



MASTERSIZER 3000 USER MANUAL

Mastersizer 3000 User Manual

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Introduction to this manual

Introduction

This manual covers the operation of the Mastersizer 3000 and Mastersizer 3000E particle characterisation systems. The instrument measures the size of particles contained within a sample, presenting data according to the user's needs.

This user manual is a companion to the **Mastersizer 3000 Basic Guide**, which gives Health and Safety, maintenance, troubleshooting and other vital information which **all users must read**.

Additional user manuals are supplied with both the Hydro and Aero dispersion units.

About this manual

The manual contains the general information required by an operator; as well as supervisor information that concentrates on administration and extension of the system's functionality.

Topics covered include:

- An overview of the Mastersizer hardware and software.
- A guide to making measurements and how to read the results.
- Sample preparation guidance.
- Reporting creating and editing reports.
- System security.
- Advanced features.



Warning!

The instrument and the samples to be measured may be hazardous if misused. Users must read the **Health and Safety** information in the **Basic Guide** before operating the system.

Access to the instrument

This manual refers to the various people who will have access to the instrument, as follows.

Malvern Instruments personnel

Malvern Instruments personnel (service engineers, representatives etc.) have full access to the instrument and are the only people authorised to perform all service procedures that may require the removal of the covers.



Warning!

Removal of the covers by unauthorized personnel will invalidate the warranty of the instrument. Unless advised within the content of this manual, only Malvern Instruments trained personnel are permitted to remove the main cover of any part.

Supervisor

The supervisor is the person responsible for the management and safety of the instrument and its operation. The supervisor is responsible for the training of the operators. The supervisor can perform all user maintenance routines identified in the **Basic Guide** and within this manual.



Warning!

Under no circumstances should the supervisor or an operator remove the main cover of the instrument.

Operator

An operator is a person trained in the use of the system. The operator can perform all user maintenance routines identified in the **Basic Guide**.

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Warning!

Failure to follow these guidelines could result in the emission of laser radiation. Laser radiation can be harmful to the body and cause permanent eye damage.

Assumed information

To make full use of this manual, the user should understand the following points.

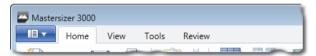
Naming convention

Within this manual:

- The Mastersizer 3000 and 3000E instruments will be referred to as "the optical unit" or "the instrument", or simply the Mastersizer.
- The sample dispersion units are referred to as "the Hydro MV" or "the Aero S", or as "the dispersion unit". "Wet dispersion units" refers to the Hydro LV, MV, EV and SM. "Dry dispersion units" refers to the Aero S.
- The combination of the optical unit, one or more dispersion units and the computer is referred to as "the system".

Ribbon commands

The control ribbon in the Mastersizer application software is divided into tabs:



The first button on this tab bar is referred to as the **Application button** and has a similar function to the File menu on older systems.

Ribbon commands are referred to in this manual in the form: ribbon tab selection, followed by option. For example, **Home-Manual Measurement** describes clicking an option that is found on the **Home** tab

Whenever a ribbon option also has a sub-item this is shown as by the presence of a down-arrow on the ribbon bar icon; as shown for the **Home-Run SOP** option shown. Ribbon commands and the names of windows are always shown in bold text.

Mastersizer 3000



The SOP Player feature, shown above, is only available with the Mastersizer 3000/Mastersizer 3000E with Extended software option.

Where to get help

This section describes the available sources of information on the system. The primary sources of information on the system are instrument and accessory manuals and the software's help system. The manuals give an overview of the system as a whole, while the online help system gives more detailed information on the Mastersizer application software. Some features are described in both the manuals and Help system.

Manuals

The following manuals are available for the Mastersizer:

Basic Guide

A concise yet essential guide that provides an introduction to the system and its usage covering **Site requirements**, **Health and Safety** and an instrument and accessory overview. Finally **a brief guide to making a measurement and viewing the results** is included.

User Guide

The primary source of information about the Mastersizer hardware and its use. See **About this manual** earlier in the chapter.

Dispersion unit guides

Separate guides are supplied with both the Hydro and Aero dispersion units. These provide essential information on the connection, operation and maintenance of the dispersion units.

Help System

Each key software window has a **Help** button giving information specific to it. Additional **Info Tips** are also available whenever is shown - simply move the mouse pointer over this icon to see a relevant tip.

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Technical support

Malvern Instruments offers several methods of technical support, which are described in this section.

Email support



Note

This is the primary support method – only use the other methods if this is not available at your location.

Customers with internet access should use the **Contact Malvern** feature in the software's **Maintenance** window. This is described fully in the **Software overview** chapter. This feature ensures that our support team have all the relevant information about your system.



If you do not have internet access from the computer operating the Mastersizer system, contact your local Malvern Instruments representative - details of all local telephone numbers are available at www.malvern.com. Always quote the following information:

- **Model and serial number of the instrument**. The serial number is shown on the Mastersizer application software status bar.
- The software and firmware version. To find this select Tools-Maintenance in the software and then examine the System information section alternatively, move the mouse over the instrument status bar icon. The Application Version is the main firmware version number to note.

Additional support

Contact the International Malvern Helpdesk if the local Malvern Instruments representative is not available:

Telephone: +44 (0)1684 891800 Email: helpdesk@malvern.com.



Note

This help line is primarily English speaking.

Mastersizer 3000

Remote support

Malvern Instruments offers a remote support service, delivered by an internet connection. Benefits include fast and efficient fault diagnosis, as well as reducing downtime and costs.

Malvern website - www.malvern.com

The Malvern Instruments website offers a comprehensive range of particle characterisation resources for use by customers 24 hours a day, seven days a week.

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Introducing the Mastersizer 3000

Introduction

This chapter provides information about the Mastersizer 3000 hardware and identifies all major features of the system.

The following topics are covered:

- A description of the purpose of the instrument and its accessories.
- A hardware overview of a typical wet and dry system.
- Detailed reference on the physical parts of the optical unit.
- Information about the parts of the cell, including how to insert and remove it.



Note

Hardware maintenance – for details on how to maintain the hardware, please refer to the **Basic Guide** and the separate dispersion unit guides.

Mastersizer 3000 and Mastersizer 3000E instruments

There are two core instruments in the Mastersizer 3000 family, these are the Mastersizer 3000 and Mastersizer 3000E. Though identical looking there are some fundamental differences between the two; in terms of their software functionality and operation with the associated dispersion units.

The differences between the two Mastersizer variants are shown in the below table.

	Mastersizer 3000E - Basic software feature set	Mastersizer 3000E - Extended software feature set	Mastersizer 3000
Hardware			
Particle size range	0.1 - 1000µm	0.1 - 1000µm	0.01 - 3500µm
Compatible with Manual Hydro wet dispersion units (Hydro EV / SM)	•	•	•
Compatible with Auto- mated Hydro wet disper- sion units (Hydro MV / LV)		•	•
Compatible with Aero dry dispersion unit			•
Software			
SOP operation	•	•	•
Customized reporting	•	•	•
Entry level legacy system result compatibility tools	•	•	•
Advanced method development and comparison tools		•	•
Advanced data quality assessment and reporting tools		•	•
Advanced measurement manager functions		•	•
Measurement sequencing / SOP player tool		•	•
New feature additions and upgrades		•	•
Ability to use the software on multiple workstations		•	•
User workspace functions		•	•

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	Mastersizer 3000E - Basic software feature set	Mastersizer 3000E - Extended software feature set	Mastersizer 3000
IQ/OQ Validation			•

CFR21 part 11 ER/ES

(additional Feature key necessary)

The **Extended software feature set** is detailed later in the **Software overview** chapter; **Customising the software interface** section.

To simplify the text throughout the manual, the 3000 and 3000E instruments will be referred to as the Mastersizer (or "the optical unit" or "the instrument"), unless it is necessary to differentiate or highlight any differences; then the instrument will be identified as Mastersizer 3000 or 3000E as appropriate.

About the Mastersizer

The Mastersizer is comprised of the main optical unit, one or more dispersion units and a measurement cell. Commonly, a dispersant such as de-ionised water is also connected directly to the dispersion unit.

Optical unit

The main optical unit, sometimes referred to as the optical bench or just the instrument, is the core of the Mastersizer system. In its simplest terms, its purpose is to transmit red laser light and blue light through a sample and then use its detectors to generate data about the light scattering pattern caused by the particles in the sample. The data is then interpreted by the Mastersizer application software to provide accurate particle size information. The blue light used on MS3000 provides improvements in the measurement resolution below 1 micron in size.

Dispersion units overview

There are two types of dispersion unit: wet and dry. Wet units (**Hydro MV/LV/EV/SM**) control the dispersion of a sample suspended within a liquid dispersant. The function of the dry unit (**Aero S**) is to ensure that a dry sample is dispersed and evenly fed to the measurement cell within a continuous stream of air.

Measurement cell

The cell is the interface between the dispersion unit and the optical unit. The sample is routed between measurement windows in the cell so that the laser can pass through it in order to make a measurement.

Computer (Mastersizer application software)

At the heart of the system lies the Mastersizer application software, which runs on a computer. This application controls the optical unit and dispersion unit hardware, and also processes the raw data gathered by the system, providing flexible data analysis and reporting features.

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Typical systems

Wet dispersion

A wet system consists of the optical unit and either the Hydro LV/MV or EV dispersion unit. These dispersion units are designed to circulate a liquid sample through the Mastersizer's measurement cell. Many materials need to be measured as wet dispersions. Typically these are materials that are used or produced wet in the manufacturing process or need to be dispersed in a liquid in order to achieve reproducible dispersion.

Hydro LV/MV

The Hydro MV and LV units are designed to create a suspension of particles in water or other liquid media. The Hydro LV can handle a liquid volume of 600ml, whereas the Hydro MV unit has a volume of 120ml. Both can be configured to automatically deliver liquid dispersants and also dispose of the sample following a measurement. This enables the cleaning and filling of the units as part of an SOP.

The large capacity of the Hydro LV makes it ideally suited to the measurement of samples which have very broad size distributions or contain large particles. In some cases, sample materials are not plentiful and the MV's smaller volume sample dispersion unit is a sensible choice to minimize sample and dispersant consumption while retaining the benefits of totally automated sample dispersion and measurement.

Hydro EV

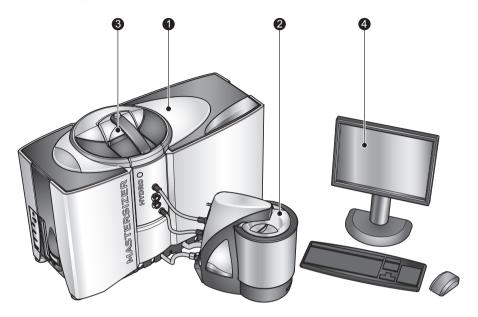
In the mineral, chemical, metal powder refining and cement industries, measurements of larger sample volumes are often most appropriate for QC of the manufacturing process and finished product. The large capacity of this unit makes it ideally suited to the measurement of such samples as well as larger materials having broad size distributions.

The Hydro EV is a wet dispersion unit designed to circulate a liquid sample held within a standard laboratory beaker through the Mastersizer's measurement cell. As such the filling and emptying of this unit is a manual process.

Hydro SM

The Hydro SM enables the Mastersizer to be used for particle-in-liquid particle sizing, where the amount of sample available limits the amount of dispersant that can be used to dilute it, or where just a small volume of dispersant is available. This is ideal when the sample or dispersant is toxic or expensive.

The following illustration shows a typical wet dispersion unit installation:



ill 8620

1	Optical unit	3	Wet cell
2	Wet dispersion unit	4	Computer running the Master- sizer application software

Optical unit (as described in the next section)

Measures the sample using red and blue light wavelengths.

Dispersion unit (in this case the Hydro LV)

Circulates the sample through the cell

Wet cell

Sample flows through the air cell so that it passes through the instrument's laser path.

Computer running the Mastersizer application software

Controls both the optical unit and the attached dispersion unit, as well as processing and presenting the measured data.

Other wet dispersion units include the Hydro MV, EV and Hydro SM.

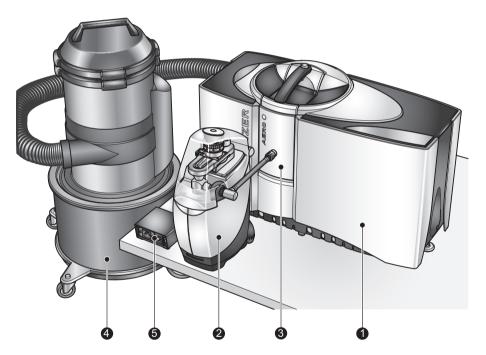
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Dry dispersion

The Aero S dry dispersion unit is only compatible with the Mastersizer 3000 optical unit.

A dry system comprises of the main optical unit plus the **Aero S** dispersion unit. The Aero S is designed for providing an even circulation of dry powder throughout Mastersizer measurement cell.

Direct software control over the feed rate and air pressure is provided, which enables flexible control of both the sample throughput and dispersion. The unit can be controlled either independently of measurement or from within a manual/SOP measurement. The following illustration shows a typical dry dispersion unit installation:



ill 8652

(1)	Optical unit	4)	Vacuum unit
2	Dry dispersion unit	⑤	Ancillary switching unit
3	Dry cell	6	Computer running the Master- sizer application software (not shown)

Optical unit (as described in the next section)

Measures the sample using red light detection.

Dry dispersion unit (in this case the Aero S)

Disperses the sample and transports it to the measurement cell.

Dry cell

Sample flows through the air cell so that it passes through the instrument's laser path.

Vacuum unit

Removes the sample from the cell exhaust once it has been measured.

Ancillary switching unit

An external control accessory that is used to switch on the vacuum unit required for the sample extraction during each measurement.

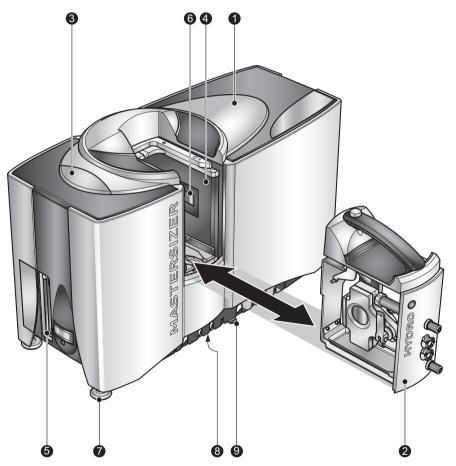
Computer running the Mastersizer application software (not shown)

Controls both the optical unit and the attached dispersion unit, as well as processing and presenting the measured data.

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Optical unit components

This diagram shows the optical unit, the main component of the Mastersizer system. The unit directs red and blue light through the sample and then collects the light scattered by the particles within the sample using a set of light-sensitive detectors. This data enables the system to calculate particle size:



ill 8622

1	Optical unit	6	Protection window
2	Cell	7	Adjustable feet
3	Instrument status LED	8	Drain
4	Cell bay	9	Tube/cable routing guide
(5)	End panel		

Cell

Two types of cell are available:

- **Wet** for the Hydro LV, MV, EV and SM units. Standard and solvent compatible versions of the wet cells are available.
- **Dry** for use with the Aero S unit (The dry cell is sometimes known as the air cell).

Instrument status LED

The instrument status is indicated as follows: **Standby** (i.e. powered on, but not making a measurement): pulsating dim; **Active** (i.e. making a measurement) - constant bright; **Error** - flashing bright.

Cell bay

The interface between the sample dispersion unit and the optical unit. The sample passes between two windows that allow it to be analysed by the optical unit's laser beam.

End panel

Provides the communication connectors for the optical unit as well as the power connection and switch. Refer to the **Mastersizer 3000 Basic guide** for further connection reference.

Protection window

Stops dust/dirt entering the system. Clean this window periodically. Refer to the **Mastersizer 3000 Basic guide** for more information on cleaning the protection window.

Adjustable feet

It is important to ensure that the instrument is levelled on the bench - adjust the feet individually in order to accomplish this.

Drain

Allows any spillages in the cell area to exit onto the laboratory bench.

Tube/cable routing guide

Neatly routes all connection pipes and cables underneath the instrument.

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End panel

The end panel of the instrument provides the communications and power connections.



ill 8623

1	Power switch	3	CAN 1-3
2	USB	4	Power input

Power switch

Press once to power on the unit, press again to power off.

USB

Provides data communication with the PC. For best performance connect to a high speed USB port on the PC:

CAN 1-3

Supplies power and data communication to the dispersion unit.

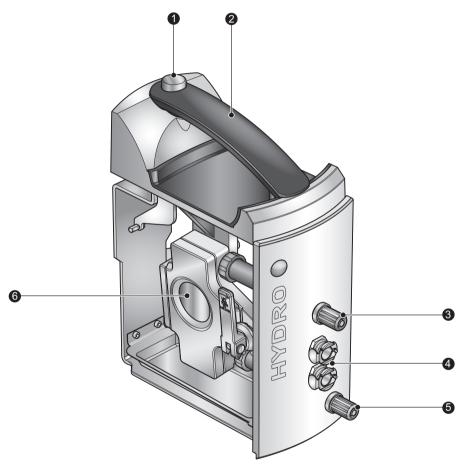
Power input

Input for 24V power supply unit.

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Cell components

The following illustration shows the key parts of a wet cell – for a more detailed description of both the wet and dry cells, refer to the Hydro or Aero Dispersion Unit Guides respectively:



ill 8625

1	Cell release button	4	Temperature regulator throughput
2	Cell handle	(5)	Sample input (yellow)
3	Sample output (blue)	6	Cell windows

Cell eject button

Ejects the cell from the cell bay locking mechanism so that it can be withdrawn from the cell bay.

Cell handle

Only lift the cell by this handle.

Sample output (blue)

Connected to the sample in (blue) on the dispersion unit.

Temperature regulator throughput

Provides throughput connections to a water jacket within the cell. This allows an (optional) heater/chiller to regulate the sample temperature to help minimise the effect of ultrasound heating.

Sample input (yellow)

Connected to the sample out (yellow) on the dispersion unit.

Cell windows

Sample passes through the window and is measured by the system. Cell windows should always be kept clean to ensure accurate measurements - see the **Hydro Wet Dispersion Units Guide**.

Inserting and removing the cell

Always follow this process when inserting or removing the cell from the instrument. This section assumes that all relevant tubing connections between the dispersion unit and cell have already been made. Refer to the dispersion unit manuals for verification of these connections.

Inserting the cell

- 1. Lift the cell by its handle ②.
- 2. Insert cell into the instrument as shown in the previous Mastersizer optical unit illustration.
- **3.** Do not force the cell into the instrument only slide it into the cell holder until it stops.
- **4.** The cell now automatically locks into place within the cell bay.

Removing the cell

1. Press the cell eject button \bigcirc to eject the cell from the cell bay.



Caution!

Do not try to remove the cell from the cell bay without first pressing the cell eject button 1 as this could damage the locking mechanism.

2. Withdraw the cell from the instrument using its handle ② in the direction shown in the previous Mastersizer optical unit illustration.

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Note

If the cell is raised and lowered too many times in a short period, the firmware will lock the cell in place for a period of time before it can be ejected again; this is to prevent damage to the locking mechanism.

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Software overview

Introduction

The Mastersizer application software serves several key functions: it controls both the optical unit and the dispersion unit; it allows users to create and edit measurement files; it enables users to run manual or SOP measurements on both wet and dry samples, and finally it provides access to powerful reporting and data analysis features.

This chapter gives details on the following:

- Powering on and starting the software.
- Description of the software user interface.
- Customising the software interface.
- Maintenance utilities.

This chapter provides a description of the key elements of the software only. For information on how to run a measurement, see the **Making measurements** chapter. Additional features of the software are also detailed in the **Advanced** chapter of this manual. Reference information for all features is provided in the Help system.



Note

This section provides a description of the key elements of the software only. For information on how to run a measurement, see the **Making measurements** chapter. Some advanced features of the software are also detailed in the Supervisor's section of this guide and reference information for all features is provided in the Help system.



Powering on and starting the software

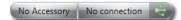
- 1. Ensure that the optical unit, dispersion unit and computer are connected (connection reference is given in the **Mastersizer 3000 Basic guide**).
- **2.** Power up the computer and printer (if available) by following the instructions in the manuals supplied.
- 3. Connect the optical unit's power supply to the mains power supply.
- **4.** Press the on/off switch on the instrument's side panel once to power on. The blue indicator on the top of the instrument illuminates, together with the blue indicator on the front of the dispersion unit, which pulsates to indicate that standby mode is active.
- **5.** Log in to the computer that is connected to the instrument and start the Mastersizer application software by double clicking on the **Mastersizer** desktop icon.



6. The main Mastersizer application software window is now displayed. **A correct connection to the instrument** shows both the instrument and accessory name in the status bar:



If the instrument is not connected correctly, the status bar indicates:



If this is the case, check the instrument's connection to the USB port on the PC and the power connections to the instrument.

If the dispersion unit is not detected, the following is shown:



If this is the case, check the connection from the instrument to the dispersion unit. Also, a blue pulsating power light on the dispersion unit indicates that it is connected correctly to the instrument.



Note

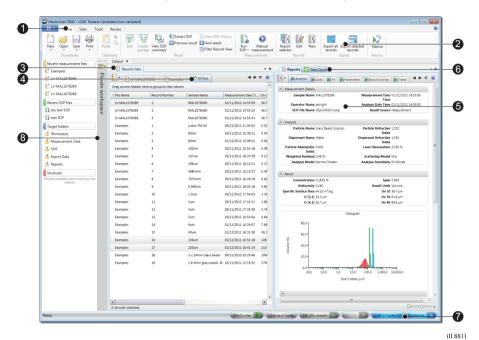
Before using the instrument to measure a sample, wait for 30 minutes for the laser to stabilise.

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Software overview Chapter 3

Software user interface

The main software window, with a measurement file loaded, is shown as follows:



① Ribbon selector tabs	⑤ Reports tabs
② Control ribbon	Data Quality tab
③ Record View panel	⑦ Status bar
All files tab	Private/Shared workspace

Ribbon selector tabs

Provides quick access to the different control ribbons. The **Application button** (leftmost) gives quick access to file, print and software options (including security) as well as software version information.

Control ribbon

Single-click access to key software functions.

Record View panel

Lists all measurements within the active measurement file. Multiple measurement files can be opened simultaneously, which are then accessible by different tabs within the Record View.

All files tab

View all records from all open measurement files in a single tab.

Reports tabs

Provides access to reports on the currently selected record. The reports shown are a function of the currently selected workspace.

Data Quality tab

Provides guidance on the measurement quality and displays a simple pass/fail data report based on selected models, together with tips on how to improve the measurement. The Data quality tab is not available in the MS3000E basic software.

Status bar

Shows the instrument mode, connected user, 21 CFR status, notifications (see later in this chapter), connected accessory type and the instrument connection status (as well as its serial number when connected).

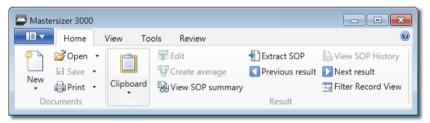
Private/Shared workspace

The workspace shows the settings and data assigned to the user or users using the instrument at the time. Individual (private) or common (shared) workspaces can be created. Workspaces are not available in the MS3000E basic software.

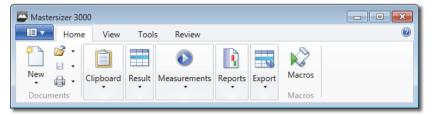
Control ribbons

Control ribbons provide quick access to key software functions.

