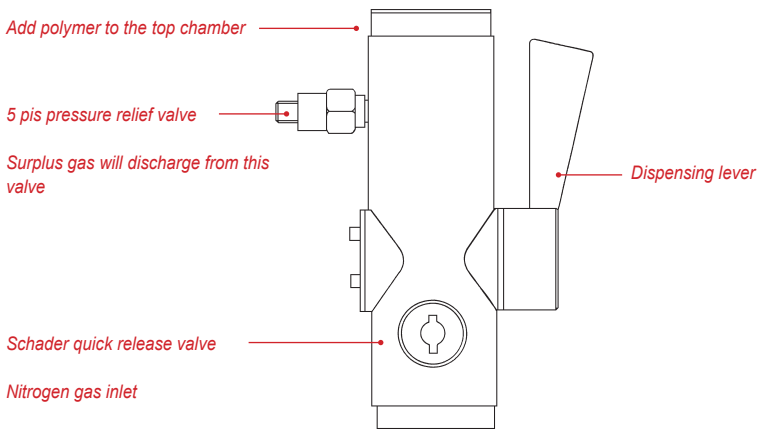
The polymer MUST be transferred to the PET Plus barrel using a procedure that ensures that the polymer does not come into contact with any external air. The Polymer Chip Transfer Vessel is designed for this purpose.

### 11.2 POLYMER CHIP TRANSFER VESSEL

This is a metal cylinder containing a central dispensing mechanism that splits the cylinder into 2 chambers. Nitrogen is passed through the two chambers to purge any air from the device. One chamber has a Schrader quick release inlet with a non-return valve. The second chamber has a 5 PSI (0.35Bar) pressure release valve.

The dispensing mechanism has an adjustable bore to preset the volume of polymer that will be dispensed with each operation of the dispensing lever.

The transfer vessel will keep enough granules dry for up to ten tests and dispense the correct amount required for each test (3.8g).



### 11.3 CHECKING THE DISPENSED VOLUME

The transfer vessel should be adjusted so that 3.8g of polymer is dispensed when the dispensing lever is turned.

Remove the top cap (cap on larger chamber) from the transfer vessel then half fill the chamber with polymer.

Refit the top cap then remove the bottom cap.

Place a container on the bench then hold the transfer vessel upright over the container.

Turn the dispensing lever until the lever is pointing upwards. This allows the polymer to enter the dispensing mechanism.

Turn the dispensing lever until the lever is pointing downwards. This will dispense a quantity of polymer into the container.

Weigh the dispensed polymer and check that it weighs  $3.8\text{g} \pm 0.1\text{g}$ . If the weight is not correct, the dispensing mechanism must be adjusted.

### 11.4 ADJUSTING THE DISPENSED VOLUME

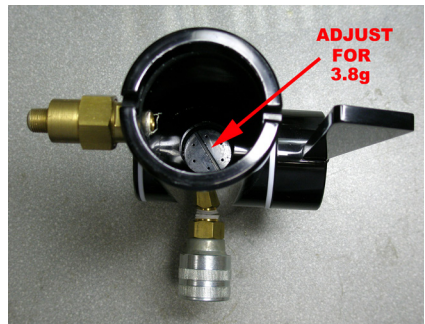
The dispensing mechanism contains a central bore with a bottom screw. The volume of the bore is adjusted by moving the bottom screw up or down.

If the dispensed weight is too low, rotate the bottom screw clockwise, i.e. screw downwards.

If the dispensed weight is too high, rotate the bottom screw anticlockwise, i.e. screw upwards.

This adjustment should only be necessary when the vessel is used for the first time or when the chip size alters, e.g. when changing suppliers.

If different sizes of polymer chip are used regularly it may be a good idea to have one transfer vessel for each chip size.



## 11.5 USING THE POLYMER CHIP TRANSFER VESSEL

Note that the production drying hopper **MUST** be fitted with a chip outlet.

This outlet may be part of the flexible hose assembly connected to the inlet of the extruder.

Turn the dispensing lever until the lever is pointing upwards.

Ensure that the top and bottom caps are fitted.

Connect a low-pressure nitrogen supply to the Schrader quick release inlet valve.

Set the external nitrogen pressure to approx 5psi (0.35 Bar) and allow the nitrogen to flow through the transfer vessel for about 10 minutes to make sure the cylinder is completely dry.

Ensure that nitrogen gas flows out of the 5psi pressure relief valve.

When ready to take a sample, disconnect the nitrogen line from the Schrader quick release inlet valve.

Do **NOT** remove the top or bottom caps.

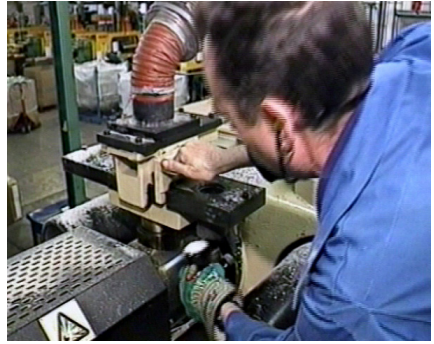
Take the transfer vessel to the production area.

If a nitrogen supply is available at the sampling site, either:

- 1 Reconnect the nitrogen to the Schrader quick release inlet valve and allow the nitrogen to flow for a minute.
- 2 Have a flexible hose that can be aimed around the chip outlet.

Remove the top cap and load with the DRY sample as quickly as possible.

The top chamber should be filled until it is between half full and full of polymer.



The picture above shows a flexible hose assembly connected to the inlet of the extruder.

**IMMEDIATELY** replace the top cap.

Take the transfer vessel containing the dry sample back to the PET Plus.

The samples do not have to be tested immediately because they will be kept dry for several hours if the top or bottom caps are not removed.

If the samples are to be stored for a time, it is recommended that the low-pressure nitrogen supply is reconnected to the Schrader quick release inlet valve.

## 11.6 TESTING THE POLYMER



**NOTE:** It is very important that the Die, the Barrel and the Follower are scrupulously clean and have been assembled correctly after cleaning.

It is also important that the PET Plus is set to the “Pre-Test” screen ready for testing.

THIS PROCEDURE IS PART OF THE MAIN PROCEDURE SHOWN IN SECTION 7.7.

Press the key under PURGE to allow a low volume of nitrogen gas to flow through the barrel to displace any air that may be present. Allow the gas to flow for a few seconds to ensure that the barrel is full of nitrogen.

Place the standard filling funnel onto the top of the barrel.

Press the key under GAS OFF to stop the flow of nitrogen gas.

Remove the bottom cap from the transfer vessel.

Place the bottom of the transfer vessel onto the top of the filling funnel.

Turn the dispensing lever until the lever is pointing downwards. This will dispense a quantity of polymer into the filling funnel

Remove the filling funnel from the top of the barrel.

Insert a clean follower into the top of the barrel.

Insert the charging tool into the top of the barrel.

Immediately press the green GO key to start the test. The “Charging” screen will be displayed.

Refit the bottom cap to the transfer vessel.

Turn the dispensing lever until the lever is pointing upwards. This allows the polymer to enter the dispensing mechanism ready for the next test.

It is recommended that the low-pressure nitrogen supply is reconnected to the Schrader quick release inlet valve.





## 12.0 MOLTEN EXTRUDATE

### 12.1 TESTING PRINCIPLE

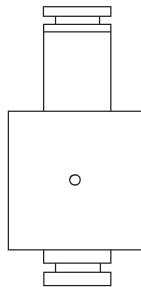
The extrudate from a spinneret or bleed point is checked by collecting molten polymer then testing it WITHOUT FURTHER DRYING.

The polymer MUST be transferred to the PET Plus barrel using a procedure that ensures that the polymer does not come into contact with any external air. The Sample Melt Pot is designed for this purpose.

### 12.2 SAMPLE MELT POT

This is a cylindrical metal block containing a PTFE insert and two tight fitting PTFE end caps.

The top of the pot has a longer stem and has a conical opening. The bottom of the pot is shaped to fit onto the top of the PET Plus barrel.



**WARNING:** Hot surface! Wear heat protective gloves.

### 12.3 USING THE SAMPLE MELT POT

Before using the sample melt pot, ensure that the top and bottom PTFE caps are a tight fit on the unit and that the sample melt pot is clean and dry.

