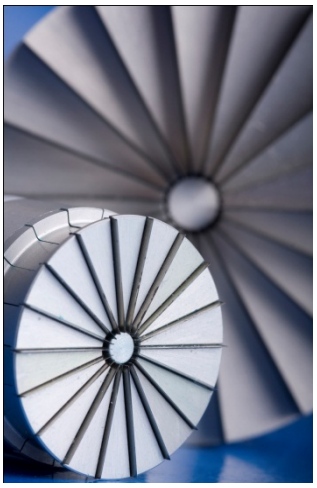
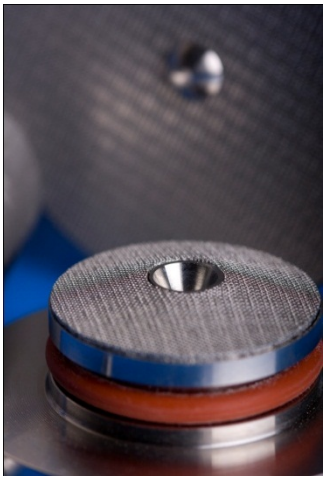
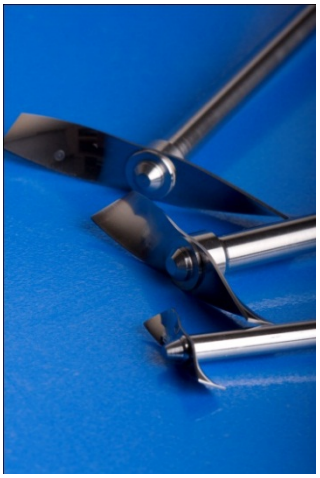
FT4 Powder Rheometer® User Manual







freeman*technology*
a micromeritics® company

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COMPANY PROFILE

Freeman Technology specialises in systems for measuring the flow properties of powders and has over a decade of experience in powder flow and powder characterisation. The company invests significantly in R&D and applications development, and provides detailed know-how alongside its universal powder tester, the FT4 Powder Rheometer®. Expert teams guide and support users around the world in addressing their individual powder challenges, focusing on delivering the most relevant information for the process. The result is world-leading solutions for understanding powder behaviour - in development, formulation, scale-up, processing, quality control, or anywhere that powders have a role.

The FT4 Powder Rheometer uses patented dynamic methodology, a fully automated shear cell (in accordance with ASTM Standard D7891) and several bulk property tests, including density, compressibility and permeability, to quantify powder properties in terms of flow and processability. Systems are installed around the world in the chemical, pharmaceutical, toners, foods, powder coatings, metals, ceramics, cosmetics, and many other, industries. They deliver data that maximise process and product understanding, accelerating R&D and formulation towards successful commercialisation, and supporting the long term optimisation of powder processes.

Founded in 1989 as a developer of automated testing systems for materials characterisation, the company has focused exclusively on powders since the late 1990s and in 2018 became part of Micromeritics Instrument Corporation. The company's R&D, manufacturing and commercial headquarters are in Gloucestershire, UK, with operations and distribution partners in key global territories.

In 2007 the company received the Queen's Award for Enterprise in Innovation and in 2012 the Queen's Award for Enterprise in International Trade.

PRECAUTIONS

⚠ CAUTION

Please note that the FT4 is supplied without anti-virus software pre-installed. Every effort is made to ensure the instrument is free from viruses at the point of delivery but it is highly recommended that your organisation's virus protection procedures are implemented prior to connecting the FT4 to your network or using electronic storage devices.

Please also be aware that this instrument contains moving parts. Bodily injury may occur if you attempt to handle the blade assembly when in operation. A safety door is in place and the interlocking system prevents movement should the door be left open.

We strongly recommend you read this manual thoroughly prior to operating the instrument.

If you require any further guidance please contact Freeman Technology or one of our approved partners.

FT4 USER MANUAL CONTENTS

SECTION 1 – GETTING STARTED

INSTALLATION

- Electrical connections are as indicated in Appendix 1. Mains AC supply may be in the range 90V to 264V, 1.6A max.
- The FT4 Powder Rheometer should be set up on a stable work surface with access to a wash basin with hot and cold water for cleaning vessels, blades, etc.
- All usual safety procedures need to be implemented when handling and testing powders. The instrument may be set up in a fume cupboard if necessary.
- When the vessels and blades are not in use, store in their respective compartments in the cases provided.

POWERING UP THE FT4

- Switch on the power using the keypad at the top of the instrument.
- The green LED should illuminate and the FT4 will boot into the Windows environment. Hard disk activity is indicated by the red LED.
- The blue ‘stop’ button at the bottom of the instrument should also become illuminated if not depressed.

RUNNING THE SOFTWARE APPLICATIONS

- Two icons should be present on the Windows desktop. Double clicking on either of these will run that application.

LOGGING ON

- After double clicking on the Powder Rheometer icon the log on screen should appear, please see Figure 1 on following page.
- The default administrator logon details are: -

  User Name: admin
  Password: admin

  Note that this password can be changed at any time, however the User Name for this account is fixed.
- An authorised user must enter their user name and password correctly to gain access.
- An authorised operator may change their password by first logging on and then selecting ‘Edit User' from the 'Create / Edit Users' menu.
- The system administrator is required to create new user accounts (see Section 6).

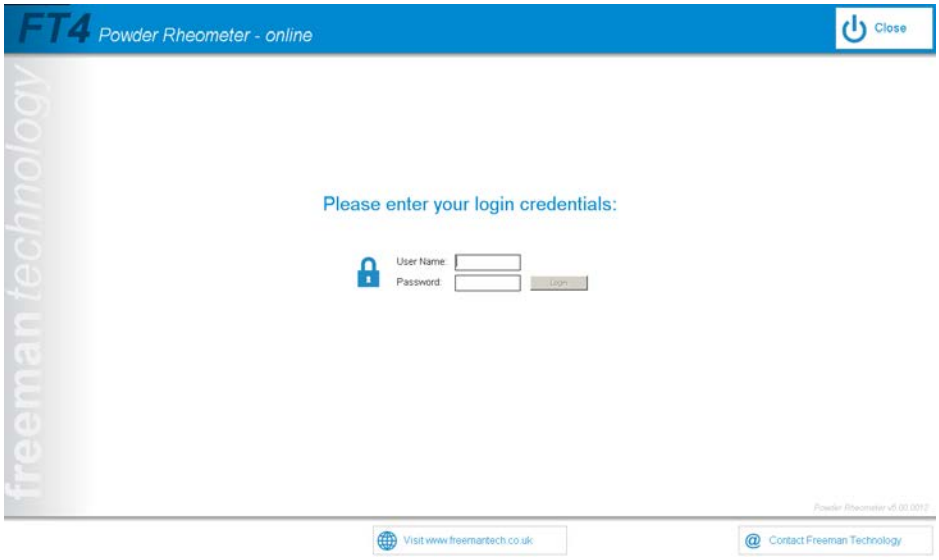


Figure 1 - Log on Screen

DIRECTORY STRUCTURE

- The Powder Rheometer and Data Analysis programs are installed into C:\Program Files\Powder Rheometer and C:\ Program Files\Freeman Technology\Data Analysis folders respectively.
- The directory structure of all the folders used by the programs is shown below in Figure 2, the Rheometer directory can be found in C:\. The structure should not be modified.

Figure 2 - Directory Structure

SECTION 2 – RUNNING A TEST

PREPARING THE SAMPLE

The instrument is very sensitive and will detect small variations in sample preparation. It is strongly suggested that a set of procedures are defined and used consistently. Some things to be considered are:

- The need for rigorous cleanliness of blades and vessels.
- Cross contamination from previous test samples.
- Condition of sample prior to test, e.g. moisture levels.

SELECTING THE APPROPRIATE VESSEL AND BLADE

- Various sizes of blade and vessel are available.
- The 50mm borosilicate glass vessel and 48mm blade is the standard combination.

PROTECTING THE BLADE

The blade and spindle (blade assembly), are precision engineered and must be protected from damage. The blade geometry is critically important. It is made from a high tensile strength stainless steel and is therefore not easily damaged. However every precaution should be taken to avoid dropping, bending or marking the blade. The repeatability of the test results is very dependent upon this.

It is recommended that the blade case should be kept adjacent to the rheometer and used to store the blade when not fitted to the instrument.

FITTING THE BLADE AND OTHER ACCESSORIES TO THE INSTRUMENT

- Insert the spindle into the hub as shown in Figure 3 below. Note that there are only two rotational positions in which the spindle can be correctly inserted.
- Ensure that the spindle is fully inserted, i.e. the circlip is in contact with the hub as shown in the drawing below.
- Tighten the nut by hand. Note that it is not possible to damage the instrument when tightening by hand.
- **WARNING** – the blade assembly should be handled by the spindle only.