 To select an option, click on the appropriate direct-access button, for example Extract SOP.



If you contract the software window horizontally, the individual ribbon options are grouped under a single icons instead. The ribbon then operates in a similar manner to menus in that the group must be selected initially to reveal the sub-items, for example **Result-Extract SOP**.



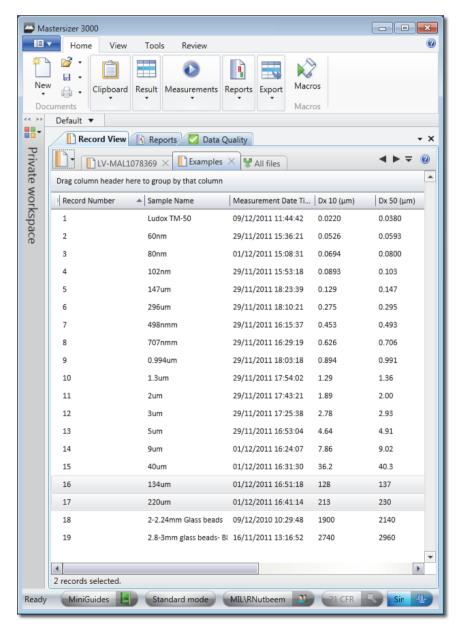
■ To select a different control ribbon, click the selector tabs above the ribbon (Home, View Tools or Audit).

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Software overview Chapter 3

Record View tab

The **Record View** allows you to view and edit the records contained in Master-sizer 3000 measurement files. It is also possible to open files that were created by Mastersizer 2000 instruments, but not edit them. Mastersizer measurement files consist of a number of individual measurement records.



Each open measurement file is given a tab in the Record View: click one of these tabs to show all of the records contained in that measurement file.



Note

You can open as many measurement files as required, and also combine results, using **All Files**, to view all records within a single tab, making graphical data overlay possible. See **Merging records** in the **Viewing the measurement results** chapter for more information.

Each measurement record consists of a set of parameters, one of which is shown in each column. (It is possible to edit the selection of parameters that are displayed if required.) Many parameters contain numerical data measured by the system, but some contain other information, such as **Scattering Model**, **Accessory Name** and so on. Some parameters also answer simple yes/no questions, such as **Has Background Alarm Triggered?** Items like this are checked if affirmative.

Powerful analysis features also allow records to be sorted, grouped and filtered so that users can focus on only those records that are of relevance to their application. See the **Viewing the measurement results** chapter, and the **Help file**, for more information on grouping and filtering.

The Parameter filters, grouping and column configuration features are only available with the Mastersizer 3000 / Mastersizer 3000E with Extended software option.

Editing which parameters are displayed

To choose different columns of data to display in the **Record View**:

- 1. Right click on a column header and choose **Column Configuration**.
- 2. Then, from the **Parameter Selection** window, select items in the **Available** column and add them to the **Selected** column. Use the **Search** feature in this window to quickly find a parameter.
- **3.** Click **OK** to complete the process.

Sorting on a parameter column

Click the column header. Click the header again to reverse-sort based on that selection - sorting is either alphabetical, numeric or boolean, depending on the parameter selected.

Locked measurement files

Any tab indicated with a padlock icon is a read only file that cannot be edited. This is the default status for any Mastersizer 2000 files that are opened with the Mastersizer application software.

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Software overview Chapter 3

Selected Records Information

At the bottom of the Records view a summary is provided that details the number of records currently selected within each measurement file.

Copying records

Records can be copied in a couple of ways:

■ Duplicating records:

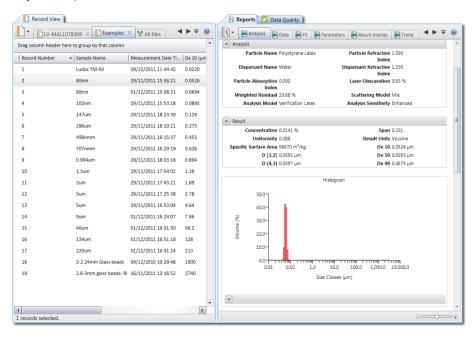
To duplicate records to a new measurement file or within the same measurement file. From the **Clipboard** group on the **Home** ribbon, select **Copy** - this will copy the records to the clipboard. The content can then be pasted back into the Mastersizer application software.

Copying records into Excel:

To quickly copy records into Excel or similar applications the selected records must be **dragged** from the Mastersizer application software, and **dropped** into the chosen application. Alternatively the data export options can be used (refer to the **Advanced features** chapter).

Reports tab

Reports are displayed automatically whenever you select a record, or number of records, from the **Record view** (assuming that the selected view is showing the Reports tab).



Chapter 3 Software overview



Note

For detailed information on the process of viewing, editing or creating new reports, see the **Reporting** chapter

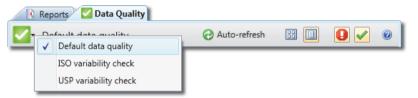
Data quality tab

The Data Quality tab is only available with the Mastersizer 3000 / Mastersizer 3000E with Extended software option.

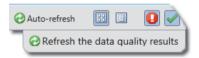
The Data quality tab presents a custom analysis of any records selected in the current measurement file.

Select the records for which you wish to view a data quality report.

Click the **Data Quality** tab (next to the **Reports** tab).



Select the **refresh** tab; this will drop-down from the menu bar when a new record is selected)

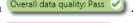


Alternatively the display can be set to **Auto-refresh**. Selecting the button will set the display to automatically refresh the data quality view whenever a new record is selected Auto-refresh

Press again to de-select.

The display is updated to show a quality review of each record selected. Click • to toggle whether items that have passed or failed are displayed.

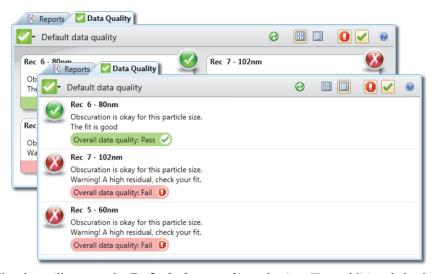
Items that have **passed** the quality check are shown Overall data quality: Pass



Items that have **failed** the quality check are shown Overall data quality: Fail

Results can be shown in a tabular 🔠 or list format 🔲

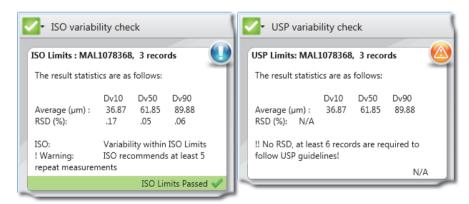
Page 3-8 **MAN 0474**



The above illustrates the **Default data quality** selection. Two additional checks are available; these are **ISO variability** and **USP variability**.



Illustrations of each are shown below.



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Workspaces

Workspaces are only available with the Mastersizer 3000 / Mastersizer 3000E with Extended software option.

This section describes the purpose of workspaces and the **Workspace** pane.

Workspaces are a collection of settings that define the information presented in the Reports and Record View, as well as which folders the system uses for accessing SOPs, measurement data and export data.

Each user can access both a Private or Shared workspace, both of which are configurable. This makes it easy for an individual to optimise the software for their own use, with the **Private workspace**, or to collaborate better across an organisation using a **Shared workspace**. The latter is particularly useful within quality controlled environments.

The following settings and data are associated with the workspace:

- SOPs and SOP Templates presented to users (i.e. their file locations).
- Record parameters shown in the Record view.
- Reports listed in the Reports view (see the **Reporting** chapter).
- Measurement data file location.
- Exported data and Export Templates file locations.
- Workspaces also control where the software will look for data quality addins, macros, user calculations, etc.



Note

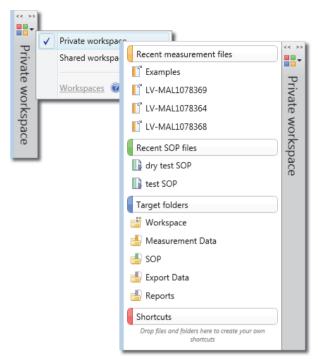
Any of these items that are created when the **Shared workspace** is active will be available to all users of that system (providing their security privileges allow them access to these functions). If the **Private workspace** is selected, these items are only available to the current user.

Selecting the workspace

Reveal the **Workspace** pane by clicking the window. Clicking the button again will close the Workspace pane. Additionally the panel splitter bar can be dragged left or right to resize the workspace pane.

To select the required workspace click the button at the top of the **Workspace** panel:

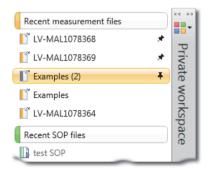
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Any work subsequently performed on the system will now use the selected workspace both to save data and to access settings.

Recent measurement files

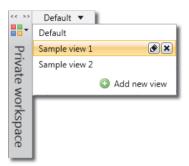
This is a simple list of the last five measurement files that have been accessed within the selected workspace.



Measurement files and SOPs within the workspace can be pinned to keep them in the windows indefinitely. To pin an item, hover the mouse over the item and select the pin button. Chapter 3 Software overview

Record and reports view selector

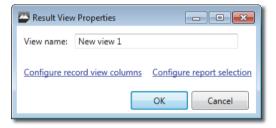
The record parameters shown in the Record View, and the reports listed in the Reports view are controlled using the **Record and reports view** selector to the right of the Workspace pane. Additionally the Records and report selector stores (and restores) any selected data quality addins.



You can add as many different views as required – these are stored within either of the currently selected workspaces.

To add a new view:

1. Click **Add new view** from the **Workspace** pane.



- 2. Enter a **View name** in the **Result View Properties** window.
- Click Configure record view columns to show the parameter selection window. Select the required parameters and then click OK.
 Similarly click Configure report selection to display the Report Selection window, select the required reports and tab order, and then click OK.
 - Refer to the **Reporting** chapter for details on report selection.
- **4.** Once you have added new views, make them the active view by clicking on them from the list. The selected view will now be shown as the record and reports view selector title.

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The Reports and the Record View are updated immediately.

5. To edit or remove a view, first select it from the list and then select the **Edit** or **Remove** icons.

Targets

The targets listed are links to Mastersizer system folders and provide quick access to all of your workspaces, reports, SOPs, measurement data, etc. Clicking on a target will open the respective folder within Windows Explorer.

The folders that are linked to from this panel are specific to the **selected** workspace.

If the **Private workspace** is selected, the folders all stem from:

C:\Users\YOURNAME\Documents\Malvern Instruments\Mastersizer
3000\Workspace\

If the **Shared workspace** is selected the folders stem from:

C:\ProgramData\Malvern Instruments\Mastersizer 3000\Workspace\

Shortcuts

Shortcuts are an easy mechanism for users to set-up a set up links to folders, e.g. on a network, that can be shared between all users on that PC.

To create a shortcut: Create new shortcuts to files/folders by dragging them into the Shortcuts panel from Windows Explorer. Shortcuts to web sites can be added by dragging a bookmark from Internet Explorer into the Shortcuts panel.

Customising the software interface

In addition to the options available as part of the Workspace specification, several further features provide control over how the software is presented to the user.

Options

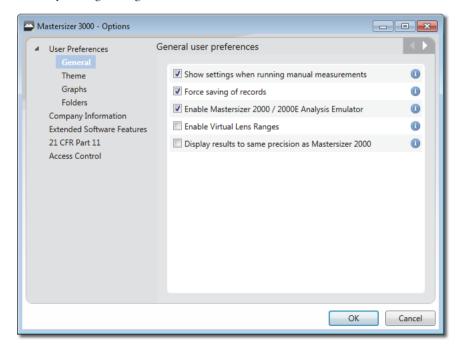
The options window provides access to various user preferences and allows security to be enabled/disabled. Selection of the options available will enable them for use in the associated software control windows. If they are not selected they will not be available for use.



Note

Some options are only available with a **feature key** installed. Please contact your Malvern representative for more information.

From the **Application button** menu choose **Options** to display the **Mastersizer 3000 - Options** window. Then click the required link on the left to view its corresponding settings.



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General Preferences

Click **General** from the **Options** window to choose the following settings:

■ Show settings when running manual measurements – this automatically displays the measurement settings window whenever a manual measurement is initiated. This can be useful during method development where the user needs to switch back into the record view after making each measurement and then make slight alterations to the measurement settings for the subsequent measurement. If this option is de-selected, the settings are still available to the user by clicking on the Manual Measurement window.

■ Force saving of records – with this option selected every measurement file will be saved on creation with temporary measurement files no longer being made. The measurement files will automatically be saved when the software is closed, so there will be no prompting for the user to make changes.

Note that this option is automatically enabled and cannot be changed when the 21 CFR feature is installed.

■ Enable Mastersizer 2000 / 2000E Analysis Emulator – Enable this option to add 3 additional emulated Mastersizer 2000 / 2000E analysis models to the list of analysis models in the **Data processing - Analysis** SOP and record editor.

The emulator converts Mastersizer 3000 results to how it would be analysed on a Mastersizer 2000 instruments, and similarly for a Mastersizer 3000E result the Mastersizer 2000E instruments is emulated.

Refer to **Data processing - Analysis** SOP description in the **Making measurements** chapter.

■ Enable virtual lens ranges – This enables the controls on the advanced analysis page of the SOP for virtual lens ranges. Virtual lens ranges limit the result analysis range to that of a legacy instrument, such as a 300mm lens used on a Mastersizer X.

Refer to **Data processing - Analysis** SOP description in the **Making measurements** chapter.

■ **Display results to same precision as Mastersizer 2000** – this option enables the software to display results to the same precision as used in the Mastersizer 2000 software. Results will be displayed to 3 decimal places.

Themes

The Themes option is only available with the Mastersizer 3000 / Mastersizer 3000E with Extended software option.

Several themes are available which modify the colour settings of the user interface. Click **Theme** from the **Options** window and then select a scheme that suits your preference by clicking **Use this theme**.



Graphs

Choose **Graphs** from the **Options** menu to access further options that relate to the presentation of graphs:

- **Line weight** specify the line weights (Normal, Light or Heavy) to be used in all graphs.
- Colour scheme choose from one of several colour schemes. The Master-sizer 2000 theme emulates the appearance of the Mastersizer 2000 graph colour scheme, Greyscale is optimized for printing on a monochrome printer, High Contrast provides better on-screen visibility.

Folders

Use this option to either view, by clicking on the folder; or edit st the workspace folders where the Malvern data is stored.



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Company Information

The **Company name** and **Logo** can be added. This information will then be displayed on all reports.

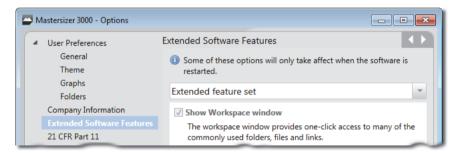
Select 🔼 to assign a logo, or 🔳 to restore the default logo image.

Extended software features

The same software is used to control both variants of the Mastersizer instruments - the Mastersizer 3000 and the Mastersizer 3000E, though some software features are removed for a 3000E installation.

The Mastersizer 3000E software focuses on just the necessary software features for operating the instrument - this is termed the **Basic feature set**.

Installing the **Extended feature set** will enable the Mastersizer 3000E to utilise the full range of the standard software and its additional features.



With the Mastersizer 3000 the extended software features are installed by default and will always be available for selection.



.The icon will be greyed out if not installed.

For the Mastersizer 3000E to have access to these features a **feature key** needs to be installed. Contact your local Malvern representative to upgrade the Mastersizer 3000E basic software to the extended software version?

With the feature key installed, three levels of configuration are available:

- **Basic feature set** disable all extended features (shown in table below) and run as a Mastersizer 3000E.
- Extended feature set enables all extended features and run as a Mastersizer 3000.
- **Custom feature selection** all extended features can be selected individually. Tick/untick the features as required and press **ok** to save.

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Extended features

Show workspace window	Dock accessory controls
Show data quality window	Show search boxes
Show trend view in measurement display window	Show split view in measurement display window
Show SOP player	Record view filter and grouping
Show documentation editor	Configurable windows

Consult the help system for a full description of each feature.

21 CFR Part 11

The 21 CFR Part 11 functionality is only available for Mastersizer 3000 users.

Select this option and input the 21 CFR feature key when requested. With the 21 CFR part 11 feature key installed these features are available:

- Enable Auditing
- Enable Electronic Signatures

Access control

Select **Enable security features** to activate the security features This option is only available once a security configuration file has been created using the optional **Malvern Access Configurator (MAC)**.

Custom layouts (Save/restore layout)

The configurable windows feature is only available with the Mastersizer 3000 / Mastersizer 3000E with Extended software option.

Save layout is a simple feature that allows you to store the exact position of all elements of the Mastersizer interface. This could be useful in situations where you have optimized the interface for your current analysis process.



Positioning elements within the user interface

To reposition items - drag any of the following panels to another position within the main window: Record View, Data Quality, and Reports.

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The following tool is shown on the screen when dragging:



This indicates that pre-set positioning options are available for the element. Whilst still holding down the mouse button, move the mouse pointer over one of the direction arrows in this tool - a shaded box is shown indicating how the element will be placed. Release the mouse button to place the element.

Saving or restoring the window layout

To save commonly used panel and window arrangements for future use:

- 1. Position any of the panels of the software as desired, for example, the Record View, Reports etc.
- 2. Click **Save layout** on the **View** ribbon.
- 3. The **New layout** file window is displayed enter a name for the layout (all layouts have the .mlay file extension) and click **Save**.
- **4.** To access a previously saved layout, click **Restore layout** and then locate the relevant .**mlay** file.



Note

By default the system initially returns to the window layout that was present when the software was last closed.

Predefined Window Layouts

The **Predefined window layouts** allow to you quickly select from a number of optimized panel arrangements within the Mastersizer user interface.



■ From the **Predefined window layouts** group in the **View** ribbon, select the required layout.

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Quick access toolbar

The Quick access toolbar provides an alternative method for accessing commonlyused functions.

1. To add any ribbon function button to the Quick access toolbar, right-click it and choose **Add to Quick access toolbar**. The button is now additionally displayed (in reduced form) in the title bar of the software:



In this example, the Manual measurement, Save and 2-pane tabbed buttons have been added to the Quick Access toolbar.

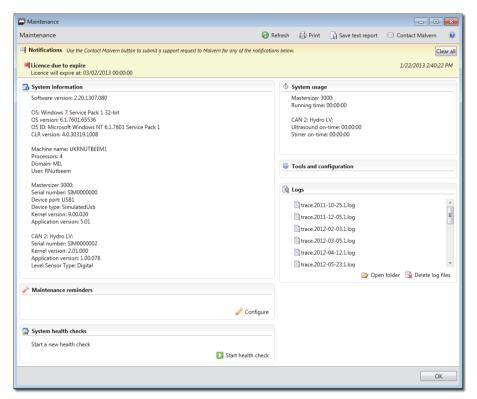
2. To remove the button, right-click it and choose **Remove from Quick Access** toolbar.

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Maintenance utilities

The Maintenance window gives access to various background information about the system that can be useful as part of an ongoing maintenance schedule or in the process of contact with the Malvern Instruments Help Desk.

Choose Tools-Maintenance from the ribbon to display the Maintenance window:



The window is broken down into the following sections:

Notifications

Notifications are brief notes that indicate any errors, problems or necessary remedial/investigative action that may be required in order to keep the system functioning correctly. Follow the advice given in any notifications and then remove them by clicking **Clear All**.

The Status bar also provides a visual reminder that notifications are available:



Click the **Notifications** icon on the status bar to view the **Maintenance** window.

System information

The main purpose of the system information area is to display details about the software, operating system and hardware connected. This information must be passed to the Malvern Instruments Helpdesk whenever you log a support call. To send this information to Malvern Instruments:

If you have email software on the Mastersizer computer:

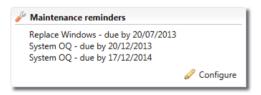
■ Click **Contact Malvern** to display the contact form. Complete the form and then click **OK**. Your default email software then starts and an email is generated containing the relevant system information. Additional fields appear in the email where you can input information about the issue you are experiencing with the Mastersizer system.

If you do not have email software on the Mastersizer computer:

- Click Save text report to save a text file containing this information. Then copy this information onto a data stick and transfer it to a computer that has an email connection in order to attach the file to send to Malvern Instruments.
- Print the information using the **Print** option and then refer to this information if you wish to contact the Helpdesk via telephone.

Maintenance reminders

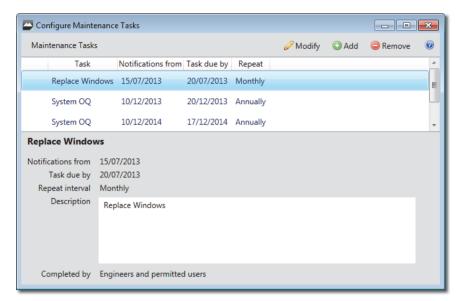
Maintenance reminders can be set up within the software to prompt users to carry out routine maintenance tasks. To configure these, access the **Tools-Maintenance** window. Within the **Maintenance** window, there is a **Maintenance reminders** section, where any active reminders are listed.



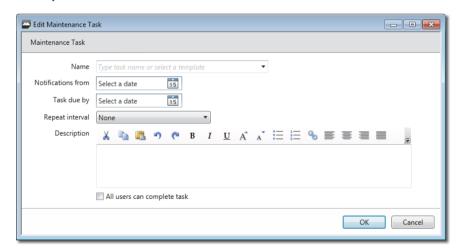
Click on the **Configure** Configure icon to open a list of active maintenance tasks, from which existing tasks can be edited, or new ones added:

Click on a maintenance task to show its full details. If the task requires editing select the **Modify** button.

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To set up a new maintenance task, click the **Add** button.



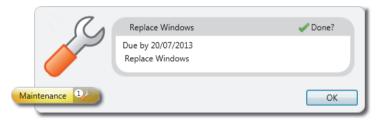
The details required include:

- Name: a task name.
- **Notifications from**: the date when users should start being prompted to carry out a task.
- **Task due by**: the date by which the task needs to be completed.
- **Repeat interval**: how often the task should be done (weekly, monthly, yearly, etc).

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- Task description: text providing guidance as to what needs to be done by the user.
- All users can complete task option: Select this checkbox to confirm that any user can complete the task. If this is left un-ticked then only users who have the Open Maintenance security permission assigned to them can set the task to done. This could, for instance, apply to tasks associated with booking a service or OQ visit, where it would be the supervisors responsibility to complete the task.

Users are alerted to tasks which are due to be carried out by a **Maintenance** icon in the **status bar**. Clicking on this will display the maintenance detail.



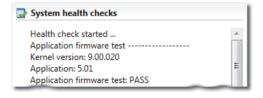
Clicking on the **Done?** Vone? icon clears the task.

If the task is not completed by the due date then a service **Notification** is generated and is displayed on the application's status bar.

System health checks

The Mastersizer application software constantly checks the status of the optical system and the dispersion units to ensure it is functioning correctly. If any issues are detected, they will be reported within this section for the maintenance report.

A health check can be performed at any time by selecting the Start health check button. The results of the tests will be reported in the System health checks section of the maintenance dialogue.





Note

Connection to a system is required for this option to work.

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System usage

The system usage section provides details on the total Running Time of the instrument and any connected accessory. Additionally details are provided on the running time of the accessory stirrer running time and ultrasound if applicable.

Logs

The **Logs** section contains links to all the logs generated in the previous 24 hour period. After 24 hours, if no further log files have been generated, only the last log file generated is retained. Log files contain no data that is of direct use to users, but may be requested by the Malvern Helpdesk when trying to resolve any issue.