Remove the top cap and using heat resistant gloves, hold the pot under either a sample bleed point or as close as possible to the spinneret face or the die of the extruder to collect a sample of extrudate.

When filling from a bleed point, allow the polymer to run into the pot until it is almost full then replace the top cap.

When filling from a spinneret, the sample should be allowed to spill over the whole top portion of the pot.

Quickly trim off the excess polymer with a pair of scissors so that the sample is wholly contained in the hollowed top of the pot.

Withdraw the pot containing the sample and allow the sample to solidify a little, forming a skin.

Push the polymer into the base of the pot using a suitably sized stainless steel rod (the barrel cleaning tool).

Repeat the sampling procedure until the pot is almost full and then replace the top plug.

## 12.4 TESTING THE POLYMER



**NOTE:** It is very important that the Die, the Barrel and the Follower are scrupulously clean and have been assembled correctly after cleaning.

It is also important that the PET Plus is set to the “Pre-Test” screen ready for testing.

THIS PROCEDURE IS PART OF THE MAIN PROCEDURE SHOWN IN SECTION 7.7.

Press the key under PURGE to allow a low volume of nitrogen gas to flow through the barrel to displace any air that may be present. Allow the gas to flow for a few seconds to ensure that the barrel is full of nitrogen.

Press the key under GAS OFF to stop the flow of nitrogen gas.

Remove the bottom plug from the sample melt pot and position the melt pot onto the top of the barrel.

Remove the top plug from the sample melt pot and push the plug of polymer into the PET Plus barrel using the charging tool.

Remove the sample melt pot from the top of the barrel.

Insert a clean follower into the top of the barrel.

Insert the charging tool into the top of the barrel.

Immediately press the green GO key to start the test. The “Charging” screen will be displayed.

## 13.0 FINISHED PRODUCT

### 13.1 TESTING PRINCIPLE

The finished product must first be broken into chips which are ground to a powder using the polymer grinder then dried using the drier and vacuum pump, all of which are optional extras.

Note that a quantity of liquid nitrogen is required to assist with the grinding process, so suitable storage and handling procedures are required.

When the polymer is dry, it MUST be transferred to the PET Plus barrel using a procedure that ensures that the polymer does not come into contact with any external air.

### 13.2 INDUSTRIAL GRINDER

High speed grinding and chipping machinery is used to pulverise samples.

The equipment must be used in accordance with the manufacturers instruction manual and ear protection must be used at all times in the vicinity of the grinding equipment.

Note that liquid nitrogen is used to pre-cool samples.

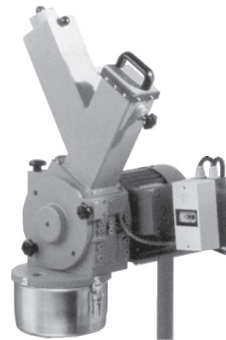


**WARNING:** ALWAYS WEAR SAFETY GLASSES AND HEAT RESISTANT GLOVES WHEN HANDLING LIQUID NITROGEN WHICH IS AT  $-196^{\circ}\text{C}$

AMETEK recommends the Fritsch Pulverisette 15, which is capable of reducing a pre-form to chip in seconds. It may be floor or bench mounted and comes complete with an 8mm sieve.



**NOTE:** A coarse sieve (8mm) is used to produce chip and very little powder, thus making cleaning easy. A sieve to produce powder could be used but this would necessitate stripping the system for cleaning after pulverising each sample. Therefore, it is better to chip the sample in the pulveriser and then to grind to powder using the Polymer Grinder.



### 13.3 USING THE INDUSTRIAL GRINDER

This is used to grind pre-forms, bottle necks, polymer plaques, pieces of polymer extrudates and thick sheet samples to approx 8mm chips.

One complete pre-form is usually sufficient for chipping and drying.

If only the bottle necks are to be used, then several bottle necks (usually about three) are required. Cut the bottle necks from the bottles as close to the threaded portion as possible using tin snips or a small clean hacksaw.

Polymer plaques, thick sheet samples or chunks of polymer extrudate should be cut using tin snips or a small clean hacksaw until they are small enough to fit into the pulverisette sample inlet port.

Using tongs, place the selected sample or samples in a Dewar Flask containing sufficient liquid nitrogen to immerse the sample completely.



**WARNING:** ALWAYS WEAR SAFETY GLASSES AND HEAT RESISTANT GLOVES WHEN HANDLING LIQUID NITROGEN WHICH IS AT -196°C

Leave the samples immersed in the liquid nitrogen until the liquid nitrogen has ceased to boil, showing that the sample has been cooled to the correct temperature.

Ensure that a clean collecting bowl is fitted to the bottom of the grinder.

Start the motor on the industrial grinder.

Carefully remove the cooled sample from the liquid nitrogen using tongs and place it in the safety interlock sample part of the pulveriser.

Open the interlock to drop the cooled sample into the sidearm and immediately lift and lower the plunger arm to push the sample onto the pulveriser blades.

Switch off the motor as soon as pulverisation has ceased (usually a few seconds after lowering the plunger arm).

Wait until the rotor has stopped then unclip the receiving bowl containing the coarsely chipped sample.

Clean the pulveriser using a jet of clean compressed air or nitrogen to remove any polymer chip remaining on the rotor blades or coarse sieve.

Follow the procedure shown in SECTION 10 - SUPPLIED PET (POWDER) to grind the chips to a powder then test using the PET Plus.

## 14.0 MAINTENANCE

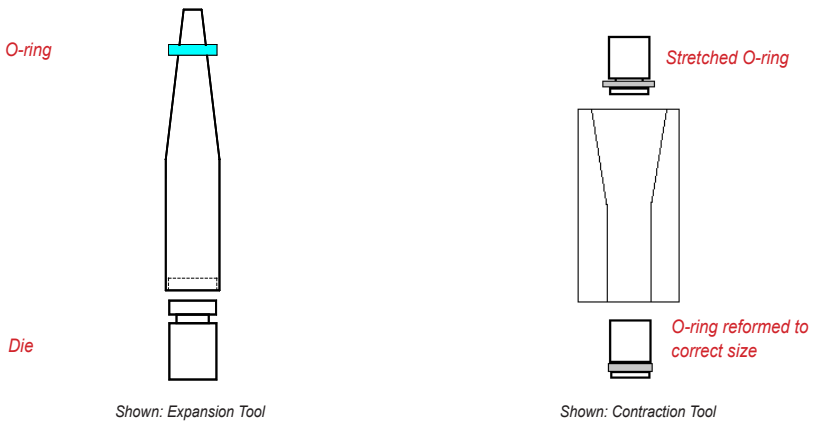
### 14.1 FITTING A NEW O RING TO THE DIE

The die “O” ring is used to prevent polymer from leaking past the die during a test.

Therefore, the “O” ring diameter should be slightly larger than the die so that it protrudes out slightly from the recess in the die.

When the “O” ring becomes worn or damaged, it should be replaced otherwise low IV measurements will be produced.

There is no hard and fast rule as to the frequency that the “O” ring should be replaced but this may need to be replaced daily if samples are tested on an hourly basis.



To remove the “O” ring make a vertical cut in the ring using a sharp pointed scalpel or Craft Knife.



**WARNING:** Always wear cut resistant gloves, such as ones made of Dyneema.

Lever out the old “O” ring with the knife.

Place the die on the bench with the “O” ring slot uppermost.

Position the Expansion Tool over the top of the die.

Place a new PTFE “O” ring over the tapered end of the expansion tool.

Slide the “O” Ring over the tool onto the die by hand.

Position the “O” ring into the recess on the die.

The “O” Ring must now be fitted into the die recess by inserting the die with the “O” ring into the wide end of the contraction taper tool and gently pushing it with the cleaning tool until it emerges from the narrower opening.

## 14.2 CLEANING THE DIE RETAINING NUT

Normally the die retaining nut does not require cleaning unless there has been a leakage of molten polymer past the die “O” ring or the extrudate has lipped back and coated the underside of the nut.

Molten polymer can be removed by wiping with a piece of cleaning cloth when the nut is hot.

If the die retaining nut becomes completely encrusted with polymer, then the nut should be burned off in a similar fashion to that described for the followers.

The sealing ring in the top of the die retaining nut should protrude above the level of the metal holder.