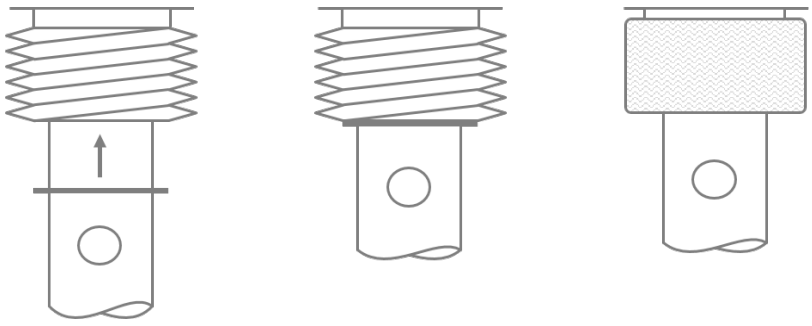


Figure 3 - Spindle Hub

SECTION 2 – RUNNING A TEST

FITTING THE VESSEL TO THE RHEOMETER TABLE

- Firstly, assemble the appropriate vessel following help files W7020, W7021 or W7022.
- Please note that it is necessary to remove the 50mm & 62mm Vessel Clamping Assembly, if fitted. To attach the 25mm vessel, insert the 25mm Vessel Clamping Assembly into the side of the table and then place the vessel centrally on the table. Ensuring that the thumb screw is loose, locate the fingers of the clamping assembly into the slots on the clamp ring. Tighten the thumb screw to secure the vessel to the table.
- Please note it is necessary to remove the 25mm Vessel Clamping Assembly, if fitted. The mechanism for the 50mm and 62mm vessels consists of a table bar and two thumb screws. If these are not already installed on the FT4, remove one of the thumb screws and insert the table bar into the side of the table. Reinstall the second thumb screw and then place the assembled vessel on the table before equally tightening the thumb screws.

USE OF THE SAFETY DOOR

- This is interlocked to prevent movement of the FT4 carriage / spindle whilst the door is open. Close the door before attempting to begin a test.
- Opening the door during a test will stop the instrument immediately. To restart, close the door and click 'OK' in the 'Door Open' message box.

THE MENU STRUCTURE

All features are accessed through the buttons on the main screen. The options available are:

- Standard Test Programs – run standard tests as supplied with the FT4 Powder Rheometer.
- Advanced Test Programs – provides access to less commonly used and user modified test programs.
- Create / Edit Users – Administer Powder Rheometer users including password management and access rights.
- Calibration / Settings Menu – allows users to carry out audit calibrations of the FT4 and ACU, back up, modify and restore system settings.

STARTING A TEST USING STANDARD TEST PROGRAMS

Click on Standard Test Programs and select a Standard Methodology from the following screen. Then proceed to the steps outlined on the next page.

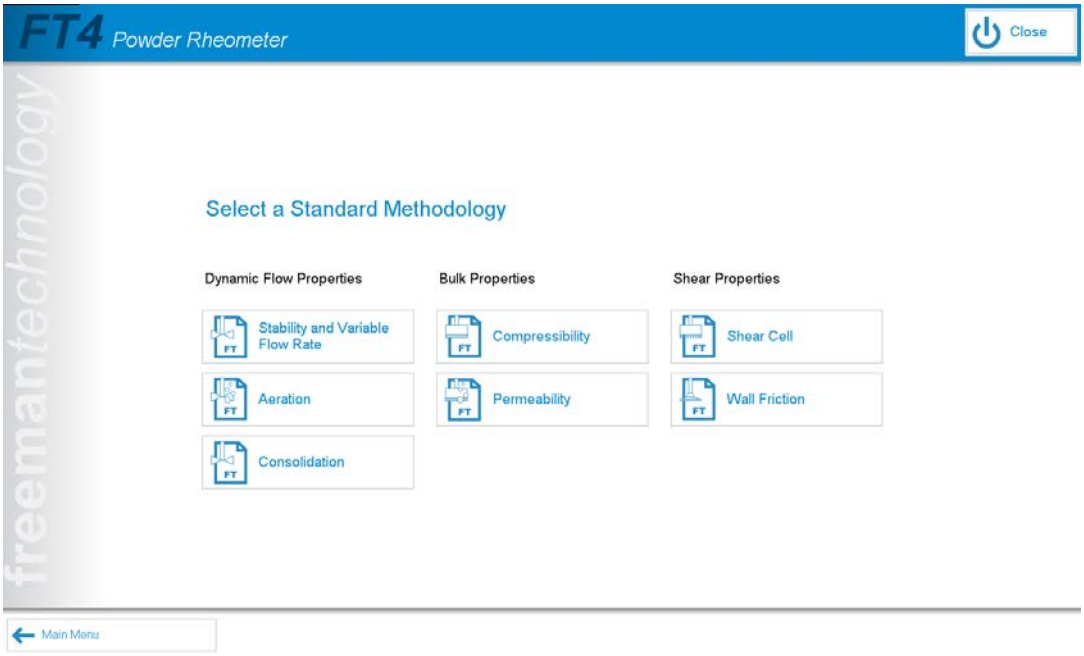


Figure 4 –Standard Methodology Selection

STARTING A TEST USING ADVANCED TEST PROGRAMS

Click on Advanced Test Programs, select a program from the program library and click 'Run'. Then proceed to the steps outlined on the next page.

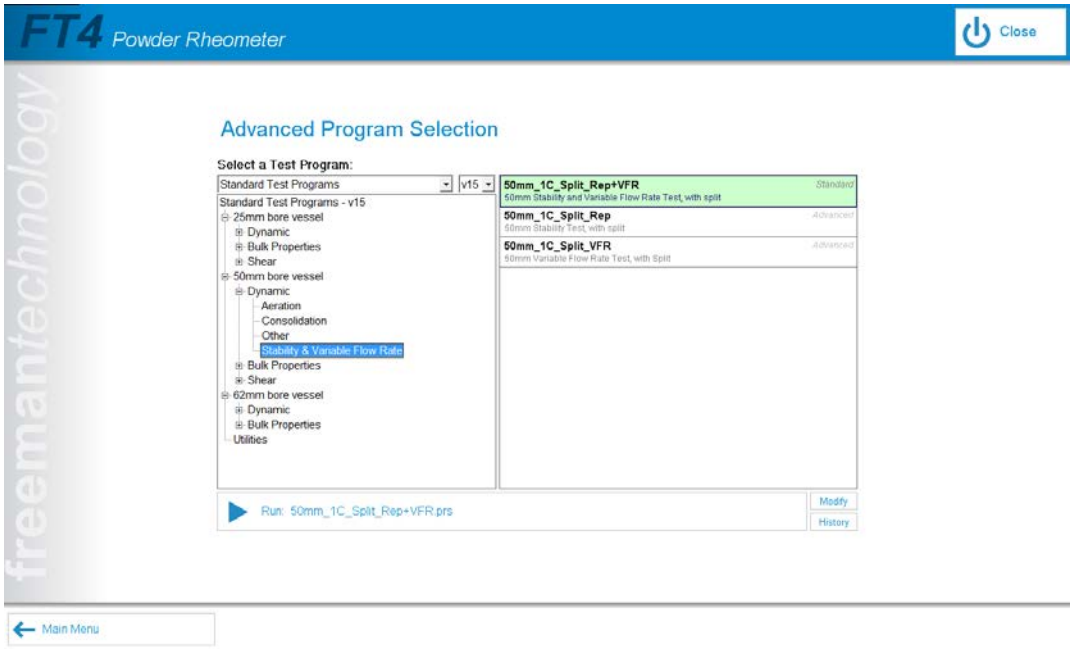


Figure 5 – Advanced Program Selection

SECTION 2 – RUNNING A TEST

RUNNING A TEST

After selecting a Standard Test Program or an Advanced Test Program proceed with the following steps:

1. Click on the required vessel size.
2. Insert the recommend accessory into the FT4 hub and click the ‘Confirm Blade’ box.
3. Assemble the recommended vessel and tick the ‘Confirm Vessel’ box.
4. Place the assembled vessel on to the FT4 table and when the balance has stabilised, click ‘Tare Empty Vessel’.
5. Fill the vessel with the sample, return the filled vessel to the FT4 table and when the balance has stabilised, click ‘Record Sample Mass’.
6. Record optional information about the test sample in the Material, Batch Code and Reference No. fields. Additional fields can be added by clicking ‘Modify Test Label Options’.
7. Click ‘Start Test’.
8. Real time measurements can be viewed during the test procedures.
9. The y-axis scale is automatically adjusted to display all recorded information. This feature can be disabled by deselecting the tick box at the bottom-left corner of each graph as necessary using the up and down arrows on the left of the plotting window.
10. Total Energy values are plotted in real time at the bottom right corner of the screen.
11. Test information is displayed in the right-hand column. Variables available to the operator, such as enable/disable ‘Shake Blade’ can be accessed via the Test Options tab.
12. 'Pause' can be selected during the test to temporarily halt the test procedure.
13. Selecting 'End Test' will immediately terminate the test procedure and the carriage will return to the parked position. Any data measured up until this point can be saved.
14. If the test program is not terminated prematurely by the operator, it will finish automatically and the operator will be required to save the test file.

NAMING AND SAVING A TEST FILE

At the end of the test, the data can be saved and the following options are available:

- Location – the directory where the test file will be saved. Click 'Change' to select an alternative location.
- File Name – the name to be assigned to the test file. This is generated automatically using the name of the test program and details entered at the start of the test. The information used in the automated naming process can be modified by clicking 'Options'.
- Run – allows the operator to allocate a run number to the file in the event that repeat tests are being performed.
- Notes – a free text box allowing any observations to be recorded.

