Mastersizer

2000

user manual

MALVERN
INSTRUMENTS

Mastersizer 2000
User Manual

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Addendum to manual

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This addendum presents new information on the Mastersizer 2000/2000E from Malvern Instruments.

Laser specification

The following information adds to the **Light Sources** information given on page A-1:

Red light

- CDRH and CE compliant (Class llla laser product (CDRH) / Class 3R laser product (IEC60825-1(1993)+A1(1997)+A2(2001)))

- Type: HeNe gas laser.

- Max. output power: 4mW.

- Beam diameter: 0.63mm ($1/e^2$).

- Beam divergence: 1.5mrad.

- Beam wavelength: 633nm.

Blue Light

For the Mastersizer 2000 and Mastersizer 2000LF this is:

- Beam wavelength: 466nm.

- Type: LED.
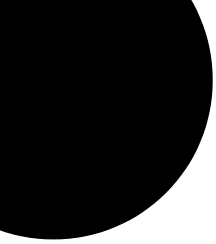
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Part 1 -
Operator's Guide

Introduction to this manual

About this manual

This manual describes the operation of the Malvern Mastersizer 2000 and 2000E particle size analysers. Information is also given on basic use of the Hydro 2000G and Hydro 2000MU sample dispersion units:

Instrument	Model number
Mastersizer 2000	APA2000
Mastersizer 2000E	EPA2000
Hydro 2000G	AWA2000
Hydro 2000MU	AWM2001

For more detailed information on the operation of any sample dispersion unit, see its individual manual.

This user manual is a companion to the **Mastersizer 2000 Essentials Manual** which gives Health and Safety, maintenance and other vital information which **all users must read**.

After reading this user manual, users should understand the basic measurement technique and be able to perform a simple measurement, analyse the data and identify the main features of the system.

For users who have never operated a Malvern particle analyser before, we recommend reading this manual fully before starting the first measurement.

Users who are more familiar with particle size analysers may wish to proceed straight to **Chapter 4** which gives details on making measurements. However, the importance of sample preparation before measurement cannot be overstated. We therefore recommend that these users read the chapter on sample preparation (**Chapter 8**) soon.

> **Warning!**
> The instrument and the samples to be measured may be hazardous if mis-used. Users must read the **Health and Safety** information in the **Essentials Manual** before operating the system.

Access to the instrument

Within this manual, reference is made to the various people who will have access to the instrument. Below is a list of these people and their responsibility:

Malvern personnel

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

Supervisor

The supervisor is the person responsible for the management/safety of the instrument and of its operation. The supervisor is responsible for the training of the operators. The supervisor can perform all user maintenance routines identified in the **Essentials Manual**, including changing the fuses.

> **Warning!**
> Under no circumstances must the supervisor or operator remove the covers of the instrument.

Operator

An operator is a person trained to use the instrument. The operator can perform all user maintenance routines in the **Essentials Manual except** for changing the fuses.

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

Assumed information

General

The Mastersizer 2000/2000E can be used with a variety of sample dispersion units that allow it to measure wet and dry samples. The function of these is identical: to prepare and deliver the sample to the optical bench for measurement. For clarity, this manual assumes that the primary dispersion unit for each system is as follows:

- For the Mastersizer 2000 the Hydro 2000G.

- For the Mastersizer 2000E the Hydro 2000MU.

For more details of any sample dispersion unit, refer to its manual.

Naming convention

Within this manual:

- The Mastersizer 2000/2000E optical bench is referred to as "the optical bench" or "the instrument". For simplicity, in general text the term "Mastersizer 2000" refers to both the Mastersizer 2000 and 2000E. Where the Mastersizer 2000E is different, this is always explained.

- The sample dispersion units are referred to in full ("the Hydro 2000G" or "the Hydro 2000MU"), or as "the dispersion unit".

- The combination of the optical bench, one or more dispersion units and the computer is referred to as "the system".

Menu commands

Menu commands from the Malvern software are referred to in the form **main menu-menu item**. As an example, the command **Configure-New SOP** refers to selecting the **New SOP** item in the **Configure** menu. The same rules apply for sub-menus of sub-menus, so that **Tools-Options-Instrument Port** refers to the **Instrument Port** item in the **Options** sub-menu, which itself is a sub-menu of the **Tools** menu. Menu commands are always shown in bold text.

Where to get help

Other manuals

Several manuals are supplied with the system, each giving information on a different aspect of it. Below is a list of the typical manuals supplied with the system, detailing the type of information in each one.

Quickstart Manual

This gives a simple introduction to the system and explains how to make a measurement.

Essentials Manual

This manual covers:

- **Site requirements** – all the physical requirements for positioning the system. Information is given on service requirements (air, water, number of power sockets, etc.), environmental requirements (temperature, humidity, etc.) and physical requirements (space needed, etc.).

> **Note**
> The system **must** be initially commissioned by a Malvern-trained representative.

- **Health and Safety** – this **must** be read by all users of the system. It details all safety issues for the optical bench and all dispersion units.

- **Maintenance** – for both the optical bench and dispersion units.

- **Installation** – this gives enough information to allow the user, for example, to move the system from one laboratory to another. Instructions are also given on how to install the Malvern software if the user upgrades the computer system.

> **Caution!**
> If the system is covered by a Qspec validation contract, we strongly recommend that it is only moved by Malvern representatives.

Autosampler 2000 manual

The Autosampler 2000 has its own manual giving details on its use.

Dispersion Unit manuals

Each dispersion unit has its own manual giving details on its use.

Online help

The primary function of the online help is to give detailed information on the software. Each dialogue has a **Help** button that, when pressed, gives help specific to that dialogue. Many dialogues also have **Advice** buttons, giving more general advice.

All help topics can be accessed through the standard help screen by selecting **Help-Help topics**. From within this screen all help topics can be searched or accessed through the contents list or index.

Help desk

All queries regarding the system should initially be directed to the local Malvern representative. Be ready to quote:

- Model number of the optical bench and dispersion unit(s).

- Serial number of the optical bench and dispersion unit(s).

- Version of the Malvern software – this can be found by starting the software and selecting **Help-About**.

The serial number of the optical bench can be found inside the sample area (remove the cell first). The serial number of a dispersion unit is found on its rear panel.

Contact the United Kingdom help desk if the local Malvern representative is not available. Its direct line is +44 (0)1684 891800.

Note
This help line is primarily English speaking.

Remote support

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

Online user training is also available, plus software updates. A high speed Internet connection is recommended for making use of this facility.

Malvern website - www.malvern.com

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

Resources include Frequently Asked Questions, a knowledge base and application notes, plus information on other material characterisation solutions that Malvern can provide.

Overview

Introduction

This chapter gives a complete overview of the system. It explains in very simple terms:

- What the Mastersizer 2000 does.

- How the Mastersizer 2000 works.

- Making measurements.

- Viewing the results.

- Saving the results.

Most of these points are explained in more detail in later chapters of this manual.

What the Mastersizer 2000 does

Take a look around. At the paint on the walls, the chocolate on a biscuit, the cement foundations of a building. The stability, chemical reactivity, opacity and material strength of these and many other materials are affected by the size and characteristics of the particles within them.

The Mastersizer has been designed to measure the size of these particles or, more specifically, the distribution of different sizes within a sample.

2

How the Mastersizer 2000 works

Scientists have for centuries tried to predict the way particles scatter and absorb light. There are many theories and models that the modern particle size analyst can use.

One of the simplest theories used is the **Fraunhofer model**. This model can predict the scattering pattern that is created when a solid, opaque disc of a known size is passed through a laser beam.

This model is satisfactory for some particles but it does not describe the scattering exactly. Very few particles are disc shaped and most particles are transparent.

The accepted theory which accurately predicts the light scattering behaviour of all materials under all conditions is known as the **Mie theory**.

Mie theory was developed to predict the way light is scattered by spherical particles and deals with the way light passes through, or is adsorbed by, the particle. This theory is more accurate, but it does assume the user knows some specific information about the particle, such as its refractive index and its absorption.

The key point about these theories is that if the size of the particle and other details about its structure are known, the way it will scatter light can be predicted accurately. Each size of particle will have its own characteristic scattering pattern, like a fingerprint, that is unlike any other size of particle.

So how does the Mastersizer measure the size of particles? It works backward from the above theories by using the optical bench to capture the actual scattering pattern from a field of particles. Then, using the theories, it calculates the size of particles that created that pattern.

There are three distinct procedures in measuring a sample on the Mastersizer:

1. The sample is prepared and dispersed to the correct concentration and then delivered to the optical bench. This is the purpose of the sample dispersion units. Sample preparation is the most important stage of making a measurement. Remember that, if the sample is poorly prepared (unrepresentative or badly dispersed) then the basic data will be incorrect; no amount of analysis of this data will give a correct answer.

2. The capturing of the scattering pattern from the prepared sample - this is known as the "measurement". This is the function of the optical bench.

   The detector array within the optical bench is made up of many individual detectors. Each detector collects the light scattering from a particular range of angles. A typical light scattering pattern is shown below.

Data Graph - Light Scattering

Background data   wet measure, 21/06/98 9:18:18 am
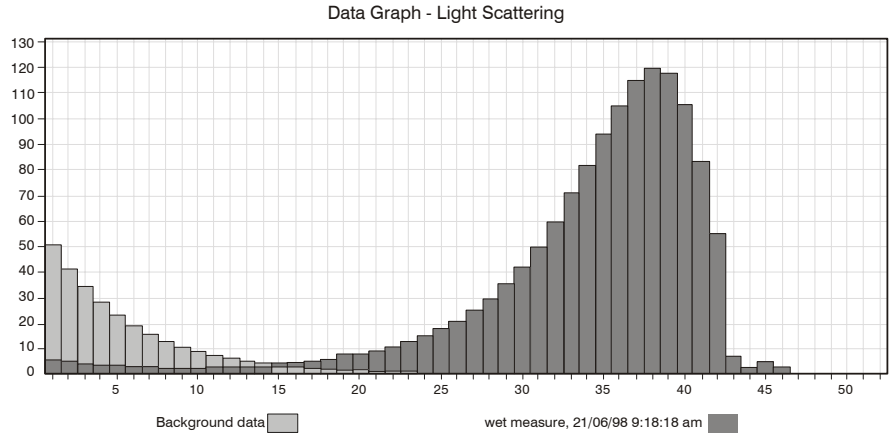
ill 4856

Each bar in the histogram represents the light scattering from one of the detector elements (known as a channel).

The detector array takes a "snapshot" of the scattering pattern. Obviously this snapshot only captures the scattering pattern from the particles that are passing through the analyser beam at that particular time. Taking only one snapshot may not give a representative reading of the scattering pattern. To overcome this, the Mastersizer takes many snapshots (known as **snaps**) and averages the result. Typically over 2000 snaps are made for each measurement, with each snap taking 1ms.

3. Once the measurement is complete, the raw data from it is analysed by the Malvern software using one of the theories mentioned above. Once the data has been analysed the information can be displayed in various ways.

Note the following points regarding the measured data and the analysis:

■   Analysing the measurement data does not permanently alter the data. The measurement data can be re-analysed using different methods over and over again.

■   The software makes a measurement and then analyses the data automatically.

Making measurements

There are two ways to make a measurement; making a **manual** measurement or using a **Standard Operating Procedure (SOP)**.

SOPs

SOPs are user-defined procedures that are easily programmed into the software to allow the same type of sample to be measured in a consistent way. Here all the difficult decisions on measurement strategy have already been made.

When the SOP is run, it automatically runs through the defined measurement procedure, prompting the operator to perform tasks (such as adding the sample). The SOP takes full control of the dispersion unit (except any manual unit), making all adjustments and settings (such as pump speed and ultrasonic power). This is the recommended method of operation and is suited to beginners or when routinely measuring the same type of sample.

Creating an SOP is very easy. The operator selects **Configure-New SOP** and the software runs the **SOP Wizard**. This wizard asks a series of questions, such as:

■   what are the optical properties of the sample?

■   what is it suspended in?

■   how fast should the stirrer be?

■   how much ultrasonic dispersion is required?

■   should any additives be added to the sample to aid dispersion?

■   how is the result to be calculated and displayed?

These all combine to define the measurement procedure.

Manual measurements

But how does a user know how to answer the questions? This information is found by referring to the advice which is available at all stages of SOP creation, as well as by experimentation; making a few preliminary measurements and observing the results. This is the primary function of a **manual measurement**. Manual measurements can be used to make "one-off" measurements or used to "get a feel" for a sample prior to defining an SOP.

More details on making measurements can be found in **Chapter 4**.

Viewing the results

Once the measurement information has been gathered and analysed, it can be displayed in many forms. The Malvern software is supplied with many standard "reports" that each display the information in a specific way. For each report that is displayed on the screen, there is an equivalent "hard copy" report for printing the report information to a printer.

Remember that the different reports show the same data in different ways.

All data from a measurement and analysis is stored in a "record". Records are stored in a measurement file. A measurement file can contain one or more records (see **Chapter 7** for details on handling measurement files).

Before measurement data can be viewed a measurement file must be opened (by selecting **File-open** and selecting a record). A typical measurement file is shown below:

The **Records** tab is shown initially. Selecting this tab shows all the records within the opened measurement file. One or more records must be selected before a report can be opened. The series of tabs at the top of the window shows the reports that are available. To change between reports simply select another report tab.

If the standard reports do not satisfy the user's specific requirements, custom reports can be built using the **Report Designer** application. See **Chapter 5** for details on using the **Report Designer**.