To send a log file:

- Click on the **Save Text Report** option on the maintenance report icon bar. This will record a new system status report.
- Click on the **Open folder** option within the **Logs** section of the report. This will open the directory containing all of the system logs.
- Copy all of the logs, and email these to the Malvern Instruments.



Note

In the case where a software crash has occurred, a series of crash dump files (.dmp) will be stored in the logs directory alongside any maintenance reports and a trace.log file. These files contain detailed information about how any crashes occurred, and should therefore be sent to Malvern Instruments along with any maintenance logs.

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Making measurements

Introduction

The Mastersizer is capable of measuring samples dispersed in a wet or dry medium, either manually as a one-off (or for developing an SOP method) or by utilising predefined settings stored within an SOP file.

This chapter is the main reference for the measurement making process and includes sections on:

- Measurement types a brief background on the overall process of making a Mastersizer measurement.
- **Measurement file basics** how to create and open measurement files.
- **Making an SOP measurement** includes how to create SOPs as well as the steps involved in running an SOP.
- **Making a manual measurement** the basic steps involved in manual measurement with a wet dispersion unit.
- **Measurement settings reference** detailed reference on all measurement settings available to both SOP and manual measurements.

Measurement types and process

The Mastersizer is capable of making two types of measurement:

■ **Manual measurements** – all of the measurement settings are specified before the measurement and some further user input is required during the running of the measurement. The measurement process is also split into key stages, which are paused after the completion of each stage.

Manual measurements tend to be used for one-off measurements or as part of method development (i.e. establishing the optimal settings for measuring the sample and then saving into an SOP file).

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■ Standard Operating Procedure (SOP) measurements – most of the measurement settings are stored within an SOP file which has been previously created by the user. Once an SOP has been initiated, the measurement sequence requires less user intervention than a manual measurement.

As SOPs lock-down most of the measurement settings they improve consistency and provide greater repeatability; features that are important in quality-controlled environments.

The process of both measurement types is broadly as follows:

- 1. Power on the system and create a measurement file.
- 2. Specify the measurement settings for a manual measurement or SOP.
- 3. Run the measurement.
- 4. Check the results, looking for any problems.
- 5. Clean the system.

Measurement prerequisites



Note

The instrument and accessory must have been connected and set up in accordance with the guidance in the Mastersizer Basic Guide and the dry or wet dispersion unit guide. This section assumes all appropriate connections have been made.

Before a measurement can be made, the instrument must be powered up and a measurement file must be created. Measurement files contain all data acquired by the Mastersizer during a measurement. Each measurement file can contain a number of individual records.

1. Switch on the optical unit. The pulsating blue light on the top of the instrument indicates that it is on. Check also that the blue light on the dispersion unit is pulsating - power to the dispersion unit is drawn from the instrument.



Note

Leave the instrument powered on for 30 minutes before making a measurement to allow the laser to stabilise.

2. When 30 minutes has elapsed, switch on the computer, log in to Windows on the PC and start the software by double-clicking on the **Mastersizer** icon on the desktop:



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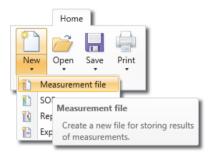
Note

If, after successfully starting the Mastersizer application software, any of the functions described in the rest of this section are unavailable to you, this will be due to your login credentials – contact your system Supervisor for assistance.

3. Ensure that the status bar indicates that the instrument is connected correctly:



- 4. If the instrument is not connected correctly (i.e. if anything else is shown in the status bar), refer to the **Software overview** chapter for more information on how to fix this.
- Create a new measurement file by selecting New-Measurement File from Home section of the control ribbon:



Alternatively new measurements can be added to an existing measurement file.

6. Choose **Save** and then name the measurement file, for example **Starter sample.mmes**.

Making SOP measurements

SOPs are predefined measurement sequences that can be used to both automate and standardise Mastersizer measurements. Widely used in the pharmaceutical industry to provide traceability, SOPs also provide non-pharmaceutical users with a reliable means of automating measurements, ensuring that a wide selection of personnel can obtain consistent results regardless of familiarity with the instrument.

SOPs can control the configuration of the optical unit and the sample dispersion unit, including the measurement parameters, timings, analysis models and other specific set-up information. They can also be configured to prompt the operator to perform specific tasks such as sample preparation steps.

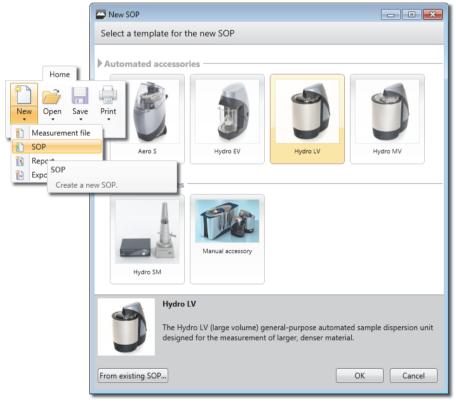
The quality and value of an SOP depends on the quality of the method development work on which it is based. A series of manual measurements is required to establish the ideal parameters for use in an SOP. A number of standard SOP templates have been prepared in advance for typical measurements, which may be used unchanged or as the basis for a new SOP.

The Mastersizer application software's powerful SOP editing facility enables users to review and refine SOPs as part of their ongoing quality control process.

Creating or editing an SOP

SOPs need to be created and saved before they can be used. To create a new SOP:

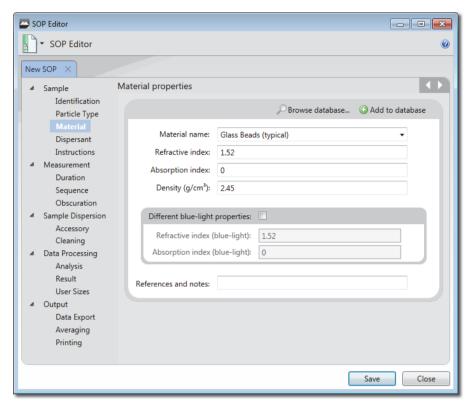
 Select New-SOP from the Home ribbon. (To edit an existing SOP, choose Open-SOP instead.)



2. Select an SOP template from the list in the **New SOP** window or click the **From an existing SOP...** button to locate an SOP in the file system that fulfils (or is close to) your required criteria.

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3. The **SOP Editor** window is now displayed as illustrated (the **Material** settings are shown in this example):



4. Step through the **SOP Editor** by clicking the section titles on the left (**Sample-Identification**, **Sample-Material** etc.) or clicking the Forward / Back arrows on its toolbar.



Note

Complete each of the sections detailed in the **Measurement Settings Reference** section in later in this Chapter.

5. When all the required settings have been specified, you can either choose to save the SOP as a file or as a template. choose **File-Save** and then give the SOP a logical name.

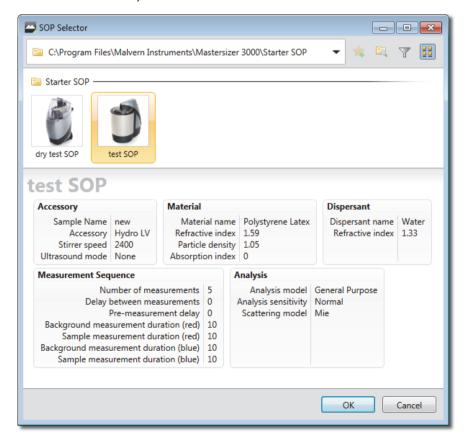
Running an SOP (Hydro units)

If you are using a wet dispersion unit (the Hydro LV/MV/EV) work through the steps in the order given here. No manual control of the dispersion units is required after loading the sample; the software controls all settings.

1. Select **Run SOP** from the **Measurements** section of the ribbon.



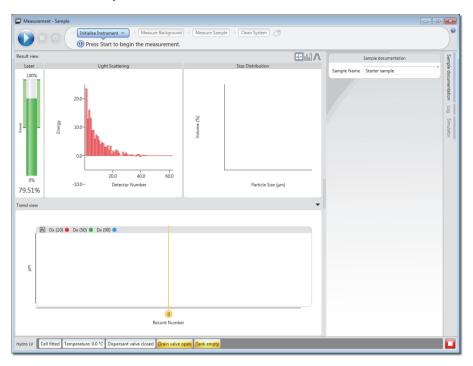
2. The SOP Selector window is shown, which initially presents all SOPs filtered for the connected accessory (Accessory filtered). Alternatively, deselect the Accessory filtered button to show those that are not specific to the connected accessory.



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Click on an SOP to display its properties in the bottom panel of this window.

- **3.** After selecting an appropriate SOP, click **OK**.
- **4.** The **Measurement Display** window is shown. The progress bar at the top of the window reports both the current status and what to do next.



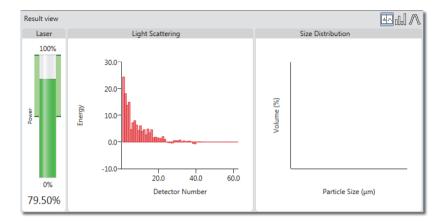
The Trend view show in the bottom half of the Measurement display illustration is only available with the Mastersizer 3000 / Mastersizer 3000E with Extended software option.

Result view panel

The **Result view** panel contains the **Laser**, **Light Scattering** and **Size Distribution** panels. The information shown is either live or, after a measurement has been completed, relates to the currently selected record (the bold yellow line) - this record number is shown at the top of the Result View panel.

Laser - the Laser display panel initially provides an indication of the current Power output of the laser, and then, during the sample addition phase, with the current obscuration level. When adding sample it is vital that the obscuration falls within the green band - this band is defined by the user in the Measurement obscuration section of the Manual measurement settings window.

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- The **light scattering** and **size distribution** views can either be shown separately or tiled next to each other by selecting the split view button.

The split view button is only available with the Mastersizer 3000 / Mastersizer 3000E with Extended software option.

5. Click the **Start** button to **Initialise the instrument**. If specified in the SOP settings, the system prompts the user to enter/confirm sample documentation details. Following this stage. the system automatically moves to the **Measure Background** stage and measures both the red and blue light values of the background.

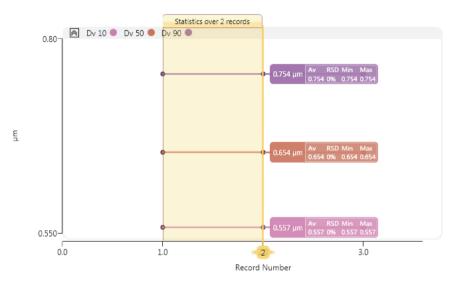


Note

To stop both the SOP and the accessory during a measurement, click the **Abort** button . This "all stop" command should only be used when a problem is suspected - this is because the sample will stop circulating, which could affect further measurements if the sample is not then refilled.

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- 6. When the Measure Background stage is complete, the SOP pauses. The system now requests that you add sample do this until the Obscuration bar indicates about 10-20% for a wet dispersion unit. (This is a rough guide only as this setting is highly sample-dependent refer to the Help system for more guidance.)
- 7. Click Start again. The measurement now commences, measuring first the red and then the blue light values. The SOP may make a number of measurements before completion, depending on the Number of measurements specified in the SOP settings. When complete, the Trend view is updated with the new measurement figures:



- 8. Complete the measurement by closing the **SOP Measurement** window.
- 9. The results are now presented in the **Record View**.

More about the Trend view

The Trend view is only available with the Mastersizer 3000 / Mastersizer 3000E with Extended software option.

The Trend view is a powerful analytical tool that shows record data in a graphical format as soon as it has been measured by the system.

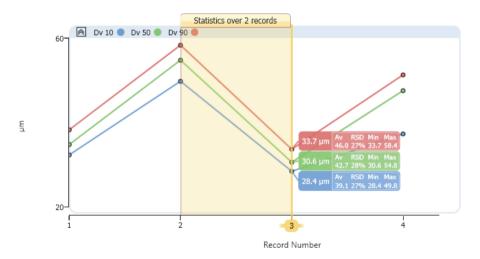
The main purpose of the default view is to indicate that sample measurements are stable. For example, if the sample was slowly dissolving then the apparent sizes may appear to be reducing. In many industries, measurement repeatability is quantified by specifying a maximum Relative Standard Deviation (RSD) over a number of measurements. The Trend view allows you to highlight a number of measurements and display the RSD, and other statistics in real time. Any other parameter

can be added to the Trend view, for example to see how Ultrasound Power settings are affecting the measurement.

By default, each measurement's Dv10, Dv50 and Dv90 figure is shown, so it is very simple to make a quick assessment of the result. The Dv50 category shows the median particle size. Further statistics and measurement parameters can also be viewed on the Trend view.

Once a number of measurements have been performed, it is possible to select multiple records in order to view combined figures for the range. It is also possible to modify the data displayed on the graph, and add entirely new graphs with a different focus of interest if required.

This is an example of the trend view after four measurements have been made. The currently selected range in this example includes just two records: Record Number 2 and 3 - the figures displayed in the coloured boxes relate only to those two records.



Statistics shown in the live trend view

The live trend view identifies each line with a key, which shows statistical information about the measurement:

578.19 μm Av RSD Min Max 578.19 0% 578.19 578.19

The data shown relates either to the individual measurement or range of measurements selected by the user. The default view shows particle size statistics. Trend plots can be configured to show any parameter – the statistics shown on a plot line relates to a single parameter only.

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- **Main figure** the actual size measurement (μ m) for the single base record that is currently selected (Dv10 blue, Dv50 green, Dv90 red).
- **Av** the average particle size (μ m) for the selected range.
- **RSD** Relative Standard Deviation (%) for the selected range.
- **Min** the minimum particle size (μ m) for the selected range.
- **Max** the maximum particle size (μm) for the selected range.

To modify which figures are shown in the Trend view:

- 1. Right-click anywhere on the trend graph and choose **Statistics**.
- **2.** Select or de-select the items required.



Tip

Click anywhere on the graph line to display data points - click again on the line to remove them. Move the mouse pointer over a data point to display its record number and size figure, for example 4, 891.09 indicates that Record 4 has a measured size of $891.09\mu m$.

Selecting a range of records in the trend view

After running several measurements, just one record is selected. Initially this is the last measurement made. This record is shown by a vertical hairline bar with the record number indicated at the bottom - for example, ³. The vertical hairline bar always shows the currently selected record.

■ To expand the range to incorporate more records, click the left or right selection arrow buttons at the top of this bar. The bar expands to cover the

records selected: Statistics over 2 records

Click and drag this header bar to the left or right to reposition the selection within the range of records. Alternatively, right-click the trend graph and choose: **Statistics range** and then select the number of records.

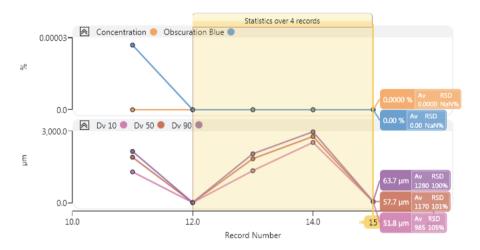
■ To contract the range, click and drag the left hairline bar that denotes the initial record in the selected range. Alternatively, right-click on the trend graph and choose: Statistics range and then select the number of records to include in the selected range. If you have manually dragged the bar to cover a number of records not listed in the Statistics range menu, Custom and the number of records currently selected is displayed.

All numerical data then provided is the arithmetic mean of the currently selected records, with the exception of the initial particle size figure, which is still the figure for the single record.

Adding further trend graphs

Add additional plots using the following method (note that the new plot needs to be configured prior to making the measurement in order to display data):

- Right-click on the trend graph and select **Add above** or **Add below** to add a new plot.
- Then choose parameters from the **Parameter Selection** window and click OK.



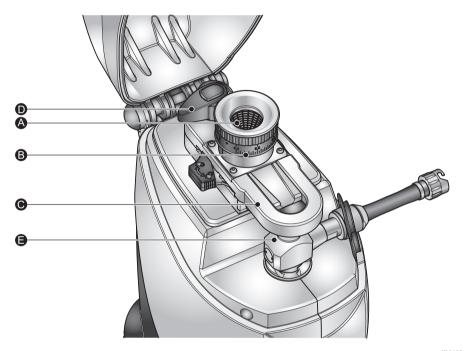
To remove the new plot, right-click it and choose **Delete**.

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Running an SOP (Aero units)

The Aero S dry dispersion unit is only compatible with the Mastersizer 3000 optical unit.

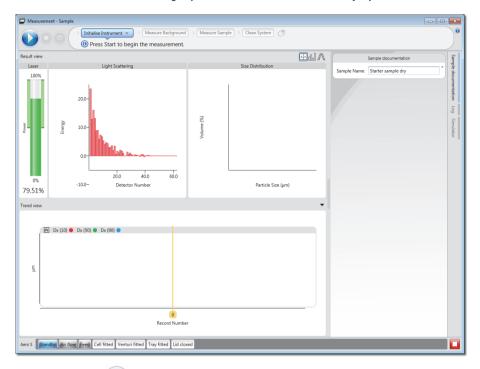
The software controls most of the dispersion unit settings except the hopper height, which is a manual adjustment. Work through the steps in the order given here.



- 1. Open the Aero S sample area lid and set the hopper flow rate [®] to an appropriate gap level using the dial (marked from 0-4mm - refer to the Aero Series Dry Dispersion Units Guide for more information).
- **2.** Empty the sample into the **hopper (A)**.
- 3. The mesh basket and ball bearings may be used on coarser samples to smooth the flow of sample, breaking up any loose agglomerates before they fall into the hopper.
- Close the lid of the Aero.
- Select **Run SOP** from the **Measurements** section of the ribbon.
- The **SOP Selector** window lists all available SOPs.

Mastersizer 3000 Page 4-13 7. Choose an appropriate SOP for the Aero dispersion unit (ensure also that the SOP is optimized for the venturi type that is fitted to your Aero unit - either stainless steel or ceramic venturi) and then click **OK**.

The **Measurement Display** window shown below is displayed:



- **8.** Click **Start** the SOP automatically aligns the optical system and then measures the background using the red light (blue light is not used in dry measurements).
- **9.** After this, the sample tray starts vibrating and the SOP-specified air pressure is applied. The overall rate at which the sample is fed into the cell is a function of the combination of air pressure, vibration rate and hopper flow-rate setting.



Note

To stop both the SOP and the accessory during a measurement, click the **Abort** button .

10. The SOP selected should be optimized for the dry dispersion unit and will only make a measurement once the obscuration is within the specified range (1-10% obscuration is the rough guide for a dry dispersion unit).

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- 11. The results are shown immediately in the **Trend view** as they are gathered. When all of the measurements have been made, the system pauses optionally, click **Clean System** on the top progress bar to initiate a cleaning cycle.
- **12.** Complete the measurement by closing the **SOP Measurement** window. The results will now be presented in the **Record View**.

Making manual measurements

In addition to SOP measurements, users can also make manual measurements. This may be useful, for instance, when investigating the most appropriate method for sample dispersion prior to setting up an SOP. This section provides details on how to run a basic manual measurement.

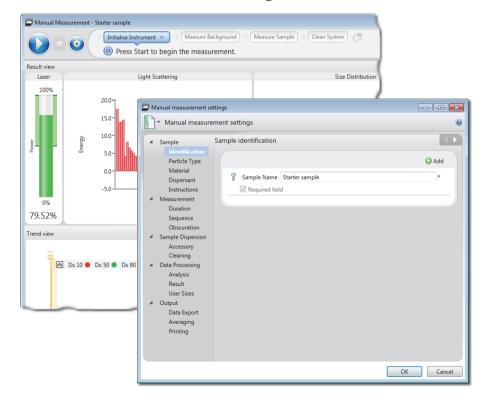
0

Note

This process description assumes that a wet dispersion unit is connected to the instrument.

The basic procedure is similar to that outlined in the SOP measurement section, but requires the user to set up the measurement settings initially, and then manually initiate more stages during the live measurement process:

From the Home ribbon click Manual measurement from the Measurements group. The Manual Measurement window is displayed, together with the Manual Measurement Settings window:



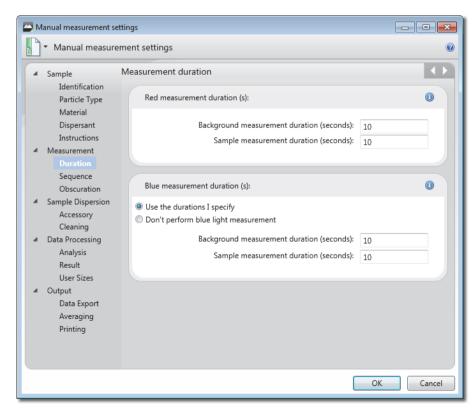
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Note

This section now provides information on how to enter only some of the key measurement settings - for complete details on all settings, see **Measurement settings reference** later in this chapter.

- Choose the Sample-Identification settings to record both the Sample
 Name details and define any further information fields (for example, Notes)
 that will help users to repeat the measurement at a later date.
- 3. Use the **Sample-Material** section to define the optical properties (the **Refractive index** and **Absorption index**) of the new material. The material may already be stored in the system's **Materials database**. Check this first by clicking **Browse database** select the material if it is present to automatically add the RI and Absorption values to the measurement.
- **4.** Use the **Measurement-Duration** section, shown below, to set up measurement times for the red and blue measurements.



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- 5. In the **Measurement-Obscuration** section define the lower and upper obscuration limits as a very rough guide, about 10-20% for a wet dispersion unit and 1-10% for a dry dispersion unit.
- 6. Click **OK** to close the **Manual measurement settings** window.
- 7. From the **Initialise Instrument** drop down menu, select **Auto Alignment**.



- **8.** Click the **Start** button to initialise the instrument when complete, the system progresses to the **Measure Background** stage.
- 9. Filling and starting the dispersion unit:

Depending on the accessory that is currently connected, the control options (shown in the panel on the right) will vary. For a wet dispersion, a steady circulation of dispersant needs to be in place before adding the sample. To do this, ensure the accessory is filled with dispersant - if using an LV/MV, click **Fill** in the **Dispersant** section of the **Accessory panel**.





Note

See Controlling dispersion units (manual mode) later in this chapter for more information.

The docked accessory control tab in the Measurement display is only available with the Mastersizer 3000 / Mastersizer 3000E with Extended software option.

Users with the Basic software feature set should control the dispersion unit using the Accessories option on the Tools ribbon.

10. When the accessory is full, set the **Stirrer speed** and start the accessory:



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- **11.** Click **Start** on the measurement progress bar at the top of the window. Both the red and blue light values for the background are measured.
- **12.** The system now requests the user to **Add Sample** add small quantities of sample to the dispersion unit until the obscuration is within range (10-20% in the case of a wet dispersion). Refer to the Help system for more guidance. With a dry dispersion, the system will skip this step.

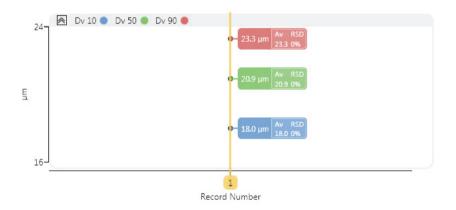


Note

With dry dispersions, the whole sample is loaded into the hopper in the Aero before the measurement process is started.

13. Click **Start** again twice to begin the measurement. First the red and then the blue light values are measured. When this is complete, the **Trend view** is updated with the new measurement figures (see **Running an SOP** in this chapter for more detailed information on the Trend view). The particle Size Distribution graph is shown in the upper right panel. In the example shown here, just a single measurement was specified in the manual measurement settings.