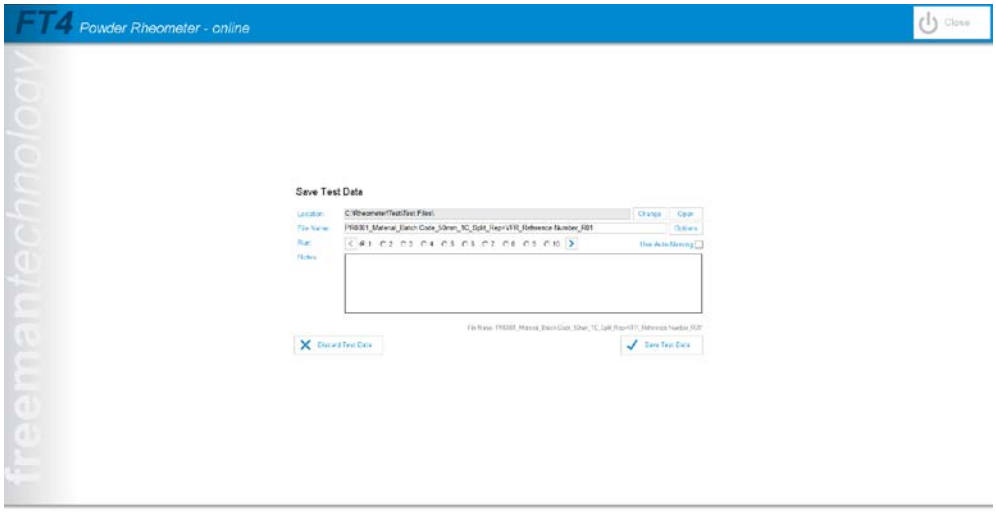


Figure 6 – Save Test Data Screen

The file is given a PRB extension and is saved as a read only file. The raw data can be viewed in Excel but must be converted into a CSV file in Data Analysis (see Section 4).

WHAT HAPPENS IF A FORCE OR TORQUE OVERLOAD OCCURS DURING A TEST?

- In the event of force or torque levels exceeding the set maximum levels, the system will stop and alert the user with a message box. Should this occur the program is terminated and a more suitable program for the material should be selected. Please contact Freeman Technology for assistance if required.
- The data recorded up to the point of overload is available and can be saved and analysed.

INSTRUCTIONAL VIDEOS

TEST	**VIDEO LINK**
Stability & Variable Flow Rate (Rep + VFR)	English | Chinese
Compressibility	English | Chinese
Permeability	English | Chinese
Aeration	English | Chinese

Shear Cell	English | Chinese
Wall Friction	English | Chinese
Tapped Consolidation	English | Chinese

SECTION 3 - THE TEST PROGRAM

TEST PROGRAM STRUCTURE

- A sequence of **Blocks** that define the Test Program – see below for further details.
- A program description that summarises what the Test Program does.
- The starting accessories (vessels, blades, pistons, shear cells, etc.) compatible with the Test Program.
- Autoturn settings.
- All equations that have been defined for the Test Program.

DEFINITION OF BLOCKS AND THEIR VARIABLES

Block	Function	Variable
Conditioning Cycle	Used to condition the powder prior to a Test Cycle or other process step (consolidation via tapping, etc.) Uses the standard blade to establish a homogenised, low stress packing in the powder.	Helix angle – angle of helical path that blade moves along on down/up traverse. Tip speed – speed in mm/s at the end of the blade tip. Note that this is not simply rotational speed, but the speed along the helical path. Height – the position in mm to which the blade will move to. Once the blade reaches this point, the test program moves on to the next step. Up Levelling – this allows the blade to adopt the settings defined here in order to produce a level powder surface once the blade has exited the powder bed. Used mainly when a Conditioning Cycle precedes a compaction step.
Test Cycle	Used to measure the Flow Energy of the powder, either after conditioning, aeration or consolidation.	Variables as Conditioning Cycle. Also includes an 'Associate Test Variable' option which allows information to be assigned to that individual test cycle (for example, air velocity during an Aeration test).
User Defined Cycle	Can be configured either as a Conditioning or a Test Cycle.	Variables as Conditioning Cycle. Main difference is that each variable can be set with finer resolution, compared to the limited standard options available with Conditioning or Test Cycles.
Traverse Zone List	A single movement or sequence of movements within one Block.	Variables as Conditioning Cycle. Provides the ability to move the blade in different flow modes during specific parts of a traverse. A traverse can therefore be made up of multiple zones, each with different variables if required.

Park	Returns the carriage to the Park position.	Vessel to be split – tells the software to recalculate the mass of powder in the vessel after the 'OK' button is clicked. Funnel to be removed – inserts a message box requesting that the funnel is removed. This step precedes the 'Vessel to be split' function in the actual program sequence and ensures that the mass calculation during the split does not include the mass of the funnel also. Change – Tells the software that the spindle accessory needs to be changed (for example after conditioning with the blade and before consolidation with the piston). Create Automatic Message – selecting this button automatically creates the message that appears in the message box during the test, based on the status of the other Park variables. If required, a message may be manually typed into this area and will overwrite any automatically created message. Restart at the following height inside the vessel – defines the height at which the spindle accessory moves to after the Park step and before starting the next Block.
Move	Moves the spindle accessory to a specific height.	Distance – moves the spindle accessory either to a specific height or by a defined amount, either upwards or downwards. Helix Angle – the angle at which the spindle accessory moves during the Move Block. Normally set to 90 degrees. Tip Speed – the speed at which the tip of the blade or outside edge of the piston moves at during the Move Block. When the Helix Angle is set to 90 degrees, this Tip Speed defines the vertical speed.
Rotate at Height	Allows the spindle assembly to be rotated at a fixed height.	Speed – can be set as Tip Speed or spindle revolutions per minute. Duration – can be set to duration of rotations, number of rotations or number of degrees.

Apply Force	Used to apply a force or normal stress to the powder. Typically uses the compaction piston.	Compact to Normal Force – sets the target force, in Newton. This can also be defined as a normal stress, in kPa, by changing the Options at the bottom of the screen. Hold – allows the target force to be held for a precise time. Alternatively, once the target force has been reached, the position is held for a precise time (this position control allows for potential force decay as the powder relaxes). The final option is to simply move to the next Block. Helix Angle – the angle at which the piston moves during the Apply Force Block. Normally set to 90 degrees. Carriage Speed – the maximum vertical speed at which the piston moves during force control. This value is automatically attenuated as the measured force reaches the target force. Do not compact below – a safety limit, which prevents the piston moving below a certain height in the event that the target force is not reached.
Shear Cell	Used to measure shear properties under a controlled normal stress (ASTM D7891).	Normal Stress – the consolidation stress imposed during the pre-shearing or shear step. This can also be defined as a normal force, in Newton, by changing the Options at the bottom of the screen. Shear for – number of degrees of rotation during the pre-shear or shear step. Control Speed - the maximum vertical speed at which the piston moves during force control. This value is automatically attenuated as the measured force reaches the target force. Shear Rate – the angular speed of shearing during the pre-shear or shear step. Auto Analyse – defines which data is automatically displayed when the test file is imported into Data Analysis.
Wall Friction	Used to measure wall friction properties under a controlled normal stress (ASTM D7891).	See Shear Cell.
Display Message Box	Inserts a message box and requires the operator to click 'OK' to continue.	Message – any text information can be typed here and will be presented during the test program. The program will not continue until the operator clicks 'OK'. Could be used to advise the operator to measure the temperature or electrostatic charge for example.
Time Delay	Inserts a time delay.	Pause – defines the period at which the instrument pauses and halts motion of the blade or accessory. During this time a message can be displayed, if entered in the Display Message box. This feature could be used if waiting for X seconds whilst the powder de-aerates, for example.

Tare Table Weight	Re-tares the Force measurement.	Used during a test program if it is necessary to re-tare the force. For example, after liquid or powder addition.
Aeration Control	Sets the air velocity provided by the Aeration Control Unit (ACU).	Turn Air Off – instructs the ACU to set the air supply to zero mm/s. Turn Air On – when enabled, allows a required air velocity to be set.
Permeability	Used to supply an air velocity through the powder whilst applying a consolidating normal stress with a compaction piston.	Measure – defines whether to measure air pressure as a function of normal force and air velocity, or air velocity as a function of normal force and air pressure. Normal Force – sets the target normal force for the measurement. This can also be defined as a normal stress, in kPa, by changing the Options at the bottom of the screen. Air Velocity / Pressure – sets the air velocity or air pressure (depending on the 'Measure' setting above) for the measurement. Control speed - the maximum vertical speed at which the piston moves during force control. This value is automatically attenuated as the measured force reaches the target force. Hold Time – the duration for which the force and air velocity are maintained. Minimum Height – a safety limit, which prevents the piston moving below a certain height in the event that the target force is not reached.

HOW TO CREATE OR EDIT AN EXISTING TEST PROGRAM

- Creating a new program is done by editing an existing program and saving it with a new program name.
- Select 'Advanced Test Programs' from the Main Menu.
- Select a program from the program library and click 'Modify'.
- Using the 'Program Editor' modify the settings as required.
- Block types can be selected from the menu on the left of the screen and inserted into the program using the 'Add' button. Using the 'Change' button changes the block type (for example, from Conditioning to Test).
- Existing Blocks can be modified by clicking on the Block in the right window and changing the parameters on the left.
- Selecting 'Copy' makes a direct copy of the block highlighted on the right hand side and inserts it into the program structure. This function is particularly useful if the program contains several repeated cycles.
- The global parameters are modified using the Test Setup tab on the left of the screen. Variables such as Autoturn and Levelling can be adjusted for the program as a whole.
- Test Setup also allows the Starting Accessories to be defined. These then become the only options available when selecting the test program to run in the 'Run a Test Program' screen.