When worn down, remove the old washer by levering it out via the slot cut into the top of the nut.

Place the new washer into the circular recess around top of nut and press down firmly until washer is evenly embedded.

## 14.3 SERVICING THE POLYMER CHIP GRINDER

Clean the grinder bowl and funnel using a vacuum cleaner or by hand using a soft brush and cleaning cloth.

Check that the lubricator on the air regulator set contains a suitable quantity of the correct grade of oil, e.g. Shell Morlina 10 or Tellus R10.

Rotate the silencer cover on the rear of the grinder slightly anti-clockwise then remove the cover from the studs.

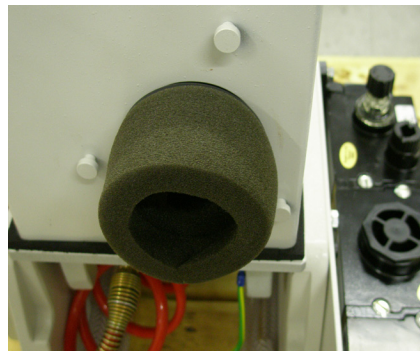
Carefully remove the foam element from the end of the silencer then wash thoroughly in paraffin.

Dry the foam element then refit onto the end of the silencer.

Refit the silencer cover.



*Shown: Silencer cover*



*Shown: Foam element in Silencer*

#### 14.4 TENSIONING THE DRIVE BELT

The polymer grinder blade is driven by an air motor via a smooth drive belt. A smooth belt is used to allow the belt to slip if the blade contacts a solid material.

The drive belt may become loose after an extended period of time and may slip under normal operation.

To check the belt tension, the top cover has to be removed from the top of the polymer grinder.

To remove the top cover, first remove the silencer cover from the rear of the grinder by twisting anticlockwise by a few degrees then lifting away.

Remove the foam element and the rubber seal from the end of the silencer.

Unscrew the brass silencer then pull out from the top cover.

Unscrew and remove the two (2) domed nuts and washers securing the top cover to the main lid assembly.

Lift the top cover away from the lid assembly.

Check that the belt can be twisted by 90 degrees between the two (2) pulleys.

If the belt can be twisted by more than 90 degrees, it must be tightened.

The motor is held in an eccentric boss so that the motor position can be moved closer or further away from the blade spindle.

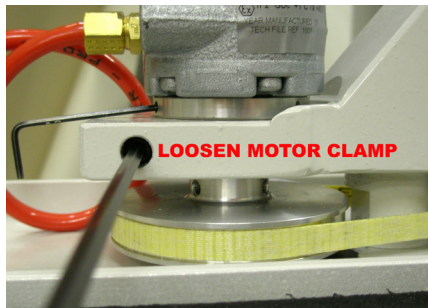
Screw the silencer to the end of the air motor.

Loosen the motor securing clamp using an Allen key.

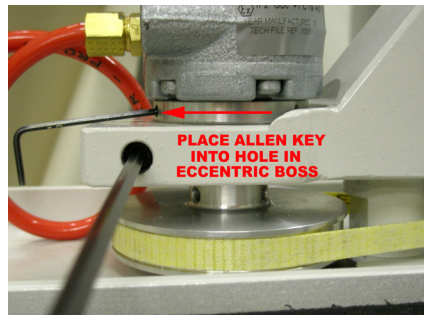
Place a smaller Allen key into one of the holes in the motor eccentric boss then rotate the motor eccentric boss clockwise to tighten the belt.



Shown: Polymer grinder top cover and belt



Shown: Loosen motor clamp



Shown: Location for loosening clamp

Ensure that the silencer remains in the same position.

Retighten the motor securing clamp then recheck the belt tension.

Unscrew the silencer from the end of the air motor.

Refit the top cover and secure using the two (2) domed nuts and washers

Screw the silencer to the end of the air motor. This is a little difficult because it is not possible to view the threaded hole in the motor body.

Refit the rubber seal and foam to the end of the silencer. Refit the silencer cover.

#### 14.5 REPLACING THE DRIVE BELT

A broken drive belt can be replaced as shown.

Remove the top cover as shown in the previous section.

Loosen the three (3) grub screws in the motor pulley.

Loosen the motor securing clamp.

Pull the motor upwards and pull the pulley downwards until the pulley can be removed from the end of the motor shaft.

Remove the motor from the casting.

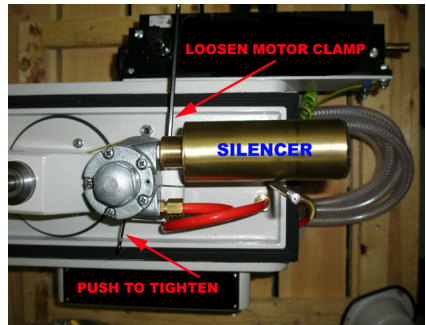
Remove the old belt (if still fitted) by holding the front end, pulling it forwards and over the blade spindle then pulling it rearwards and away from the casting.

Position the rear of the new belt under the motor casting.

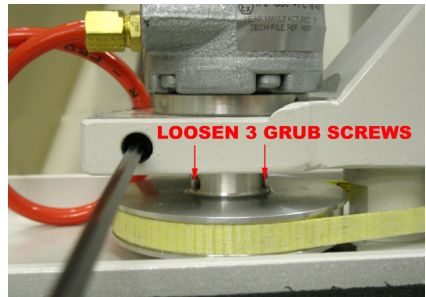
Hold the front end of the new belt, pull it forwards and over the blade spindle then lower it onto the blade spindle pulley.

Refit the motor and motor pulley, noting that the 3 grub screws should be located into the 3 slots machined into the motor shaft.

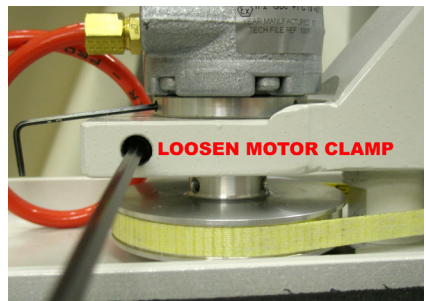
Check the belt tension as shown earlier.



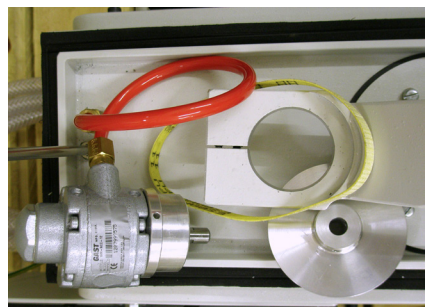
Shown: Location of Silencer



Shown: Location of grub screws



Shown: Location of motor clamp



Shown: Loosened drive belt



## 14.6 RESULT DISCREPANCIES

Insufficient drying is by far the most common problem, which leads to low results and high variance between results.

The following is a list referring to some items that can also contribute to unexpected IV results from the equipment:

- Sample condition or size, resulting in variable results.
- Dirty or hot follower - will give false high results.
- New die "O" ring required - low IV
- Inadequate gas pressure, e.g. empty bottle.
- Poor vacuum to Dryer tubes - will give low and variable results.
- Poor drying procedure - will give low and variable results.
- Dryer temperature incorrect.
- Holes in probe rod blocked.
- Barrel inadequately cleaned - will give variable results and erratic graph.
- Hot funnel used for sample.
- Purge and full gas supplies not reaching the barrel.
- GO key not pressed immediately after sample has been loaded into the barrel - will give variable results.

The PET Plus is basically simple in operation and construction. No form of regular maintenance is required except that the unit is kept in a clean condition.

## 14.7 POSSIBLE FAULTS IN PRESSURE CONTROL SYSTEM LOSS OF PRESSURE

1. Gas cylinder empty.
2. Malfunction of gas supply regulator.
3. Check all external screwed and compression joints using a soapy water solution if necessary, especially where the supply pipe enters the equipment. Unscrew leaky joints and wrap PTFE tape around male end, re-connect and tighten.
4. Check the condition of the seals at the top and bottom of the barrel, i.e. PTFE washer in die retaining nut and PTFE washer where probe seals top of barrel.

## 14.8 BASIC ELECTRICAL CHECKS

Your equipment has been completely tested and checked for electrical safety before leaving the factory.

Regular checks should be carried out on the instrument to establish that the instrument is safe to operate.