It is possible to zoom into a graph report. Simply hold down the left button, then slowly move the mouse to draw a "marquee" (from top left to bottom right) around the area to be enlarged. To zoom back out, simply left mouse click on the graph.

Details on interpreting the reports and an explanation of the standard reports are given in **Chapters 5** and **6**.

Saving the results

After making a measurement the user can save the result in a measurement file by selecting **File-Save** or **Save as** (an SOP can be configured to automatically save the results to a measurement file). A measurement file has the extension **.mea**.

3

Identifying the features

Introduction

This chapter describes all the physical features of the system. Users will probably not use all of the features described as some are only used for specific dispersion units or applications.

This chapter covers:

■ Typical systems – identifies the main modules of typical systems.

■ Optical bench components – examines the features of the optical bench in more detail.

■ Software – identifies the main features of the Malvern software.

Detailed information on the dispersion units can be found in their own manuals.

Typical systems

These diagrams show typical systems with their key modules:

Mastersizer 2000
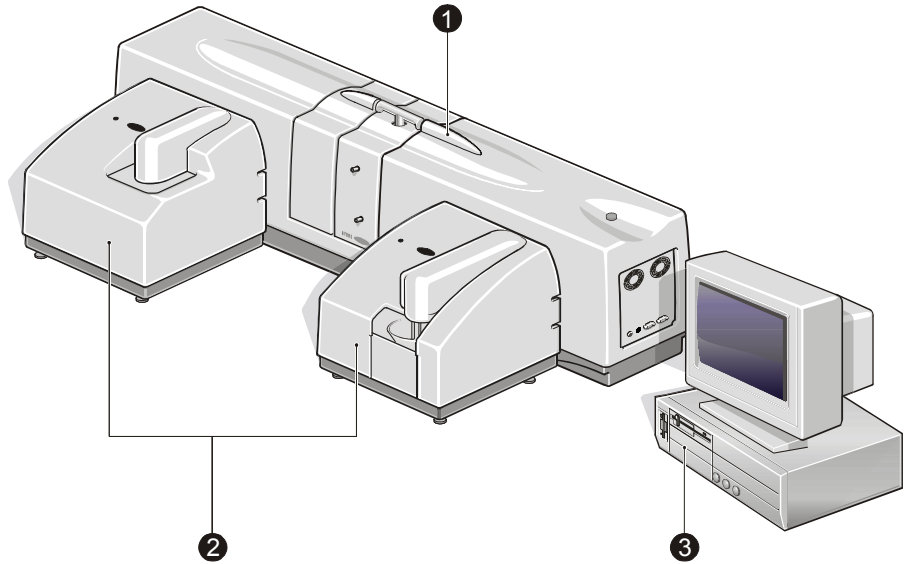
ill 7917

Mastersizer 2000E
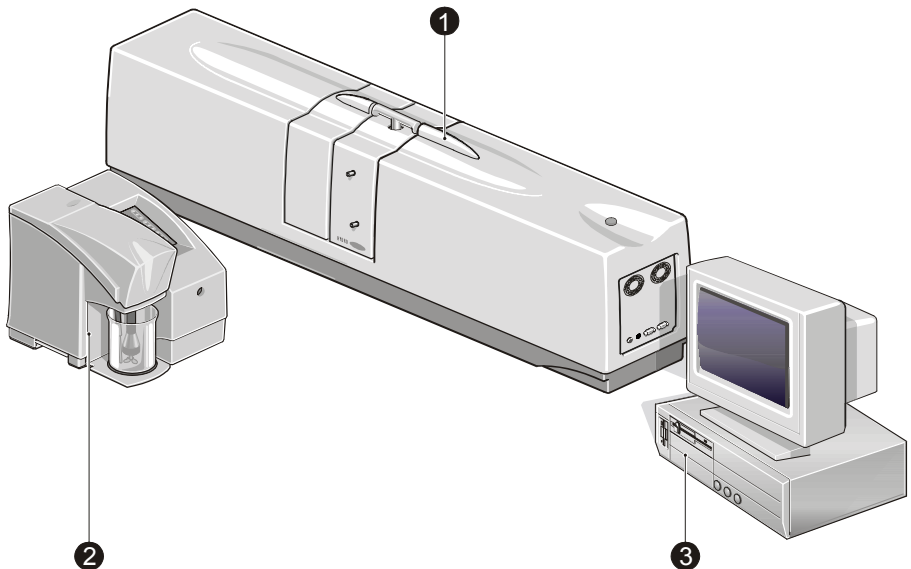
ill 7918

The features shown are:

Optical bench ①

The optical bench is used to collect the raw data used to calculate the size of particles in a sample.

One or more sample dispersion units ②

The sole purpose of a sample dispersion unit is to prepare the sample then deliver it to the optical bench so that it can be measured. Malvern makes many sample dispersion units to handle many forms of sample, including dry powders and samples dispersed in a liquid. A user may have one or more sample dispersion units, depending on their particular requirements.

In the diagrams above:

■   The Mastersizer 2000 is shown with two dispersion units, a Hydro 2000G on the left and a Hydro 2000 S on the right.

■   The Mastersizer 2000E is shown with a Hydro 2000MU.

The individual dispersion unit manuals identify the dispersion units' features.

Computer system ③

The computer system is a stand-alone computer that runs the Malvern software. The Malvern software controls the optical bench and dispersion units (apart from the Hydro 2000MU and Scirocco 2000M which are manually controlled). It also analyses the raw data from the optical bench to determine the size of the particles.

Optical bench components

The main features of the optical bench are shown below:
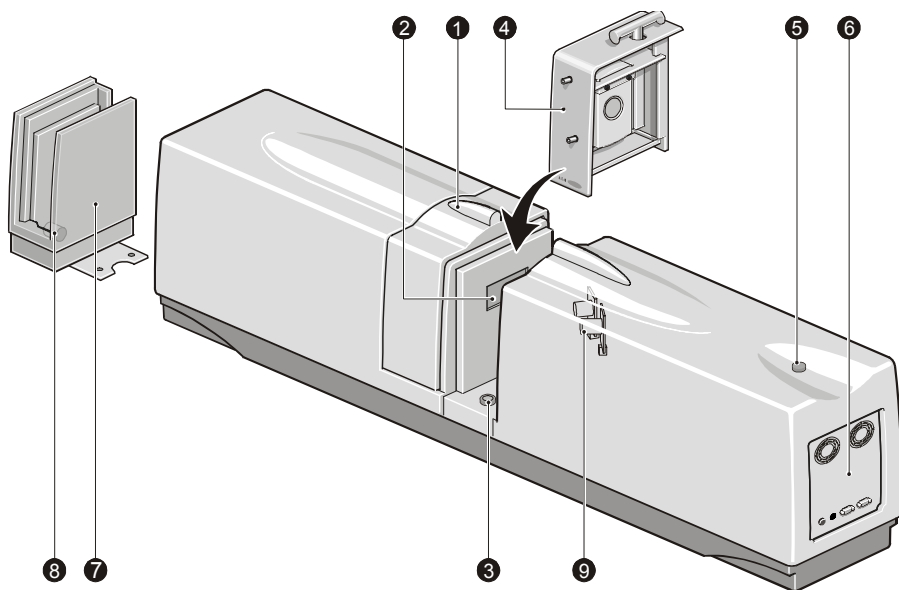
ill 7919

The features are:

① Cell area protection cover

The cell area protection cover can be swung into position when the cell is not in place to prevent dust and dirt contaminating the cell area optics. It is moved by a lifting action rather than a sliding action.

② Protection window

The protection window stops dust and dirt entering and damaging the internal detectors. Clean this window periodically (see the **Essentials Manual**).

③ Drain

The drain allows any spillages within the cell area to exit onto the bench.

④ Cell

The cell is the interface between the sample dispersion unit and the optical bench. The sample measured is passed between two windows that allow it to be analysed by the laser beam.

Three types of cell are available:

- A wet cell used on the Hydro 2000G/S/SM/MU, detailed later in this chapter.

- A specialised wet cell (the "micro cell") used on the Hydro 2000μP, detailed in its manual.

- A dry measurement cell used on the Scirocco 2000/2000M, detailed in their manuals.

⑤ Power indicator

This indicator illuminates when the optical bench is turned on.

⑥ End panel

The end panel contains all communication connectors for the optical bench. It is detailed below.

⑦ Cell holder

The cell holder is a "parking" area for the cell when it is not in use. This is supplied when more than one dispersion unit is used on the system. It allows the cell to be removed and stored without the need to disconnect it from the dispersion unit.

⑧ Window tool

The window tool is used to remove the windows from the cell if they need cleaning or replacing (see the **Essentials Manual**).

⑨ Backscatter detectors

These are two detectors that collect some of the light scattering information from the sample. Ensure that these are kept clean and undamaged; the **Essentials Manual** has details.

End panel

The features of the end panel are detailed below:
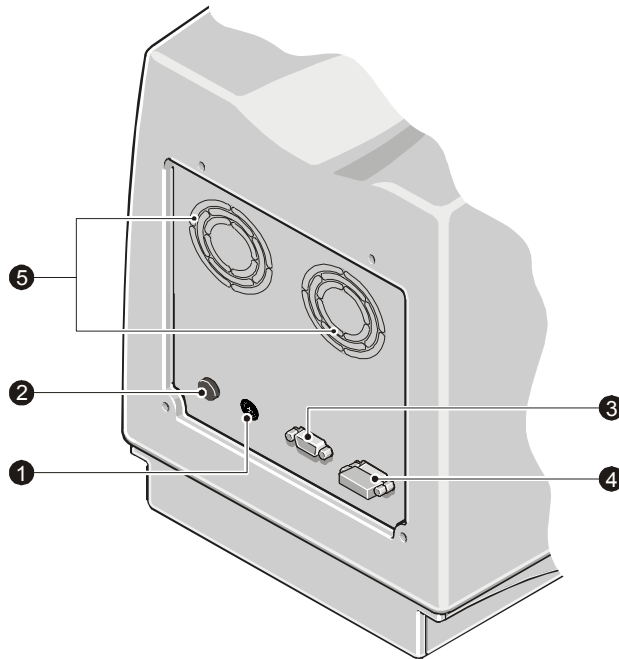
ill 7920

① Power input socket

This is the power input socket to the optical bench from the external PSU.

② Optical bench power switch

This is the on/off power switch for the optical bench.

③ COMPUTER connector

The RS232 cable from the computer is plugged in here. Data from the optical bench and control commands from the computer are transmitted down it.

④ ACCESSORY connector

Dispersion unit communications connector. The first sample dispersion unit is connected here. It allows the dispersion unit to be controlled by the software.

This connector is not used if only manually controlled dispersion units (Hydro 2000MU and Scirocco 2000M) are used.

⑤ Air holes

These holes allow air to cool the unit. **Do not obstruct them**.

Wet cell

The features of a wet cell are detailed below:
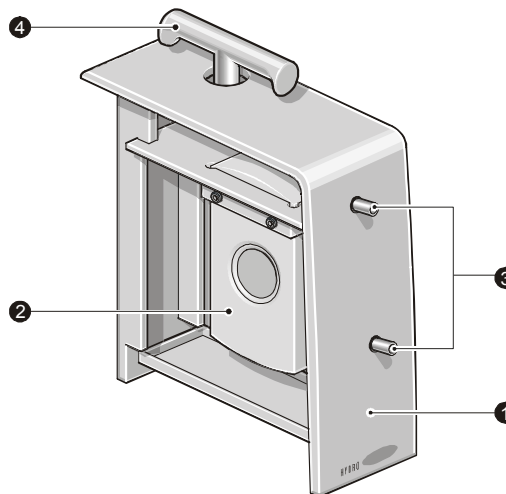
ill 4860

① Light shroud

This shroud stops light entering the cell area during a measurement. More importantly, it protects the user from laser emissions. **Never operate the system if any of the covers is damaged**.

② Cell

The cell has a pair of windows that allow the laser to pass through the sample. The windows can be removed for cleaning/replacement (see the **Essentials Manual**).

③ "Cell in" and "Cell out" connectors

The sample is recirculated through the cell and back to the dispersion unit. These connectors attach the cell to the dispersion unit. The tubing must be connected correctly. The tube from the dispersion unit to the cell must be connected to the **Cell in** connector. This allows any trapped air bubbles to rise through the cell. The connectors are colour coded to aid correct connection.

④ Locking handle

The locking handle is rotated anti-clockwise to lock the cell in place.

⚠ **Caution!**
   Never try to lift the optical bench by this handle.

Software

The Malvern software controls the system during a measurement and processes the measurement data to produce a size distribution. There are two software modules:

■   The main application, described below. This main interface allows the user to define an SOP, run a measurement, display the results and print reports.

■   A secondary module, the **Report Designer**, allows the user to create custom reports to display the results. The **Report Designer** is detailed in **Chapter 5**.