- Program type defines the type of test and instructs Data Analysis (DA) on how to present the results.
    - Dynamic - used for all tests where a flow energy is measured using the blade and where the flow energy is to be plotted in DA against any variables in the test program, e.g. air velocity, tip speed, etc.
    - Shear - used for Shear Cell or Wall Friction tests, where shear stress is plotted against applied normal stress in DA.
    - Compressibility – used for all Compressibility tests and ensures percentage volume change (compressibility) is plotted against applied normal stress in DA.
    - Permeability – used for all Permeability tests and ensures pressure drop across the powder bed is plotted against either applied normal stress or air velocity.
- Equations can be created and defined using the Equations tab on the left of the screen. These will be saved with the program and will automatically appear in Data Analysis.
- Select ‘File’ / ’Save As’ and allocate a new name for the modified program. Then click 'OK' to save to the Test Program directory.
- Close the Program Editor by clicking the cross in the top right corner.

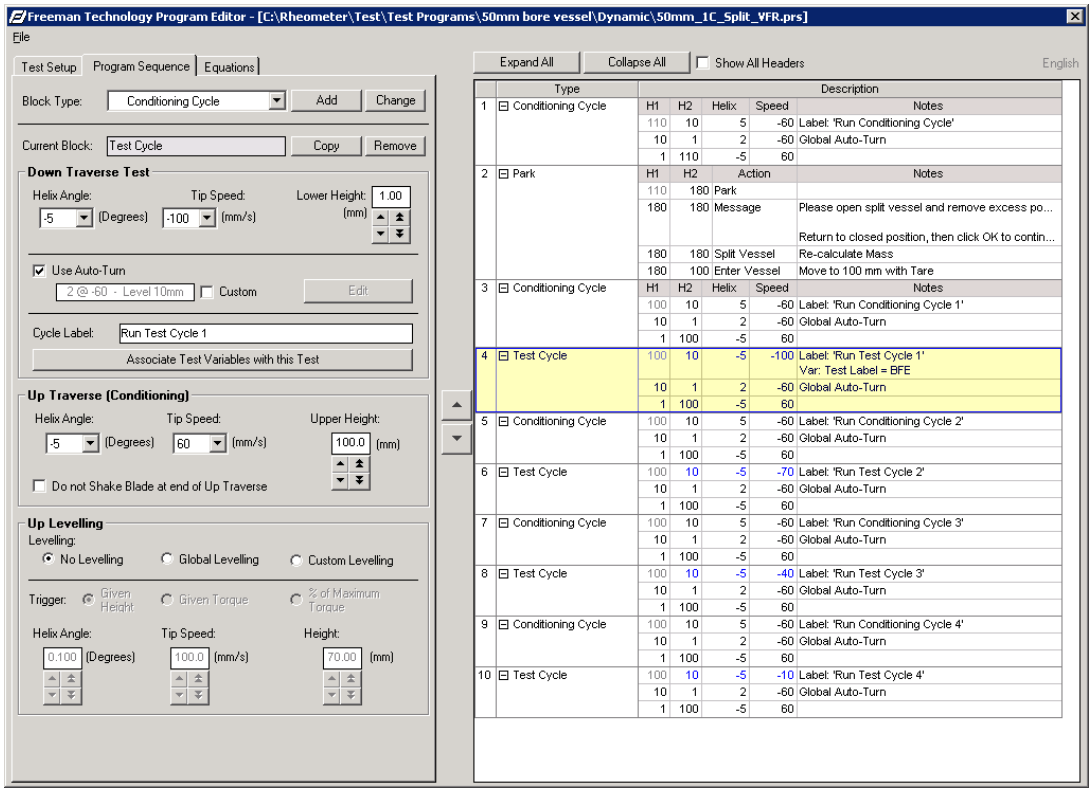


Figure 7 – Program Editor

SETTING AND USING AUTOTURN

- Autoturn allows any preset combination of Helix Angle and Tip Speed to be effective below a predetermined height, irrespective of the settings for the particular downward traverse. It is typically used to avoid the generation of very high stresses that would occur between 10mm and 1mm during a Test Cycle. Adopting the Autoturn settings (similar to a Conditioning Cycle) at 10mm ensures that the powder is still disturbed all the way to the bottom, but the high compaction stresses are avoided.

SECTION 4 – DATA ANALYSIS

OVERVIEW

This guide provides an introduction to the basic operation of Data Analysis v4.0 (DA4) and an overview of the main features. It is not intended to provide a complete description of all of the available functions. The software has been written so that a 'right mouse click' in the appropriate area will reveal a context sensitive menu so that users can manipulate and display data as desired.

There are a number of significant improvements over previous versions including:

- A clearer user interface with intuitive, context sensitive menu system
- The ability to analyse results from different test methodologies in a single session
- More display options and greater flexibility

If you have any questions or would like more detailed advice on specific aspects of Data Analysis, please contact Freeman Technology on +44 (0)1684 851551 or via support@freemantech.co.uk.

IMPORTING TEST FILES

When starting DA4, the window shown in Figure 8 will appear. If a DA4 session is already running, select 'Import Files' from the 'File' menu.

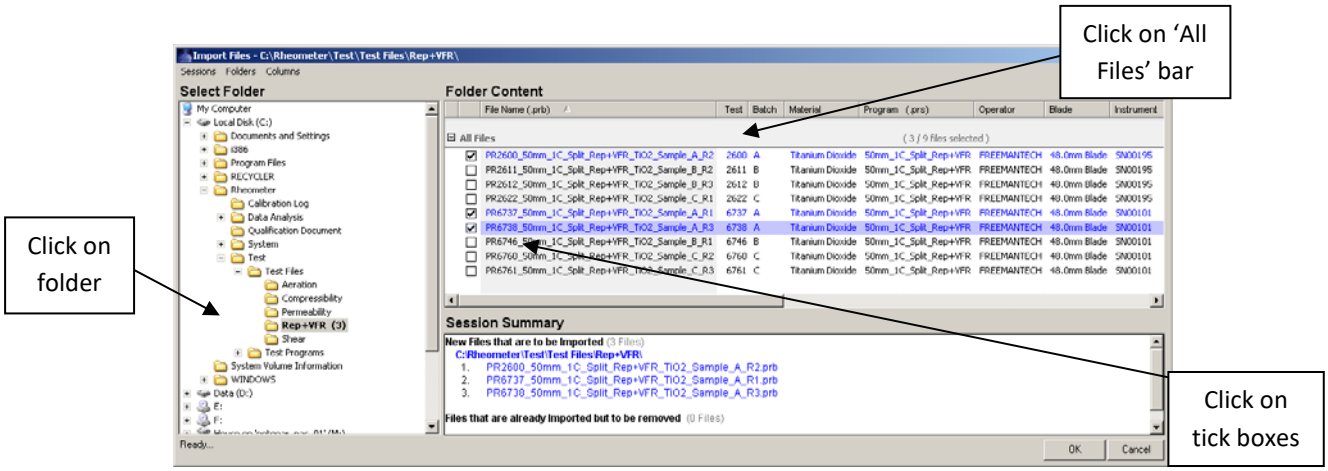


Figure 8 - DA4 File Import Screen

Use the left hand column to navigate through the directory structure and find the required folder. Any Powder Rheometer (xxx.prb) files that exist in the selected folder will appear in the upper right-hand window. Several options are then available:

- Right-click on a folder to import all files in that folder and/or sub-folders.
- Right-click in the 'All Files' border of the upper right-hand window to select files in groups.
- Select individual files from the list by ticking the associated box in the upper right-hand window. Please note that you can use SHIFT and CTRL to create multiple selections.

Any files that have been selected will appear in the Session Summary. To import these files for analysis, click 'OK'.

To remove files, go to the File Overview tab in Overview (see **Reviewing Test Data**) and right-click on the file to be removed.

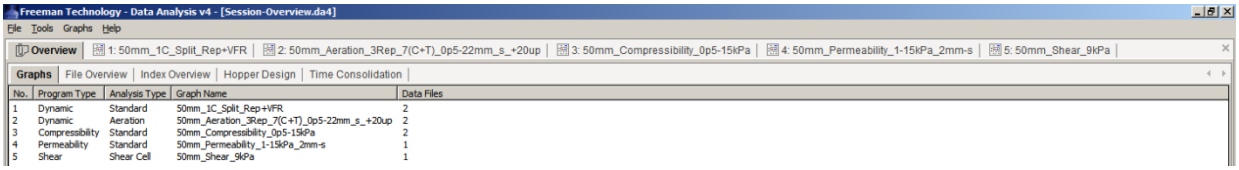
To remove all files and start a new session, click 'Clear Session' from the File menu.

SECTION 4 – DATA ANALYSIS

REVIEWING TEST DATA

The selected test data will be imported and sorted according to test methodology. The default display setting shows an Overview tab alongside a tab for each methodology. Each tab is then broken down into sub-tabs as described below.

OVERVIEW TAB



- **Graphs** – provides a summary of the type and number of test methodologies used in the imported test files.
- **File Overview** – provides data on each individual test file including variables entered by the operator and parameters derived from the test.
- **Index Overview** – shows derived parameters grouped by Material and Batch Code. Includes the option to display error values.
- **Hopper Design** – allows the user to generate Hopper Half Angle and Outlet Size of an axisymmetric and plane flow hopper if suitable test files have been imported.
- **Time Consolidation –** allows the user to evaluate the effects of extended storage on the shear behaviour of a sample by comparison to a standard shear test (at the same consolidating stress) once suitable test files have been imported.