Trend view



The Trend view is only available with the Mastersizer 3000 / Mastersizer 3000E with Extended software option.

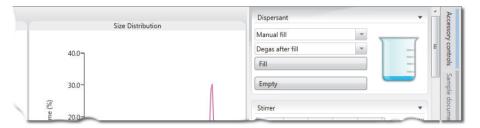
14. Continue this final **Measure Sample** process as many times as required by clicking **Start** again. To complete the measurement, just close the **Manual Measurement** window. The new records are now added to the **Record View**.

Manually controlling the dispersion units

The docked accessory control tab in the Measurement display is only available with the Mastersizer 3000 / Mastersizer 3000E with Extended software option.

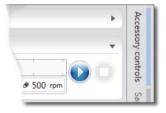
Users with the Basic software feature set should control the dispersion unit using the Accessories option on the Tools ribbon.

Use the **Accessory controls** panel from the manual measurement mode to configure the currently attached accessory.



Normally, an SOP or Manual measurement controls the accessory's operation within the context of a measurement. Alternatively, the **Accessory controls** panel allows you initiate the current accessory before the measurement is actually made. This is essential when observing the effects of variation to the accessory's settings on the live **Laser** and **Light Scattering** panels, in order to optimise the sample's concentration and circulation prior to making a measurement. This option could also be used as part of a manual cleaning process.

■ To access the Accessory control panel: the instrument must be in Manual Measurement mode, then click the Accessory controls tab on the right of the Manual Measurement window.





Tip

The same controls are also available, when not making a measurement, from the **Active accessory control** feature (choose **Tools-Accessories** from the main control ribbon). This option could be useful, for example, if you wanted to keep a sample in circulation whilst undertaking a report analysis.

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The options available on this panel depend on the connected accessory. These are described in the dispersions units respective user manual.

- Hydro series wet dispersion units manual for the Hydro MV, LV, EV and SM dispersion units
- **Aero series dry dispersion units manual** for the Aero dispersion units

Manual control accessories

Manually controlled accessories may also be connected to the optical unit. These accessories do not have an automated or manual software control, but will be controlled directly from the control interface on the accessory itself.

Like the automatic controlled dispersion units, manual accessories are selected from the SOP Editor:

On the **Home** ribbon, select **New-SOP** from within the **Documents** group. ribbon. (To edit an existing SOP, choose **Open-SOP** instead.)



■ Complete the SOP Editor as described in the Mastersizer 3000 User Manual.

Information on the manually controlled accessories is described in the dispersions units respective user manual.

Refer to the **Hydro series wet dispersion units manual** for the Hydro SM dispersion units.

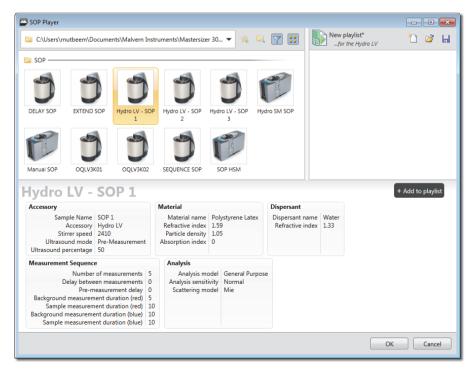
SOP Player

The SOP Player is only available with the Mastersizer 3000 / Mastersizer 3000E with Extended software option.

With SOPs created and saved, users are now able to define an SOP playlist sequence where results can be automatically collected for a single sample using different measurement and/or analysis settings - this is done using the **SOP player**.



In order to set up an SOP playlist, users first need to create a set of SOPs for the dispersion unit they want to use that contain the measurement settings required for the sequence. They then select the **SOP Player** option from the **Home** ribbon bar, the SOP Player dialogue will open.

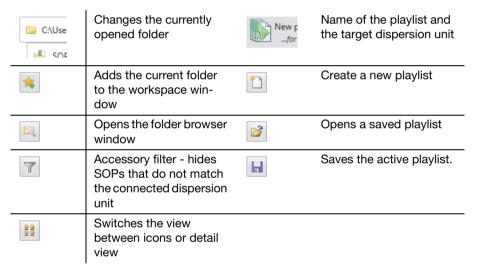


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SOPs available for use in a playlist are shown on the left of the SOP Player window, with the current playlist is shown on the right. The details for any selected SOP are provided in the bottom section of the player, allowing users to confirm the settings the SOP file contains.

To add SOPs to the player, select them from the available SOPs list and drag them

into the SOP playlist, or use the **Add to playlist* button. SOPs can then be dragged up and down into the final position or deleted from the list using the **Subtraction*. As the playlist is constructed, messages will appear to help the users organise the finished playlist (e.g.: Drag and drop SOPs here to create a playlist / The SOP does not match the target accessory type).

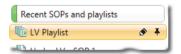




In the above example:

■ The SOP at the top of the playlist (Hydro LV - SOP 1) is used to initialize the Mastersizer system and request sample details.

- The final SOP in the list (Hydro LV SOP 3) is used to control the clean sequence. The dispersion unit, measurement and analysis settings for each SOP in the list are applied in sequence when the playlist is run.
- SOP playlists can be opened and saved using the options at the top of the SOP playlist. Playlist do not have to be saved before they are run, but a prompt will be displayed to ask if saving the playlist.
- Once the list is set up, the sequence can be run by clicking OK.
- The new playlist will be saved into the workspace window.



When the sequence is now playing, the current SOP name is reported in the window bar of the measurement manager:



Apart from the additional playlist bar, the measurement will run in a similar way to standard SOP measurements.

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Measurement settings reference



Note

SOPs and Manual Measurements involve specifying a number of settings before the measurement can be run. This section relates equally to both.

The Measurement settings window is displayed whenever you create a new SOP or manual measurement. This window groups settings into logical sections in which various parameters for the measurement are defined as follows:

- **Sample** identify the sample and provide more information to users of the SOP. Define the physical characteristics of the sample and dispersant.
- **Measurement** set both the red and blue light measurement durations, the number of, and delay duration between, measurements. Specify the obscuration levels for the measurement.
- **Sample Dispersion** control the behaviour of the currently connected accessory.
- **Data Processing** specify the scattering model, latex analysis options and modify the light detectors used in a measurement. Define any Analysis Smoothing, Size Range/Resolution options and set size banding for all measurement histograms and tables.



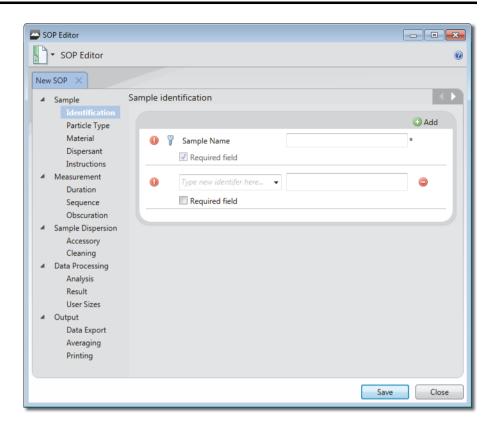
Note

When progressing through the settings, any 0 icon next to a field indicates that the user must specify a value for the field before continuing to the next section.

The rest of this section goes through each of the measurement settings in more detail.

Sample - Identification

The Sample Identification settings section allows you to enter details that will both identify the sample and provide more information to users. When the measurement is run, the user is prompted to confirm or alter the fields specified here.

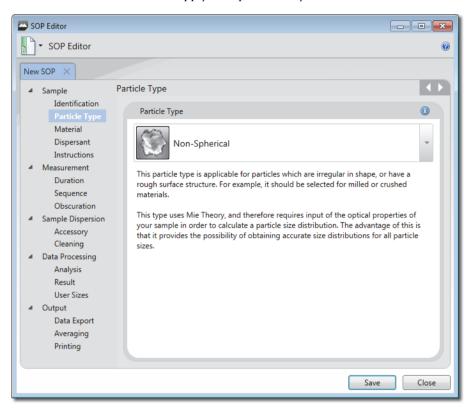


Section/option	Description
Sample Name (mandatory)	A descriptive name for the sample, such as "Batch 1A" or "Series 3, Sample 1". This is the only default sample identification field.
Adding a Field Add	To add further fields for example, Notes, Batch, Lot, Group etc., click the Add button. The new field is added to the list. Enter a name for the field in the drop down menu to the left of the field. Check Required field if this is mandatory for the operator to enter.
	To re-order the list of fields, drag the item up or down by clicking and dragging the [®] icon next to the item.
	To remove unwanted identification fields, click the
	icon adjacent to the field.

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Sample - Particle Type

The **Particle Type** settings allow you to specify the shape of particles under analysis in order for the software to apply the optimal analysis model.

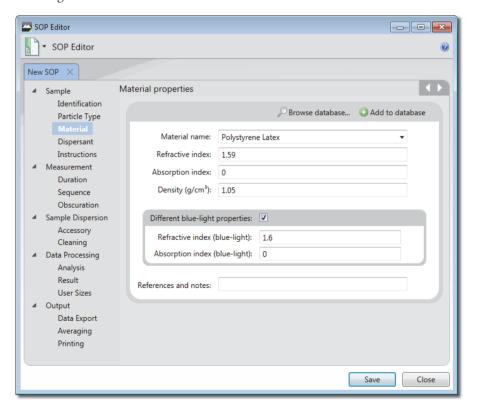


Section/Option	Description
Non-Spherical	This analysis mode is applicable for particles which are irregular in shape, or have a rough surface structure. For example, it should be selected for milled or crushed materials. This mode uses Mie Theory, and therefore requires input of the optical properties of your sample in order to calculate a particle size distribution. The advantage of this is that it provides the possibility of obtaining accurate size distributions for all particle sizes.
Spherical	This analysis mode is applicable for particles which are perfectly spherical in shape. For example, it should be selected for polymer latex samples or for emulsions.

Section/Option	Description
Spherical (continued)	This mode uses Mie Theory, and therefore requires input of the optical properties of your sample in order to calculate a particle size distribution.
	The advantage of this is that it provides the possibility of obtaining accurate size distributions for all particle sizes.
Opaque Particle (Fraunhofer Approximation)	The Fraunhofer Approximation can be used to calculate particle size distributions in cases where the particle size is large and where the particles can be assumed to be completely opaque. It is the easiest mode to use, as you do not have to provide any optical properties in order to calculate a size distribution. However, its use may lead to inaccurate results, particularly for small particles below 50 microns in size or for those which are transparent.

Sample - Material

Use the **Material properties** section to enter physical characteristics of the material being measured.



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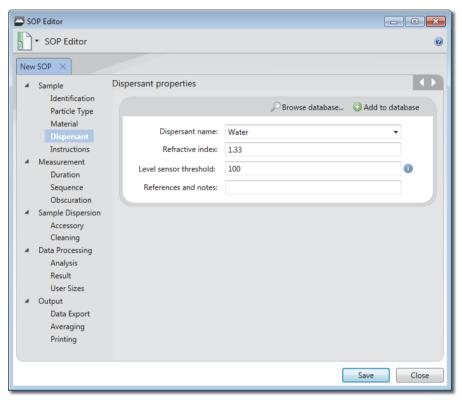
To select a previously added material from the Materials Database, click **Browse database**, then select an item from the database and click **Use these values**. See the **Help** system for more on the Materials and Dispersants databases. Alternatively, manually complete the fields detailed below.

Section/option	Description
Material name	Descriptive name for the material.
Refractive index	Value of between 0 and 5. This value relates to the speed of light within the material, which in turn allows the degree of refraction (light bending) to be predicted when light passes from one medium to another.
Absorption index	Value between 0 and 10, which is a measure of the quantity of light absorbed by the particles. Generally, transparent samples will have a low or zero absorption while coloured or black samples will have a higher value.
Density	Value of between 0.001 and 25g/cm ³ .
Different blue- light properties (Wet dispersion units only)	If required, select the check box and add the Refractive index (blue-light) and Absorption index (blue-light). See Help System for more information.
References and notes	Any further comments that help to describe the material.

Sample - Dispersant

This is a unique window for Wet dispersion SOPs.

Use the **Sample Dispersant** section settings to specify the properties of the dispersant used in the measurement.



It is possible to specify the dispersant details manually or to choose from the **Dispersants Database**. To select a previously added material from the **Dispersants Database**, click **Browse database**, then select an item from the database and click **Use these values**.

Alternatively, manually complete the sections detailed below.

Section/option	Description
Dispersant name	Descriptive name for the dispersants, for example, Ethanol.
Refractive index	Value of between 0 and 5. This value relates to the speed of light within the material, which in turn allows the degree of refraction (light bending) to be predicted when light passes from one medium to another.

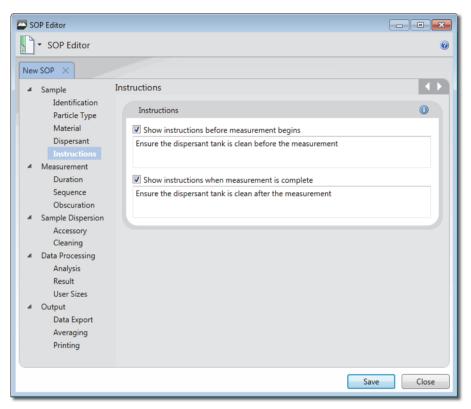
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Section/option	Description
level sensor threshold (Hydro MV only)	A value can be inputted that is suitable for the level sensor to detect the dispersant level when filled. The value will be different depending upon the dispersant used.
	The level sensor in the dispersion unit automatically stops the tank being overfilled above a certain level. If the tank fails to fill properly then the level sensor threshold, for the dispersant being used, may need adjusting. This value can be changed either manually (for the current measurement only) or by using the dispersants database, to store the new values for future use.
	Configuring this threshold value will be done using the accessory controls window; this is described in the Hydro series wet dispersion units guide.
References and notes	Enter any further comments that help to describe the dispersant.

If the newly added dispersant will be used in the future, add it to the Dispersants Database by clicking **Add to database**.

Sample - Instructions

Use the **Sample Instructions** settings to specify pre-measurement and post-measurement instructions that need to be drawn to the attention of the user.

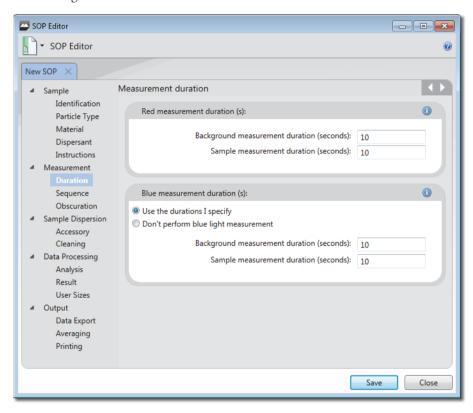


These are then shown in the Sample Documentation panel/window before and following the measurement respectively. In this example pre-measurement instructions have been specified and are displayed above the Sample Name field in the Sample Documentation window.

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Measurement - Duration

The Measurement Duration settings can be used to separately specify both the red and blue light measurement durations.



Section/option

Description

Red measurement duration(s)

Background measurement duration (seconds)

This facility will make a measurement using only clean dispersant – this is then subtracted from the sample measurement by the system in order to 'clean' the data. This is often set to the same duration as the sample measurement.

Sample measurement duration (seconds)

The optimum measurement time depends on the size of the sample, its particle size distribution and the dispersion accessory used. If a material is monomodal its essential particle size characteristics can be captured in less time than a material with a broad particle size distribution, which will need to be measured longer to ensure that the coarser particles have been properly represented.

Section/option Description

Blue measurement duration(s)

(Wet dispersion units only)

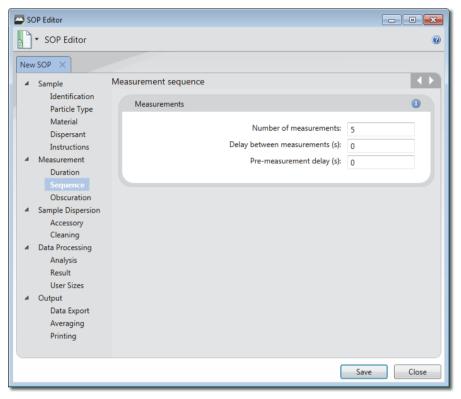
Blue light measurement is more effective when measuring particles under approximately 1µm in size. There is a gradual reduction in the effectiveness (and hence significance to the overall measurement result) of the use of blue light on samples containing particles above this size.

Some red and yellow samples (such as pigments) can absorb light in the blue spectrum. This may lead to problems with the validity of the data acquired during the blue light part of the measurement. If you have such a sample, the blue light measurement can be disabled, so limiting the measurement to the main red laser.

Either select **Use the durations I specify**, or If no blue light measurement is required, select **Don't perform blue light measurement**. This will speed up the measurement time.

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Measurement - Sequence



The Measurement Sequence settings window allows you to specify the number of measurements and the duration of any delay between measurements.

Section/option Description

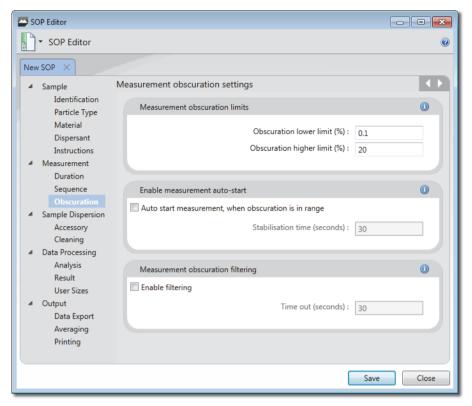
Measurements

If the material is difficult to disperse, requiring ultrasonication, surfactant or both to achieve a stable dispersion, several measurements of the same sample may be required in order to determine the ideal dispersion time. In general, repeat measurements are performed until the last measurement obtained agrees closely with the previous one.

Enter the **Number of measurements** (up to 999) and **Delay** between measurements (up to 9999 seconds). Specifying several measurements, e.g. 5, allows repeatability to be assessed.

In wet measurements a Pre-measurement delay (s) can be specified. This delay occurs before the very first measurement but after Ultrasound has been stopped, assuming the pre-measurement ultrasound option is selected.

Measurement - Obscuration



The Measurement Obscuration settings enable you to specify the obscuration levels between which the measurement will be conducted. This is critical for ensuring the correct amount of sample is added to the measurement system.

The optimal obscuration settings for a measurement are both sample and dispersion unit dependent. As a rough guide, use a range of 10-20% for a wet dispersion unit and 1-10% for a dry dispersion unit.



Note

For more advice on obscuration settings see the **Help**.

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Section/option	Description	
Measurement obscuration limits	Set the following parameters:	Laser
	Obscuration lower limit (%) - Enter a percentage value from 0.1 to 50 above which the measurement should be run.	100%
	Obscuration higher limit (%) - Enter a percentage value from 0.1 to 50 below which the measurement should be run.	Power
	This information is subsequently indicated on the Laser pane of the measurement sequence:	0% 79.52%
Enable measure- ment to auto- start	If required, select Auto start measurement tion is in range . If selected, it is also necess bilisation time delay in seconds - this will the before the measurement initiates after the objective is first detected within the specified band.	ary to set a Sta - nen add a delay
Measurement obscuration fil-tering	Select the Enable filtering option to optional ware to only record data where the obscurate the specified range.	-
	The Time out option forces the measuremer such data are recorded within the specified onds.	•

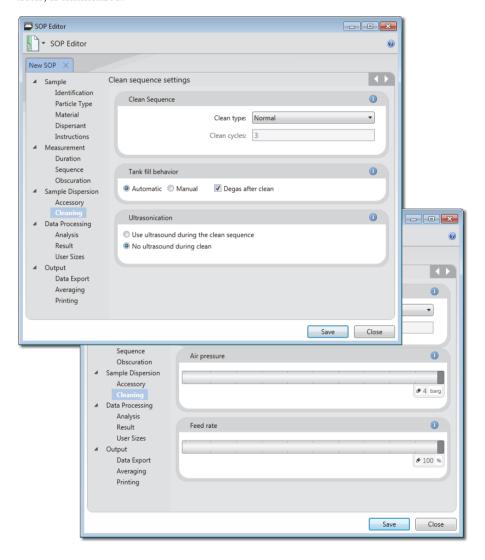
Sample Dispersion - Accessory

The Accessory control settings allows you to set up the behaviour of the currently connected accessory. The options available on this Accessory control depend on the connected accessory. These are described in the dispersions units respective user manual.

- Hydro series wet dispersion units manual for the Hydro MV, LV, EV and SM dispersion units
- Aero series dry dispersion units manual for the Aero dispersion units

Sample Dispersion - Cleaning

The **Cleaning** options allow you to specify both a clean sequence and whether to use ultrasound as part of the sequence. This ensures that all traces of the sample just measured are removed so that no contamination of the next sample occurs. Cleaning following a measurement is essential to ensure that background noise, consisting of particles agglomerating within the system and forming accumulations, is minimized.



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Section/option	Description
Clean Sequence	Each Clean type specifies a set number of cycles (nothing further is changed in the cleaning method). Select a Clean type of either Quick, Normal, Extensive or Custom. If Custom is selected the user must also manually specify the number of Clean cycles.
	If measuring the same sample type in succession a Quick clean may be sufficient.
Clean Sequence (continued)	If making the last measurement of the session, it is prudent to perform an Extensive clean to ensure that the system is as free as possible from contamination in readiness for the next measurement session.
Tank fill behaviour (Hydro LV/MV only)	The tank can be filled with dispersant manually or automatically. When set to Automatic the dispersant is plumbed into the dispersant inlet port at the rear of the accessory and a solenoid valve controls the flow of dispersant into the tank.
Only)	However, if a solvent is to be used as a dispersant, it is unlikely that the solvent will be plumbed into the accessory. In these situations the filling of the tank can be set to Manual . Under manual filling the solenoid valve is switched off (this will mean that the dispersant inlet does not have to be disconnected during a manual fill) and the tank can be filled by hand.
	The software will tell the operator when to fill the tank during an SOP measurement:
	Filling the tank manually: Fill the tank slowly until the tank light flashes - this will indicate the correct fluid level for adding sample and running measurements. If the tank is filled higher than this, sample and particles may be lost via the over-flow system (drain), leading to an incorrect particle size distribution being reported.
	Degassing
	If dispersant is stored under pressure or at low temperature, consider de-gassing before use. The pressure release or temperature rise reduces the solubility of gases, resulting in possible bubble formation in the pipes and tank.
	Select the Degas after fill check box to remove bubbles and dissolved gases from the dispersant before use. This pulses the ultrasonics for a period of time after the tank is filled.

Note: It is preferable that all dispersants are degassed before being added to the system. Degas by storing the dispersant at room temperature and pressure before use.

Section/option	Description
Ultrasonication	Ultrasound can reduce agglomeration, which may help fur-
(Wet dispersion units only)	ther with cleaning. Select whether to Use ultrasound during the clean sequence or No ultrasound during clean .
Air pressure	Use the slider bar to set the air pressure at which any
(Dry dispersion units only)	remaining sample is removed (from 0-4 bar, in 0.1 bar increments).
Feed rate	Use the slider bar to set the rate at which the remaining sam-
(Dry dispersion units only)	ple is removed - effectively this controls the vibration speed of the feed tray.