Features of the window

The features of the window are detailed below:
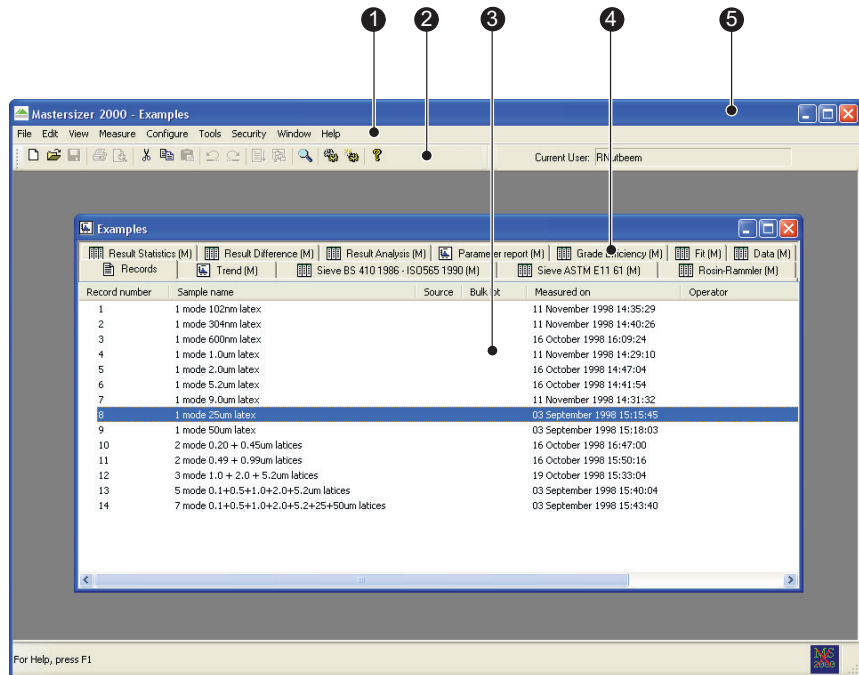
ill 7752

① Menu bar

The menu bar contains the main menu headings for all software functions. There are several ways to select an item from the menu bar:

■   Using the mouse – to select an item from the menu bar, click the left mouse button once on the menu. When the menu list drops down, select an item from the menu list by clicking once on it.

- Using the keyboard – to select an item from the menu bar, hold down the **Alt** key and press the letter which is underlined in the command required. For example to use the **Measure** menu hold down **Alt** and press **m**. When a key is used in this way it does not matter if it is upper or lower case; **M** and **m** both work, for example.

- Using keyboard accelerators – to the right of a menu item name there may also be a note of the accelerator for this item. This is a key or combination of keys which can be used to by-pass the menus. For example, users can press **Ctrl** and **O** together to select **File-Open...** without having to use the main menu and drop down menu.

Clicking an item which ends with a row of dots (**...**) opens a dialogue. Items shown in grey are those which are not currently available.

② Toolbar

The toolbar contains a selection of buttons for performing common operations.

Each button has an equivalent menu command. For example using the button is equivalent to using the **File-Open** command.

To help users identify a button, a short **tooltip** description of its action is displayed in the status bar when the cursor is moved over it.

As with the menu bar, if a button is not available it is shown "greyed out".

③ Measurement window

The Measurement window displays all the information for a measurement file. Multiple measurement windows can be displayed at a time. The contents of the display area change depending on which report tab is selected.

④ Report tabs

Selecting a report tab displays the data for the selected record(s) in that format. Malvern supplies standard reports that allow the user, among other things, to view the list of records within a measurement file, view the measurement data and view the size distribution as a graph and table. Custom reports can be designed using the **Report Designer.**

Details on interpreting reports can be found in **Chapter 6**.

⑤ Title bar

The title bar displays the file name of the currently selected measurement file.

4

Making measurements - a tutorial

Introduction

This chapter describes how to make a measurement. It describes:

- General measurement advice – the importance of sample preparation and cleanliness.

- The basics of making a measurement – describes both manual and Standard Operating Procedure (SOP) measurements. This section details the procedure for each type of measurement in detail.

- Making measurements on a Mastersizer 2000E.

- Measuring new samples – what to do for a new material.

- Measurement options – describes the series of dialogues which comprise the **SOP Wizard**.

The best way to learn how to use the system is to make a measurement. To get the most benefit from this chapter we advise reading it once, then reading it a second time while following the instructions to make a measurement.

To do this, obtain a suitable sample to measure and a suitable dispersant to disperse it in. Ordinary single dairy cream dispersed in water is readily available and should give good predictable results.

General measurement advice

Before making a measurement, note the important general advice given here.

Sample preparation

Good sample preparation is critical. A representative sample must be taken. Dry powders, for example, tend to separate out if stored for some time or vibrated. The larger particles tend to rise to the top and the smaller particles collect at the bottom of the container. If the sample is taken from the top of the container it will not contain the smaller particles, giving a biased measurement. The sample should be correctly mixed before a measurement is taken.

Wet samples also have to be correctly dispersed in a liquid dispersant. Using the wrong dispersant can cause the sample to stick together in clumps, float on the surface, or even dissolve. Check the sample and dispersant to see if they are suitable before a measurement is made. There are many ways to prepare the sample to ensure a perfect measurement.

Details on sample preparation are given in **Chapter 8**.

> **i** **Note**
> Over half of the problems encountered in measuring a sample are caused by bad sample preparation. If the user prepares badly for the measurement, no amount of subsequent analysis will give a good result.

Adding the sample

The software reports exactly what level of signal the sample generates and whether this is ideal, too low, too high, etc. The technique has a wide range of concentrations that are ideal so concentrations do not have to be precise.

The sample concentration is controlled by monitoring the obscuration of the laser beam caused by the sample. The obscuration is simply the fraction of light "lost" from the main beam when the sample is introduced. For example, an obscuration of 30% means that 30% of the incident laser beam (recorded during the **Measure Background** step) has been lost through scattering or absorption.

The range of concentrations over which the instrument can be used can be expressed in obscuration terms as the following table shows.

Obscuration range (%)	Obscuration graph colour	Notes
<5	red	Add more sample.
5-10	orange	Low but usable with a good signal to back-ground ratio.
10-20	green	Ideal.
20-50	orange	Usable but try to add more dispersant - there's a danger of multiple scattering.
>50	red	Too high.

Cleanliness of the optical system

Laser scattering is a high resolution optical method in which the detectors and windows are an integral part of the measurement zone. Dust or smears on the windows will scatter light that will be measured with the sample scattering. In general the process of measuring both a background and a sample and then subtracting the background corrects for such contributions. However, this correction is first order only and excessive dirt on the optics will degrade the instrument's sensitivity.

We recommend ensuring that detectors and windows are clean at all times. See the **Essentials Manual** for advice on cleaning the system. If in doubt about the optical cleanliness, the user can use the live display of the background measurement. By viewing this screen they can judge whether the optics are clean or not.

The basics of making a measurement

There are two ways to make a measurement:

- Using a **Standard Operating Procedure (SOP)** – SOPs are user defined procedures that are easily programmed into the software to allow the same type of sample to be measured in a consistent way with all the difficult decisions on measurement strategy already having been made.

  When the SOP is run, it automatically runs through the defined measurement procedure, prompting the operator to perform tasks such as adding the sample. The SOP takes full control of the dispersion unit (unless it's a manual unit), making all adjustments and settings (such as pump speeds and ultrasonic power). This is the recommended method of operation and is suited to beginners or when routinely measuring the same type of sample.

- **Manual measurements** – used either to make one-off measurements or to try out a sample prior to defining an SOP for it.

Follow the section below for the instrument used.

Measurements on a Mastersizer 2000

Instrument preparation

Before starting a measurement, ensure that the correct cell is installed in the cell area then power up the system as outlined below. (If an SOP is run, the software will check that the correct cell is connected, informing the user if it is not).

▶ **To power up the system:**

1. Switch on the dispersion unit(s).

2. Switch on the optical bench.

3. Switch on the computer and start the software by double-clicking on the Mastersizer 2000 icon.

Making a basic SOP measurement

Start an SOP by selecting **Measure-Start SOP**. In the dialogue which appears select an SOP. The **Measurement Display** dialogue will appear:

ill 7753
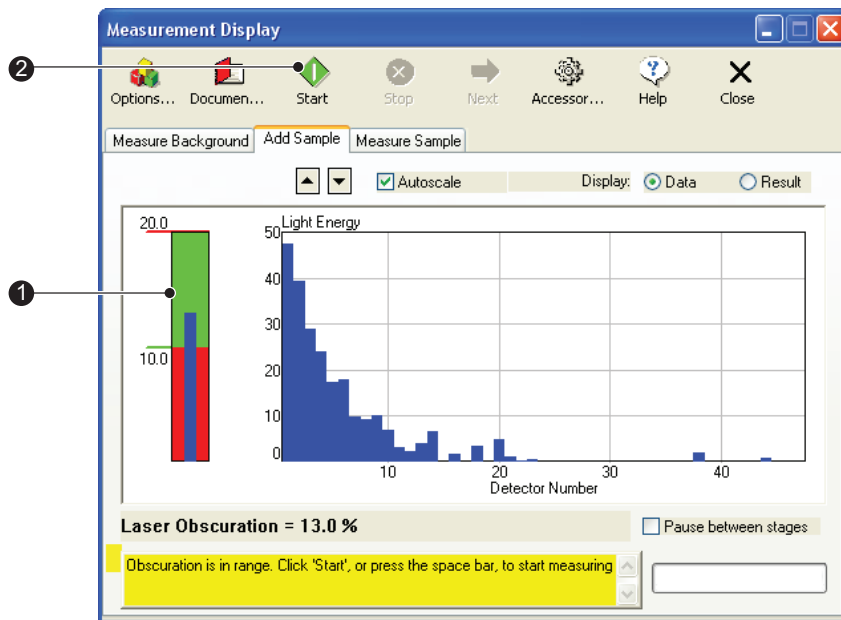
The **Status area** at the bottom left of the dialogue reports what is going on and what to do next. The information in the dialogue changes depending on the stage of the measurement. The SOP measurement runs as automatically as possible. In a typical SOP measurement there are only three things the user has to do:

1. Part way through a measurement the dialogue asks the user to add sample to the dispersion unit's tank. Depending on the SOP design, the user is either asked to add a specific quantity to the tank or add sample to the tank until the obscuration bar ① is in the green section.

2. Once the correct amount of sample has been added, press the **Start** button ② to start the actual measurement.

3. Depending on the SOP design, a dialogue may appear asking the user to document the measurement (add comments and observations).

If specified, an SOP measurement will automatically clean and fill the dispersion unit, measure the background, align the optics, calculate the size distribution, and save the record to a measurement file. All the user has to do is wait for a prompt from the SOP to perform the actions detailed above.

Try making an SOP measurement using single dairy cream. Make sure that the system is connected and powered up, then:

1. Select **Measure-Start SOP** and browse for the SOP file.

2. Select **Dairy cream using Hydro 2000G.SOP** (or **Dairy cream using Hydro 2000S.SOP** if using the Hydro 2000S) from the directory `C:\Program files\Malvern Instruments\Mastersizer 2000\SOP`.

3. Select **OK**. The SOP measurement will start by cleaning the tank.

4. The SOP automatically measures an electrical background, aligns the optical system and measures the optical background.

5. The **Add Sample** tab appears and the SOP asks the user to add the cream to the tank. Add enough sample (one drop at a time) to make the **Laser Obscuration** bar show green (try and achieve a mid value).

6. Once satisfied that enough sample has been added, press the **Start** button. The sample is measured and the result (size distribution) calculated. The measurement record is automatically saved to a measurement file.

The measurement is complete. Now select the record in the Measurement window and view the results by selecting one of the report tabs.

Making a basic manual measurement

To make a manual measurement select **Measure-Manual**. The **Measurement Display** dialogue shown below will appear:

ill 7754
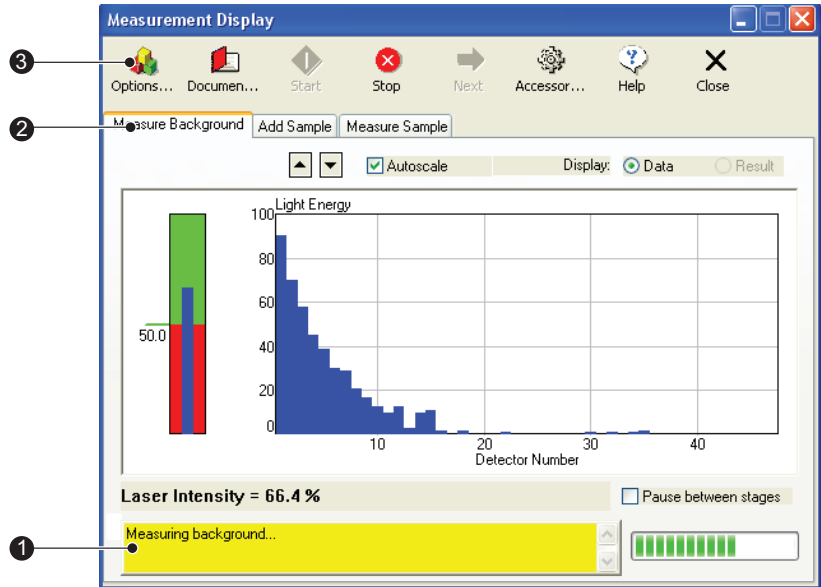
The software will automatically make an electrical background measurement and an optical background measurement then align the optics. The main features of this display are:

① The **Status area** at the bottom left of the dialogue reports what is going on. The information in the dialogue changes as the measurement progresses.

② The tabs show information on the four stages of making a measurement:

- **Measure Background** – the measurement data from a particle field is contaminated by background electrical noise and also by scattering data from dust on the optics and contaminants floating in the "clean" dispersant. The **Measure Background** facility automatically makes a measurement of the system containing only clean dispersant when a manual measurement is started, as well as a measurement of the electrical background. This background information is subtracted from the sample measurement in order to "clean" the data.

- **Add Sample** – this tab shows how much sample has been added to the tank for measurement. The system measures how much sample is added by monitoring the "obscuration" of the beam caused by the sample being added to the dispersant. The obscuration is simply the fraction of light "lost" from the analyser beam when the sample is introduced. For example

an obscuration of 30% means that 30% of the analyser beam has been lost by either scattering or absorption.