Check all connections for mechanical tightness, an earth loop impedance check must be carried out to all components which should be at earth potential. This should conform to local safety standards and AMETEK recommends that this be carried out at least annually.

## 14.9 CHANGING A DAMAGED PROBE ROD

If the probe rod becomes damaged and will no longer slide freely inside the probe bush, then it must be replaced as shown below.

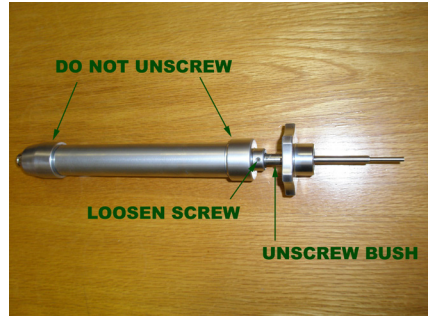
Loosen the grub screw securing the probe bush to the main body of the probe then unscrew and remove the probe bush.

Remove the probe rod and replace with a new one.

Re-assemble the probe then calibrate as shown in the calibration section.

The part number of the probe rod is 750/1042.

A probe repair kit is also available, part number 778/103.



*Shown: Probe*

## 15.0 CALIBRATION

### 15.1 SELECTING THE CALIBRATION MODE

Display the "Select Test" screen to display one of the pre-defined test set-ups.

```
      Select the required test
      20THOU_284C (0 Samples)
      Date and Time 01/05/2008 14:44
      Select      >      Edit
```

Press the key under EDIT to display the edit test screen.

```
      Press a key to select an option
      20THOU_284C
      Rename      Global      Delete      Setup
```

Press the key under GLOBAL to display the first "Global Options" screen.

```
      Press a key to change a parameter
      Change      Current
      Password Language Calibrate
      <None>      English      Mode      >
```

Press the key under CALIBRATE MODE to display the "Calibration" screen.

```
      Calibration Mode
      Cal      Cal      Cal
      Temp Pressure Probe
```

## 15.2 TEMPERATURE CALIBRATION

Note that an accurate reference thermometer is required for this procedure.

The barrel temperature is calibrated by measuring the actual temperature and entering this value into the system.

Press the key under CAL TEMP to display the "Temperature Calibration" screen.

Set Temp 295.0C			
Measured Temp 294.8C			
Set	Cal	View	View
Temp	Point	Points	Readings

Press the key under SET TEMP then enter 300°C. This is the recommended calibration temperature.

Set Temp 300.0C			
Measured Temp 300.0C			
Set	Cal	View	View
Temp	Point	Points	Readings

Fit the die to the bottom of the barrel then insert approx. 2 grams of PET.



**NOTE:** Use Polythene instead of PET if using a mercury in glass thermometer. PET will break a glass thermometer when it cools and contracts.

Insert the reference thermometer into the melt.

Allow the barrel temperature to stabilise (approx 5 minutes) as shown by the "measured temperature" display.

Measure the actual barrel temperature using the reference thermometer and compare with the requested temperature of 300°C.

If the actual temperature is not 300°C  $\pm 0.1^\circ\text{C}$ , press the key under CAL POINT to display the "cal point" screen.

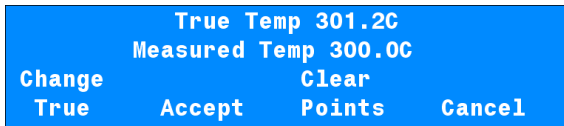
True Temp 300.0C			
Measured Temp 300.0C			
Change		Clear	
True	Accept	Points	Cancel

Press the key under CHANGE TRUE to display the “change true temperature value” screen.



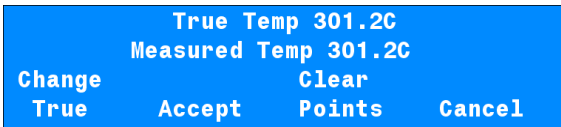
Enter the true temperature value as indicated on the reference thermometer.

The display will now show the true temperature as measured by the reference thermometer.

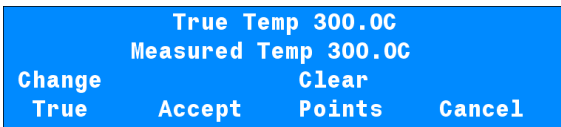


Press the key under ACCEPT to correct the measured temperature value.

If a calibration point had already been entered at 300C (or approx. this value) a warning message will be displayed. Press the key under OVERWRITE POINT to accept this new calibration point. The measured temperature value will now change to the entered value.



The system will now cause the barrel temperature to change and stabilise at the corrected temperature value.



Press the ENTER key two times to exit the temperature calibration screen.

### 15.3 PRESSURE CALIBRATION

Note that an accurate pressure gauge is required for this procedure.

The barrel pressure is calibrated by measuring the actual pressure and entering this value into the system.

Press the key under CAL PRESSURE to display the "Pressure Calibration" screen.

<b>Nitrogen Gas is OFF</b>			
<b>Measured Pressure 0.4Bar</b>			
<b>Gas</b>	<b>Cal</b>	<b>View</b>	<b>View</b>
<b>On</b>	<b>Point</b>	<b>Points</b>	<b>Readings</b>

This screen indicates the gas status (Nitrogen Gas is Off) and also displays the measured pressure. Check that the measured pressure is 0Bar  $\pm$ 0.1Bar.

Note that the "Zero" reading can vary slightly with changes in atmospheric pressure.

If the measured pressure is not 0Bar  $\pm$ 0.1Bar, press the key under CAL POINT to display the "cal point" screen.

<b>True Pressure 0.0Bar</b>			
<b>Measured Pressure 0.4Bar</b>			
<b>Change</b>		<b>Clear</b>	
<b>True</b>	<b>Accept</b>	<b>Points</b>	<b>Cancel</b>

The true pressure should automatically display 0.0Bar.

If it does not show 0.0Bar, press the key under CHANGE TRUE then enter 0.0Bar.

The display will now show 0.0Bar for the true pressure.

Press the key under ACCEPT to correct the zero pressure reading.

<b>Nitrogen Gas is OFF</b>			
<b>Measured Pressure 0.0Bar</b>			
<b>Gas</b>	<b>Cal</b>	<b>View</b>	<b>View</b>
<b>On</b>	<b>Point</b>	<b>Points</b>	<b>Readings</b>

If a calibration point had already been entered at 0Bar (or approx. this value) a warning message will be displayed. Press the key under OVERWRITE POINT to accept this new calibration point.

Press the ENTER key to return to the "Pressure Calibration" screen.

Fit the barrel blanking nut and washer to the bottom of the barrel.

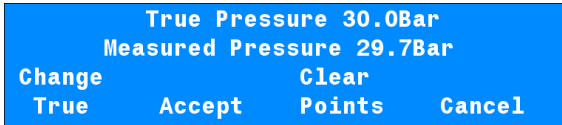
Connect a reference pressure gauge to the top of the barrel.

Press the key under GAS ON to apply pressure to the barrel.

Adjust the gas regulator on the right hand side of the equipment to apply approx. 30Bar as measured by the reference pressure gauge.

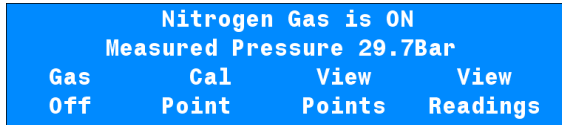
Allow the pressure to stabilise (approx 1 minute) then measure the actual pressure using the reference pressure gauge and compare with the measured pressure.

If the measured pressure is not the same as the reference pressure +/- 0.1Bar, press the key under CAL POINT to display the "cal point" screen.



Press the key under CHANGE TRUE then enter the pressure measured by the reference pressure gauge, e.g. 29.9Bar.

The display will now show the entered value for the true pressure. Press the key under ACCEPT to correct the pressure reading.



If a calibration point had already been entered at 30Bar (or approx. this value) a warning message will be displayed. Press the key under OVERWRITE POINT to accept this new calibration point.

Press the ENTER key to return to the "Pressure Calibration" screen.

Press the key under GAS OFF to release the pressure inside the barrel.

Rotate the pressure regulator on the right hand side of the equipment fully anti-clockwise.

Press the key under GAS ON to re-apply the pressure.