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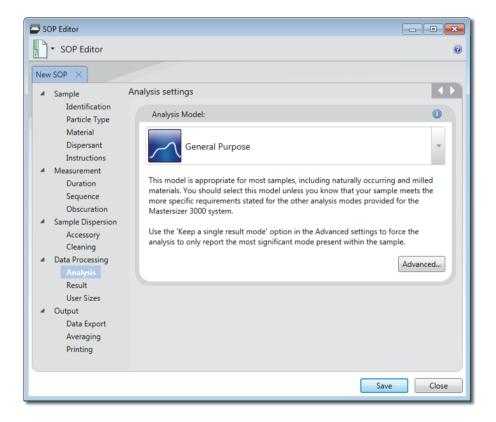
Data Processing - Analysis

The Analysis settings allow you to specify the scattering model, latex analysis options and modify the light detectors used in a measurement.



Tip

If you are unsure which analysis settings are best suited to your application, try reapplying different analysis settings to the measurement records. To do this, use the **Edit Result** feature after the first set of measurements has been made – see **Viewing the measurement** chapter for more information.



Analysis model

Section/option

Description

Analysis Model - standard

General Purpose – this model is appropriate for most samples, including naturally occurring and milled materials. You should select this model unless you know that your sample meets more specific requirements stated for the other analysis modes provided for the system.

Narrow Modes – this model is appropriate for sample consisting of one or more narrow modes, where each mode spans much less than a decade in size. It is not intended for use with broad distributions which exhibit more than one peak.

Verification Latex (Wet dispersion units only) – this model is designed to enable the analysis of one or more very narrow Latex size standards, such as those used during verification of the optical unit performance.

Note: For all of the analysis models – use the 'Keep a single result mode' option in the **Advanced** settings to force the analysis to only report the most significant mode present within the sample.

Analysis Model -Mastersizer 2000/2000E emulator mode

With the Enable Mastersizer 2000 / 2000E Analysis Emu-

lator options enabled (options menu emulated Mastersizer 2000 / 2000E analysis models are added to the list

The emulator converts a Mastersizer 3000/3000E results to how it would be analysed on a Mastersier 2000/2000E instruments, The extra analysis modes are:

General Purpose (Emulated MS2000 / MS2000E) – this model is appropriate for the majority of milled and naturally occurring samples.

Multiple modes (Emulated MS2000 / MS2000E) – this model is appropriate for sample consisting of one or more very narrow modes, such as polystyrene lattices.

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Section/option Description

Analysis Model -Mastersizer 2000/2000E emulator mode (continued)

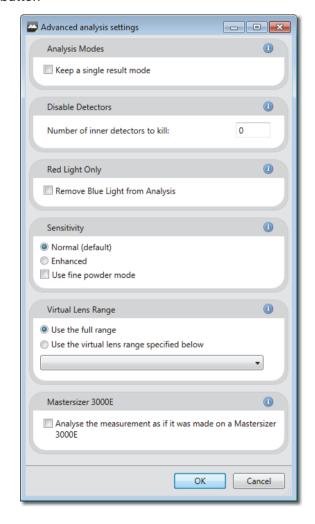
Single mode (Emulated MS2000 / MS2000E) – this model is appropriate for sample consisting of one very narrow mode, such as polystyrene lattices.

Fine Powder Mode

(Dry dispersion units only)

This option should be enabled for samples containing a significant proportion of material below 10 microns in size. It can help improve the result reproducibility, and can be used with any of the Mastersizer 3000 analysis modes.

Advanced button



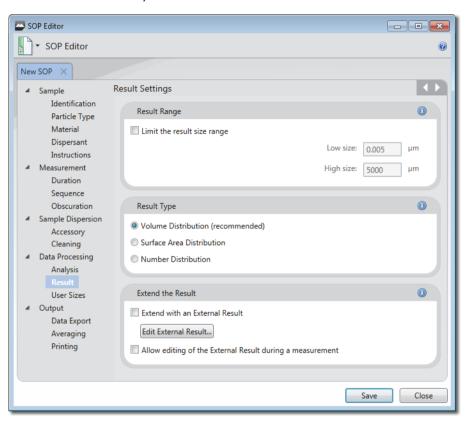
Click the **Advanced** button to reveal the following additional options:

Section/option	Description
Analysis Modes	Use Keep a single result mode if you know your sample has a single mode size distribution. This option eliminates any small modes produced by noise on the measurement.
Disable Detectors	Set the number of inner (low scatter-angle) detectors to be ignored by the analysis. Thermal fluctuations in the dispersant can cause signals in the inner channels to be interpreted as large particles. Set the number of detectors to remove the effect of these fluctuations, at the expense of reducing the sensitivity to the presence of genuinely large particles.
Red Light Only	Remove Blue Light from Analysis allows a measurement made with red and blue light to be analysed as if the blue light was not used. (wet only)
Sensitivity (Wet dispersion units only)	Enhanced sensitivity has maximum sensitivity to any small modes that are separated from the main particle mode; while Normal sensitivity (default) subtracts more of the background and reduces small extra modes.
Virtual lens range	Virtual lens ranges limit the result analysis range to that of a legacy instrument, such as a 300mm lens used on a Mastersizer X, and are used to help with method transfer from older Malvern laser diffraction systems:
	Selecting each lens range will truncate both the detector range and the size range of the Mastersizer 3000 system to match the selected system.
	Select this feature for use from the options menu
Mastersizer 3000E	This option will force a Mastersizer 3000 measurement to be analysed as if it was made on a Mastersizer 3000E.
	Selecting the Mastersizer 3000E analysis mode will override some of the other advanced settings. For example, the number of virtual lens ranges available will be reduced.

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Data processing - Result

The results settings allow you to specify the displayed size range and the type of distribution that the analysis has used in the measurement.



Section/option

Description

Result range

Reducing the measurement range is not usually a recommend option, especially when conform to good laboratory practice. This facility is useful though if a sample includes two or more distinct size populations which may need to be monitored individually.

Adjusting the **low** and **high size** range will alter the fit applied to the fundamental result, where the intermediate values are then read from the curve allowing interpolation of percentile points. This adjustment will not alter the analysis bands that are used in the measurement.

Result range (continued)

Note: The software will automatically flag the use of any range reduction facility to preserve the integrity and traceability of the results.

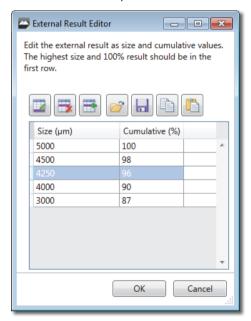
Result type

Set the result type for the particle size distribution - choices are **Volume distribution** (recommended), **Surface area distribution** or **Number distribution**. By default the Mastersizer measurement is fundamentally a measurement of the volume distribution - transforming the result into number distribution is a mathematical process that may amplify any error in the original result. Small volumes of small size particles may be transformed in to significant numbers of particles. Use the size range controls above to limit this effect.

Extend the result

Result extension can be used to extend the Mastersizer result to sizes beyond 3500µm.

To enable a result extension, first click the **Extend with an External Result** option. Then, click the **Edit External Result**... button to open the External result editor window.



To enter the size band data to add to the Mastersizer result; use the control icons at the top of this Editor window to add or remove size bands, as required.

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Extend the result (continued)

To enter the size band data to add to the Mastersizer result; use the control icons at the top of this Editor window to add or remove size bands, as required.

The data entered will be dependent upon the technique you are using to extend the result. Values for the size of each band and the cumulative percentage below each size band can then be entered by clicking within the table and adding the value. The only requirement is that 100% of the volume of material must be present below the largest size specified in the table.

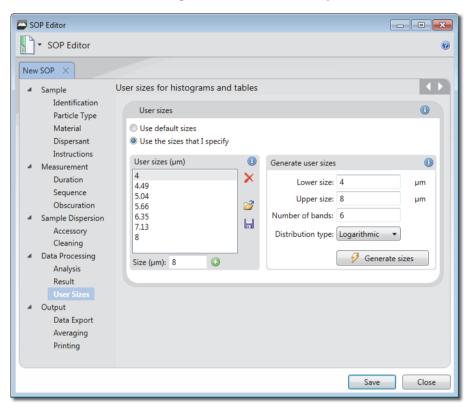
Clicking Ok will store the extended result data as part of the SOP settings.

Alternatively the **Allow editing of the external result during a measurement** option will allow the external result to be modified for each measurement of an SOP. Each time the SOP is run, users will be able to enter new undersize data bands

Note: When the extended size band set overlaps the Mastersizer result, it is assumed that the extended result will be correct. Therefore the Mastersizer result will be truncated and renomalized to fit with the new extended result data set.

Data Processing - User Sizes

The **Data Processing: User Sizes** settings allow size banding for the measurement to be defined for all histograms and tables used in reports.



Section/option Description

User sizes

A practical use of size bands is when using sieves that conform to specific standard sizes - size bands could be set to corresponding ranges.

The system can either automatically set size bands for histograms and charts, or these can be user-specified.

Use default sizes - Select this check box to automatically select appropriate high and low sizes based on the measurement result.

Use the sizes that I specify - Select this check box to manually define size bandings. The User sizes (μm) and Generate user sizes panels become active.

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User sizes (continued)

Creating size bands

- From the Generate user sizes panel, enter the required Lower size and Upper size limits (in μm).
- 2. Then enter the **Number of bands** into which the table or histogram should be divided.
- 3. Select a Distribution type:

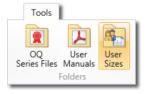
Linear: Divides the bands into equal sizes.

Logarithmic: Bands are represented by multiples of a constant factor that accounts for the size range specified.

- Click Generate sizes to populate the set of figures in the User sizes panel.
- 5. To add more size bands, enter a figure into the Size (μm) field and then click the button. The new figure is added to the list in the correct numerical position.
- To delete one of the size bands, select the figure you wish to remove and then click X.
- To save size band information that you wish to re-use within other SOPs, click to save the current sizes. To use previously saved sizes, click and then locate the relevant .siz file.

Note: refer to description below for reporting standard sieve sizes.

Retrieving User sizes. Any size file can be retrieved using the User sizes button on the Tools ribbon (Folders group).



Reporting standard sieve sizes

Result tables can be set up to display results using **standard sieve MESH** sizes. This is configured using the **User Sizes** option.

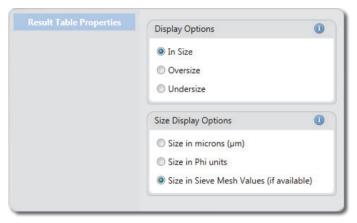
To display the MESH sizes, users must first load in the required sieve size data - select the **Use the sizes that I specify** option described above and then load in the file containing the data for the sieve set required.

Sieve sizes are available for all standard ASTM, BS, ISO and Tyler sieve sets.

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- The ISO sieve size sets includes a list of the micron sizes for all of the available sieves; these sieves are in the same format as the standard Mastersizer size class files.
- The ASTM, BS and Tyler sieve size class files include a MESH reference for each size class. In this case the list includes the standard microns size, along with the MESH size in brackets.

To report the MESH sizes in the report it is necessary to configure the result table in a report to use the MESH sizes stored in the User Sizes file rather than microns sizes. This is done by creating a new result table in a report, and then changing the properties of the table to report the **Size in Sieve Mesh Values**.



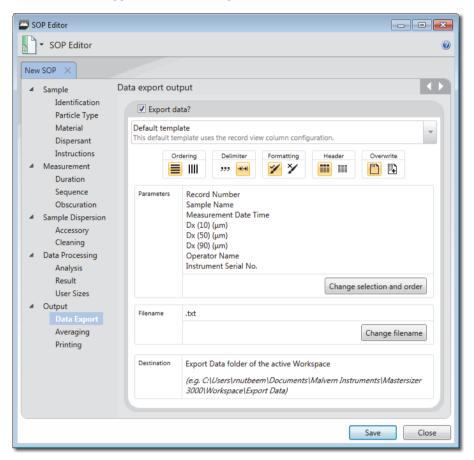
This will cause the result table to show MESH sizes.

Refer to the **reporting** chapter for details on creating and saving reports.

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Output - Data Export

The **Output - Data Export** window allows specific date to be selected for analysis in other software applications, for example Microsoft Excel.



Alter the parameters as required.

Section/option	Description
Export data?	To enable exporting select the Export data? check box.
Custom/Default	Select either the Default template or create a Custom one.
template	Note The Default template will reflect the current workspace settings and use the record view column configuration.

Export settings

Choose either rows or columns from the **Ordering** selector to present the data horizontally or vertically respectively.

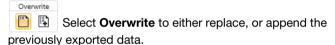


From the **Delimiter** selection, choose either

Comma separated (csv) or Tab separated. Your choice will depend on the requirements of the target application.

Use the Formatting option to select either Format values as displayed in software, which displays numeric information to a limited number of decimal places (this may be preferable for presentation purposes); or choose Unformatted values to show all data as recorded by the system.

Select **Header on/off** to specify whether the export should contain the names of the fields as a header row (or column).



Parameters

Click the **Change selection and order** button to show the **Parameter selection window**, then select the required parameters as follows:



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Parameters (continued)

Locate parameters by manually expanding the tree structure in the **Available** list, or search for them by typing their first few characters into the **Search...** field.

- - -

The search feature is only available with the Mastersizer 3000 / Mastersizer 3000E Extended software option.

- - -

Select a parameter in the Available list and then click the

Add button to transfer it to the Selected list. Similarly, select an item from the Selected list and click the Remove

button to transfer it to the **Available** list. It is also possible to select multiple items using the standard Windows selection methods (i.e. SHIFT and click, CTRL and click).

When all of the required parameters are shown in the **Selected** list, choose an order of presentation so that they are shown in the desired left-to-right order. Select a parameter and then click the un/down buttons to make its

ter and then click the up/down 📦 buttons to move its position in the list. Items at the top of the list are shown on the left, items at the bottom are shown on the right.

Click **Apply** to apply the settings without closing the window, or click **OK** to apply the settings and also close the **Parameter Selection** window.

Filename

Name the exported as required. Select the Change filename button and select the parameters, if any, that are to be added to the filename.

The filename will be saved as a .txt or .csv file as determined by the delimiter selection.

Destination

The exported file will be saved in the Export Data folder of the current workspace.

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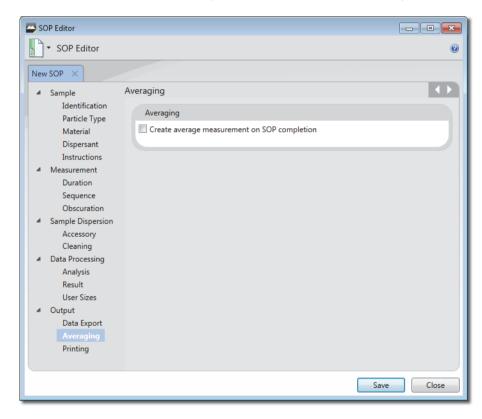
Output - Averaging

An SOP can be set to run through a number of measurements, with an average result then being created at the end; as set by the **Output - Averaging** window.

Section/option Description

Averaging

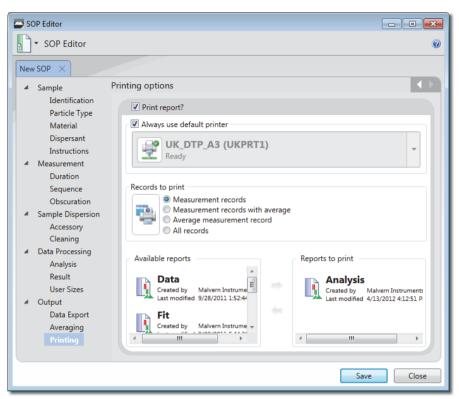
After the measurements have finished it may be advantageous to see the average value produced from all the measurements run. Selecting the **Create average measurement on SOP completion** check box will enable this option.



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Output - Printing

The Output - Printing window allows specific reports to be selected for printing



Section/option	Description	
Print report?	To enable printing select the Print report? check box	
Always use default printer	Select the Always use default printer check box if a certain printer is to be used, otherwise select from the list.	
Records to print	Select the radio button as required for selecting the records.	
Available reports	Select the reports required for printing.	
	This window contains a list of Available Reports on the left	
	and Selected Reports on the right. Reports shown with are reports that were created by Malvern Instruments.	
	Move reports from the Available Reports column to the Selected Reports column (and back as required) by select-	
	ing them and then clicking the Add 🍑 button or Remove	
	button as appropriate	

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Viewing measurement results

Introduction

The Mastersizer delivers powerful features for manipulating and presenting the data gathered in measurement files.

This chapter provides the following information:

- An overview on working with measurement files.
- Features of the Analysis report.
- Fundamental concepts to help understand measurement data.

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Working with measurement files

To make a measurement it is necessary first to open, or create, a measurement file. Measurement files can be thought of as containers for the individual records created by each measurement.

A measurement file can contain any number of individual records. Often these will have been made at a similar time, but equally they could have been generated on different dates. Similarly, a measurement file could contain records that are either the result of measurements from one or more dispersion unit types.

Multiple measurement files can be opened at the same time, each of which is displayed in its own tab (see the **Software overview** chapter). When working with multiple measurement files, it can also be useful to compare data by merging all of the files into a single list; the **All files** tab offers this feature. Additionally, if required, individual measurement records can be copied between files or copied into an empty measurement file.

Opening existing measurement files

This section provides information on how to open and close existing Mastersizer 3000 (.mmes files) and Mastersizer 2000 (.mea files).

Files from the Mastersizer S/X/Micro/Microplus instruments can also be opened (.sam files). These first have to be saved to an Mastersizer 3000 measurement file format. However, no reanalysis is possible.



Note

Although Mastersizer 2000 files can be opened for analysis, they cannot be edited directly. They are shown with a differently coloured file icon and also a lock, which indicates their read-only status (like the Examples file shown here):



Mastersizer 2000 files can only be edited if they are **first** saved in Mastersizer 3000 format. This can be done by selecting **Save-As.**, or by copying and pasting the Mastersizer 2000 results into a Mastersizer 3000 result file.

■ To open a Mastersizer 3000 measurement file: from the Home ribbon's **Documents** section, choose **Open-Measurement File**. Mastersizer 2000 measurement files can be opened by selecting **Open-Mastersizer 2000 Measurement File**.

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- Scroll the open measurement file tabs left and right using the buttons.
- Use the button for quick selection of any open measurement file.
- To close a measurement file: click \times in the right corner of the tab of the measurement file you wish to close.



diT

To view more information about an open measurement file, move the mouse pointer over its tab - information about the file type, access mode and location are provided.

Creating and saving measurement files

- Select **New-Measurement file** from the **Home** ribbon.
- From the **Home** ribbon choose **Save As** and then enter a new file name.

Alternatively, choose **Save** from the **Home** ribbon if the file has already been saved and you wish to just overwrite the last saved instance.

If multiple measurement files are simultaneously open, select **Save All** to save the changes of all modified files.

Filtering the Record View

The parameter filters feature is only available with the Mastersizer 3000 / Mastersizer 3000E with Extended software option.

This section provides information on how to set up parameter filters within the Record View.

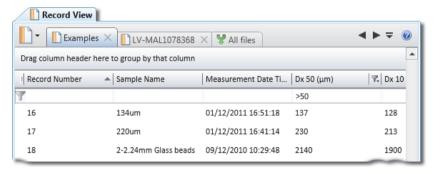
To simplify the analysis of large numbers of records (perhaps when using the **All files** feature or a large measurement file), it is possible to set up parameter filters. These are used to hide or make visible entire groups of records as required. It is possible to filter on any parameter, e.g. operator name, measurement date, sample name or a value range of a particular measurement parameter, like "Dv50 > $50\mu m$ ".

1. From the **Record view**, move the mouse pointer over one of the column headings until a down arrow is displayed next to the title of the column, for example:

Scattering Model

- 2. Click the down arrow to display a list of available parameter values upon which to filter (these are the values shown in the record).
- 3. Select any of the values to show only those records with values matching the checked filters. When a filter has been set, the column header shows the following icon: 8. As many filters as required can be set up in this way.
- 4. It is also possible to extend the functionality of this grid by manually typing text into the sorting header area filtering criteria can include numerical information, date ranges and strings.
- **5.** Select **Filter Record view** from the **Result** group of the Home ribbon.
- 6. Click on the column header and then enter text the display updates immediately to show only those items that are within the scope of the filter see Parameter filtering logic in the Help system.

In the example shown here a filter of >50 has been applied (meaning, show only those items whose $Dv50(\mu m)$ value is greater than 50).



The filters can be removed by:

- Deselecting the Filter Record view from the Result group of the Home ribbon
- Right-click on the icon and either de-select the individual check box from the filter menu or choose the Clear filter option.
- or simply delete the text.



Note

Check box filters: having <u>all</u> filters either selected or deselected has the same effect, namely that all of the records are shown.

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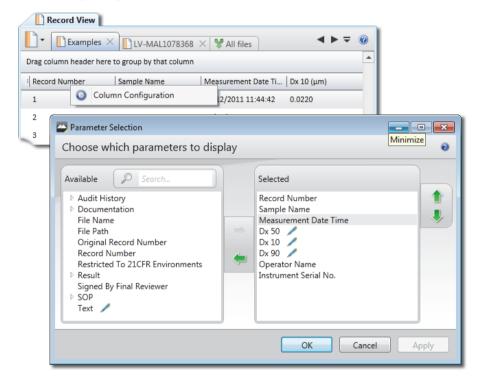
Column configuration

The column configuration feature is only available with the Mastersizer 3000 / Mastersizer 3000E with Extended software option.

The columns can be changed as required to indicate various parameters.

Right click the mouse in the header field of the record view to display the **Column configuration** button, then select the button to display the **Parameter selection** window.

Alter the required parameters as follows:



- Locate parameters by manually expanding the tree structure in the **Available** list, or search for them by typing their first few characters into the **Search...** field.
- Select a parameter in the **Available** list and then click the **Add** button to transfer it to the **Selected** list. Similarly, select an item from the **Selected** list and click the **Remove** button to transfer it to the **Available** list. It is also possible to select multiple items using the standard Windows selection methods (i.e. SHIFT and click, CTRL and click).

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- When all of the required parameters are shown in the **Selected** list, choose an order of presentation so that they are shown in the desired left-to-right order. Select a parameter and then click the up/down buttons to move its position in the list. Items at the top of the list are shown on the left, items at the bottom are shown on the right.
- Click **Apply** to apply the settings without closing the window, or click **OK** to apply the settings and also close the **Parameter Selection** window.

Grouping records

The grouping records feature is only available with the Mastersizer 3000 / Mastersizer 3000E with Extended software option.

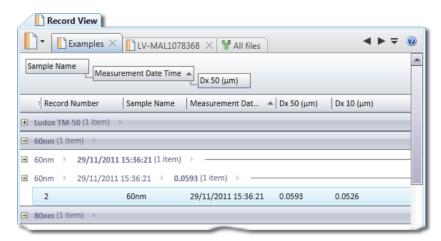
Read this section for details on how to group records by parameter type.

The **Record view** allows records with the same parameter values to be grouped into collapsible sections. Any parameter can be selected on which to group. It is then possible to add further sub groups. Examples of the use of groups include, grouping by Operator Name, Measurement Date, Accessory Type etc.

- 1. To group items by a parameter, drag the required column header into the area above the columns, titled **Drag a column header here...** The records are now presented in collapsible groups based on groupings of the chosen parameter of the same value. For example if Scattering Model was used as the grouping parameter, items would be grouped either in the Fraunhofer, Fraunhofer Extended or Mie groups.
- 2. To collapse the groups, click next to the group section's title. To expand a group that has been collapsed, click next to the title.
- **3.** Add further groupings in the same way these are then indicated in the area above the records in the order in which the grouping was applied. The parameter that is leftmost is applied first.