KEY FEATURES OF THE OVERVIEW TAB

- In File Overview and Index Overview, right-click on the table to open the data in Microsoft Excel or to export the data to the clipboard to paste into other applications.
- In Index Overview, right-clicking on the data will allow you to show the Standard Deviation and select how this is displayed.

TEST METHODOLOGY TABS

- **Main Graph** – shows the test points plotted in their familiar format as described in the various methodology help files. For further information see **Working with the Main Graph**.
- **Index Graphs** – provides a flexible method for plotting information derived from the test files. Graphs can be configured to a user's specific requirements. See **Creating Index Graphs** for more detail.
- **Report** – allows the user to create a report for printing purposes which contains all of the information relevant to a group of tests.
- **Raw Data** – provides a method of further analysing the raw measurements made during a test. See **Reviewing Raw Data** for additional information.
- **Series Value Table** – displays the parameters derived from a test. Right-clicking on the table allows you to select the desired parameters and export data.

WORKING WITH THE MAIN GRAPH

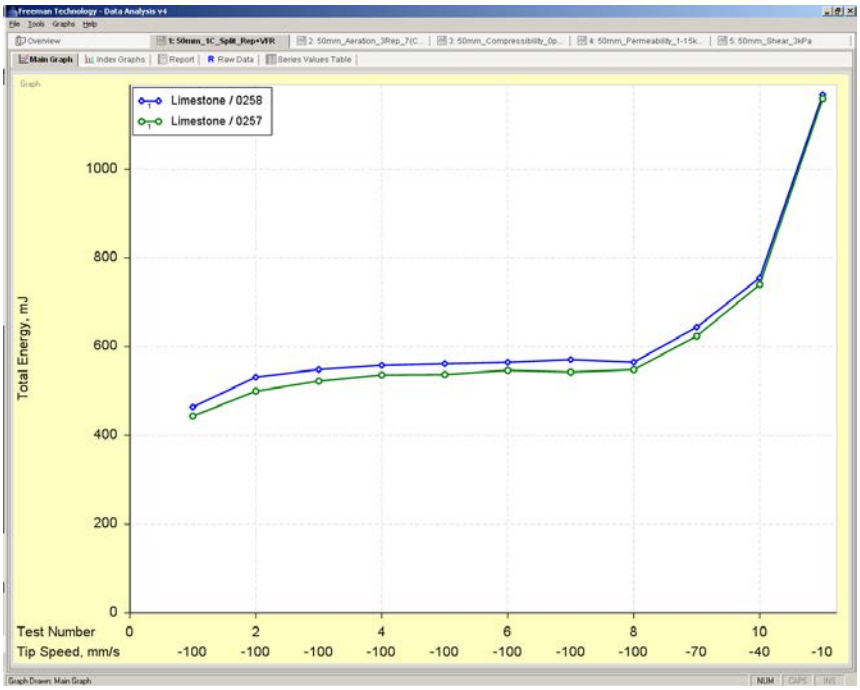


Figure 9 - DA4 Main Graph

When reviewing data on the Main Graph, holding the mouse cursor over a data point will generate a window containing detailed information. Simultaneously pressing the 'D' key will also display the raw data plots for those data points. Holding the cursor between two data points and simultaneously pressing the 'D' key will display both the raw data plots allow direct comparison of the data. If the cursor is held on a specific data point, pressing the 'M' key will reveal additional information that was collected during the test.

As with most areas of DA4, context sensitive menus are available by clicking with the right mouse button. In the Main Graph, the key features can be accessed by right-clicking the main body of the chart, (shown in white in Figure 9). The main features are described below.

- **Manage Graph** – this is a key feature of the Main Graph and allows users to describe how the test data is displayed.
  - **Variables** – select the values to be displayed on the x- and y-axes.
  - **Data / Groupings** – define how the test data is grouped and displayed. Also allows features such as error bars, data cluster and Mohr Circles to be toggled.
  - **Legends** – provides the option for user defined legends and colour schemes.
- **Add or Remove Data** – this feature allows the user to show files generated from different test methodologies on the same graph by ticking the box next to the required file name. By expanding the information (click on the plus sign) specific data points or groups of data points can also be included or removed.
- **Add New Object** – select this option to add a text label, a line or arrow or a data table. The data table can be configured by the user to show the required data. Once in place, right-click on the table to amend or to remove it.
- **Export Image or Data** – provides options for exporting data to other applications.

CREATING INDEX GRAPHS

By following the steps below, the Index Graphs feature allows users to create a customisable series of graphs displaying a range of application relevant test parameters.

- Select the relevant test methodology tab and click on the Index Graphs tab.
- Click the 'Create Index Graphs' button to display the window shown below.

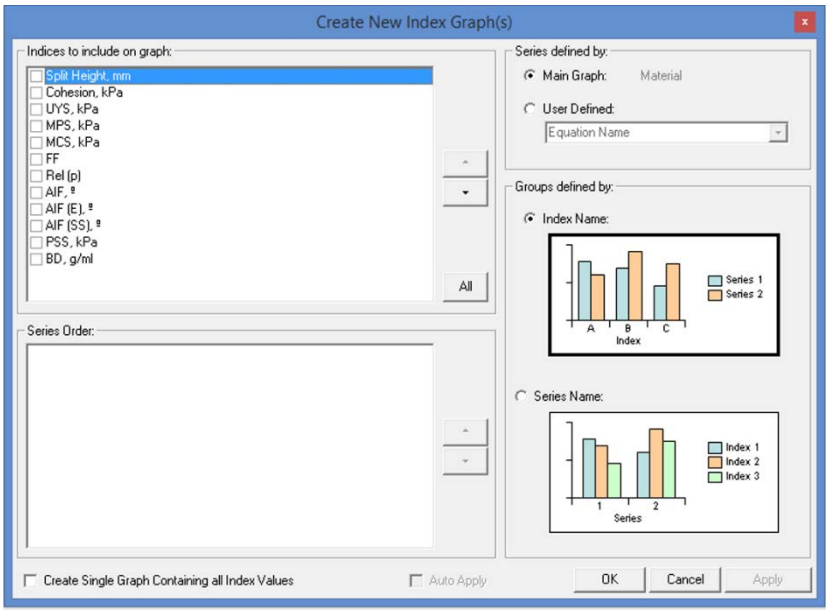


Figure 10 - Index Graphs Menu

- Tick the indices that are to be displayed in graphical format.
- Use the 'Groups defined by' box to select if the indices are grouped by parameter or by series name.

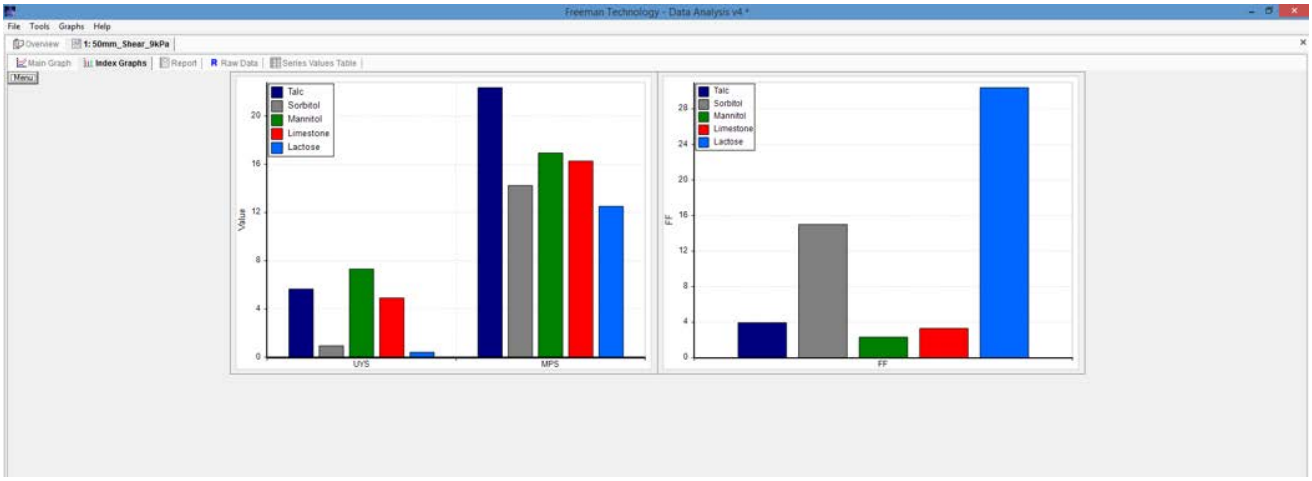


Figure 11 - Example of Index Graphs

Each individual graph can then be maximised by clicking the 'Max' button that appears in the top right-hand corner. Right-clicking on a graph in its maximised or minimised state will bring up a menu of relevant operations.

The Menu button in the top left-hand corner provides overall management of the Index Graphs.