Adjust the pressure inside the barrel using the pressure regulator on the right hand side of the equipment and check the readings at approx 5Bar, 10Bar, 15Bar, 20Bar and 25Bar.

If any of the readings differ from the true pressure measured on the reference pressure gauge by more than  $\pm 0.1$ Bar, add a calibration point as shown earlier.

Finally, press the key under GAS OFF to release the pressure inside the barrel.

#### 15.4 PROBE CALIBRATION

Note that a special probe calibration tool (part number 750/35) is required for this procedure.



Also note that the probe must be connected to the PET Plus and the equipment MUST be left switched on for at least 15 minutes BEFORE performing this calibration. The probe is calibrated by moving the probe rod a known distance then indicating this distance into the system.

Press the key under CAL PROBE to display the "Probe Calibration" screen.

Probe Serial Number 105102			
Measured Probe 12.671mm			
Serial	Cal	View	View
Number	Point	Points	Readings

Set the calibration micrometer to 0.000mm then lock the micrometer into its holder so that the micrometer tip is in line with the two reference grooves.

Place the probe into the holder at the right hand side of the vertical column.

Smear petroleum jelly onto the end of the probe rod then attach the calibration tool to the Probe.

Check that the measured probe is 0mm  $\pm 0.01$ mm.





<b>True Probe 1.000mm</b>			
<b>Measured Probe 1.673mm</b>			
<b>Change</b>		<b>Clear</b>	
<b>True</b>	<b>Accept</b>	<b>Points</b>	<b>Cancel</b>

The true probe should automatically display 0.000mm.

If it does not show 0.000mm, press the key under CHANGE TRUE then enter 0mm.

The display will now show 0.000mm for the true probe.

Press the key under ACCEPT to correct the zero probe reading.

<b>True Probe 0.000mm</b>			
<b>Measured Probe 0.000mm</b>			
<b>Change</b>		<b>Clear</b>	
<b>True</b>	<b>Accept</b>	<b>Points</b>	<b>Cancel</b>

If a calibration point had already been entered at 0mm (or approx. this value) a warning message will be displayed. Press the key under OVERWRITE POINT to accept this new calibration point.

Press the ENTER key to return to the "Probe Calibration" screen.

Set the calibration micrometer to 25.000mm.

Check that the measured probe is 25mm +/- 0.01mm.

If the measured probe is not 25mm +/- 0.01mm, press the key under CAL POINT to display the "cal point" screen.

<b>True Probe 25.000mm</b>			
<b>Measured Probe 24.829mm</b>			
<b>Change</b>		<b>Clear</b>	
<b>True</b>	<b>Accept</b>	<b>Points</b>	<b>Cancel</b>

The true probe should automatically display 25.000mm.

If it does not show 25.000mm, press the key under CHANGE TRUE then enter 25mm.

The display will now show 25.000mm for the true probe.

Press the key under ACCEPT to correct the zero probe reading.

<b>True Probe 25.000mm</b>			
<b>Measured Probe 25.000mm</b>			
<b>Change</b>		<b>Clear</b>	
<b>True</b>	<b>Accept</b>	<b>Points</b>	<b>Cancel</b>

If a calibration point had already been entered at 25mm (or approx. this value) a warning message will be displayed. Press the key under OVERWRITE POINT to accept this new calibration point.

Press the ENTER key to return to the "Probe Calibration" screen.

Reset the calibration micrometer to 0.000mm.

Check that the measured probe is 0mm +/- 0.01mm. If the measured probe is not 0mm +/- 0.01mm, enter a new calibration point, overwriting the previous point.

Reset the calibration micrometer to 25.000mm.

Check that the measured probe is 25mm +/- 0.01mm. If the measured probe is not 25mm +/- 0.01mm, enter a new calibration point, overwriting the previous point.

Reset the calibration micrometer to 0.000mm.

Check that the measured probe is 0mm +/- 0.01mm. If the measured probe is not 0mm +/- 0.01mm, enter a new calibration point, overwriting the previous point.

Set the calibration micrometer to 2.500mm.

Check that the measured probe is 2.5mm +/- 0.01mm. If the measured probe is not 2.5mm +/- 0.01mm, enter a calibration point, overwriting the previous point if requested.

Perform the complete calibration by entering points at 2.5mm intervals, i.e. at 5mm, 7.5mm, 10mm, 12.5mm, 15mm, 17.5mm, 20mm, 22.5mm and a new calibration point at 25mm.

## **16.0 NEXYGEN PLUS SOFTWARE**

### **16.1 NEXYGEN PLUS PET**

The optional NEXYGEN Plus PET software (part number 40/0780) is an accessory for use with the PET Plus equipment.

It is a multi-tasking 32 bit Windows 2000 / Windows XP / Windows Vista program which has been designed to be easy to use by all operators including those who have little computer knowledge.

When a TEST SET-UP has been selected, the NEXYGEN Plus PET program will control the barrel temperature, display the test graphs and automatically calculate the required test results, e.g. IV, Degradation factor and MV.

User specified Pass/Fail (specification) Limits can be set for the IV, Degradation factor and / or MV and the software will indicate which samples have passed or failed.

When several tests have been performed, the batch statistics can be viewed either as a data table, an X Bar Range chart or a Histogram.

The NEXYGEN Plus PET program can print test graphs, SPC charts and simple test reports with statistics from its built-in statistical and print features and can also produce custom reports when used together with Microsoft Word 2000 or later.

Therefore, Microsoft Word SHOULD be installed on the computer and it is HIGHLY RECOMMENDED that Office 2000 or Office XP or later is installed if data is to be exported to Microsoft Excel and / or Microsoft Access.

### **16.2 ADVANCED SECURITY**

The NEXYGEN Plus PET software has an optional Security and Audit Trail Feature, which can be configured to prevent unauthorised personnel from using the software.

The benefits of the security features are that the Quality Manager can define the “controlled tests” required for the QC or test department and only authorised operators can perform these tests.

Whenever a test, or sequence of tests, is to be performed, the operator has to “log-on” to the software using a previously specified log-on name and user password. This logging on and off is stored in a separate audit database, which can be viewed by the Quality Control manager to monitor who has been performing the tests.

This ensures that only trained operators can perform tests so minimises the chances of the test being incorrectly performed and incorrect data being reported. The NEXYGEN Plus PET also enters the log-on name into the test file against each test.

The Quality Control manager can configure the tests to ensure that the operators can only work within specified test conditions. These restrictions can be used to prevent the operator from creating new “uncontrolled tests”, changing the test temperature or permanently deleting test data etc.

If the Quality Manager subsequently changes any “controlled test”, the changes are stored into the audit database to provide an audit trail. This database can also store the reason for the changes so that full traceability of test changes is recorded.

### 16.3 INSTALLING THE SOFTWARE

Insert the CD and the set-up wizard will be automatically shown:

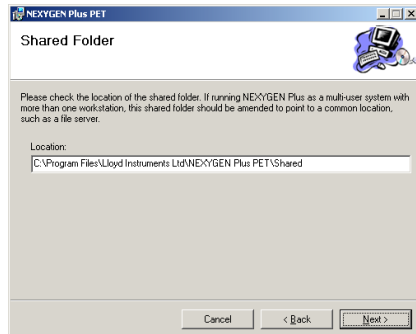
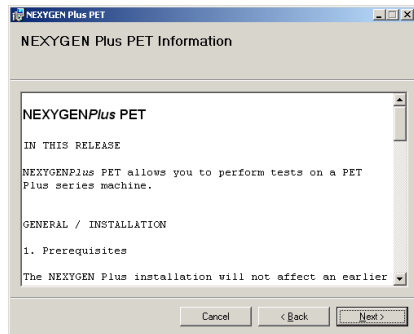
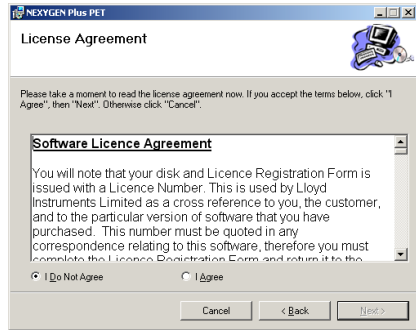
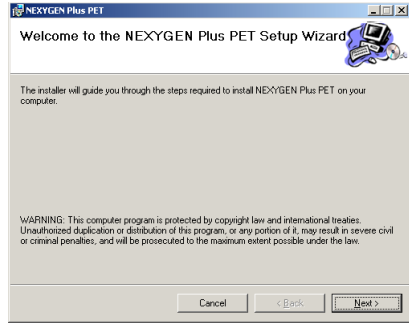
Click on the NEXT button to proceed.