The image below shows several groupings applied; the up arrow in **Measurement Date Time** indicates that sorting is applied on that field:

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The grouping hierarchy can be edited by dragging and dropping the group parameter headers left or right as appropriate.

4. To remove groupings, drag the group parameter header back into the records area.

Merging records

This section provides information on how to use the **All files** feature of the **Record View**.

When working with multiple Mastersizer measurement files, it can be useful to compare data by merging all of the files into a single (virtual) measurement file so that Mastersizer's powerful sorting, grouping and reporting features can be exploited.

- 1. Open more than one measurement file.
- **2**. Click the **All files** tab (rightmost):



The records contained in **all** of the open measurement files are shown combined into a single file. It is now possible to sort, group, filter and report on the records displayed in this view. If graphically analysing multiple records in reports, each record's data is overlaid using a different colour.

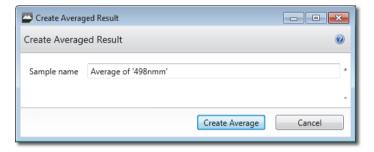
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Creating an averaged record

The **Create Averaged Record** feature allows you to create a new record that contains the average values of all selected records.

1. From the **Record View**, select the records from which to create an average and choose **Create average** from the Home ribbon. Alternatively, right-click a selection of records and choose **Create Averaged Record**.

The **Create Averaged Result** window is displayed:





Note

This feature is not available from the **All files** view.

- 2. Enter a **Sample name** and a **Reason** for creating the new result.
- Then click the Create Average button.
 A new record is created within the current measurement file. To retain the newly created record, save the measurement file.

Editing results

This section provides help on the **Edit Result** feature of the Records view.

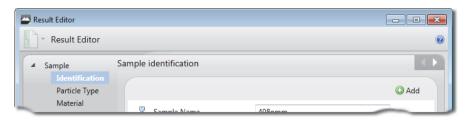
Each Mastersizer record is the result of the processing of raw measurement data. By editing a record it is possible to re-apply new values to some of the parameters used in the original measurement settings in order to generate a completely new record.

This can be used to explore the effect of using different settings from those chosen when the measurement was made. Typically adjustments are made to improve the 'fit' of the calculated result to the data. Use the **SOP summary report** (see the **Advanced features chapter**) to show which parameters have been altered.

 From the Record View, right click on a record, or group of records, and choose Edit Result.

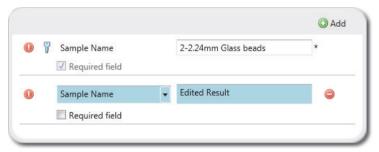
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2. The **Result Editor** window is displayed.



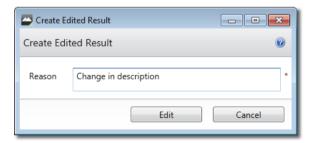
This contains all of the information set up in the original measurement. Some of the options are unavailable because they relate only to the physical set up of the original measurement, for example, all of the **Measurement Duration** options.

3. Edit any available sections of the **Result Editor** as required.



Any edited information is clearly highlighted by a different background colour (in the example shown here the lower field has been edited).

4. Click **OK** when all settings have been altered to your specification, then input a reason for the change in the Edited result text window. Select **Edit** to finish and the newly-set values will be applied to the existing raw measurement data.



5. When the result has been edited, the edited result is then displayed as the last item in the record view (this is dependent upon the view configuration).



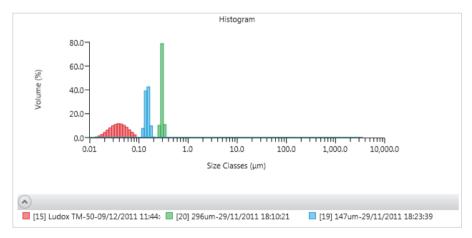
Note

If you save the measurement file, the new settings will overwrite the original file settings. To create a result-edited version of a file and still retain the original file settings, just save the file with a different name before editing.

Selecting records to display in a report

Mastersizer reports display data from all measurement records that are currently selected in the **Record view**. It is possible to select either single or multiple records for report analysis. Records can also be sourced from different measurement files and still compared in the same report – see **Merging records** for more information.

- 1. To view a report of the data contained in a single record, click on it within the list of records. The display is updated immediately to reflect the selection (assuming that you are using a view that shows a report and records simultaneously see the **Software overview** chapter).
- 2. To select multiple records to display in a report, use the standard Windows selection methods; either hold SHIFT and click to select multiple contiguous records or hold CTRL and click to select non-contiguous records. When more than one record is selected, and a graph is displayed in the currently shown report, the graph indicates the data taken from individual records by using a different colour for each. A key to the colours used is presented beneath the graph.

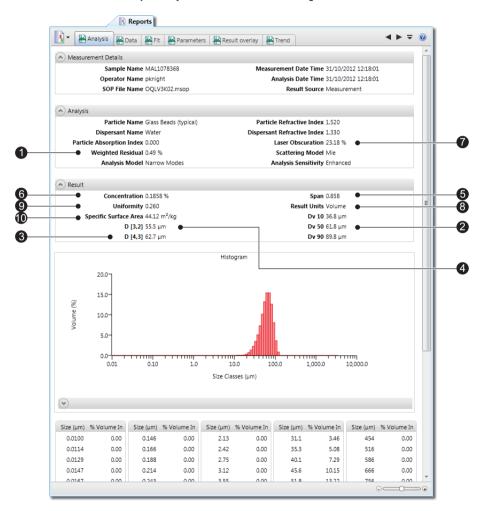


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Analysis report

The Analysis report provides a useful breakdown of the measurement data.

■ To display this report, select the record(s) to analyse in the **Record View** and then click the **Analysis** report from within the **Reports** tab.



The following information is displayed in the report:

① **Weighted Residual** – an indication of how well the calculated data was fitted to the measurement data. A good fit is indicated by a residual of under 1%. A

- residual of over 1% may indicate use of incorrect refractive index and absorption values for the sample and dispersant.
- The statistics of the distribution are calculated from the results using the derived diameters D[m,n] an internationally agreed method of defining the mean and other moments of particle size. See British standard BS2955:1993 for more details.

$$D[m, n] = \left[\frac{\sum V_i d_i^{m-3}}{\sum V_i d_i^{n-3}}\right]^{\frac{1}{m-n}}$$

Dv 50, Dv 10 and Dv 90 are standard percentile readings from the analysis.

- $\mathbf{Dv} \mathbf{50}$ the size in microns at which 50% of the sample is smaller and 50% is larger. This value is also known as the Mass Median Diameter (MMD) or the median of the volume distribution. The v in the expression $\mathbf{Dv} \mathbf{50}$ shows that this refers to the volume distribution. This can be replaced by \mathbf{s} for surface, $\mathbf{1}$ for length or \mathbf{n} for number distributions.
- **Dv 10** the size of particle below which 10% of the sample lies.
- **Dv 90** the size of particle below which 90% of the sample lies.
- ③ **D[4,3]** the **Volume Weighted Mean** or Mass Moment Mean Diameter, also known as the De Broucker mean.
- D[3,2] the Surface Weighted Mean, also known as the Surface Area
 Moment Mean Diameter or Sauter mean.
- ⑤ **Span** is the measurement of the width of the distribution. The narrower the distribution, the smaller the span becomes. The **span** is calculated as:

$$Span = \frac{d(x, 0.9) - d(x, 0.1)}{d(x, 0.5)}$$

The \mathbf{x} is replaced by any of the letters \mathbf{v} , \mathbf{s} , \mathbf{l} or \mathbf{n} that define the distribution type.

- © Concentration the volume concentration. This is calculated using Beer-Lambert's law. For full details see the following section.
- Obscuration this helps the user to set the concentration of the sample when it is added to the dispersant. It is a measure of the amount of laser light lost due to the introduction of the sample into the analyser beam.

The obscuration term can be expressed mathematically:

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$$Ob = 1 - \frac{L_s}{L_b}$$

 L_s is the light intensity measured in the central detector when a sample is present in the cell, L_b is the same but with clean dispersant (i.e. with no sample). Obscuration is usually expressed as a percentage: $100 \times Ob$.

An ideal range is between 3 and 20%, depending on the sample and dispersion unit used.

- ® Distribution shows the type of distribution the analysis has used. Options include change from volume to surface area, length or number. Remember that the Mastersizer measurement is fundamentally a measurement of the volume distribution transforming the result into a surface, length or number distribution is a mathematical process that may amplify any error in the original result, especially at the fine end of the size distribution.
- 9 **Uniformity** a measure of the absolute deviation from the median.

$$Uniformity = \frac{\sum X_i |d(x, 0.5) - d_i|}{d(x, 0.5) \sum X_i}$$

Here d(x,0.5) is the median size of the distribution (where x is replaced by v, s, l or n) and d_i and x_i are respectively the mean diameter o f, and result in, size class i.

Specific Surface Area (SSA) – the total area of the particles divided by the total weight.

$$SSA = \frac{6\sum \frac{V_i}{d_i}}{p\sum V_i} = \frac{6}{pD[3, 2]}$$

where V_i is the relative volume in class i with mean class diameter of d_i and p is the particle density.

If the SSA is used, it is important that the density of the material is defined (in the SOP's **Material** settings). This figure is a mathematical calculation based on the assumption that the particles are both spherical and non-porous.

Fundamental concepts

To understand the meaning of the results from the Mastersizer, a number of fundamental concepts require explanation. These are:

- How the concentration is calculated.
- The results are volume-based.
- The result is expressed in terms of equivalent spheres.
- How the distribution parameters are derived.

Calculating the Concentration (Cv)

Beer-Lambert law

The software uses the Beer-Lambert law to calculate the concentration of the sample. This may be expressed as:

$$\frac{I}{I_0} = e^{-\alpha b}$$

Where:

- I is the intensity of light at a distance b in the particle field of absorbance α .
- \blacksquare I_0 is the intensity of the light beam as it enters the particle field.
- I/I_0 is the relative transmission T of the beam (measured directly by the instrument). I_0 is the intensity of the laser beam measured at the receiver when no sample is present and I is the intensity with sample in the beam.

Expressing the Beer-Lambert law in terms of relative transmission and re-arranging to solve for absorbance gives:

$$\alpha = -\frac{1}{h} Ln \langle T \rangle \tag{1}$$

Volume concentration

The term α contains information about the concentration and size of the particles. From scattering theory the light attenuated by a particle *i* may be described by:

$$a_i = Q_i \pi r_i^2 n_i$$

Where:

• Q_i is the efficiency of light extinction (by scattering and absorption), calculated from Mie theory for a particle of radius r_i .

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■ The second term is the cross-sectional area of the particle and the final term n_i is the number of particles of radius r_i .

In terms of the volume of particles $V_i = \frac{4}{3}\pi r_i^3 n_i$, the equation above becomes, for an ensemble of particles:

$$\alpha_i = \frac{3}{4} \sum \frac{Q_i V_i}{r_i}$$

The size of the particles is expressed by the diameter d and the volume terms can be separated into a relative volume distribution v and a total concentration Cv (total volume of particles in a unit volume of dispersant). The equation then becomes:

$$\alpha_i = \frac{3}{2}C_v \sum \frac{Q_i V_i}{d_i}$$

Substituting the above into equation (1) and solving for the concentration gives:

$$C_{v} = \frac{-2Ln(T)}{3b\sum \frac{Q_{i}v_{i}}{d_{i}}}$$

If *d* is measured in μ m and *b* in mm, and ν is the relative concentration of the size distribution (such that $\sum v_i = 1$) then:

$$C_{v}(ppm) = \frac{-2000Ln(T)}{3b\sum \frac{Q_{i}v_{i}}{d_{i}}}$$

This equation provides the concentration in parts per million (ppm). To calculate the value as a percentage volume concentration, the final value is divided by 10,000.

In the above equation:

- The Transmission (*T*) is a value between 0 and 1 and is measured directly by the instrument.
- The particle size distribution v_i is the relative volume in size-class i with mean diameter d_i .
- $extbf{Q}_i$ the mean extinction term for size-class i is calculated from scattering theory and is a function of the optical properties of the particle and dispersant media.

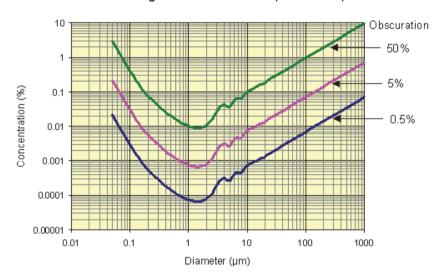
Concentration at different obscurations

This advice gives an approximate idea of the obscuration ranges to use during a measurement.

It is possible to convert the obscuration limits into an equivalent volume concentration but there is a strong size dependence that makes it difficult to use the data at the time of measurement. The relative volume concentrations of monomodal (single sized) particles that would give a certain obscuration is illustrated below. The actual curves differ according to the presentation being used. It will be clear that there is a size dependence; an obscuration of 30% means a widely varying volume concentration dependent on size.

Concentration at different obscurations

Calculated for glass beads in water (n=1.54 +i0) in water



In the more realistic situation of a polydisperse (or multimodal) sample it is not possible to provide simple graphical correlations of volume concentrations and obscuration. The system calculates concentration, however, using the size distribution result and the obscuration.

For a polydisperse sample the curve of the above figure can be used if the **Surface Weighted Mean (D[3, 2])** of the material is taken as single size.

Volume-based results

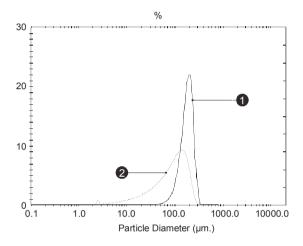
The first, and probably most important, point to remember in interpreting results is that the fundamental size distribution derived by this technique is volume-based. This means that when a result indicates, for example, that 11% of the distribution is

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in the size category 6.97-7.75 microns, this means that the **volume** of all particles with diameters in this range represents 11% of the total **volume** of all particles.

It is useful to consider a numerical example to illustrate this point. Suppose, for simplicity, that a sample consists of only two sizes of particle, 50% by number having a diameter of 1 micron and 50% by number a diameter of 10 microns. Assuming that the particles are spherical, the volume of each of the larger particles is 1000 times the volume of one of the smaller ones. Thus, as a volume distribution, the larger particles represent 99.9% of the total volume.

The graph below illustrates this for a more realistic distribution:



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Equivalent spheres

Mie theory presumes that the particles measured are perfect spheres. In practice they are very rarely so. This causes a problem in the definition of the term "measure the particle's size": if the particle is an irregular shape, which particular dimension should be measured?

As an example, imagine that I give somebody a matchbox and a ruler and ask them to tell me the size of it. They may reply by saying that the matchbox is 50mm x 25mm x 10mm. They cannot say that "the matchbox is 25mm" as this is only one aspect of its size. It is not possible to describe the three dimensional matchbox with one unique dimension. Obviously the situation is even more complex for irregular shaped particles such as grains of sand or the pigment particles in paint.

Most people want a single measurement to describe their sample, for example, they wish to say that their sample is made up of 50 micron particles. What is required is a unique number that describes the particle. There is only one shape that can be described by one unique number and that is a sphere. If we say we have a sphere of

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50 microns, this describes it exactly. We cannot do the same even for a cube as 50 microns can refer to its edge or to a diagonal.

One way to get a single unique number to describe an irregular shaped particle is to compare some feature of the actual particle to an imaginary spherical particle.

Some typical methods of doing this are:

- **Equivalent surface area** calculating the diameter of a theoretical sphere that has the same surface area of the original particle.
- **Equivalent maximum length** this is where the diameter of a theoretical sphere is the same as the maximum dimension of the original particle.
- **Equivalent minimum length** this is where the diameter of a theoretical sphere is the same as the minimum dimension of the original particle.

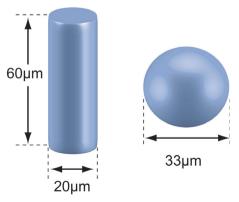
There are many other methods available to do this. This technique is known as "equivalent spheres".

The Mastersizer uses the **volume** of the particle to measure its size. In the example above, the matchbox has a volume of $50 \times 25 \times 10 \text{mm} = 12500 \text{mm}^3$. If the Mastersizer was able to measure this size of "particle" it would take this volume and calculate the diameter of an imaginary sphere that is equivalent in volume - in this case it will be a sphere of about 30mm diameter.

Obviously the answer will be different if the surface area or maximum dimension of the matchbox is used to calculate an equivalent sphere. All of these answers are correct but each is measuring a different aspect of the matchbox. We can therefore only seriously compare measurements that have been measured using the same technique.

Consider a cylindrical particle of diameter 20 microns and length 60 microns. The volume of the cylinder is:

$$V = \pi (10\mu m)^2 (60\mu m)$$



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The sphere of equivalent volume would have a diameter centred on:

$$\sqrt[3]{\frac{6V}{\pi}} = 33\mu m$$

with a spread from 20 to $60\mu m$.

It is interesting to compare this with other techniques. Sieving would pass the particles through a $20\mu m$ aperture and classify them as $20\mu m$. Sedimentation would give a result related to the total surface area, in this case reporting a diameter of around $40\mu m$.

Derived distribution parameters

The particle size distribution reported by the software is expressed in a set of size classes which are optimized to match the detector geometry and optical configuration, giving the best resolution. All parameters are derived from this fundamental distribution.

Distribution parameters and derived diameters are calculated from the fundamental distribution using the summation of the contributions from each size band. In performing this calculation the representative diameter for each band is taken to be the geometric mean of the size band limits:

$$\sqrt{d_{i-l}}di$$

This number will be slightly different to the arithmetic mean:

$$\frac{d_{i-l} + d_i}{2}$$

For example, the size band 404.21 - 492.47 microns has a geometric mean of 446.16 microns and an arithmetic mean of 448.34 microns. In most cases the difference is small but the geometric mean is chosen in these calculations as more appropriate to the logarithmic spacing of the fundamental size classes.

The same principle of calculation applies to the distribution statistics standard deviation, skewness and kurtosis, as shown below:

standard deviation

$$\sigma = \sqrt{\frac{\sum X_i (d_i - \bar{d})^2}{\sum X_i}} = \sqrt{D[K + 2, K]^2 + D[K + 1, K]^2}$$

skewness

$$Skew = \frac{\sum X_i (d_i - \bar{d})^3}{\sigma^3 \sum X_i} = \frac{(D[K+3, K]^3 - \bar{d}(\bar{d}^2 + 3\sigma^2))}{\sigma^3}$$

kurtosis

$$Kurt = \frac{\sum X_i (d_i - \overline{d})^4}{\sigma^4 \sum X_i} - 3 = \frac{(D[K + 4, K]^4 - \overline{d}(4D[K + 3, K]^3 + 3\overline{d}^3 + 6\overline{d}\sigma^2))}{\sigma^4} - 3$$

In the above formulae *K* and *X* depend on the distribution being calculated as shown in this table:

Distribution	K	X_i
Volume	3	V_i
Surface	2	V_i/d_i
Length	1	V_i/d_i^2
Number	0	V_i/d_i^3

The various derived diameters are related by:

$$D[m, n]^{m-n} = \frac{D[m, 0]^m}{D[n, 0]^n}$$

For "mono-size" distributions such as latex, the distribution mean is reported as the geometric mean of the size class and **standard deviation**, **skewness** and **kurtosis** are reported as zero.

The procedure used for other parameters of the distribution is to create a spline fit to the fundamental result. Intermediate values are then read from this curve allowing interpolation of percentile points which do not coincide with the measurement size band boundaries.

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Optical models

This section contrasts the Fraunhofer approximation used in some instruments with the Mie theory which underpins Mastersizer operation. Although Mie is the preferred scattering model, users can also select Fraunhofer if required from the **Data Processing - Analysis** measurement settings.

Fraunhofer approximation

Older instruments and some existing instruments rely on the Fraunhofer approximation only. This assumes that:

- The particle is much larger than the wavelength of light employed. ISO 13320 defines this as being greater than 40x wavelength ($25\mu m$ when a He-Ne laser is used).
- All sizes of particle scatter with equal efficiencies.
- The particle is opaque and transmits no light.

These assumptions are incorrect for many materials and for small particles they can give rise to errors approaching 30%, especially when the relative refractive index of the material and medium is close to unity, or when the particles are transparent. When the particle size approaches the wavelength of light the scattering efficiency becomes a complex function with maxima and minima present.

Mie theory

The Mastersizer uses the full Mie theory which completely solves the equations for interaction of light with matter. This allows completely accurate results over a large size range.

Requirements for using Mie

The Mie theory assumes the particle is spherical, as opposed to Fraunhofer, which is a projected area prediction. The penalty for this complete accuracy is that the refractive indices for the material and medium must be known and the absorption part of the refractive index must be known or estimated. However, for the majority of users this will present no problems as these values either will be known or can be measured.

A standard set of materials is available for selection in the SOP (presented in the **Sample - Material** section of the measurement settings). Further materials can be defined by a user within the materials database, but the following parameters must be specified accurately:

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- **Refractive Index (Real)** this value relates to the speed of light within the material, which in turn allows the degree of refraction (light bending) to be predicted when light passes from one medium to another.
- **Absorption Index** an imaginary number that describes the amount of absorption that takes place as the light enters the particle.
- **Density** the density in g/cm³. This is used to calculate the **Specific Surface Area** (**SSA**). Users who want to include the SSA as a derived parameter must complete this field.

The **residual** shown in the **Parameters** report indicates how well the calculated data fitted the measurement data.

Refractive index of medium

This value is needed by the software in order to provide accurate calculations and is specified within Mie theory.

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Sample preparation guidance

Introduction

Preparation of the sample before it is added to the system can be critical. Over half the problems encountered when measuring a sample are caused by poor sample preparation. If the sample is sticking together, dissolving or floating on the surface, or if it is not a representative sample, the result will be incorrect.

There are many techniques available to ensure that the sample is prepared successfully. Once a suitable dispersion technique is found for the sample, standardise the procedure (using an SOP) so that comparisons can be made between samples.

This chapter gives information on:

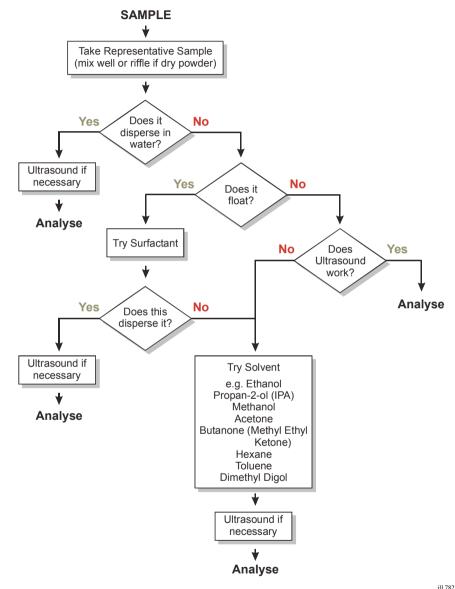
- Sample preparation flow chart summarises the process.
- Representative sampling.
- Considerations for dry samples.
- Considerations for wet samples.
- Symptoms of poor sample preparation.

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6

Sample preparation flow chart

This flow diagram shows the route taken to prepare an unknown sample:



Using liquid-borne particle suspensions, such as latex and emulsions, is usually straight forward. Use the suspension phase as the dispersant, mix the sample well and add directly to the Mastersizer beaker. Ultrasound is not usually necessary and will almost certainly affect the size of liquid particles.