REVIEWING RAW DATA

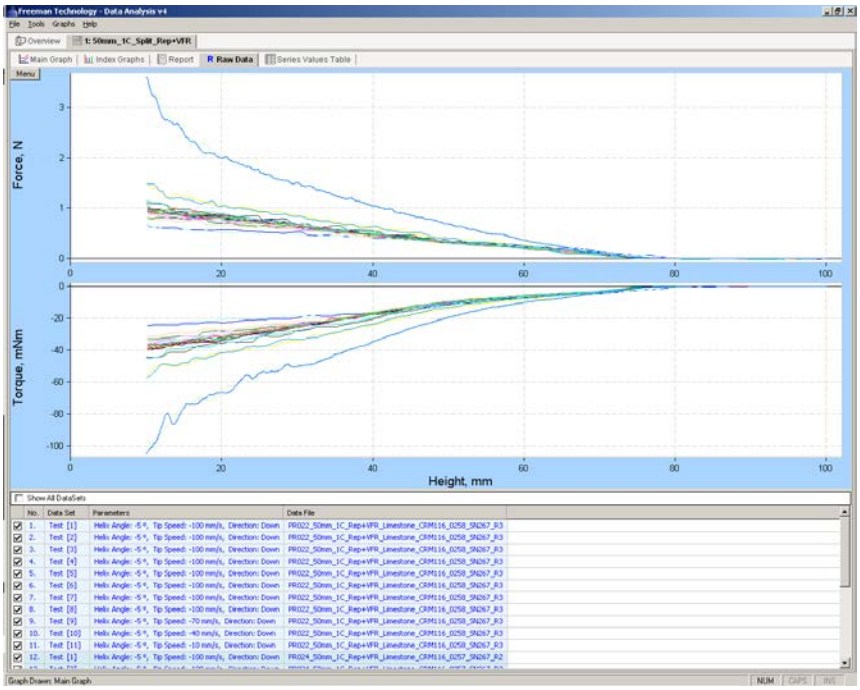


Figure 12 - Raw Data Display

Options for reviewing and analysing raw data are mostly available via location specific menus which are accessed by right-clicking on the relevant area of the screen. Some of the key functions are highlighted below.

- **Managing the Display** – the main graph area is managed by clicking the 'Menu' button in the top left-hand corner. This menu allows users to select how many graphs are displayed and how. There is also the option to export the raw data graph as a range of image files.
- **Changing X and Y Axes** – right-clicking on the axis variable name, e.g. Force, Torque, Height, etc. brings up a list of other variables available to display.
- **Viewing Detailed Information** – hovering the mouse cursor over a specific trace will highlight that data set and display any relevant information. Double-clicking on the trace will zoom into that data set.
- **Zoom Function** – to zoom in on a specific area of the graph, drag the mouse cursor to create a rectangle over the area of interest and select the required option from the list that appears.
- **Selecting Data** – the list below the graphs allows users to select which data sets are displayed. Simply toggle the corresponding tick box to add or remove a data set. The tick box above the list allows additional data sets to be selected, e.g. conditioning cycles or upward traverses. Further options are available by right-clicking on a data set.

SESSION FILES

DA4 Sessions (xxx.da4) can be saved to prevent the user having to manipulate the data each time the same files are imported for analysis. Please note that Session files only record information on which files were used and how data was displayed and not raw test data. Subsequently moving the original test files (xxx.prb) to a different location will prevent the Session File from loading. The **Save Session** and **Open Session** functions are available from the File menu.

OPTIONS

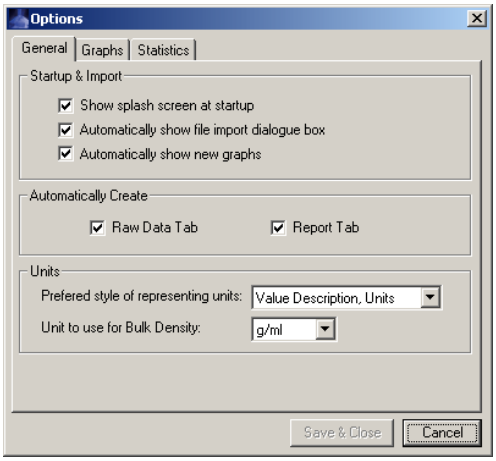


Figure 13: DA4 Options Menu

DA4 **Options** can be accessed via the **Tools** menu and allows the user to define preferences for features such as the start-up screen, how units are displayed, default colours and error calculations.

CREATE NEW MAIN GRAPH

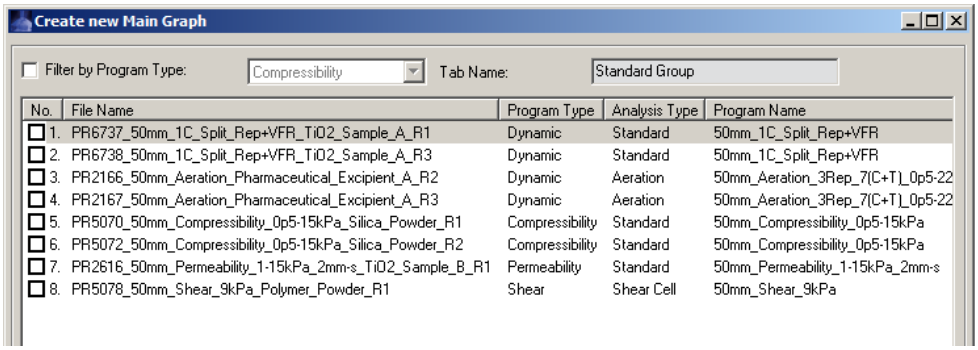


Figure 14: Create New Main Graph Menu

The **Create New Main Graph** function in the **Graphs** menu provides another method of creating a user defined graph. By ticking the relevant box, the results of different test methodologies can be displayed and analysed together.

HELP

The **Help** menu provides quick access to this user guide and allows the preferred language to be selected. Information is also provided on the current installed version and allows the user to check for and download updates.

SECTION 5 – CALIBRATION

CALIBRATION OF THE RHEOMETER

Regular calibration of the measured and controlled parameters is recommended. A calibration kit is supplied for this purpose. The calibration procedures are fully supported by the software and are accessed via the Calibration / Settings Menu.

The calibration process is split into two groups, as follows: -

System Calibration – to provide the control software with the necessary calibration coefficients. Completed at the factory and should only be run after consultation with Freeman Technology.

Audit Calibration – a procedure that may be carried out in order to verify the accuracy of coefficients derived during a System Calibration. This procedure should be run at least once every 90 days.

On screen instructions are provided for each stage of each calibration procedure and it is important that these are followed precisely. A comprehensive help file is also available which can be followed during the calibration sequence. This is available from the FT4 Support Documents.

The following comments provide background information relating to calibration.

SYSTEM CALIBRATION (FORCE AND TORQUE)

- Two load cells are used in the instrument to measure the force and torque required to move the spindle accessory (blade, shear cell, etc.) through the material under test.
- Calibration requires known forces and torques to be applied accurately whilst electronic measurements are taken. Calibration coefficients are derived from this and used during normal testing.
- A calibration kit is supplied for this purpose and it must be assembled and used as described on the user screen and in the calibration help file.
- Important things to note are: -
    - The small pulleys attached to the pulley assembly need to be carefully handled to avoid damage. It is important that the pulleys are free running. When not attached to the instrument, keep in the calibration case.
    - Correct insertion of the drum spindle into the instrument is essential. Ensure that the spindle is fully inserted by checking that the circlip is against the spindle housing (see Section 2).
    - After fitting the drum and the pulley support, check that the drum rotates freely and does not contact the pulleys in any way.
- The nylon cord that supports the weight carrier must be undamaged and installed around the pulleys so as to run freely. In all cases the cords should hang vertically. If they do not then check and correct.

SECTION 5 – CALIBRATION

SYSTEM CALIBRATION (HEIGHT)

- This refers to the measurement of spindle accessory height in mm above the table. The zero position is defined as the position when the head of the blade assembly touches the surface of the vessel base.
- Two cylindrical height gauges are provided in the calibration kit. These are placed on the table in turn, when calibrating. The gauges have a difference in height of 100mm.
- The height calibration procedure takes two measurements, one near zero and another at a position precisely 100mm above this zero. The slope coefficient is calculated from these readings and used by the software to determine height.

SYSTEM CALIBRATION (SPEED)

- The spindle rotational speeds and the carriage vertical speeds are controlled by software in response to parameters defined in the Test Program. Both are driven by servo systems comprising motors and gearboxes and use encoder feedback to enable the control of speeds during testing.
- The encoders provide 500 pulses per revolution of the motor and speeds are calibrated by measuring the pulse rate of each servo system at the calibration speeds. (For example, one revolution of the blade produces 33000 pulses, with a 66:1 gearbox).
- Tip speed and helix angle setting and control accuracy are dependent directly upon the accuracy figures of the above servo speeds.

AUDIT CALIBRATION (FORCE, TORQUE & HEIGHT)

- The Audit Calibration is required to be run at least once every 90 days.
- The Audit Calibration procedures are identical to those of the System Calibrations, however the main difference is that the coefficients measured in the Audit Calibration are just compared to the existing, master coefficients on the FT4 (derived during the initial System Calibration). Therefore the Audit Calibrations are simply a verification that the FT4 is within calibration. They do not reset or overwrite anything.
- At the end of an Audit Calibration procedure, an Excel document is created and the results are presented alongside the acceptable pass / fail criteria. This Audit Log should be saved and retained as a record of calibration.
- In the event that an Audit Calibration results in one or more points outside acceptable limits, reported as a ‘Fail’ in the Audit Log, then it is recommended that the Audit Calibration is repeated. If two consecutive Audits result in failure, please contact Freeman Technology and do NOT complete a System Calibration.
- If the Audit Calibration confirms the FT4 is within calibration, denoted by a ‘Pass’ in all rows of the Audit Log, then that part of the calibration is compete and does not need repeating for 90 days. The date is recorded into the Admin form and a tick indicates it is within calibration.
- After three months have elapsed since the last Audit Calibration, a warning message is displayed reminding the operator that the Audit Calibrations need completing.
- Height Audit Calibration is also an identical procedure to the Height System Calibration, but again is solely for verification.