Select the "I Agree" option then click on the NEXT button.

Click on the NEXT button to proceed.

The dialog shows the default shared folder and this can be changed if required.

Click on the NEXT button to proceed.



The dialog shows that the Security Feature is not enabled.

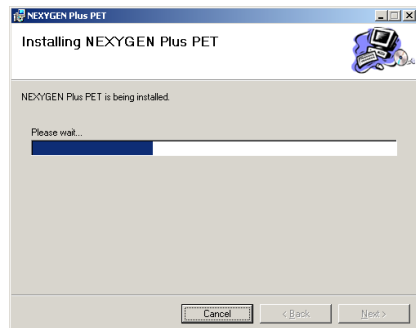
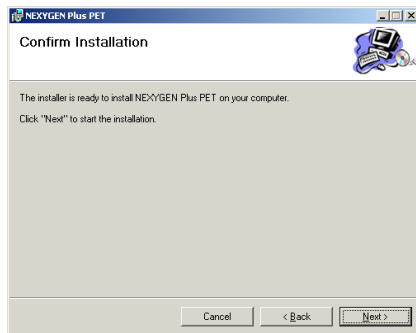
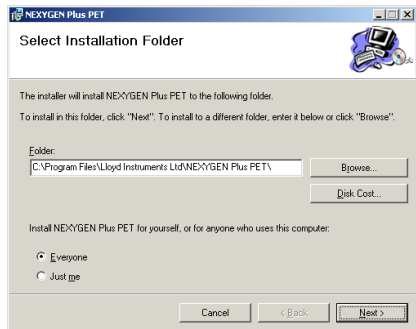
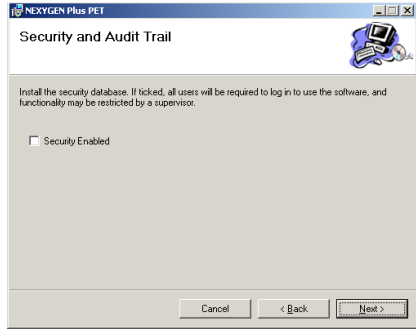
Click on the NEXT button to proceed.

The dialog shows the default program folder and this can be changed if required.

Select the "Everyone" option to create a Start Menu item for all users then click on the NEXT button to proceed.

Click on the NEXT button to install the main part of the program.

The dialog loading status bar will be displayed during the installation process.



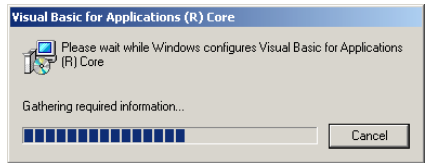
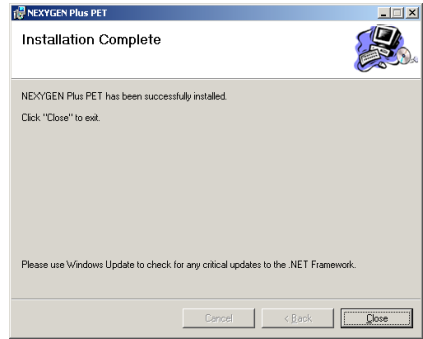
When the NEXYGEN Plus PET has been installed, a confirmation screen will be displayed as shown.

Click on the CLOSE button then the installer will automatically install the VBA components that are required for NEXYGEN Plus PET.

When the screen closes, the NEXYGEN Plus PET program is installed.

To complete the configuration, leave the CD in the drive then start the NEXYGEN Plus PET using the Windows Start button.

A screen similar to that shown will be displayed while the VBA is automatically configured.



#### 16.4 CONFIGURING THE SOFTWARE CONSOLE

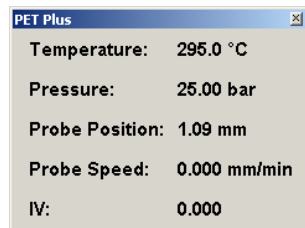
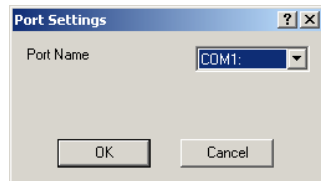
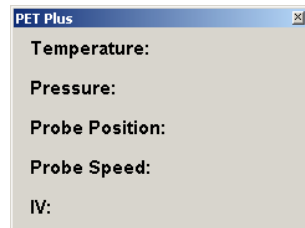
Connect the supplied RS232 cable (part number 09/0639) between the rear of the PET Plus machine and an unused COM port on the computer.

Start the Software Console by clicking on the Windows START button then selecting NEXYGEN PLUS PET then selecting PET PLUS SERIES CONSOLE.

Right mouse click on the software console then select PORT SETTINGS.

Select the appropriate COM port then check that the console shows the Temperature, Pressure and Probe Readings.

The NEXYGEN Plus program is now ready for use.

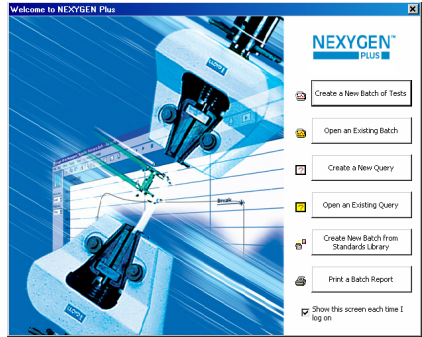


## 16.5 CREATING A TEST FILE

Note that the test parameters (test temperature, die dimensions and pass/fail limits) MUST be set in the NEXYGEN Plus PET test set-up.

The parameters stored in the PET Plus are NOT used when performing a test using the NEXYGEN Plus PET software.

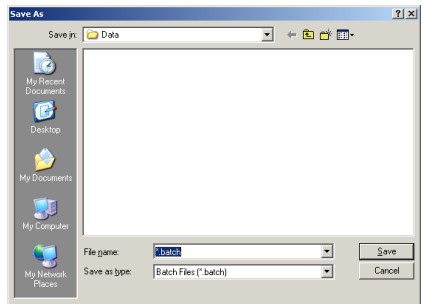
Click on the CREATE A NEW BATCH OF TESTS button on the Welcome screen shown.



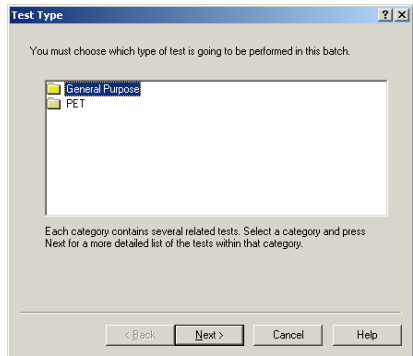
Note that if the Welcome screen is not displayed, it can be displayed by clicking on the 9th Icon from the left on the toolbar.

A SAVE AS dialog will be displayed as shown.

Enter the required name for this test file noting that the file can be saved in any location, e.g. a networked folder.



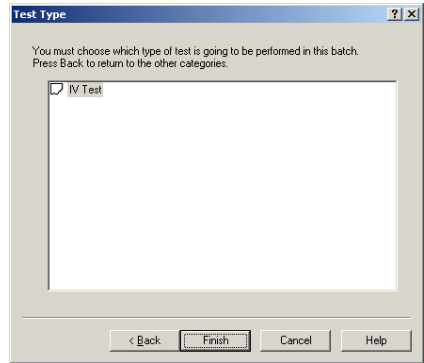
Click on the SAVE button to display the TEST TYPE screen shown.



Click on the PET icon then click on the NEXT button.

Click on the IV TEST Icon then click on the FINISH button.

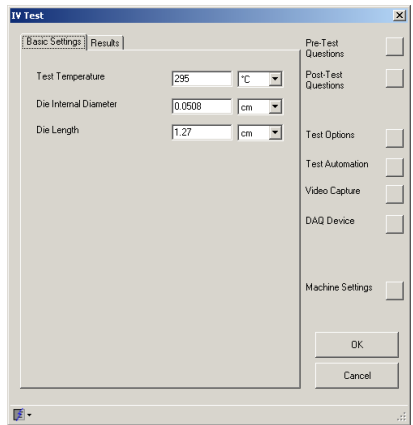
This action will automatically create the Test File then display the TEST CONFIGURATION screen.