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Representative sampling

When taking a sample for a measurement, it is vital to ensure that the sample used is representative of the whole sample. This section gives guidelines.

If the sample is taken from a bottle or container, take care to ensure that it is thoroughly mixed. When the sample is a powder, large particles tend to rise to the top of the container as smaller ones work their way to the bottom.

In most samples there are some large and some small particles, but the majority fall between these extremes. If a sample is taken from the top of the container, mainly large particles will be measured. If this is compared to a measurement with the sample taken from the centre of the container the results will differ noticeably.

If the sample is stored in a container this should be mixed thoroughly before measurement. Do not shake the container as this often increases the separation of the particles. Instead, hold the container in both hands and gently roll the container, continually changing its orientation for about 20 seconds. This method works better if the container is only half full.

Spinning rifflers

If the distribution of particles within a sample is particularly broad, representative sampling can be difficult. If problems continue, using a **spinning riffler** may help. This is a vibrating hopper which vibrates the sample down a chute.

The act of vibrating the sample causes the larger particles to separate out and travel down the chute first. At the end of the chute a set of rotating pans collects the sample evenly. When all of the sample has passed down the chute each collecting pan will contain a representative sample.

Liquid samples

Liquid samples can also separate out if stored in containers, with larger particles sinking to the bottom. Again, it's necessary to mix the sample thoroughly to get a representative sample. Sample splitters/rifflers are also available for liquid samples. Beware of using a magnetic stirrer to mix a liquid sample. Large particles tend to move to the outside of the container due to centrifugal separation, and this can lead to sample biasing.

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Considerations for dry samples

The first step when analysing a sample for the first time is to decide whether to analyse the sample in a wet or dry state. This is usually determined by the nature of the end use of the sample. If the product is to be used and stored in a dry form, a dry analysis may be preferred.

Some samples can only be measured in a dry state as they react with all wet dispersants, for example they may dissolve or the particles may swell when in contact with a liquid.

Another consideration is whether the material in its dry state is free flowing. Good pouring characteristics indicate a non-cohesive powder which will usually disperse well in a dry powder feeder without any difficulties, whereas a highly cohesive material tends to stick and clump together, giving biased measurements.

Sample clumping together can often be overcome by drying the sample in an oven. Obviously care should be taken with delicate materials where drying in the oven may damage the sample. To drive the moisture from the sample, set the oven to the highest temperature possible, but not above its melting point!

If it is obvious that using an oven is going to damage the sample, use a desiccator.

A fresh sample that has not had time to absorb moisture from the atmosphere is always preferable and usually gives better results. If hygroscopic samples need to be conveyed to the system over some distances, they should be sealed into tubes as soon as possible with a silica gel bag if this is practicable.



Note

Application Notes describing how to develop a method for dry sample analysis are available at **www.malvern.com**.

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Considerations for wet samples

This section covers sample preparation considerations for wet samples. When analysing a sample in a wet medium there are even more choices to be made than for dry samples.

Choice and preparation of the dispersant

The majority of samples will allow water to be used as the dispersant. If the particles are soluble in water or react chemically an alternative must be used. For example, flour added to water will float on the surface but it disperses well when added to propan-2-ol (IPA).

The first choice to consider when measuring a wet sample is the choice of suspension medium (dispersant). When analysing a sample for the first time, it is always best to check the dispersion beforehand. Add the selected dispersant (for an initial measurement it is usual to use water) to a little of the sample in a beaker and visually note the result. The sample may dissolve; this can usually be seen visually. If unsure, analyse the sample and observe the obscuration figure. If the obscuration figure decreases the sample is probably dissolving.

The dispersant may itself contain impurities or particles that could be significant. We recommend filtering the dispersant before use either with an in-line pipe filter or, for small quantities, a syringe based disposable type. Filtration to 1 micron is generally adequate with 0.22 microns being commonly available and an ideal size.

If the dispersant is stored under pressure or at low temperature it may be necessary to consider degassing before use. A pressure release or a temperature rise will reduce the solubility of gasses, resulting in the formation of bubbles in pipes and cells, etc. Bubbles are a problem as they are measured with the sample and are counted as particles, biasing the results. This is particularly a problem with some mains water supplies. The simple answer is to store sufficient dispersant at room temperature and pressure for several hours before use to allow the degassing to occur. Also see the section on bubbles later in this chapter.

Note that the use of cold dispersant in a warmer environment can also give rise to condensation on the outside surfaces of the cell windows. For systems plumbed into the mains supplies a small header tank may overcome this problem. Filter this water prior to use. Another solution is to warm the dispersant (for water typically to 60-80 °C) and then allow it to cool before use.



Warning!

Do not warm a dispersant to allow re-gassing if the dispersant is volatile. Never allow dispersants to reach their boiling points.

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If condensation is a problem in a **water-based** system, a quick remedy is the addition of warm water to the sample tank. The increase in obscuration caused by the condensation will quickly disappear.

When analysing particles suspended in a liquid dispersant, one of the most important decisions is which liquid to use. The dispersant can be any clear (at 633nm wavelength), optically uniform liquid that does not interact with the sample causing it to change its size. Clearly users wish to use the safest, lowest cost liquids that are effective. Particles that give problems, such as dissolution, in one medium may be quite suitable in another.



Warning!

If a volatile solvent has to be used as dispersant, leave the sample area door open during use to aid ventilation.

Whenever difficulties in dispersion are experienced, consider using another dispersant. This list gives commonly used dispersants with their refractive indices:

Dispersant	Refractive index	
Water	1.33	
Ethanol	1.36	
Propan-2-ol (Isopropyl-alcohol)	1.39	
Dimethyl Digol	1.41	
Butanone	1.38	
Hexane	1.38	
Acetone	1.36	

The cost of some of the organic dispersants may limit their use to the Hydro MV, which typically only uses 120ml of dispersant. The problem of the safe disposal of the sample after measurement must also be considered. Always adopt the correct procedures for disposing of the sample and dispersant, following any local guidelines. Most local regulations forbid hazardous samples and dispersants to be tipped down the drain, allowing them to enter the water system.



Note

Application Notes describing how to develop a method for wet sample analysis are available at www.malvern.com.

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Surfactants and admixtures

If experiencing problems such as the sample floating on the surface of the dispersant, addition of a surfactant or admixture may help.

Surfactants

Adding a surfactant may help in preparation of the sample it will lower the surface tension, which promotes wetting of the particles.

Surfactants have to be added in minute quantities, typically one drop per litre of dispersant. If too much surfactant is added the action of stirring and pumping the sample may cause it to froth, causing bubbles to be introduced into the system. Bubbles are seen by the system as particles which can bias the measurement. Antifoaming agents may be added to prevent the formation of bubbles but, owing to the fact that these may contain particulates, they should be added to the dispersant **before** the background is measured.

Try adding a drop of surfactant to a quantity of sample and dispersant mixed in a small beaker. If the sample sinks to the bottom of the beaker in large clumps, dispose of the sample and start again. This time add the sample to a dry beaker, add a drop of surfactant and mix thoroughly to a thick paste. Add the dispersant and mix well. This usually avoids the agglomeration caused by adding the dispersant first.

A list of recommended surfactants in order of common use is given below:

Surfactant	Nature	
Igepal	Non-ionic	
Teepol L	Non-ionic	
Synperonic N	Non-ionic	
Aerosol OT	Anionic (solid)	
Sodium dodecyl sulphate	Anionic	
Hyamine 2389	Cationic	

Admixtures

Admixtures work by causing the particle surface to become charged, causing particles to repel each other (i.e. providing a zeta-potential to aid dispersion). Admixtures are added in larger quantities, typically 1g/l. A list of commonly used admixtures is given below:

- Sodium Hexametaphosphate (e.g. Calgon)
- Sodium Pyrophosphate
- Trisodium Phosphate
- Ammonia

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- Sodium Oxalate
- Calcium Chloride

As many of these are solid materials that are dissolved into the dispersant, the solution should be filtered after preparation to remove impurities.

Slurries

The act of mixing up a small quantity of concentrated sample, dispersant and additives before it is added to the dispersion unit tank is known as preparing a slurry.

Once the particles have been successfully dispersed into a slurry, the sample may be added to the dispersion unit without any further additions of surfactants etc. The problem of the sample settling out within the beaker can be solved by using a pipette to continually stir the sample. While stirring, continually fill and empty the pipette. Use the pipette to add the sample to the dispersion unit tank.

Use of ultrasonics

In addition to the processes described above, ultrasonics can be applied to help the dispersion, whether or not it contains a surfactant.

When mixing the sample in the suspension medium, a visual inspection often shows whether ultrasonics are necessary. If large agglomerates of particles sink to the bottom of the beaker, try applying two minutes of ultrasonics by placing the slurry and its beaker in an ultrasonic bath. It will be immediately apparent if this has been effective. Further ultrasonics can be applied when the sample is added to the tank, if necessary. This often prevents re-agglomeration, but is not always needed.



Note

Be wary of using ultrasonics with fragile particles; the ultrasonic action may break up the particles themselves. If in any doubt, microscopic observation before and after the ultrasonics can establish whether it has helped.

Samples with unstable concentrations

When adding the sample to the dispersion unit tank using the software's **Add sample** facility, users may experience obscurations that change during the dispersion period. Most samples disperse very quickly so users will not notice the obscuration value rising. With some samples the slow dispersion of the sample can be clearly seen. Do not make a measurement until the obscuration has stabilised, indicating that the sample has properly dispersed. When adding very fine material and relying on the action of the ultrasonics to achieve a good dispersion, add less sample than you would expect. The action of the ultrasonics will cause the obscuration to rise.

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The obscuration and its behaviour during the dispersion of the sample can also warn of other potential problems.

If the obscuration decreases the size of the particles within the sample may be increasing; either the sample is sticking together or the particles are actually swelling due to the dispersant. Other causes could be the larger particles settling out due to inadequate pumping and stirring or even the particles dissolving.

If obscuration increases rapidly, particles may be attaching themselves to the cell windows due to surface charges. This means material is in the laser beam continuously and the obscuration appears to increase. To solve this, use an admixture.

Bubbles

The Mastersizer sees bubbles as particles and measures them. Bubbles vary in size but are typically around 100 microns in size. In many cases these bubbles can be seen clearly as a second and separate peak when the measurement data is analysed. Always be wary of bubbles in the system.

When the dispersant has been added to the dispersion unit and circulated, we advise stopping the unit briefly (using the **Stop all operations** button in the **Active Accessory control**) to allow trapped air to escape the system. Also, during installation, ensure there are no twists or loops in the connected sample tubing.

When adjusting the pump and stirrer speed for a particular sample, ensure the speed is not so fast that it introduces air into the system.

When a surfactant is added to the sample, excessive speed of the stirrer/pump may cause frothing. This will force bubbles into the system.

Degassing

If dispersant is stored under pressure or at low temperature, consider de-gassing before use. The pressure release or temperature rise reduces the solubility of gases, resulting in bubble formation in the pipes and tank etc. This is a problem with some mains water supplies. The best answer is to store enough dispersant at room temperature and pressure for several hours before use to allow de-gassing to occur. For more information see **Choice and preparation of the dispersant** above.

Pulses of ultrasonics can help to degas the dispersant after the tank has been cleaned and filled with fresh dispersant. To do this, use the **Active accessory control** panel (**Tools-Accessories**) and then select **Enable pulsed sonication**.



Note

Ensure that all dispersants are degassed before being added to the system.

Symptoms of poor sample preparation

Use this table to identify sample dispersion problems:

Problem	Symptom	Action
Sample dissolving	Obscuration decreases.	Try another dispersant.
Dispersant contains impurities	Poor background read-ings.	Filter the dispersant before use.
Bubbles within the dispersant	Bubbles typically show as a secondary peak at 100 microns.	Degas the system.
Sample floating on the surface of the dispersant	Sample seen on the surface of the dispersant in the tank.	Add surfactant or admixture.
Sample clumps together	Obscuration decreases.	Add surfactant or admixture, or use ultrasonic action.
Sample sinks to the bottom	Obscuration decreases as the larger particles settle out.	Increase the pump/stir- rer speed.
Sample swells in dispersant	Obscuration decreases.	Try another dispersant.
Particles sticks to the windows	Rapidly increasing obscuration.	Use an admixture or surfactant.
Condensation on the cell windows	Rapidly increasing obscuration.	If (and only if) the dispersant is water, add a quantity of hot (not boiling) water to the tank. If the obscuration falls, condensation is the problem.
Bubbles sticking to windows	Difficulty in obtaining low background despite many rinses.	Drain the sampling tank. The bubbles will burst. Fill the system carefully with degassed dispersant. In a new system, adding Decon 90 to the tank overnight and rinsing 7 or 8 times will wet out the cell surface and reduce bubble formation.

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Reporting

Introduction

Reports help to make sense of all the data that is generated in a Mastersizer measurement. They enable you to graphically analyse measurement data and also isolate areas that are of interest using tables and graphs.

This chapter provides the following information:

- About reports.
- Viewing and using reports.
- Creating and editing reports.

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Chapter 7 Reporting

About reports

Mastersizer's reports consist of information sections that are displayed together in a single report for easy on-screen analysis or printing. Reports can be configured so that only those items that are of interest to the user are displayed. It is also possible to alter the order in which the report elements are displayed.

Additionally data in the reports can be directly copied and exported to other Windows applications.



Note

It is only possible to edit user-defined reports, not those supplied with the system. It is possible however to copy the system reports and create new reports that are based on them.

Malvern reports

In addition to the ability for users to build reports tailored to their own requirements, Malvern Instruments supplies a number of standard reports with the software. These are:

Analysis – a report that provides a mixture of key parameter sections and a particle size distribution graph. See **Viewing the measurement results** chapter for more information on this report.

Data – this report mainly focuses on the raw light scattering data gathered by the instrument during the measurement. It includes a light scattering graph.

Parameters – a summary of the key parameters measured by the system.

Fit – shows the scattered light data together with a curve calculated from the result distribution. Differences between these two curves come from deviations in the calculated result from the actual result and may be due to measurement errors or poor choice of optical properties and calculation model.

Additionally, **Result overlay** and **Trend** reports are also available.



Note

Although the Malvern Reports cannot be modified directly, they can be opened for editing and saved under a different **Report name** to create a new report.

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Viewing and using reports

This section describes how to view and manipulate reports about the records contained within measurement files. It is assumed that a measurement file is open and that it contains several records.

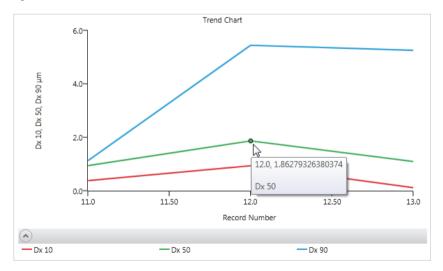
Displaying reports

- 1. Select a record or number of records from the **Record View**.
- 2. If it is not already shown in the current view, select the **Reports** tab. One of the reports for the current workspace is then shown (by default this is the Analysis report).

It can be easier to work in **2-pane Vertical Split** view whilst initially selecting the records. Any selections or de-selections of records are then updated immediately in the visible report.

Graphs

Three types of graph are used within the reports: **result**, **trend** and **data** graphs. Whenever a graph is displayed, click on the plot lines to display data points. Move the mouse pointer over the data lines to see numerical information relating to that data point.



Collapsing/expanding report sections

To collapse the individual sections within a report, click the up arrow icon to the left of the report section title. To expand an already collapsed report section, click the down arrow icon to the left of the report section title.

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Chapter 7 Reporting

Scrolling layout

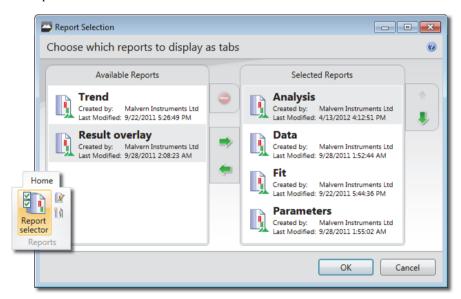
Reports are presented in sections that are stacked vertically. To reveal all of the information on the report requires the use of the scroll bar, or **collapse** individual sections as required.

Zooming in and out of reports

To zoom in and out use the **Zoom slider** on the bottom right of the report pane. In **Paging Layout** view, the number of pages in the report is adjusted accordingly.

Selecting reports to display in a workspace

The set of reports that are displayed are associated and saved with the workspace that is currently selected. See **Workspace** in the **Software overview** chapter for more information. This section gives information on how to choose the set of reports that are shown in the current workspace and how to arrange the order of the report tabs shown.



- **1.** From the **Workspace** pane, select the workspace for which you wish to select reports.
- From the Reports section of the Home ribbon, choose Report Selector to display the Report Selection window.
- 3. This window contains a list of **Available Reports** on the left and **Selected Reports** on the right. Reports shown with are reports that were created by Malvern Instruments.

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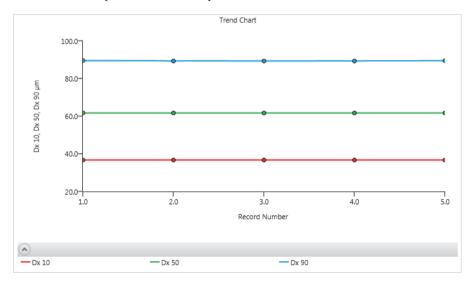
4. Move reports from the **Available Reports** column to the **Selected Reports** column by selecting them and then clicking the **Add** button. Similarly, move **Selected Reports** back to the **Available Reports** list by selecting them and then clicking the **Remove** button.

- 5. To move a report up or down within the Selected list, select it and then click the up or down arrow icons as required. The order of this list corresponds to the left/right position of the Reports tabs, the leftmost tab being the top report in the Selected Reports list.
- **6.** Click **OK** when you have organised the reports as required.

Trend Graphs (in reports)

Similar to the trend plot shown in Manual Measurements, the reporting trend chart shows a graphical plot for each record from a measurement that is currently selected in the **Record View**.

By default trend graphs plot Dv10, Dv50, Dv90 data against record number. Each parameter is given a different colour. Click on the graph line to reveal data points - move the mouse pointer over those points to reveal numeric values.



Editing a Trend Graph

To alter the axis scaling limits:

1. Move the mouse pointer over the X or Y axis to display the scale adjustment feature.

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2. Click **MANUAL** and then enter the required lower and upper limits (click anywhere outside the input box to confirm the figure).

3. To return the graph to the automatically-adjusted setting, click **AUTO**.

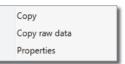


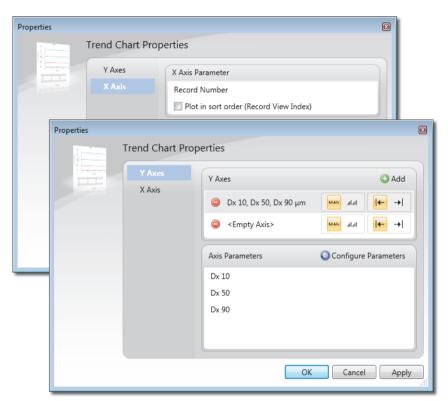
Tip

This feature is also available in the live **Trend View**, **Light Scattering** and **Size Distribution** panels in the measurement window.

To alter the information shown on the graph:

- 1. Right click on the graph and select properties from the pop up.
- This will open the Trend Chart properties window.





3. Select either the **X** or **Y Axis** button on the left. The data currently plotted is shown in the top panel of the window.

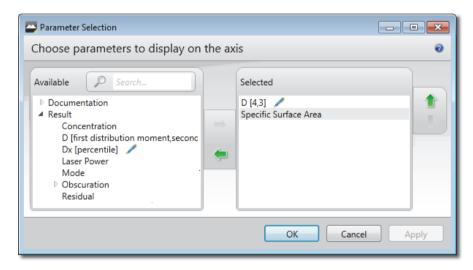
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4. To **remove** the currently plotted data, click on next to the parameter description.

To **add** or select a new data item against which to plot, click the Add button. A new **Empty Axis** element is added into the top panel.

- Select the new **Empty Axis>** element to display its properties in the lower panel.
- **6.** Click the Configure Parameters button to add a parameter from the **Parameter Selection** window.



7. Select the required parameters and press Ok to confirm selection.



Note

Parameters with numeric values (such as percentiles) are editable and indicated with \checkmark . To edit the parameter numeric value, click the parameter and amend the value. Any value entered that is inappropriate for the parameter is flagged with an exclamation mark icon - amend the value accordingly if this is the case. It is possible to enter multiple variants of the same parameter with different values associated (for example Dv10, Dv50 and Dv90). These are then indicated on the graph with different colours.

- 8. Select the **Position** in which the axis should be labelled

 left or right.
- 9. Finally, choose a **Scale Type of Linear** or **Logarithmic**.

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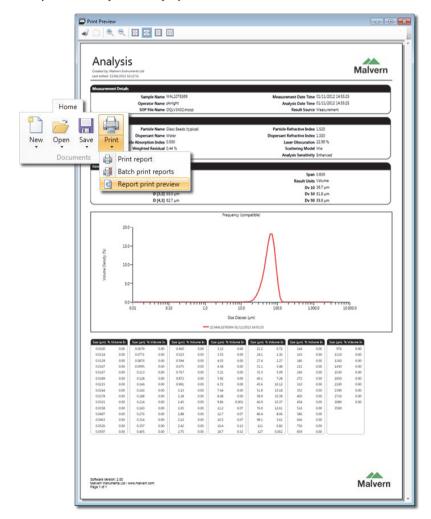
Chapter 7 Reporting

10. Click **OK** when you have completed the settings.

Printing reports

Before sending a report to the printer, it can be helpful to view a preview initially. The **print preview** option shows how a report will appear when printed on paper, enabling the effect of alterations to a report to be easily established without wasting paper.

1. Choose **Print-Report Print Preview** from the ribbon. A preview of the currently selected report is displayed:



The following display options are available:

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■ **Zoom** – to zoom in and out of the print preview, click the [®] icons respectively. When zoomed in it may be necessary to use the scroll bars in order to see all of the elements on the report.

- 100% shows the report at 100% zoom.
- **Page width** stretches the page to view the width of the page within the current window.
- **Whole page** zooms out to so that an entire report page can be viewed in the window.
- **Two pages** zooms out so that two pages of the report can be seen side by side.



Note

Use of these options does not have any impact on the final report printout.

2. To print the report, click the **Print** icon $extit{ extit{def}}$.

Copying data from reports

Report elements and data can be copied directly form the reports and quickly exported data to other Windows applications.

There are two ways to do this, right click on the report element (e.g. Analysis, Result, etc) and either select **Copy** or **Copy raw data**.



This will enable the following data to be copied to the clipboard, which can then be pasted into the secondary application:

- **Size distribution graphs**: copies the complete size distribution data set, including data for all results which are over-plotted on the graph.
- **Result tables**: copies all of the size distribution data from the table.
- **Parameter grids**: copies all values for the parameters listed in the selected grid, including measurement details, analysis settings and results.

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■ **Data and fit graphs**: copies a data set reporting the measured signal per detector.

■ **Trend tables**: copies all of the table data.

The copied report element data will either be exported as text (**Copy raw data**),

Result

Concentration 0.0034%

Uniformity 0.076

Specific Surface Area 118.0 m²/kg

D [3,2] 50.9 μm

D [4,3] 51.3 μm

Span 0.213

Result Units Volume

Dv (10) $46.1 \,\mu\text{m}$

Dv (50) $51.0 \,\mu\text{m}$

Dv (90) $57.0 \,\mu\text{m}$

or as a simple screen copy of the report element (Copy).