The "obscuration bar" ④ gives a visual indication of how much sample is added. If the bar is in the green zone the concentration is in the correct range. If it is in the red the concentration is out of range. The exact obscuration is given at the bottom left of the dialogue. The instrument has a wide range of concentrations that are acceptable so concentrations do not have to be precise.

The target obscuration values can be set by the user; see **Chapter 9**.

■ **Measure Sample** – this tab displays the scattering information during the actual measurement.

■ **Result** – this tab is shown as the software calculates the size distribution.

③ The toolbar buttons – these allow the user to control the measurement process and document the measurement, control the dispersion unit and set up all the measurement parameters. The buttons are:

■ **Options** – this button gives access to the measurement options. For example, the user can specify what sample and dispersant to measure, how the size distribution is to be calculated, and where to save the results.

■ **Document** – pressing this button displays a dialogue where the user can document the measurement i.e. make notes on the pre-dispersion techniques, sample quantity, etc. This allows the measurement to be repeated at a later date.

■ **Start** – starts a measurement process. For example, after adding the sample and once satisfied with the quantity, the user can start the measurement process by pressing **Start**.

■ **Stop** – this button stops the current measurement process.

■ **Next** – pressing **Next** automatically moves to the next logical stage of the measurement. For example, if the user has just completed a background measurement (and the **Pause between stages** check box is enabled), pressing **Next** takes them to the **Add Sample** tab.

■ **Accessory** – pressing this displays a dialogue allowing the user to control the dispersion unit (pump and stirrer speeds, etc.). The system automatically detects which dispersion unit and cell are fitted and changes this dialogue to suit.

■ **Help** – pressing this button displays help information.

■ **Close** – pressing this button closes the measurement window.

A manual measurement test

Try making a manual measurement using ordinary single dairy cream. Make sure the system is connected and powered up, then:

1. Select **Measure-Manual**. The software will automatically make an electrical and optical background measurement as well as aligning the optics.

2. Press the **Options** button to open the **Measurement Options** dialogue:
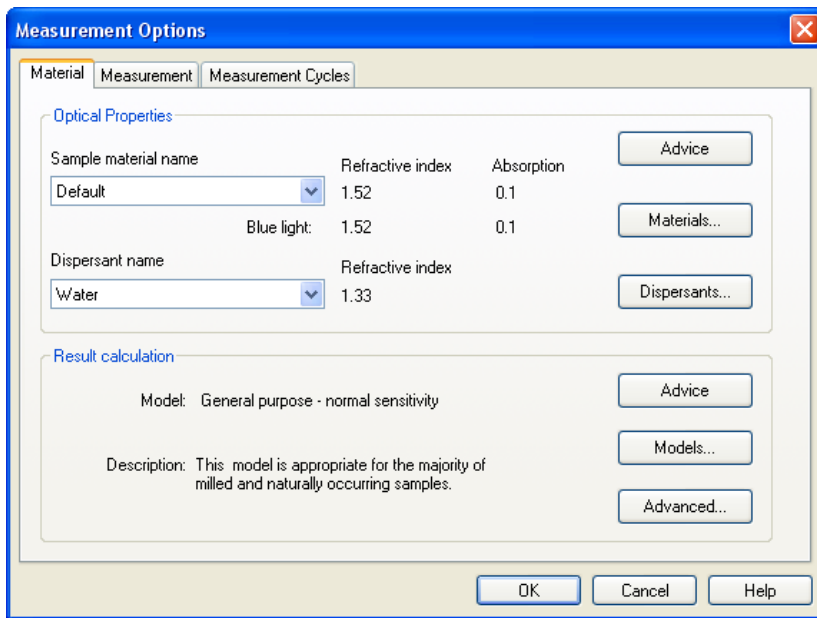
Select the **Material** tab to specify the materials measured. Select **Dairy cream** from the **Sample material name** box. (When measuring their own samples, the user would specify a new material by pressing the **Materials** button).

3. This sample is an aqueous emulsion so it should be dispersed in water. Select **Water** in the **Dispersant name** box.

4. Select the **Measurement** tab (shown below). Change both the **Sample measurement time** and **Background measurement time** boxes to 5 seconds.
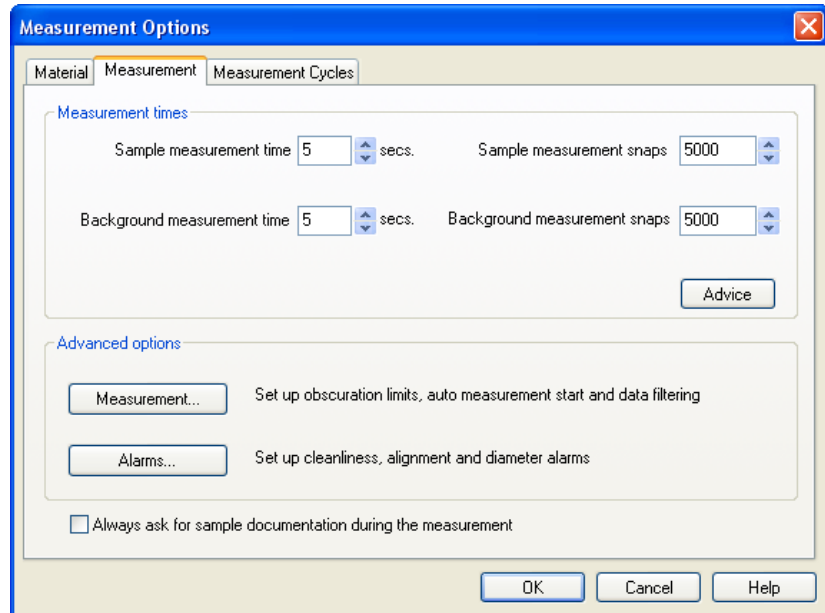
Select **OK**.

5. In the **Measurement Display** window press the **Next** button to add the sample. The display will change. Add one drop of the cream to the tank and wait for it to diffuse through the system. Add enough drops to make the blue **Laser Obscuration** bar move into the green area (try and achieve a mid value).

6. Once satisfied that enough sample has been added, press the **Start** button. The sample is measured and the result (size distribution) calculated. The measurement record is automatically saved to the records buffer.

7. At the end of the measurement a dialogue appears, asking if the user wants to make another measurement. Select **Finished measuring**. The **Measurement Display** disappears and the software returns to the main window.

8. The measurement is now complete. Click on the **Records** tab, select the record and then select one of the report tabs to view the results in detail.

Measurements on a Mastersizer 2000E

Instrument preparation

Before starting a measurement, ensure that the correct cell is installed in the cell area then power up the system as outlined below. (If an SOP is run, the software will check that the correct cell is connected, informing the user if it is not).

▶ **To power up the system:**

1. Switch on the dispersion unit(s).

2. Switch on the optical bench.

3. Switch on the computer and start the software by double-clicking on the Mastersizer 2000E icon.

Making a basic SOP measurement

Start an SOP by selecting **Measure-Start SOP**. In the dialogue which appears, select an SOP. The **Measurement Display** dialogue will appear:

ill 7753
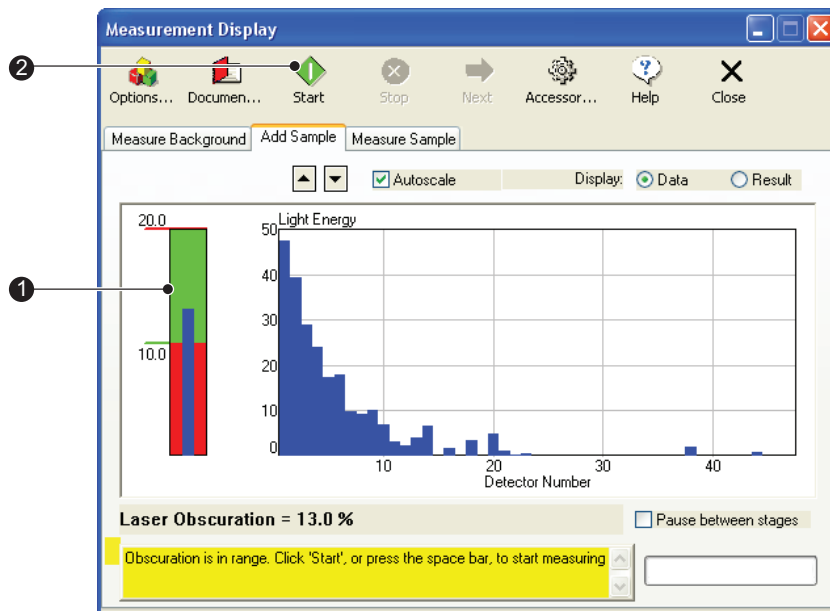
The status area at the bottom left of the dialogue always says what is going on in the measurement. The information in the dialogue will change depending on the stage of the measurement. The SOP measurement runs as automatically as possible. In a typical SOP measurement there are only a few things the user has to do:

1. Pump clean dispersant through the cell using the pump and stirrer controls on the dispersion unit, before starting the measurement.

2. Part way through a measurement the dialogue asks the user to add sample to the dispersion unit's tank. Depending on the SOP design, the user is asked to add a specific quantity to the tank or add sample to the tank until the obscuration bar ① is in the green section. Depending on the SOP design, the user may be asked to alter the dispersion unit controls.

3. Once the correct amount of sample has been added, press the **Start** button ② to start the actual measurement.

4. Depending on the SOP, a dialogue may appear asking the user to document the measurement (add comments and observations).

If specified, an SOP measurement will automatically measure the background, align the optics, calculate the size distribution, and save the record to a measurement file. All the user has to do is wait for a prompt from the SOP to perform the actions detailed above.

SOP summary

To summarise, an SOP measurement generally follows the steps indicated below. Make sure that the system is connected and powered up before starting.

1. Select **Measure-Start SOP**.

2. A browse dialogue appears; select an appropriate SOP file from this directory:
    `C:\Program files\Malvern Instruments\Mastersizer 2000E\SOP`

3. Select **OK**.

4. The SOP measurement asks the user to clean the cell and then pump in fresh dispersant using the dispersion unit controls.

5. Press **Start** when the system is clean and ready to start a measurement.

6. The SOP automatically measures an electrical background, aligns the optical system and measures the optical background.

7. The **Add Sample** tab appears and the SOP asks the user to add the sample to the tank. Add enough sample (add one drop at a time) to make the blue **Laser Obscuration** bar show green (try and achieve a mid value).

8. Once satisfied that enough sample has been added, press the **Start** button. The sample will be measured and the result (size distribution) will be calculated. The measurement record will automatically be saved to a measurement file.

9. The measurement is now complete - the user can now select the record and view the results by selecting one of the report tabs.

Making a basic manual measurement

To make a manual measurement select **Measure-Manual**. First fill the beaker with clean dispersant, then use the dispersion unit to pump the dispersant through the cell - this will be prompted by the software. The **Measurement Display** now appears and the software automatically makes an electrical background measurement, followed by an optical background measurement and alignment of the optics. It is worth spending some time to familiarise yourself with this display. The display is shown below and its main features identified.
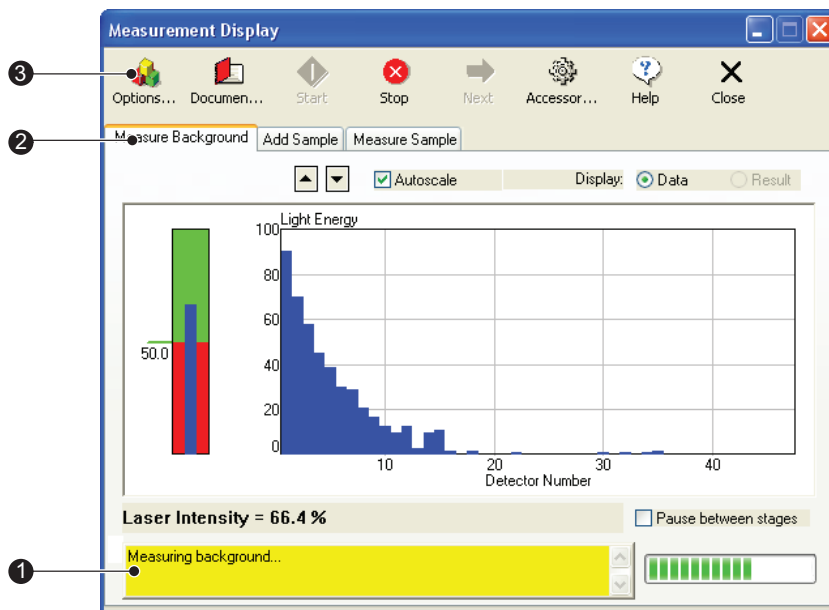
ill 7754

Its main features are:

① The status area at the bottom left of the dialogue reports what is going on. The information in the dialogue changes as the measurement progresses.

② The tabbed displays show information on the four stages of making a measurement:

- **Measuring a background** – the measurement data from a particle field is contaminated by background electrical noise and also by scattering data from dust on the optics and contaminants floating in the "clean" dispersant. The "measure background" facility makes a measurement of the system with only clean dispersant in as well as a measurement of the electrical background. This background information is subtracted from the sample measurement in order to "clean" the data.

A background measurement is automatically performed when a manual measurement is started.

- **Add Sample** – this tab shows how much sample has been added to the tank for measurement. The system measures how much sample is added by monitoring the "obscuration"of the beam caused by the sample being added to the dispersant. The obscuration is simply the fraction of light "lost" from the analyser beam when the sample is introduced. For example an obscuration of 30% means that 30% of the analyser beam has been lost by either scattering or absorption.