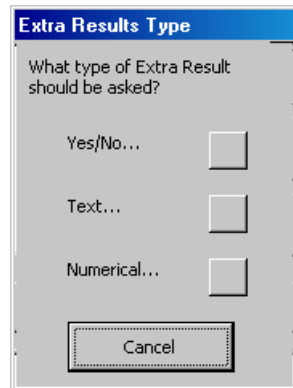
Enter the required test temperature and the die dimensions as shown on the die certificate.

If sample identification is required for traceability of results, a pre-test dialog can be displayed to enter this information.

To define any pre-test questions, click on the PRE-TEST QUESTIONS button on the right hand of the screen to display the EXTRA RESULTS screen shown.



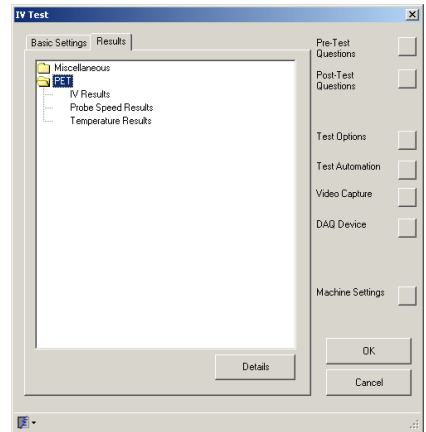
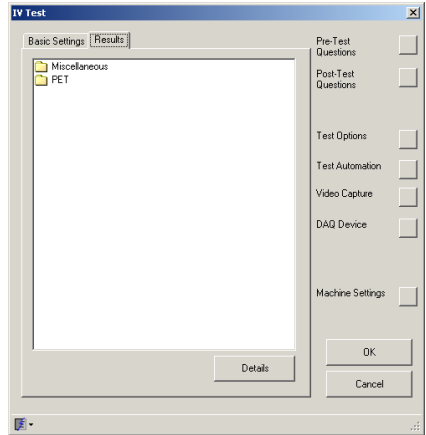
Click on the NEW button to display the EXTRA RESULTS TYPE screen then select which type of question is required.





Define as many questions as required noting that each question can be configured to accept Boolean (True/False - Yes/No etc), text, numbers or quantities (numbers with units, e.g. 10g) as required.

Click on the RESULTS tab then click on the PET Icon to display the available test results.

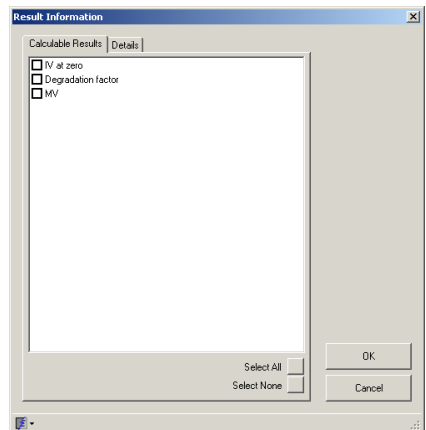


Click on the required result title (e.g. IV Results) to display the available results.

Select the required results using the checkboxes.

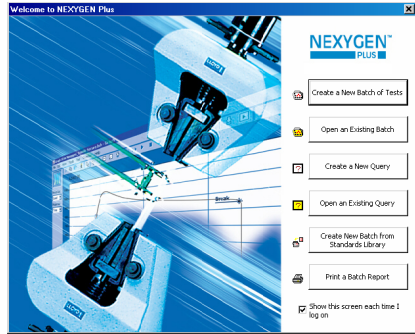
Note that the selected results can be modified at any time after the test has been performed.

Click on the OK button 2 times to close the TEST CONFIGURATION screen.



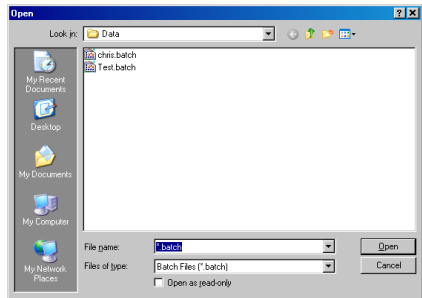
## 16.6 PERFORMING A TEST

Click on the OPEN AN EXISTING BATCH button on the Welcome screen.



Note that if the Welcome screen is not displayed, it can be displayed by clicking on the 9th Icon from the left on the toolbar.

An OPEN dialog will be displayed as shown.

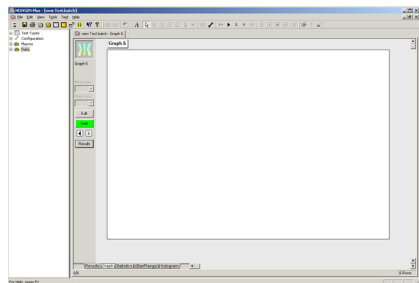


Select the required test file then click on the OPEN button.

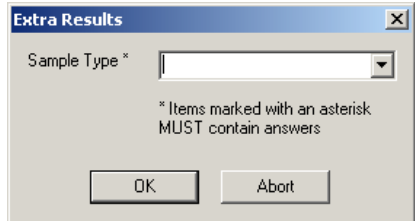
Note that the above steps do not have to be performed if the test is run immediately after the test file has been defined as shown in the previous section.

The graph screen will be displayed as shown.

Click on the green START TEST button at the left hand side of the graph to start the test.



If any pre-test questions have been defined, a dialog will be displayed requesting the answers.



The display on the PET Plus will automatically change to the “PC Pre-Test” screen.

```
Set Temp 295.0C   Barrel Temp 295.0C
Pressure 0.0Bar   Probe 15.85mm
Insert sample + follower then press GO
Gas Off   Purge   Full
```

Note that the “PC Pre-Test” screen will NOT be displayed if the probe is not electrically connected to the probe socket on the rear of the vertical column. If the probe is not connected, the warning screen below will be shown.

```
ERROR CONDITION
Probe NOT Connected

Press ENTER to Cancel
```

Also note that the intermediate screen shown below will be displayed if the barrel is not at the required temperature.

```
Set Temp 295.0C   Barrel Temp 292.8C
Under Computer Control
Waiting for barrel temp to stabilise
Suspend
```

This screen will automatically change to the “PC Pre-Test” screen when the barrel has stabilised at the required temperature.

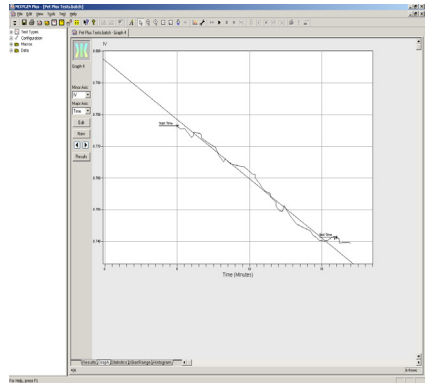
Note that the equipment MUST be set to the “PC Pre-Test” screen BEFORE trying to test a sample.

The test is performed in exactly the same way as for a stand-alone system, i.e. the sample is inserted into the barrel then the GO button is pressed.

Note that the computer now requires no user intervention until the end of the test.

NEXYGEN Plus PET will initially plot the barrel temperature and nitrogen pressure and will then plot the probe position, probe speed and instantaneous IV at the relevant times during the test.

At the end of the test, the "Line of Best Fit" will be drawn through the IV values and the IV at Zero will be calculated. The selected test results will also be calculated and stored in the data table.



To perform the next test, click on the NEW button at the left hand side of the graph.

The button will change to a green START TEST button and clicking on this button will perform the next test.

When 2 or more tests have been performed in a batch, the graphs can be quickly displayed sequentially by clicking on the LEFT or RIGHT buttons at the left hand side of the graph.

To view the data table, click on the RESULTS button at the left hand side of the graph or click on the RESULTS tab at the bottom of the screen to data table will display the results as shown.