Note

An alternative way to export data to other Windows applications is to use the Exporting data functions. This is described in the **Advanced features** chapter.

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Creating and editing reports

In addition to the standard Malvern reports shipped with the system, the Mastersizer provides a quick and simple way to define and modify reports to a user's specification. This section gives information on how to define and modify reports.

To create a new report (or edit an existing report)

To create a **new** report select **New** from the **Reports** group on the **Home** ribbon.



To **edit** an existing report, first select the report from the **Report** tab and then choose **Edit** from the **Reports** group of the ribbon. The example below shows that a Data report has been selected for editing.



The report is now displayed in **edit** mode. To alter a graph From the **Report Editor** controls, click the properties button next to the graph you wish to alter.



Note

Actual data from the selected measurements is still shown whilst in edit mode. This greatly simplifies the process of visualising the final output.

2. Enter an appropriate name for the report in the top field, **New Report** is shown by default.

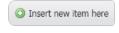


The **report name** is used to identify the report in the Reports tabs and can be modified later if required.

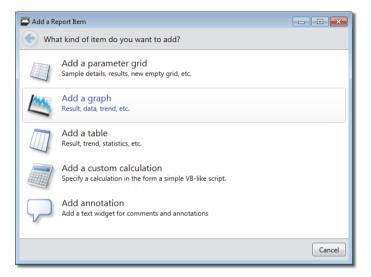
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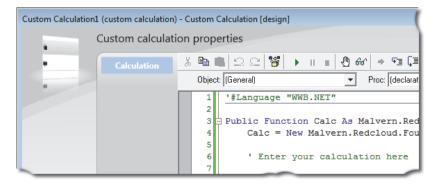
3. To add a new item to a report, click the **Insert new item here** button. If **editing** an existing report, this button is placed between each section of the report.



4. The **Add a Report item** window is displayed, from which the following choices are available:



- Add a parameter grid a parameter grid contains just parameter information, it is also possible to then select the individual parameters that are required.
- Add a graph choose from either the Result, Trend or Data graph type.
- Add a table choose from either the Result, Trend, Data or Fit table.
- Add a Custom calculation choose a Text calculation. Once the Calculation section is added to the report template, select the edit icon and construct the calculations using Custom calculation properties editor.



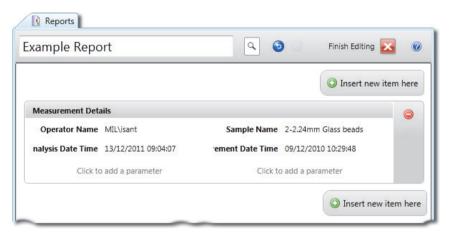
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Reporting Chapter 7

■ Add annotation - use this option to input basic text and comments about the report, measurement or other associated information. Once the annotation area is added to the report template, select the edit icon and add comments as required.

Choose one of these options and then select a more specific kind of report element from the window that follows.

The new section is now added to the report in the main report editing window. In this example, a parameter grid has been added:



- **6.** To undo the last change made to the report, click the **Undo** button . If required, continue to click **Undo** until all edits in that session have been undone. To re-do a change that has been undone, click the **Redo** button .
- 7. Click anywhere in the report section's heading area to activate the cursor, then edit the heading for the report section as required.
- **8.** To move items up or down in the report, click the arrow buttons on the right of the item.





Tip

To move items around a large report it is often easier to zoom out initially (so that more of the report is visible) and then drag the report section to the required area of the page. The mouse pointer must not be positioned over any of the buttons when clicking and dragging.

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9. With all report elements positioned, alter the font style and color as required refer to **Changing the font style for report parameters** below.

- **10.** When you have finished editing the report, click the **Finish Editing** button at the top of the window.
- 11. The software asks whether you would like to save (changes to) the report click **Save**, **Don't Save** or **Cancel** as required.



Tip

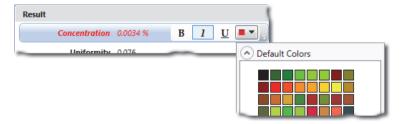
To display a thumbnail preview of the complete report move the mouse pointer over the \(\alpha \) icon.

Changing the font style for report parameters

It is possible to change the font style and color used for parameters included in report parameter grids and highlight them to give key parameters more emphasis in the final report. To do this, place the report into **edit** mode and hover the mouse pointer over the parameter to be configured. This will cause a **font edit** icon **t** to appear as part of the parameter selection area.



Clicking on this icon will open a menu where the color and font style for the parameter can be changed.



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System security

About security

By default the Mastersizer application software uses Windows user security. That is, only a user logged into the computer that is physically connected to the instrument can access the system. For many environments this level of security is sufficient.

For quality controlled environments however, the optional **Malvern Access Configurator** (or MAC) adds a fully-featured security system to the Mastersizer application software. The MAC not only provides control over an individual user's access to the various features of the software, but also logs an audit trail of significant system events, who performed them and when.

A full description of the functionality and features of the **Malvern Access Configurator** (or MAC) is provided in the MAC Help file.

Activating security



Note

This process should only be undertaken **after** setting up security privileges for the Mastersizer in the MAC.

 Choose Options from the Application button menu to display the Mastersizer 3000 - Options window and then choose Access Control.



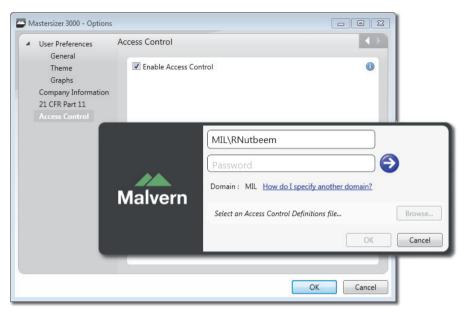
2. Select **Enable Access Control** to activate the security features

This option is only available once a security configuration file has been created using the optional Malvern Access Configurator (MAC).

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Once selected, the user is prompted to enter a user name and password:



The user who logs in must be a user with both administrative rights on the computer and also one who has been set up with Disable/Enable Security permission in the MAC.

Click the Validate credentials button



- Select **Browse** and then locate the MAC security definitions file. This file will be found in the location identified with the MAC application software, during its creation and export.
- Click **OK** to finalise the process.

Following this, only users with valid permissions that have been set up in the MAC will be able to access the functions of the system.

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Software licensing

Mastersizer 3000E basic software users are restricted to use of the software only on the computer that is attached to the Mastersizer 3000E. Licences cannot be shared.

If multi-workstation use is required, upgrading to the extended software is required.

Without a valid licence, the Mastersizer application software will not function. The software automatically generates a licence on the computer that is connected to the instrument. After first installation of the software you will be prompted to accept this license.



 Click Accept licence to accept the license file. If the software was set up by your administrator or Malvern Instruments, you may not see this window this means that the software is already activated.

Sharing a licence file

You can share a Mastersizer application software licence so that other computer can use the software without having an instrument connected. This could be useful if you wanted to undertake analyses on measurement files whilst away from instrument.



Note

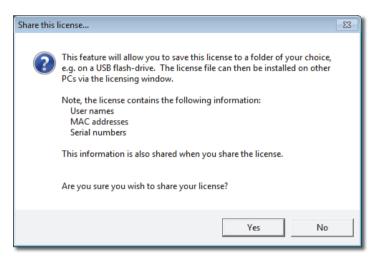
The licence file contains the identification of the user and the instrument from which it was generated. Do not share the licence outside of your authorised user group.

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1. From the **Application button** menu , choose **About**



- **2.** A Malvern Mastersizer application software info page will appear. Click the **View licence** button to display details of the active licence.
- **3.** To export the licence, click **Share this licence** the software provides details about what is included in the licence.



- 4. Click Yes and then specify a file name and location in which to save the licence. Then just send a copy of this .licence file to the Mastersizer application software user without a licence.
 On the first attempt to run the software, the user is prompted with the an install licence message.
- **5.** Click **Install licence** and then browse to the location of the .**license** file.
- **6.** The credentials of the licence file are then displayed **-** click **Install licence**.

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System security Chapter 8

21 CFR part 11

The 21 CFR Part 11 functionality is only available to users of the Mastersizer 3000 system.

With the 21 CFR part 11 feature key installed these features are available:

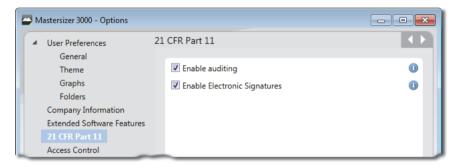
- Enable Auditing
- Enable Electronic Signatures

These can then be enabled from the **Application button** menu **______**, and then

choosing **Options**



and 21 CFR Part 11.



Audit history



Note

Auditing is only available when 21 CFR Part 11 mode is enabled. This requires a special feature key, which can be purchased from Malvern Instruments.

After you have purchased a feature key from Malvern Instruments:

- 1. From the **Application button** menu, select **Options**.
- 2. Choose 21 CFR Part 11. and then enter the key you have been sent.
- 3. Click **OK** (the OK button is only available if you have entered a valid key).
- 4. Ensure that **Enable auditing** is selected.

Once enabled, the audit feature logs every system event and measurement record amendment.

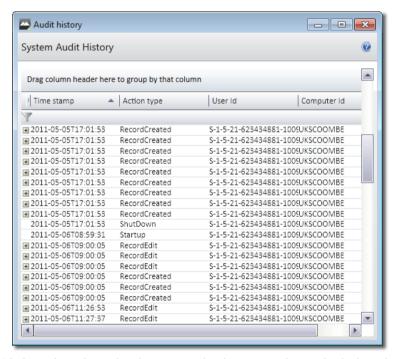
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Viewing the audit history

To view the **Audit history** log; on the **Review** ribbon, select **System** from the **Auditing** group.



2. The **Audit history** window is displayed, which shows all non-archived system events that have been recorded.



3. Click on the column headers to sort the data or use the methods described for filtering the record view (see **Filtering the Record View** in the **Viewing the measurement results** chapter).

This log shows:

- **Time stamp** the time in the following format: YYYY-MM-DDTHH:MM:SS (e.g. 2011-05-05T12:55:35).
- **Action type** a self explanatory name for the action performed, for example RecordCreated, Record Edited, RecordDeleted, Startup etc.

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- **User ID** the unique ID of the individual user logged onto the system.
- **Computer ID** the network name of the computer that accessed the system.



Tip

Right click a record from the **Record View** and choose **View Record Audit History** to view a history of the selected record only.

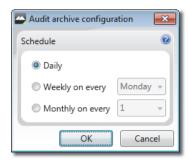
For edited records, the following properties are shown in the expanded tree view:



- Property the record property (for example, Sample Identifiers) that was altered.
- **Old Value** the previous value recorded for the property.
- **New value** the new value recorded for the property.
- **Reason for change** this is for averaged records only where a Reason was specified by the user at the time the averaged record was created.
- **Modified file name** the (.rmd) file name of the record that was modified.
- Modified record the record number within the measurement file that was modified.

As the audit history records all significant system events, the log can grow quite quickly, making it more difficult for a user to visually analyse the data. The **Archive schedule** feature allows you to set the frequency for archiving for the audit trail. The archived data is still available, but is stored separately to the active archive. You can view archived data by selecting **System (including archived entries)**.

4. On the **Review** ribbon, select **Schedule** from the **Auditing** group.



5. Choose a frequency of either **Daily**, **Weekly** or **Monthly** as required and then click **OK**.

Electronics signatures

The electronics signature option allows any changes done on the system to be checked and signed to acknowledge any work that has been done.

- 1. From the **Application button** menu, select **Options**.
- 2. Choose 21 CFR Part 11. and then enter the key you have been sent.
- 3. Click **OK** (the OK button is only available if you have entered a valid key).
- **4**. Ensure that **Enable Electronic Signatures** is selected.

Record Signing

Withe the electronics signature enabled, the record sign-off features are available.

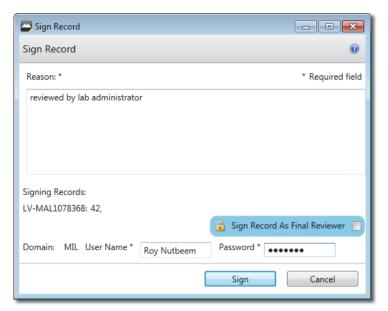
Select a measurement record and either right click and select **Sign-off result**; or from the **Review** ribbon, select the **Sign selected records** button in the **Electronic signatures** group.



The sign-of result window will appear.

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Enter a **Reason** for the change and signature, then enter your **User name** and **Password**. The **Final reviewer** check box can also be selected, this will mean that no further changes or updates will be allowed on that record.

For more information consult the MAC documentation

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Advanced features

Introduction

Advanced users can exploit additional features of the Mastersizer application software that are not normally required as part of the basic measurement process. These features enable a supervisor to set up options that may help operators use the system in a more standardised way.

This chapter focuses on the following areas:

- Doing more with SOPs, including how to set up SOP templates and analyse the development history of an SOP.
- Editing records that have already been made.
- Exporting data and working with export templates.
- Maintenance utilities.

Doing more with SOPs

Extracting an SOP from a measurement

From time to time it can be useful to extract the settings used to make a measurement into an SOP, so that the same settings can be applied to further measurements.

- 2. The **SOP Editor** is shown containing all of the settings used at the time the measurement was created. If more than one record was selected, multiple tabs are shown in the SOP Editor.

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3. Save the SOP by choosing **Save** from the SOP Editor's menu.



Alternatively, save the SOP as an SOP Template by choosing **Save as template**. Refer to Creating and Editing an SOP in **Making measurements** chapter for more information.

SOP Templates

SOP templates allow you to create SOPs based on predefined settings, improving both the speed of creation and consistency. Effectively SOP templates are just SOPs with additional, optional, metadata attached to them; a Name, Description, Category and image. It is a good idea to create a template whenever the core settings of an SOP are to be used repeatedly.

The process of creating an SOP template is very similar to creating a standard SOP. You can either create an SOP from scratch and then save it as a template, or open a previously created SOP/template, make amendments to it and then save it. SOP templates can be edited, saved as new templates or just saved as standard SOPs.

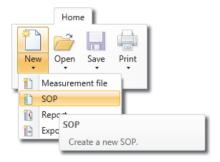


Note

SOP Templates are stored as .msot files in the Windows file structure. If required, use the Workspace shortcut from the Workspace panel to locate the SOP Template folder (the templates are organised in folders based on any categories that users have assigned to the templates). If any associated image is removed from the SOP Template folder, a default image is used instead.

Creating an SOP template

1. From the **Home** ribbon select **New-SOP**.

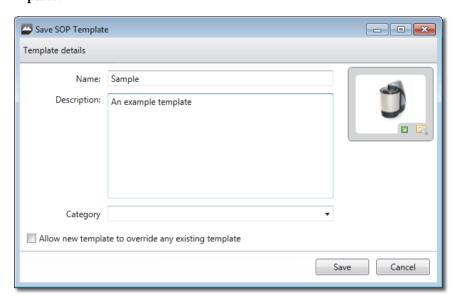


2. Then select the appropriate template from the list (or **From existing SOP...**) and click **OK**.

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3. Edit the SOP details as required and then choose File-Save as template.



4. In the **Save SOP Template** window, give the new SOP template a: **Name** (a descriptive name for the SOP template), a **Description** and, if required, assign a **Category** (previously added categories are available from the drop down menu). Categories could be of assistance when your environment has multiple SOP templates.

Additionally an image can be assigned to the template:

- a to assign a custom image.
- to restore the template default image.
- 5. To save over an existing SOP template (i.e. to edit an existing template), select Allow new template to override any existing template from the Save SOP Template window. Also, ensure that the new template uses the same name as the existing template.



Tip

To view a set of pre-selected SOP templates relevant only to the currently attached dispersion unit, choose **File-New** from the **Manual Measure-ment settings** window.

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SOP Summary Report

The SOP Summary report is only available with the Mastersizer 3000 / Mastersizer 3000E with Extended software option.

The SOP Summary Report feature provides a simple means of quickly observing the SOP settings that were used for an individual or multiple records in a measurement file.

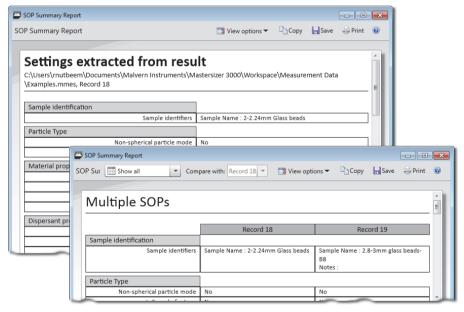
 In the records view, select the record(s) for which you want to review the SOP summary and then either right click on the record and choose View/Compare SOP settings, or click View SOP summary on the Home ribbon

₩ View SOP History

Alternatively, from the **SOP Editor**, choose **Summary report** from the drop-down.

The SOP Summary report window is displayed. This lists all settings applied by the SOP at the time of measurement.

If more than one record is selected from the records view, each record is shown in its own column.



- **2.** To **Save**, **Copy** (to clipboard) or **Print** the information shown, click the appropriate option from the SOP Summary Report window toolbar. The date that the SOP summary was created is appended to all three options.
- **3.** Select the following options from the **SOP Summary Report** window toolbar as required:

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- Show all this is the default view and shows all SOP settings from all items.
- **Highlight differences** shows all items (as the default view), but highlights using different colours, areas where different values or SOP settings were applied.
- Show only differences shows all items, but only those SOP settings where differences exist.
- **Compare to** becomes active when either Highlight Differences or Show only differences is selected. This option then allows you select a 'source' item against which to compare the others any field in another item that differs from the source is highlighted or isolated (as selected).
- **4.** Select any required **View options** from the list as follows:
 - **DragDrop mode** ensures that the columns are kept to a minimal size to enable the display of as much data as possible on the printed page, and allows elements to be dragged and dropped easily into other applications such as Microsoft Word. If de-selected the columns are only constrained to the width of the current window.
 - Column headers in each section adds each record name to each section's header.
 - **Move source column to the left** moves the column selected in the Compare to field to the leftmost position for easier analysis.

SOP History

Each time an SOP is edited and re-saved, its history is recorded for future audit purposes. The SOP History feature exposes any differences between versions of the SOP.

This feature only applies to SOPs that have been revised and re-saved. If the **Save As** option is used the SOP's history is not maintained.

- 1. Select **View SOP History** from the Home ribbon and then choose the required file. The **SOP Version History** window is displayed. This lists all parameters as applied by the SOP. Each version of the SOP is shown in its own column.
- **2.** To **Save**, **Copy** (to clipboard) or **Print** the information shown, click the appropriate option from the **SOP Version History** window toolbar. The date that the SOP history summary was created is appended to all three options.
- **3.** Select the following options from the SOP Version History window toolbar as required:

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 Show all – this is the default view and shows all SOP settings from all items.

- **Highlight differences** shows all items (as the default view), but highlights using different colours, areas where different values or SOP settings were applied.
- Show only differences shows all items, but only those SOP settings where differences exist.
- Compare to becomes active when either Highlight Differences or Show only differences is selected. This option then allows you select a 'source' item against which to compare the others any field in another item that differs from the source is highlighted or isolated (as selected).

Exporting data

Data recorded by the system can be exported for analysis in other software applications, for example Microsoft Excel.

It is possible to export either selected records or all records contained in a single measurement. Additionally, by selecting the **All Files** tab when multiple measurement files are open, a selection of, or all records from, multiple measurement files can be exported.

The ability to create export templates further enhances the flexibility of the system, allowing you to select only those parameters required for your analysis, as well as providing control over other data formatting options.



Note

An alternative way to export data is to use the **Copy data / Copy raw data** functions. Refer to the **Reporting** chapter for more information

Export data settings

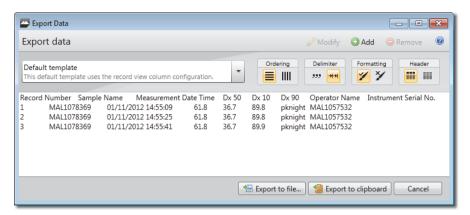
 To export selected records only, select the required records and choose Export Selected Records from the Home ribbon. To export all records from the currently active record choose Export All Records.



The **Export Data** window is displayed, which shows a preview of the data to be exported as specified by the currently selected export template.

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- **2.** Select an appropriate export template from the drop-down list the preview pane is updated accordingly.
- 3. Change the presentation of the data using the **Ordering**, **Delimiter**, **Formatting** and **Header** options (see **Export templates** for more information).
- 4. Click **Export to file** to save the data as a separate (.txt or .csv) file, or select **Export to clipboard** to copy the data to the clipboard.



Tip

Drag and drop record data from the **Record View** straight into Microsoft Excel/Word. The parameter values copied are all of those currently shown in the records view.

Export templates

Export templates provide a means of storing, and quickly accessing, commonly-used sets of parameters and format settings for exporting data. You can build and store any number of export templates.



Note

Each template is associated only with the workspace in which it was constructed. This means, for example, that a template defined in the **Malvern default** workspace, will not then be displayed in a custom workspace.

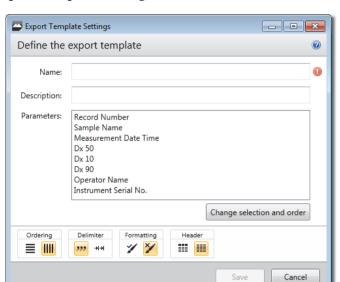
Creating or modifying an export template

 To create a new export template: from the Home ribbon select New-Export template (or click Add from the Export Data window).

To modify an existing template: select it from the drop down in the **Export Data** window and click **Modify**.

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The **Export Template Settings** window is shown:

- 2. Enter a **Name** and then a short **Description** that explains the purpose of the template. The list of parameters associated with the template is displayed in the **Parameters** panel. Initially this consists of the whole set of system parameters.
- **3.** Click the **Change selection and order** button to show the **Parameter selection window**, then select the required parameters as follows:



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Locate parameters by manually expanding the tree structure in the Available list, or search for them by typing their first few characters into the Search... field.

- Select a parameter in the **Available** list and then click the **Add** button to transfer it to the **Selected** list. Similarly, select an item from the **Selected** list and click the **Remove** button to transfer it to the **Available** list. It is also possible to select multiple items using the standard Windows selection methods (i.e. SHIFT and click, CTRL and click).
- When all of the required parameters are shown in the **Selected** list, choose an order of presentation so that they are shown in the desired left-to-right order. Select a parameter and then click the up/down buttons to move its position in the list. Items at the top of the list are shown on the left, items at the bottom are shown on the right.
- Click **Apply** to apply the settings without closing the window, or click **OK** to apply the settings and also close the **Parameter Selection** window.
- 4. Choose either rows or columns from the **Ordering** selector to present the data horizontally or vertically respectively.
- 5. From the **Delimiter** selection, choose either **Comma separated** (csv) or **Tab separated**. Your choice will depend on the requirements of the target application.
- 6. Use the **Formatting** option to select either **Format values as displayed in software**, which displays numeric information to a limited number of decimal places (this may be preferable for presentation purposes); or choose **Unformatted values** to show all data as recorded by the system.
- 7. Select **Header on/off** to specify whether the export should contain the names of the fields as a header row (or column).
- 8. Click **Save** when complete.

Header

Deleting an export template

From the **Export Data** window, select the template you wish to remove and click **Remove**. The software asks for confirmation of the action before permanently removing the template.

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