SECTION 5 – CALIBRATION

- Servo velocity audit requires external and independent instrumentation to measure the carriage displacement and blade rotations during an accurately timed interval. Most methods used will be less sensitive and accurate than the built in pulse counting system used for system calibration.

SPECIFICATION

- A specification for the FT4 Powder Rheometer is available in Section 9.
- The accuracy of measurement of all parameters is as follows: -

  Better than 1% of reading or 0.25% of full scale, whichever is greater.

INSTRUCTIONAL VIDEOS

AUDIT TYPE	**VIDEO LINK**
Force	English | Chinese
Torque	English | Chinese
Height	English | Chinese

ADD USER

New users can be added by selecting the 'Administer Users' option in the 'Admin' then 'Users' screen. The following screen then appears enabling the 'Add User' button to be selected.

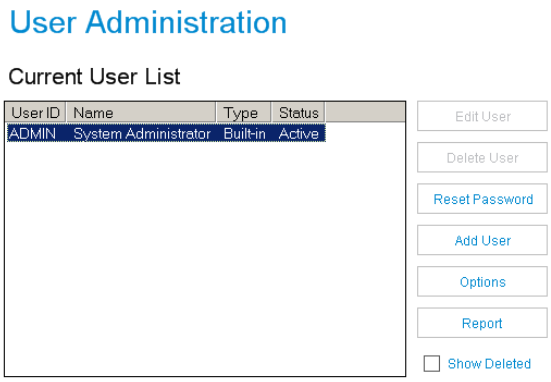


Figure 15: User Administration

When creating a new user, the Administrator is required to enter certain details describing the user to be added. As a minimum, they must define the User ID, Name and Password, as well as the level of permission the user will have.

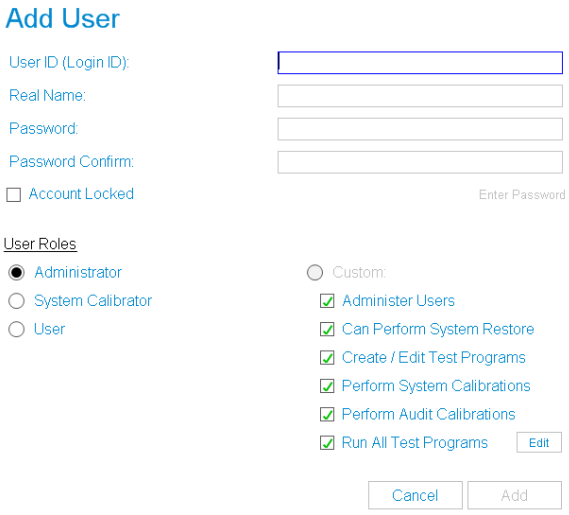


Figure 16: Add User

- Once saved, the new user can logon using their User ID and assigned Password.
- A user can be configured with various permissions. Default rights for System Calibrators and Users are defined, although a Custom user can be configured with any combination of permissions.
- De-selecting 'Run all Test Programs' allows the Administrator to configure which Test Programs are available for the User to run.

EDIT USER

The Administrator can edit user properties after they have been created. Everything except the User ID and the user's Password can be re-configured in this way. This option is also used to unlock previously locked accounts, arising from failed logon attempts.

DELETE USER

A user account can be deleted using this option. The account is made inactive and unavailable to the user, although a record of its existence is available in the Report (see below). If the Administrator tries to create a new account with the same User ID as a deleted account, the account will be reactivated allowing changes to be made to the user properties.

RESET PASSWORD

This option allows a system Administrator to reset the password of any of the accounts contained in the system.

REPORT

Produces a text report defining the attributes of all the accounts in the system.

EDIT OPTIONS

This allows the Administrator to define the Password Policy that will be employed by the system.

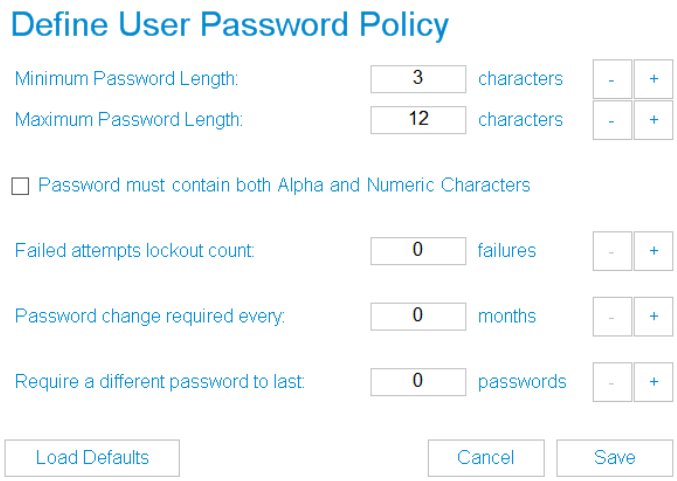


Figure 17: Define User Password Policy

SECTION 6 – USER ADMINISTRATION

The following attributes can be defined:

- Minimum and Maximum Password Length.
- Whether the password is required to contain both Alpha and Numeric characters.
- How many times the account can be unsuccessfully logged onto before it is locked out (this does not apply to the Administrator account – this account is never locked).
- How frequently the users are required to change their passwords.
- If the user is required to have a different password from previous passwords.
- How many previous passwords are remembered.

SECTION 7 – SYSTEM BACKUP & RESTORE

SYSTEM BACKUP

The Powder Rheometer system files can be backed up in case there is a computer failure resulting in the computer's operating system needing to be reinstalled. This option can be found on the Calibration / Settings Menu.

Clicking Backup System opens the following screen:

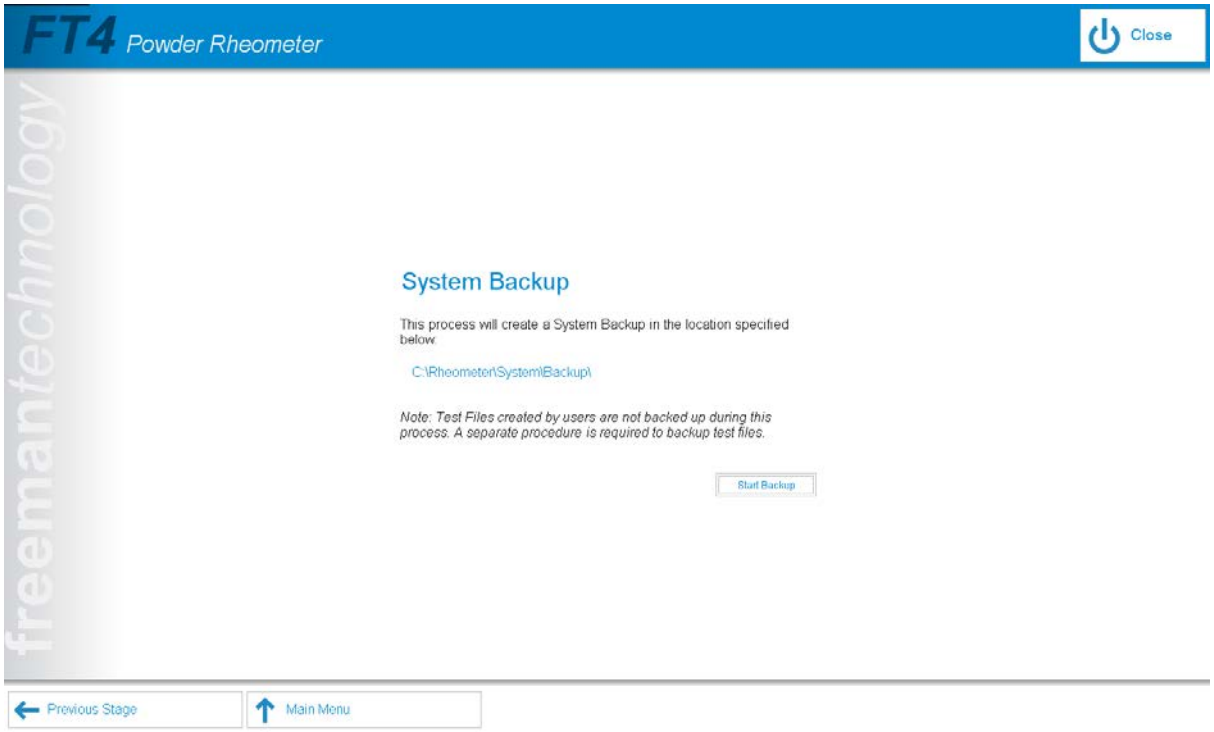


Figure 18: System Backup

The default location for this file to be saved will appear in the 'Backup Location' dialog box. If required, this can be changed to a different location enabling storage directly to a network drive for example. The backup procedure produces a single file with all the necessary information to restore the Powder Rheometer system files. The following information is backed up:

- System Registry (containing calibration information).
- User Account details.
- Blades and Vessels Settings.
- All Test Programs contained in the 'C:\Rheometer\Test\Test Programs' folder, including all sub folders.
- Powder Rheometer system INI file.