	Date	Time	Sample Type	IV at zero	Degradation factor	MV
1	01/07/2...	09:33:37	Eastman 0.8	0.789	-0.004	543.00
2	01/07/2...	10:00:11	Eastman 0.8	0.790	-0.003	544.00
3	01/07/2...	13:39:59	Eastman 0.8	0.805	-0.004	600.00
4	01/07/2...	14:00:45	Eastman 0.8	0.797	-0.004	570.00
5	01/07/2...	14:34:58	Eastman 0.8	0.787	-0.003	534.00
6	01/07/2...	15:06:48	Eastman 0.8	0.798	-0.003	575.00

Results | Graph | Statistics | X-Bar | Range | Histogram

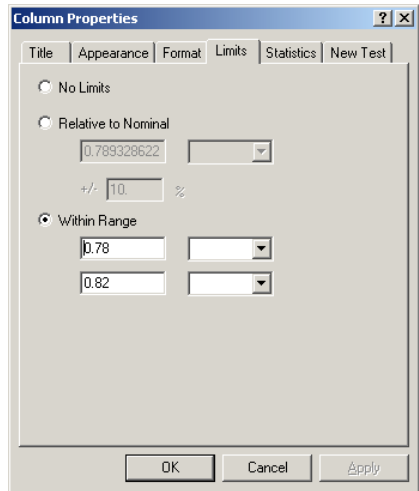
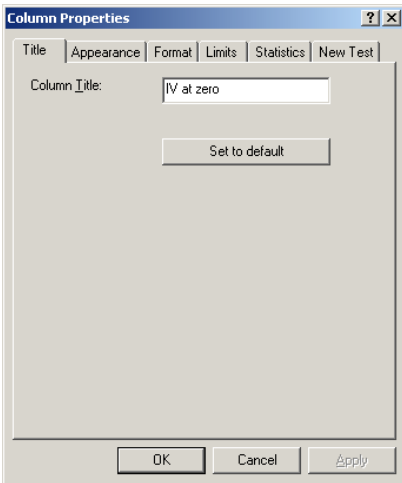
6 Rows

A PASS/FAIL indication can be provided by specifying the PASS/FAIL limits for one or more columns.

Right mouse click on the required column, e.g. "IV at Zero" then select PROPERTIES to display the dialog shown.

Click on the LIMITS Tab at the top of the screen then set suitable PASS/FAIL limits, e.g. between 0.78 and 0.82.

Note that BOTH limits MUST be defined.



Click on the OK button to close the dialog and display the data table in colour. Any rows shown in green pass the PASS/FAIL parameters and any rows shown in red fail the parameters.

Date	Time	Sample Type	IV at zero	Degradation factor	MV	
1	01/07/2...	09:33:37	Eastman 0.8	0.789	-0.004	543.00
2	01/07/2...	10:00:11	Eastman 0.8	0.790	-0.003	544.00
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4	01/07/2...	14:00:45	Eastman 0.8	0.797	-0.004	570.00
5	01/07/2...	14:34:58	Eastman 0.8	0.787	-0.003	534.00
6	01/07/2...	15:06:48	Eastman 0.8	0.798	-0.003	575.00

Results (Graph) (Statistics) (X-Bar/Range) (Histogram) 6 Rows

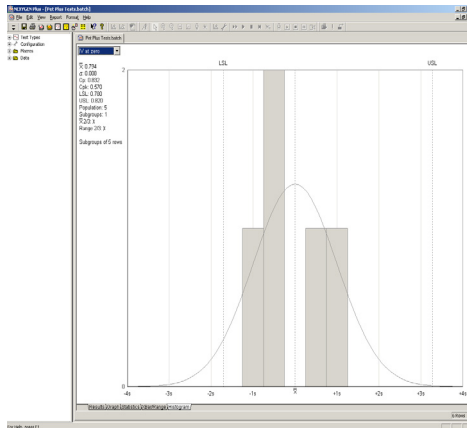
The statistics can be viewed by clicking on the STATISTICS Tab at the bottom of the screen.

	Maximum	Minimum	Mean	Median	Coefficient of Variance	Standard Deviation (N)	Standard Deviation (N-1)
IV at zero	0.805	-0.787	0.794	0.793	0.81%	0.006	0.007
Degradation factor	-0.003	-0.004	-0.004	-0.004	12.26%	0.000	0.000
MV	600.00	534.00	561.00	557.00	4.08%	22.876	25.060
Number of Rows that Passed	6						
Number of Rows that Failed	0						

The XBAR/RANGE and/or HISTOGRAM charts can also be viewed. Note, however, that 10 or more rows are normally required to view the XBAR/RANGE chart because this normally groups the test results into groups of 5 rows.

Note that the XBAR/RANGE and HISTOGRAM charts can ONLY be displayed if PASS/FAIL parameters have been defined for one or more test results.

A histogram chart is shown.



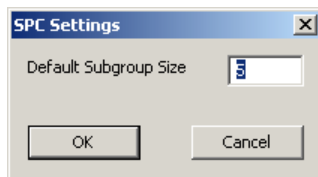
This chart shows the LSL and USL values along the top of the chart and the grey blocks indicate the data distribution. The “Bell Curve” is also displayed for information.

The Cp and Cpk values are calculated and are displayed at the left hand side of the chart.

The “Default” Sub-Group size is 5 but this can be changed by selecting the Main Menu item of EDIT, SPC SETTINGS as shown.

To save the test file, select FILE, SAVE from the Main Menu.

For further information regarding the many features of the program, please refer to the comprehensive on-line HELP within the software.



## 17.0 TECHNICAL SPECIFICATIONS

### IV Measurement

Range	Minimum 0.45 using 0.015" Bore Die Maximum 1.40 using 0.040" Bore Die
Barrel	Insulated stainless steel barrel with replaceable stainless steel liner
Dies	One die is supplied with the equipment (size specified at time or order) but additional dies may be purchased as required. Dies are available with bores of 0.015", 0.020", 0.030" and 0.04"
Heaters	3 x 250 W cartridge type symmetrically positioned for optimum temperature control
Temperature Range	50°C - 400°C
Temperature Control	Solid state, electronic 3 term controller with set point adjustment
Over temperature Device	Secondary overheating safety limit, factory set to 400°C
Nitrogen Gas	Oxygen free and less than 5ppm of water specification (White Spot). Recommended 200 Bar capacity bottle
Pressure Input	30 Bar (440 psi) Maximum 35 Bar (510 psi)
Working Pressure Range	7 - 30 Bar
Pressure Measurement	Electronic pressure transducer
Mounting	Bench mounting. Enough bench space will also be needed for the optional computer, key board and possibly printer
Optional Computer	PC compatible. Optional software supplied on CD
Probe Unit	Linear Voltage Displacement Transducer encased in non-magnetic canister
Ancillary Equipment (optional)	Polymer Drier, Vacuum Pump, Polymer Grinder, Polymer Chip Transfer Vessel, Industrial Grinder and Melt Pot
Electrical Supply	230V AC $\pm$ 10% 50/60Hz or 115V AC $\pm$ 10% 50/60Hz Not adjustable. <b>Mains Power Must be specified at time of order.</b>
Power Consumption	
PET Plus	800 VA (max)
Polymer Dryer	1000 VA (max)
<b>Fuses</b>	
PET Plus	Mains In 230V AC, 2 x T5AH250V Mains In 115V AC, 2 x T10AH250V
Polymer Dryer	Mains In 230V AC, 2 x T10AH250V Mains In 115V AC, 2 x T10AH250V
Operating Temperature	- 5°C to 35°C
Storage Temperature	-20°C to 55°C (must be substantially dry)
Relative Humidity	0 to 80% (non-condensing)
<b>Net Instrument Weights</b>	
PET Plus	34kg
Polymer Dryer	15kg
Polymer Grinder	41kg
<b>Dimensions (HxWxD)</b>	
PET Plus	64cm high (94cm with probe fitted) 47cm wide, 42cm deep
Polymer Dryer	45cm high (with tubes fitted), 36cm wide, 45cm deep
Polymer Grinder	40cm high (52cm high when open), 40cm wide, 54cm deep



#### International Symbols

##### WEEE Directive

This equipment contains electrical and electronic circuits and should not be directly disposed of in a landfill site.



##### RoHS

This product is RoHS and China RoHS compliant. This symbol indicates the equipment contains some restricted hazardous substances above the recommended level, and may offer potential harm to the environment after 15 years from date of manufacture.

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