  The "obscuration bar" ① gives a visual indication of how much sample is added. If the bar is in the green zone then the concentration is in the correct range. If it is in the red then the concentration is out of range. The exact obscuration is given at the bottom left of the dialogue. The instrument has a wide range of concentrations that are acceptable and thus concentrations do not have to be precise.

  The target obscuration values can be set by the user; see **Chapter 9**.

- **Measure Sample** – this tab displays the scattering information during the actual measurement.

- **Result** – this tab is displayed when the software is calculating the size distribution.

③ The measurement control buttons allow the measurement process to be controlled and allow the user to document the measurement, control the dispersion unit and set up all the measurement parameters. The buttons are outlined below.

- **Options** – this button gives access to the measurement options. For example, the user can specify what sample and dispersant to measure, how the size distribution is to be calculated, and where to save the results.

- **Document** – pressing this button displays a dialogue where the user can document the measurement i.e. make notes on the pre-dispersion techniques, sample quantity, etc. This allows the measurement to be repeated at a later date.

- **Start** – starts a measurement process. For example, after adding the sample and once satisfied with the quantity, the user can start the measurement process by pressing **Start**.

- **Stop** – this button stops the current measurement process.

- **Next** – pressing **Next** automatically moves to the next logical stage of the measurement. For example, if the user has just completed a background measurement (and the **Pause between stages** check box is enabled), pressing **Next** takes them to the **Add Sample** tab.

■ **Accessory** – pressing this displays a dialogue allowing the user to control the dispersion unit (pump and stirrer speeds, etc.). The system automatically detects which dispersion unit and cell are fitted and changes this dialogue to suit.

■ **Help** – pressing this button displays help information.

■ **Close** – pressing this button closes the measurement window.

A manual measurement test

Try making a manual measurement using ordinary single dairy cream.

1. Make sure the system (optical bench and Hydro 2000MU) is connected and powered up.

2. Fill the sample beaker with clean dispersant and, using the dispersion unit controls, pump the dispersant through the cell.

3. Select **Measure-Manual**. The software will automatically make an electrical and optical background measurement as well as aligning the optics.

4. Press the **Options** button to open the **Measurement Options** dialogue:
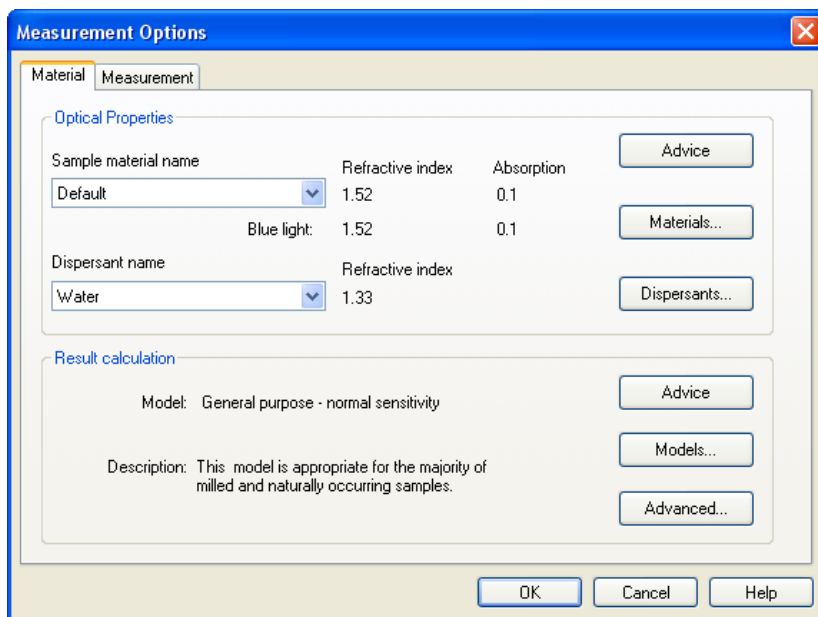
Select its **Material** tab and specify the material measured. Select **Dairy cream** from the **Sample material name** box. (For their own samples, the user would specify a new material by pressing the **Materials** button).

5. This sample is an aqueous emulsion so it should be dispersed in water. Select **Water** from the **Dispersant name** box.

6. Select the **Measurement** tab (shown below). Change both the **Sample measurement time** and **Background measurement time** boxes to 5 seconds.
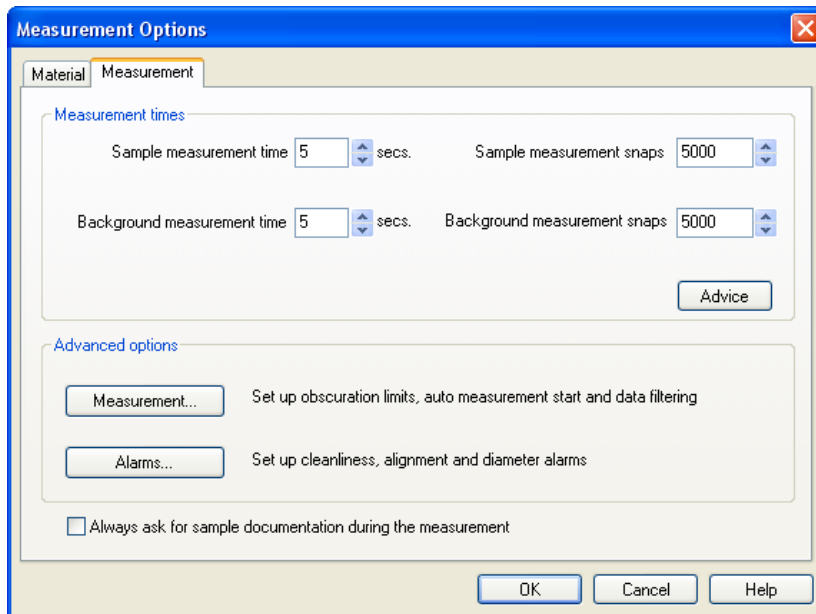
7. Use the controls of the dispersion unit to set the pump, stirrer and ultrasonics settings as appropriate (refer to the **Hydro 2000MU manual**).

8. Select **OK**. The status bar on the bottom left of the window always says what is happening and what to do next.

9. Press the **Next** button to add the sample. The display will change. Add one drop of the cream to the tank and wait for it to diffuse through the system. Add enough drops to make the **Laser Obscuration** bar move into the green area (try and achieve a mid value).

10. Once satisfied that enough sample has been added, press the **Start** button. The sample is measured and the result (size distribution) calculated. The measurement record is automatically saved to the records buffer.

11. At the end of the measurement a dialogue appears, asking if the user wants to make another measurement. Select **Finished measuring**. The **Measurement Display** disappears and the software returns to the main window.

12. The measurement is now complete. Click on the **Records** tab, select the record and then select one of the report tabs to view the results in detail.

Measuring new samples

The above procedures are fine if the SOP is set up or all the parameters for a manual measurement specified. For a new material, either create or modify an SOP or set up the options for a new manual measurement. This section outlines how to measure a new sample.

Measuring new samples: manual measurement

The basic procedure is the same as the demonstration outlined above. When the **Options** button in the **Measurement Display** is pressed, the **Measurement Options** dialogue appears. This dialogue controls all aspects of the measurement:

- The **Materials** tab allows the user to select a different material and dispersant or define the optical properties of new materials. It also allows the user to specify options regarding the result calculation.

Note

If a material is created or modified, the computer calculates a new scattering model. This usually takes one to 10 minutes, depending on the computer.

- The **Measurement** tab allows the user to set up measurement times and set measurement alarms, etc.

Press the **Document** button in the **Measurement Display** and use the **Document** dialogue to add relevant details that will allow users to repeat the measurement in future.

All the options available are basically the same as when setting up an SOP. For more details of these options see **Measurement options** later in this chapter.

Measuring new samples: SOP measurements

Creating an SOP is easy. The operator selects **Configure-New SOP** and the software runs through a **SOP Wizard**. The **SOP Wizard** asks a series of questions that combine to define a measurement, such as:

- what are the optical properties of the sample?

- what is it suspended in?

- how fast should the stirrer be?

- how much ultrasonic dispersion is required?

- do any additives need to be added to the sample to aid dispersion (and what quantity)?

- how will the result be calculated and displayed?

Anyone can create an SOP but typically it is the responsibility of the supervisor. An SOP can be created in one laboratory and then distributed to all Mastersizer 2000 users within an organisation. (All the sites must have the dispersion units the SOP refers to.)

Remember that no instrument has to be connected to the computer to write an SOP. The Malvern software can be installed on a remote computer and SOPs created and edited before they are tried out on an instrument. See the **Essentials Manual** for details on installing the software on another computer.

Editing an SOP

An existing SOP can be easily edited, either to correct that SOP or to create a new SOP based on it.

▶ **To edit an SOP:**

1. Select **Configure-Existing SOP**.

2. A tabbed dialogue appears, each tab corresponding to a **SOP Wizard** dialogue.

3. Select a tab and change any of the entries as required.

4. Select **OK** – the changes will be saved.

To create a new SOP based on an existing one, the user must first modify the existing SOP using **Configure-Edit SOP**. Once all the changes have been made, the **Save-As** dialogue will appear. Save the SOP under a different name

Deleting an SOP

An existing SOP can also be deleted using **Configure-Existing SOP.**

▶ **To delete an SOP:**

1. Select **Configure-Existing SOP**.

2. When the **Open** dialogue appears select the SOP to delete.

3. Right-click and select **Delete**.

4. When asked to confirm deletion of the file, select **Yes**.

Alternatively, use Windows Explorer to delete the SOP.

Measurement options

This section shows each of the dialogues that appear when the **SOP Wizard** is run and explains the basic options available. As stated above, the choices for the options of both manual and SOP measurements are basically the same; most information in this section applies equally to manual measurements.

More details on how to select these options can be found in the online help, accessed by pressing the **Help** or **Advice** button in any dialogue.

A series of dialogues, listed below, is displayed in the **SOP Wizard**. Identical tab versions of these dialogues are displayed during a manual measurement.

Sampler Selection dialogue

Use this dialogue to select a sample dispersion unit:
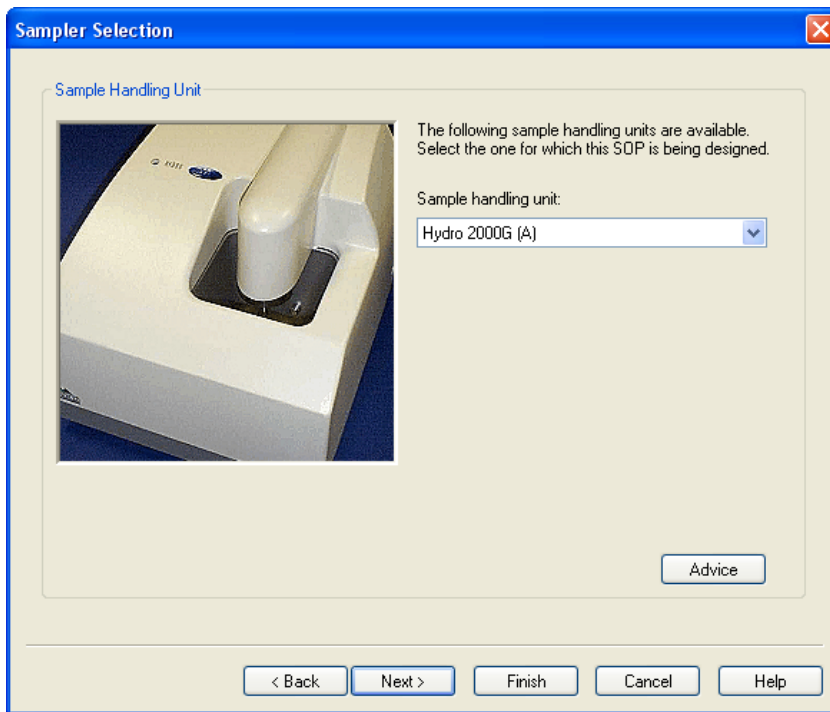
Up to two samplers (designated A and B) of each type can be selected, allowing for two different configurations. This may be appropriate, for example, if a specific sampler is dedicated to one sample material or dispersant. Clicking on the **Sample handling unit** list box displays a list of available samplers. Selecting one of these closes the box. For advice on the choice of sampler, click the **Advice** button.

All dispersion units are listed - even those that are not attached to the system. This allows an SOP to be written remotely from the system.

> **Note**
> The versions of the following dialogues actually seen may differ slightly, depending on the dispersion unit selected.

Material dialogue

This dialogue has two sections, **Optical properties** and **Result calculation**:

Optical properties

Use this section to define the optical properties of the material measured. The system has a large database of commonly used materials, from which a Frequently Used list may be created. The material and dispersant to be used in a measurement are selected from the frequently used sample/dispersant list boxes. The **Refractive Index** and **Absorption** of each item listed is displayed next to the list box. Changes to the frequently used lists can be made by clicking on the **Materials** or **Dispersants** buttons which open up the material/dispersant databases. This is also the area where additional materials can be defined or existing ones modified.

Materials and dispersants listed may have a small icon beside their entry in the lists:

- A red star – these items cannot be deleted or modified. They have special significance either because they are commonly used, such as water, or they have been generated in a special way, such as the Fraunhofer particle type.

- A small Malvern logo – these items can be deleted or modified but a warning is displayed first. Items marked in this way have been added by Malvern Instruments and are thought to have universal application. Examples are Polystyrene latex and Propan-2-ol.