It is recommended that the backed up file is stored on durable media separate from the FT4 for future restoration if needed. This procedure should be run on a monthly basis as a minimum.

Test Files are NOT backed up during this procedure and should be stored on a secure network drive on a regular basis.

SYSTEM RESTORE

From the Calibration / Settings Menu, it is possible to run the Restore System procedure.

Selecting this option allows the user to choose the directory of the backup file and begin restoration of the system files by clicking 'Start Restore'.

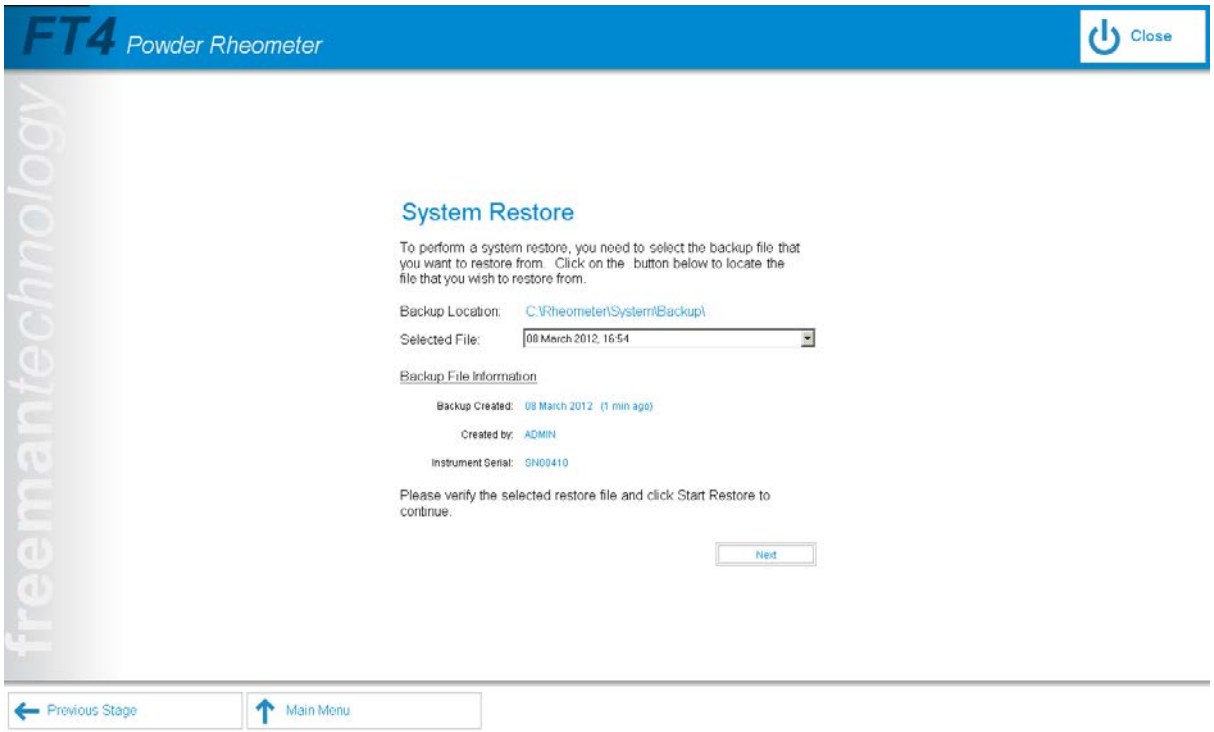


Figure 19: System Restore

Information contained in the selected restore file, such as date, user and instrument serial number is displayed at the bottom of the screen and can be used to confirm whether the file selected is the required one.

The restore process begins by backing up all existing parameters before the new files are installed. These are saved to the directory listed in the 'Restore Backup Location' box.

Once the restore process has successfully completed, the application will automatically shut down to allow the restored settings to take effect.

The Administrator (Admin) password is always reset to 'admin' after a restore to prevent system lockout. It should be changed by the Administrator immediately after the restore process.

SECTION 8 – UPGRADING SOFTWARE

INSTALLING NEW SOFTWARE

- Copy the installation file to a memory stick or similar device.
- In Windows, select the 'Start' button at the bottom left corner of the screen.
- Select 'Run' then 'Browse', to open Windows Explorer.
- Select the relevant drive to view the contents of the memory device and follow the instructions below:
- **Installing Powder Rheometer**
    - Double click on 'Setup_PR*_v**.exe' where * represents version numbers.
    - Follow on screen instructions
- **Installing Data Analysis**
    - Double click on 'Setup_DA*_v**.exe' where * represents version numbers.
    - Follow on screen instructions

SECTION 9 – SPECIFICATION

SYSTEM: -

FT4 Powder Rheometer intended for use in a laboratory environment for measuring the rheological properties of powders, pastes and semi-solids.

Complies with the following EMC specifications and ASTM International standards:

- EN61000-3-2:2001
- EN61000-3-3: 1995
- EN61326: 1997 + A2:2001
- ASTM D7891

Certificates of conformity available on request.

PERFORMANCE: -

Force	+/- 50N maximum 0.0001N resolution
Torque	+/- 900mNm maximum 0.002mNm resolution
Vertical travel	185mm
Rotor speed	120 rpm maximum
Axial speed	30 mm/sec maximum
Residual energy level in air	< 2mJ

COMPUTER SPECIFICATION: -

The instrument incorporates an integrated high specification processor and operates on a Microsoft Windows Embedded operating system. It has built in networking capability and a universal serial bus to provide serial daisy chaining of all automated accessories.

CONSTRUCTION: -

Working zone:	316 stainless steel
Contact parts:	316 stainless steel Borosilicate glass Delrin and Peek plastics

DIMENSIONS: -

Main instrument	306 x 306 x 760mm high

WEIGHTS: -

Main instrument	22kg net

POWER REQUIREMENTS: -

Supply voltage range:	90 to 264VAC
Input current range:	1.6A at 120VAC 0.8A at 230VAC
Input frequency range:	47Hz to 63Hz

Minimum fault protection limit: 30mA

ENVIRONMENTAL CONDITIONS: -

Humidity range	20-80% non-condensing
Temperature range (operating)	10˚C to 40˚C
Temperature range (storage)	0˚C to 50˚C

VESSELS: -

Precision bore, borosilicate glass tube.

Standard sizes:-

25mm x 10ml Split Vessel
25mm x 25ml Split Vessel
25mm x 35ml Vessel

50mm x 85ml Split Vessel
50mm x 160ml Split Vessel
50mm x 260ml Vessel

62mm x 137ml Split Vessel
62mm x 240ml Split Vessel
62mm x 400ml Vessel

BLADES: -

Hardened stainless steel.
Standard sizes:-

23.5mm diameter x 6mm wide
48.0mm diameter x 10mm wide
60.0mm diameter x 10mm wide

CALIBRATION KIT: -

Force, torque, height, carriage velocity and spindle speed are configured for calibration.

Calibration fixtures, weights and height gauges are supplied as part of the calibration kit.

A calibration log is automatically kept of the current and all previous calibrations.

ACCESSORIES: -

25mm Accessories Kit
50mm Accessories Kit
62mm Accessories Kit
Aeration Control Kit

24mm Shear Cell
48mm Shear Cell
24mm Wall Friction Kit
48mm Wall Friction Kit

1ml Shear Cell

SOFTWARE: -

All Control and Data Acquisition Software is supplied and configured with the instrument and includes Microsoft Office.

APPENDIX 1 – ELECTRICAL CONNECTIONS

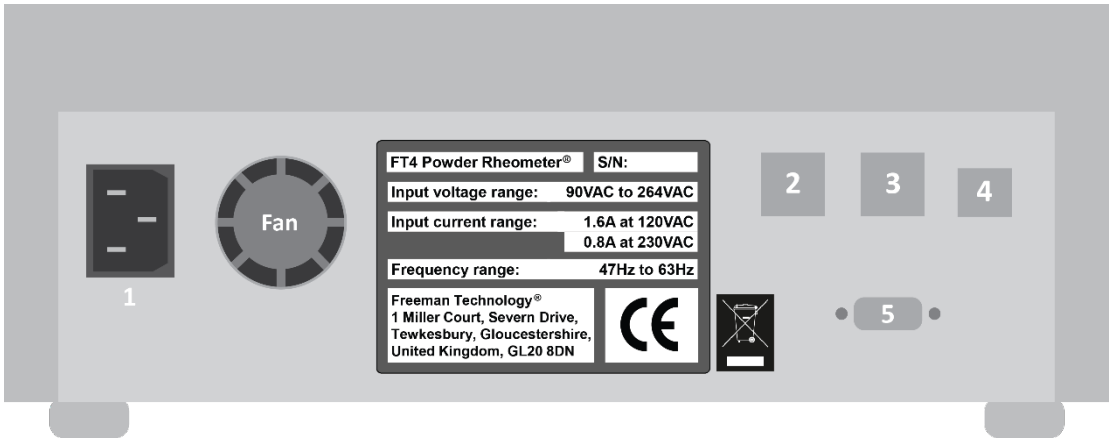
POWER

Supply voltage range: 90VAC to 264VAC

Input current range: 1.6A at 120VAC
0.8A at 230VAC

Input frequency range: 47Hz to 63Hz

REAR OF FT4



Key	
1	Mains Power Cable
2	USB (Dual)
3	USB (Dual)
4	Ethernet (RJ45)
5	VGA

WARNING

The FT4 must be shut down and power removed before connecting / disconnecting cables.