The initial installation of the Mastersizer 2000 software includes other particle materials and dispersants not marked with the icons described above. These are examples used at Malvern Instruments; delete these if they are not appropriate.

Result calculation

The process of result calculation can be configured in a number of ways, depending on what is known about the sample. Clicking on the **Models** button enables selection of one of three models, **General purpose (default)**, **Multiple narrow modes** or **Single mode**.

Clicking on the **Advanced** button allows the selection of reduced ranges or specific result transformations. Click on the relevant **Advice** button for advice.

Note (Mastersizer 2000 only)
The Mastersizer 2000 uses red and blue light to measure samples. For certain materials (most notably certain inks and pigments) the sample has substantially different refractive indices in red and blue light. This is normally due to the material being highly absorbing in one of the two wave lengths. In these cases it is necessary to enter both refractive indices.

For most materials, there is no need to enter a blue refractive index. If a blue refractive index is not given, the Mastersizer assumes that it has the same value as the red refractive index. Blue refractive indices can be found from papers and textbooks in the same way as red refractive index information. When no information is available, determine the most appropriate choice of refractive index by looking at the fit and residual reports and comparing the results obtained with other observations, e.g. microscope images.

Labels dialogue

This dialogue allows an identifier for the sample to be added, uniquely identifying its source. This allows the sample to be traced in the future.

This dialogue also allows messages to be displayed for the operator before and/or after a measurement. For example, it may be necessary to give details on how to prepare the sample before a measurement starts, or how to dispose of it after the measurement.

Report/Saving dialogue

The **Report/Saving** dialogue can be set to allow the results to be printed once a measurement has been made.

Use the **Reports page** list box to select the report to use. If distributing the SOP to other users, ensure that they have the requested report installed.

This dialogue also allows results to be exported to a spreadsheet or other external application. See **Exporting results** in **Chapter 7** for more details.

Measurement dialogue

Use the **Measurement** dialogue to set the measurement and background times. Changing the measurement time automatically changes the **Measurement snaps** box (there are 1000 measurement snaps per second).
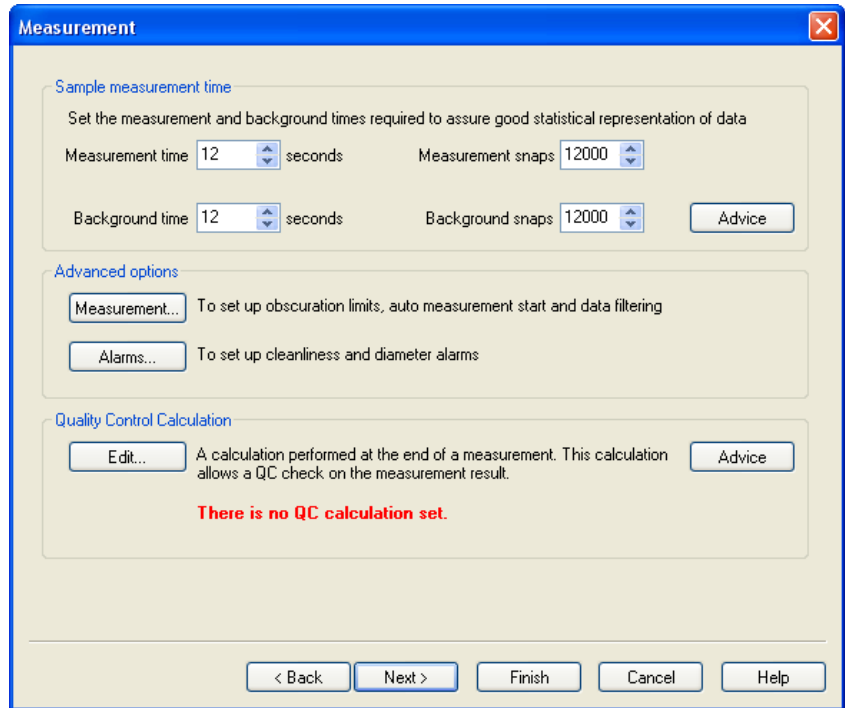
The optimum measurement time depends on the size of the sample, its particle size distribution and the dispersion unit used.

If a material is monomodal, its essential particle size characteristics can be captured in fewer snaps than a material with a broad particle size distribution which will need to be measured for longer to ensure that the coarser particles have had time to flow through the measurement beam.

This dialogue also gives access to some advanced features and alarms. See **Chapter 9** for more details.

Sampler Settings dialogue

The contents of this dialogue depend on which dispersion unit was selected in the **Sampler Selection** dialogue:

Use the dialogue to set all dispersion unit control settings. See the appropriate **dispersion unit manual** for more details.

Measurement Cycles dialogue

Use the **Measurement Cycles** dialogue to set up repeat measurements and set up tank cleaning and dispersant degassing. This diagram shows the features:

ill 7760
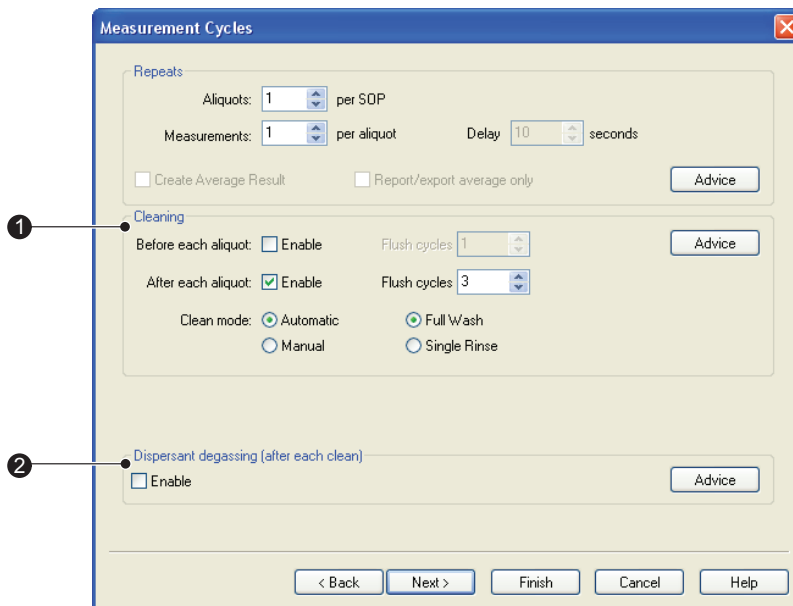
① Cleaning

It is possible to set up the SOP so that the tank and cell are cleaned together:

■ Before each aliquot.

■ After each aliquot.

■ Before **and** after each aliquot (select both check boxes).

Selecting the relevant **Enable** box causes a clean sequence to be performed.

If the sample/dispersant used is not normally removed with a single flush, enter a number in the **Flush cycles** box to run the flush sequence that number of times.

The user can set the **Clean mode** to either **Automatic** or **Manual**. **Manual** enables the user to clean the system easily using a beaker and a bottle of clean solvent. A series of prompts guides the user through the cleaning cycle.
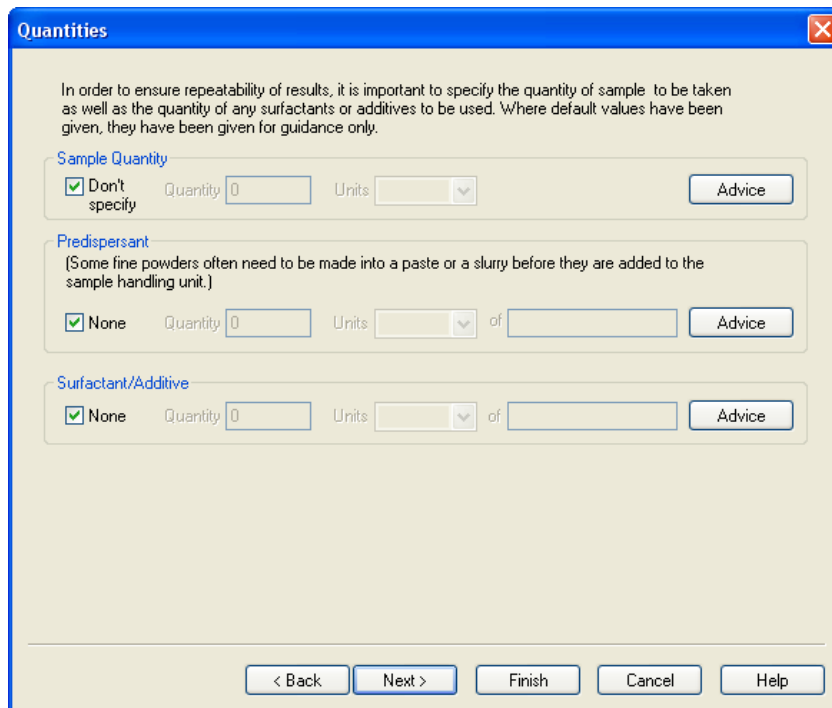
② Dispersant degassing

Selecting the **Enable** check box causes a degassing routine to be performed each time the tank is cleaned. The degassing routine uses ultrasonic action to help remove dissolved gases from the dispersant.

Quantities dialogue

Use this dialogue to define the quantities of sample/additives to be added during a measurement.

Viewing the results

Introduction

Once the result has been calculated, the data can be displayed in many forms. Each way of displaying the data is known as a report. Each type of report has a tab in the **Measurement window**; to view the data in another report simply select the appropriate tab.

This chapter covers:

■ Displaying the information – the reports provided and the use of the **Records** tab.

■ Using the **Report Designer** to create custom reports.

> **i** **Note**
> All the tabs in a **Measurement window** display the same measurement data but in different formats.

Malvern provides several default reports, identified by the "(M)" in their name, that will be sufficient for most users. The user can also create their own custom reports as required.

Generally a computer monitor displays information in a landscape format while a printer prints in a portrait format. Therefore, each report is split into two formats-one for displaying the report on the screen, and another configured for a printer. It is possible for the screen and print formats to be configured to report different information.

A report file has the extension **.pag**. The file holds both the screen and print varia-tions.

Chapter 6 gives details on interpreting the information in a report.

Displaying the information

Open a measurement file by selecting **File-Open**. Once a file has been selected, a **Measurement window** will appear.

A typical **Measurement window** is shown below. The information displayed for a measurement is controlled by the tabs. The first tab is always **Records**. This lists all the measurement records (individual measurements) that are held in that measurement file. The user must select a record before it can be displayed.

To view the record's data in a report, select the record then select a report tab.

Some reports allow the user to compare or display more than one record. For example, if the user selects more than one record in the **Records** tab and then selects the **Sample data report** tab, a graph will show the sample data for all selected records over-plotted on a single graph.

The format of the information in a report can often be changed by selecting an item and right-clicking the mouse. For example, selecting the table in a **Result Analysis** report and right-clicking lets the user change the report to a "Result-in %", "Result-over %" or "Result-under %", format (see **Chapter 6** for details of the reports). Changes to the graph and table format will be reflected in any printouts of the selected report.

Default reports

The default reports supplied by the software are the following.

Result Analysis report

This report displays the calculated particle size distribution for the selected record.

Parameter report

This report displays the measurement parameters for a record. It contains items such as sample details, measurement options, whether any alarms have been triggered, etc.

Sample fit report

This report shows how well the analysis data was fitted to the measurement data. It gives an indication of how well the optical properties for the material were described. For example, if the user had to predict the absorption value for a sample, a bad choice will result in large differences in the fit.

Sample data report

This report displays the basic measurement data. It shows the background signal measured and the actual measurement data. This report can be used to identify problems with measurement data. For example, dirty optics will result in a poor background signal (i.e. one with high values).

Result Difference report

This report compares two records and displays the difference. The first record must be set as the reference by selecting it, then right-clicking and selecting **Set reference result**.

Sieve reports (British standard and ASTM)

These two reports convert the measurement data so that they can be directly compared to the equivalent sieve standard.

Result Statistics report

This report displays various statistics comparing two or more measurements.

Fit report

This report shows how well the analysis data was fitted to the optical model.

Grade Efficiency report

This report shows grade efficiency data. The online help gives more information on this.

Changing the Records tab

By default the **Records** tab displays seven fields which include **Record number, Sample name**, **Source**, **Measured on**, etc.

The record tab can be customised by adding or removing any of the fields that are stored with the measurement record. The order in which the fields are displayed can also be rearranged.

Adding a field to the Records tab

There are two ways to add a field to the **Records** tab:

■   Using the **Show Fields** dialogue – this is accessed by selecting **View-Select Fields** (or right-clicking on the list header). The dialogue is split into two sides. The list box on the left shows all fields available that can be added to the records tab. The **Show these fields in this order** list shows all the fields that are currently displayed in the **Records** tab. The order in which they are listed is the order in which they appear in that tab.
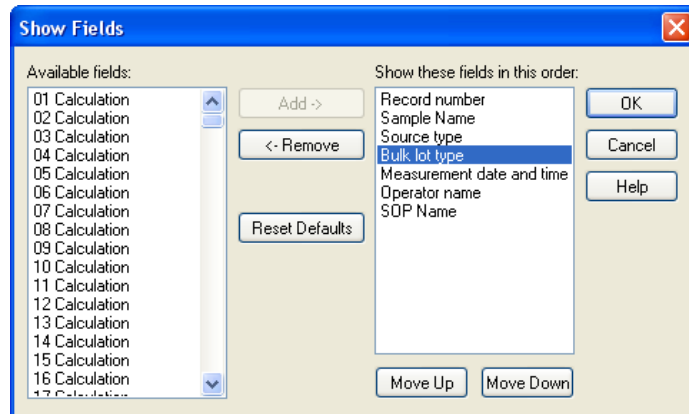
To add a field to the **Records** tab, select a field from the **Available fields** list box and press the **Add** button.

■   Using **Field Chooser** – this is a drag and drop facility that allows the user to select a field from a list and then drop it directly onto the records tab. The field chooser can be accessed by selecting **View-Field Chooser** (or right clicking on the list header).

Removing a record from the Records tab

There are two ways to remove a record from the **Records** tab:

- Using the **Show Fields** dialogue, select the field to remove from the right-hand list, then press the **Remove** button.

- Selecting the header of the field, dragging the header off the page and then releasing the mouse button.

Changing the order of records in the Records tab

There are two ways to rearrange the records in the **Records** tab:

- Using the **Show Fields** dialogue, select the field to move in the right-hand list, then use the **Move Up** and **Move Down** buttons to change its order.

- Directly from within the **Records** tab by selecting the header of the field and dragging it to a new position. A red arrow will show the insertion point. Be careful to only release the header while a red arrow is displayed, otherwise the field may be removed from the **Records** tab.

Changing views - the Report Designer

Using the **Report Designer**, custom reports can be designed by the user. These provide a standardised output for the result of a selected measurement record which can be tailored to the user's needs.

Two views must be edited due to the different aspect ratios of the printed page and the computer screen. The **Page Layout** shows the printed version and the **Screen Layout** shows the screen display. This allows addition of picture objects such as company logos to printed reports while not showing them on the screen display.

To add items to a report, use this **Tools** box or **Control Palette**:

Use the following buttons on the palette to add the listed elements to a report:

Button	Use this to add:
A	Text.
	Picture – bitmaps, GIFs, JPEGs, metafiles and icons.
X	Parameter (any parameter used by the Mastersizer system).
f(x)	Conditional expression – can be used to make pass/fail decisions.
$D_{x,y}$	Calculated quality parameter.
	Custom calculation.
	Grade efficiency parameter.
	Sample pot barcode.
	Graph – several types are available, as described below.
	Table – several types are available, as described below.

The toolbox can be moved anywhere on the screen. It can be switched on and off using **View-Control Palette**.

Opening a report

Select **Tools-Report Designer** to start the **Report Designer** application. Reports can be saved and opened using the menu options **File-Save** and **File-Open**. The default file extension is ⋆**.pag**.

A report must be saved in the ../**pages** directory if the report is to be listed in the report tabs of a measurement file.

Creating a report

To add an item, click on its button in the **Control Palette**. When a button is selected an area can be dragged on the report which will then contain the selected item.

Once objects are placed on the report screen they can be manoeuvred by selecting and dragging. They can also be reduced or expanded in size using the cursor and tag boxes on the selected object.

The **Report Designer** has many size and alignment tools, available from the tool-bar or using the **Layout** option on the menu.

The following functions operate on two or more selected objects. The last object selected becomes the reference object.

- Align left

- Align right

- Align top

- Align bottom

- Make same width

- Make same height

- Make same size

The following two functions can be used when two or more objects are overlaid. Using these functions, the selected object can be moved to the front or back of the overlay:

- Send to front

- Send to back

Objects can be selected individually by a left click on the mouse button, cumulatively by holding down the **Shift** key while clicking the mouse button or in a group

by "marquee selection", dragging a box over a group of items. If an item has been overlaid by another item and cannot be selected, use the tab key to step through all the items one-by-one until it appears.

Changing an item's properties

Set the properties of any added element by selecting it and double-clicking to open the **Properties** dialogue(s). The online help in the dialogues details how to use each one.

Graphs and reports

The font type and size of text within a report can be altered using the **Properties Box** for the selected item.

Graphs

Multiple data graphs can be added to a report. The following graph types can be added:

- **Data graph** – shows the data produced by the measurement. The data can be displayed in a number of ways which can be altered using the properties box.

- **Result Difference graph** – shows the difference in result between two or more measurement records. A reference record must first be selected.

- **Result graph** – shows the particle size result of the measurement. It can be displayed in different ways using its properties box.

- **Statistics graph** – shows statistics of the result. The parameters available are mean, standard deviation, maximum and minimum.

- **Trend graph** – allow the measurement data from multiple records to be compared to investigate any trends in the information.

- **Grade efficiency graph** – grade efficiency curves created from particle sizing results are used to establish the efficiency of separators such as hydrocyclones, sedimenting centrifuges or gravitational clarifiers.

The properties of a graph can be altered using its **Properties Box**. The properties are:

- **Colour** – the colour of the background and/or grid can be modified using the colour sets available. A custom colour can also be created.

- **Axes** – the axes of the graph can be modified. The X and Y axes have optional grid lines. The Y axis can be set to autoscale.

- **Display** – the display can be set to show combinations of data, background and fit. The display can be shown as a histogram or curve.

Selecting the **graph tips** check box allows the setting of pop up tips (flags showing data points on the graph). These appear on the report itself (not on the screen shown in the **Report Designer**).

Selecting the **legend** check box places a summary table, showing date and name, at the foot of each graph.

Tables

A number of data tables can be added to the report. The data is taken from the current selected record or, for the **Difference** and **Statistics** tables, group of selected records.

The following data tables can be added:

- **Data** – displays the raw data produced by the system.

- **Difference** – shows the difference between the results in a reference measurement record and the currently selected measurement record.

- **Data Fit** – gives a table showing the fit. This shows the difference between the actual light energy distribution measured for a sample and the theoretical prediction of the light energy distribution according to the Mie or Fraunhofer scattering model selected for that material.

  The closeness of the fit is an indicator of the correctness of the light scattering data used and the quality of the measurement (cleanliness of the system, etc.).

- **Result** – shows the raw result data.

- **Statistics** – shows the mean, standard deviation, maximum and minimum of a group of selected measurement records. The statistic shown on the table can be altered using the **Properties** box.

- **Sieve** – presents the data from the current measurement record as it would be displayed if a stack of sieves had been used for the measurement. A number of standard sieves can be selected using the **Properties** box.

- **Phi** – soil scientists often use this size classification to present size data. This phi notation is based on a power of two series where the class boundaries are labelled by the index.

- **Grade efficiency** – grade efficiency curves created from particle sizing results are used to establish the efficiency of separators such as hydrocyclones, sedimenting centrifuges or gravitational clarifiers.

The table's colour, font and number of rows and columns can be altered using its **Properties** box.

Understanding
the views

Introduction

This chapter gives guidelines on the interpretation of the results from the Master-sizer. It covers:

- Understanding the display – runs through some of the terms and expressions used in the standard views.

- Report features – explains the statistics which reports show.

- Fundamental concepts – explains some important concepts the user should understand before proceeding. These include:

    - How the concentration (Cv) is calculated.

    - Use of volume-based results.

    - How the result is expressed in terms of equivalent spheres.

    - How the distribution parameters are derived.

- Optical models – contrasts the Fraunhofer approximation used in some instru-ments with the Mie theory which underpins Mastersizer 2000 operation

Understanding the display

The result from the analysis is the relative distribution of volume of particles in the range of size classes. From this basic result the statistics of the distribution are calculated. The result is also interpolated so that the result at points in between the size class boundaries is available. The cumulative undersize and oversize, frequency curves and histograms are obtained from the interpolated result.

Once the data has been analysed, the information can be displayed in various ways. Usually the display shows a graph of the result or a table showing the same information in a tabular form. This diagram shows four of the more common graph types for displaying the result:
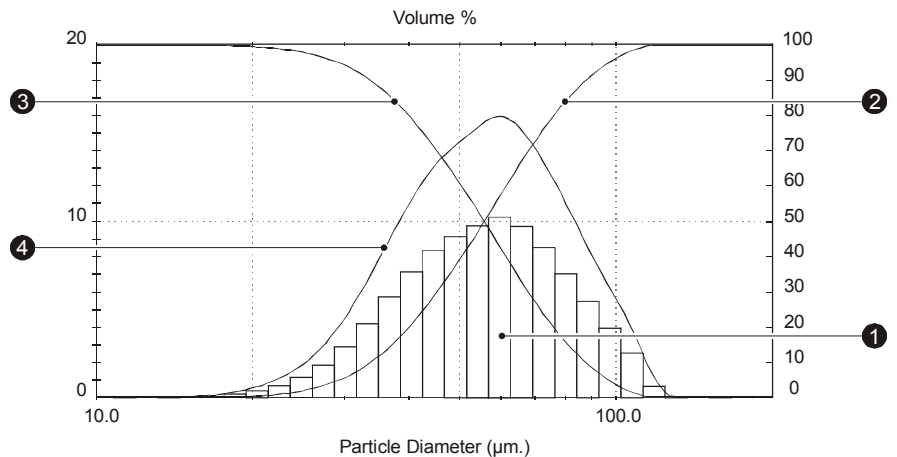
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① **Histogram** – displays the result in the form of "in band" percentages. Each bar in the graph represents a size band of particles (52 - 59 microns for example) and the height of the bar represents the percentage of the sample that is within that band. Read the percentage from the left-hand axis. Unless the size bands are changed (see **Chapter 9**), the initial analysis uses the size bands that are set by the physical design of the detector.

② **Result-under plot** (also known as **cumulative undersize** or **result less than**) – displays the result in the form of "% of sample below a certain size of particle". For example, by reading the values from the graph (right-hand axis) the user may be able to determine that 10% of the sample is under 23 microns.

The exact value can be read from the table accompanying the graph or using a graph tip (position the mouse pointer over the graph and a tooltip appears, showing the size data). The result-under plot is calculated from the initial size bands by fitting a curve to the analysis data so that values within a size band can be read.

③ **Result-over plot** (also known as "cumulative oversize") – similar to the result-under plot except that the result is in the form "% of sample above a certain size of particle". For example by reading the values from the graph (right-hand axis), the user may be able to determine that 90% of the sample is above 23 microns.

④ **Frequency curve** – this is calculated by differentiating the result-under/ cumulative undersize curve. The frequency curve is particularly useful for displaying the results to show the "modes" or peaks in the graph.

The peak of the frequency curve gives the modal diameter, the most commonly occurring particle diameter. Note that the frequency curve is scaled to be approximately the same height as the analysis size band histogram.

Several peaks in the graph indicate that there are distinct sizes of particles within the sample. This "at a glance" inspection of the results is difficult if the result is shown as a cumulative plot.

Another use for frequency curves is to compare results from different measurements. Other graph types can be over-plotted but the results may be confusing.

Report features

A typical report is shown below, followed by a brief description of the key features:.

RESULT ANALYSIS

Sample Name: Glass beads
Sample Source & type: Supplier = Malvern
Sample bulk lot ref: 1

SOP Name: Glass beads (typical)
Measured by: A. Operator
Result Source: Measurement

Measured: 28 January 1999 10:10:10
Analysed: 09 September 1999 10:00:20

Particle Name: Glass beads (typical)
Particle RI: 1.520
Dispersant Name: Water

Accessory Name: Hydro 2000G (A)
Absorption: 0
Dispersant RI: 1.330

Analysis model: General purpose
Size range: 0.020 to 2000.000 um
Weighted Residual: 0.876 %

Sensitivity: Enhanced
Obscuration: 7.47 %
Result Emulation: Off

Concentration: 0.1889 %Vol
Span: 0.651
Uniformity: 0.206
Result units: Volume

Specific Surface Area: 0.0143 m²/g
Surface Weighted Mean D[3,2]: 171.372 um
Vol. Weighted Mean D[4,3]: 182.042 um

d(0.1): 128.404 um   **d(0.5):** 176.273 um   **d(0.9):** 243.215 um

Particle Size Distribution — Volume (%) vs Particle Size (µm)

Legend: Glass beads, 28 January 1999 10:10:10

Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.010	0.00	0.363	0.00	13.183	0.00	478.630	0.00
0.011	0.00	0.417	0.00	15.136	0.00	549.541	0.00
0.013	0.00	0.479	0.00	17.378	0.00	630.957	0.00
0.015	0.00	0.550	0.00	19.953	0.00	724.436	0.00
0.017	0.00	0.631	0.00	22.909	0.00	831.764	0.00
0.020	0.00	0.724	0.00	26.303	0.00	954.993	0.00
0.023	0.00	0.832	0.00	30.200	0.00	1096.478	0.00
0.026	0.00	0.955	0.00	34.674	0.00	1259.925	0.00
0.030	0.00	1.096	0.00	39.811	0.00	1445.440	0.00
0.035	0.00	1.259	0.00	45.709	0.00	1659.587	0.00
0.040	0.00	1.445	0.00	52.481	0.00	1905.461	0.00
0.046	0.00	1.660	0.00	60.256	0.00	2187.762	0.00
0.052	0.00	1.905	0.00	69.183	0.00	2511.886	0.00
0.060	0.00	2.188	0.00	79.433	0.11	2884.032	0.00
0.069	0.00	2.512	0.00	91.201	0.97	3311.311	0.00
0.079	0.00	2.884	0.00	104.713	4.92	3801.894	0.00
0.091	0.00	3.311	0.00	120.226	9.85	4365.158	0.00
0.105	0.00	3.802	0.00	138.038	17.84	5011.872	0.00
0.120	0.00	4.365	0.00	158.489	21.27	5754.399	0.00
0.138	0.00	5.012	0.00	181.970	20.04	6606.934	0.00
0.158	0.00	5.754	0.00	208.930	14.15	7585.776	0.00
0.182	0.00	6.607	0.00	239.883	7.38	8709.636	0.00
0.209	0.00	7.586	0.00	275.423	2.89	10000.000	0.00
0.240	0.00	8.710	0.00	316.228	0.55		
0.275	0.00	10.000	0.00	363.078	0.04		
0.316	0.00	11.482	0.00	416.869	0.00		
0.363	0.00	13.183	0.00	478.630	0.00		

Operator notes:
Glass beads added directly to tank

ill 7755

① The **residual** is 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.

② 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.

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

D(v, 0.5), D(v, 0.1) and D(v, 0.9) are standard **percentile** readings from the analysis.

- **D(v, 0.5)** is 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 ν in the expression **D(v, 0.5)** shows that this refers to the volume distribution. This can be replaced by **s** for surface, **l** for length or **n** for number distributions.

- **D(v, 0.1)** is the size of particle below which 10% of the sample lies.

- **D(v, 0.9)** is the size of particle below which 90% of the sample lies.

③ **D[4,3]** is the **Volume Weighted Mean** or Mass Moment Mean Diameter.

④ **D[3,2]** is the **Surface Weighted Mean**, also known as the Surface Area Moment Mean Diameter or Sauter mean.

⑤ The **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 **x** is replaced by any of the letters **v**, **s**, **l** or **n** that define the distribution type.

⑥ **Concentration** – the volume concentration. This is calculated using Beer-Lambert's law. For full details see the following section.

⑦ **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:

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

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 x Ob.

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

⑧ **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 of, and result in, size class i.

⑩ **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 (on the **SOP Wizard**'s **Material** dialogue). 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.

■ 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:

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

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.

- 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 μm and b in mm, and v 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.

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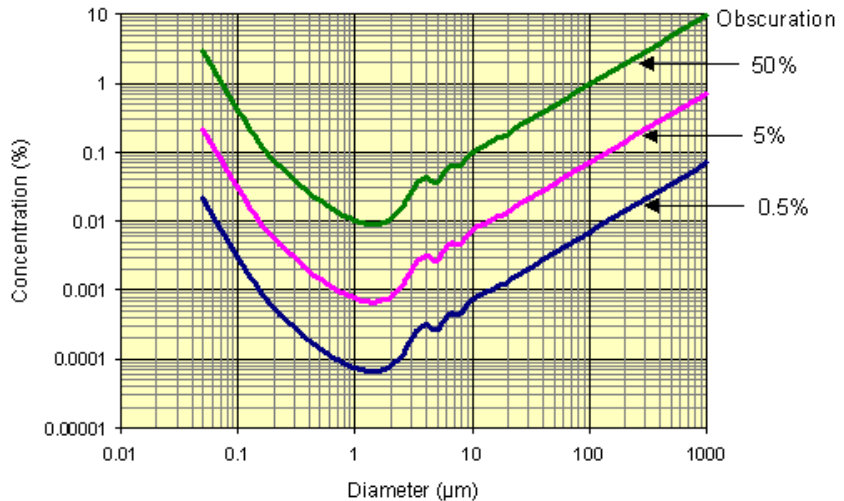
In the more realistic situation of a polydisperse (or multimodal) sample it is not possible to provide simple graphical correlations of volume concentrations and obscuration. The system calculates concentration, however, using the size distribution result and the obscuration.

For a polydispersed 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 in the size category 6.97-7.75 microns, this means the total **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:
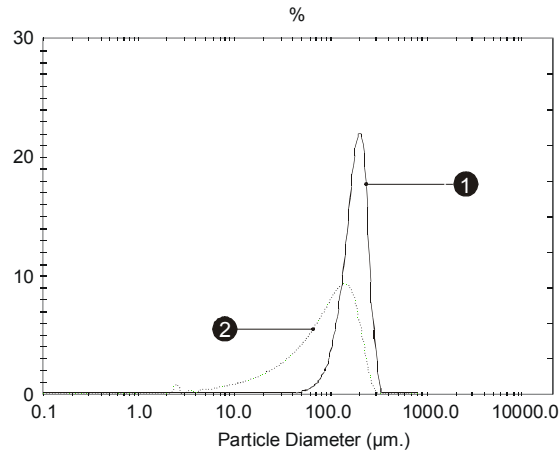
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Equivalent spheres

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 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 x 25 x 10mm $= 12500mm^3$. 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:

$$V = \pi(10\mu m)^2(60\mu m)$$
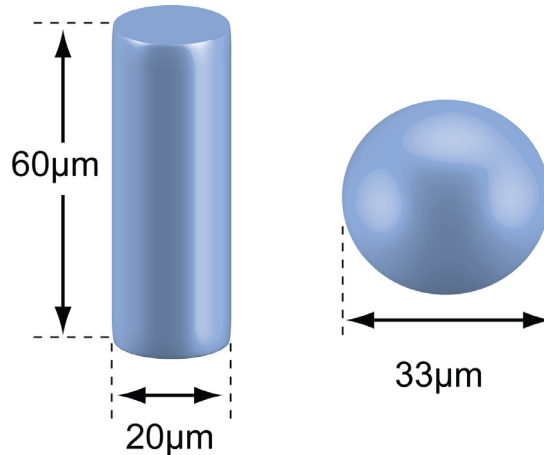
The sphere of equivalent volume would have a diameter centred on:

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

with a spread from 20 to 60mm.

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

If there's a need to correlate laser diffraction results with values from some other technique, consider applying a shape correction using the **Result Modification** procedure built into the Malvern software.

Derived distribution parameters

The third point is that the analysed distribution is expressed in a set of size classes which are optimised 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:

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

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

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

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.

Optical models

This section contrasts the Fraunhofer approximation used in some instruments with the Mie theory which underpins Mastersizer 2000 operation.

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 (25μm 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 2000 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 particles is available for selection in the SOP (presented in the first **Analysis** dialogue). More can be defined by a user, but the following parameters must be specified accurately:

- **Refractive Index (Real)** – describes the amount of scattering that takes place as a result of light interacting with the particle.

- **Refractive Index (Imaginary)** – the imaginary or complex refractive index. This describes the amount of absorption that takes place as the light enters the particle.

- **Density** – the density in g/cm^3. 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 **Measurement Parameters** view indicates how well the calculated data fitted the measurement data.

Refractive index of medium

This value is needed by both the Fraunhofer approximation and the Mie theory.

Handling measurement files

Introduction

This chapter outlines the storage, sorting and exporting of measurement data. It covers:

- Records and files.

- Searching and sorting records.

- Exporting results – how to export information to other applications.

- Extracting an SOP.

Records and files

The fundamental unit of storage is the "record". A record contains all the information for an individual measurement, including the original measurement data and all parameters that have been set to calculate the size distribution.

All records are stored in a measurement file which may contain one or more records.

As a practical demonstration, consider a cement manufacturer who is asked to measure a sample from the production line every hour for five hours. They may wish to save the measurement data in a measurement file called **Cement.mea** (measurement files always have the extension **.mea**). When they make the first measurement they can save the result as a record (possibly called "Hour1") in the **Cement.Mea** measurement file.

Once all measurements are completed the user will have a measurement file with the following structure:

```
Cement.Mea
Hour 1
Hour 2
Hour 3
Hour 4
Hour 5
```

Alternatively they could have created five measurement files that each contain one record.

A record can be deleted but it can never be altered. However, a record can be re-analysed and the resultant record saved under a new record name.

For example, imagine a user who has a record called **Record1** and has found out that the refractive index they set for the dispersant is incorrect. They can select **Record1**, edit the parameters and re-analyse the result. A new record will be created (**Record2**) that is based on **Record1**. **Record1** will still exist unaltered.

▶ **To re-analyse a record:**

1. Select the record to re-analyse.

2. Select **Edit-Result** (or right-click and select **Edit result**).

3. A dialogue appears allowing changes to the parameters.

4. Make the changes and select **OK**.

Searching and sorting records

The software has powerful search capabilities that allow users to find records within measurement files that match a search criterion.

Records in a measurement file can also be sorted into alphanumeric order by selecting a field header in the **Records** tab.

Searching records

Users can search many measurement files to find records matching a set of search criteria they define. Any parameter contained in the file can be a search clue. For example, it is possible to search all measurement files for records measured after a certain date that contain a D(v, 0.5) value of more that 50 microns.

▶ **To search measurement files:**

1. Select **Tools-Find measurements** to open this dialogue:
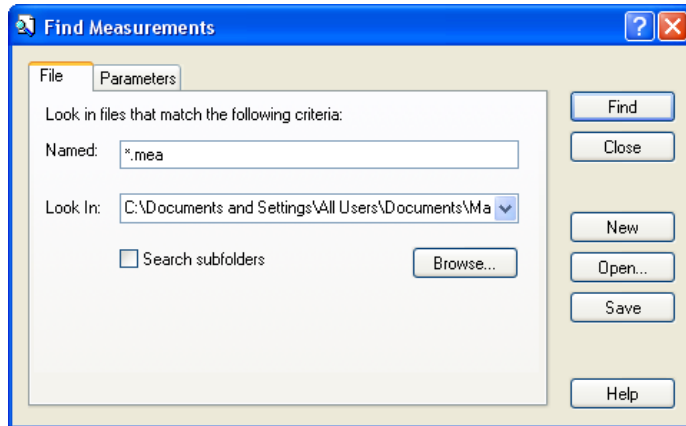
2. Use the **Files** tab to specify which measurement files to search. Use the **Browse** button to select a directory which contains the measurement files (enabling the **Search subfolders** check box extends the search to all sub-directories of this directory).

   In the **Named** box specify the name of the measurement files to be searched. By default this is set to ★.**mea**, which will search for all measurement files in the selected directory. Wild cards can be used to narrow down the search. For example selecting "Titanium★.mea" will search for all measurement files beginning with "Titanium".

3. Select the **Parameters** tab to specify the search criteria.

ill 7756
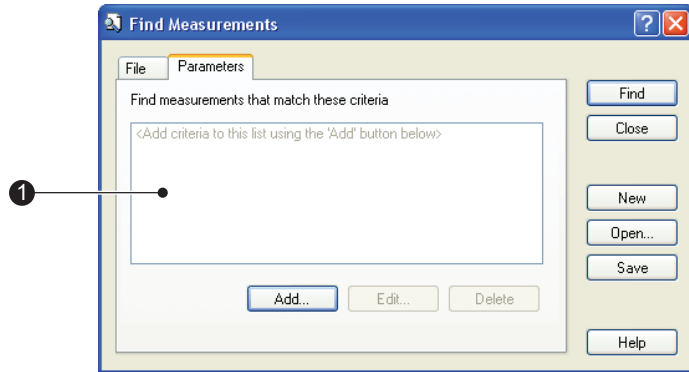
The main list box ① displays all the search criteria that have been set (when starting a new search this will be blank). To add search criteria to the list, select the **Add** button. More than one criterion can be specified. When the search is initiated all records that match all search criteria are displayed.

4. Selecting the **Add** button displays this dialogue:
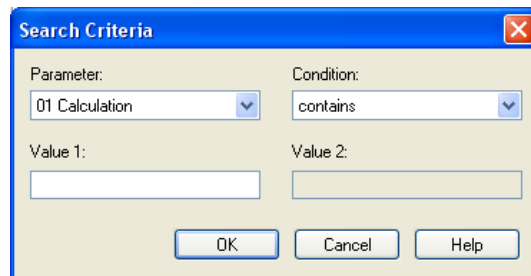
The **Parameter** list box lists all parameters available in the measurement file. Select one and use the **Condition** and **Value** list boxes to set the conditions of the search. Select **OK** to add the search criterion to the list box in the previous dialogue.

A single search criterion can be altered or deleted by selecting the **Edit** and **Delete** buttons in the **Find Measurements** dialogue.

5. Start the search by pressing the **Find** button. Once the search is completed, all records found will be displayed in a window similar to the normal **Records** tab, except that all the headers for the fields of the records will change to reflect the parameters set in the search criteria.

> **Note**
> If a large number of records are found in the search, it may take some time to display the search results as all measurement files must first be loaded.

The results of the search combination can be saved. Saving consolidates all the records found into a single measurement file, effectively making a copy of each record from its original measurement file.

Reusing search criteria

The search criteria can also be saved so that they can be opened and used at a later date. This is governed by these search buttons:

- **New search** - clears all criteria currently set.

- **Open search** - opens a previously saved search.

- **Save search** - saves the current search criteria to a new file.

Sorting records

Records in a measurement file can be sorted into alphanumeric order. From a measurement file window select the **Records** tab. Selecting any of the field headers will rearrange all records into alpha-numeric order based on that field. For example, selecting the **Measured on** header lists all records into numeric **Measured on** date order.

Exporting results

There are several ways to export information from the Malvern application to other applications. These are:

■ One or more records can be selected in the **Results** tab and "Dragged and dropped" directly into another application (provided that the application supports tab separated formats e.g. Excel).

■ A table or graph can be dragged and dropped into another application by selecting the item, then dragging while holding the **Ctrl** key.

■ A table or graph can also be copied by selecting the item then selecting **Edit-Copy Table** or **Edit-Copy Graph**. The item can then be pasted into another application.

■ Any of the parameters from one or more records can be exported as a text file. This file can then be loaded into a word processor or spreadsheet. A template is used to specify which parameters are exported. Use the **Configure-Data export templates** command to create and modify templates.

■ A measurement file can be attached to an email. Use the command **File-Send**, which opens the computer's default mail program, and attach the file.

Exporting results

▶ **To export a record:**

1. Select one or more records from a measurement file.

2. Select **File-Export data**. This dialogue will appear:
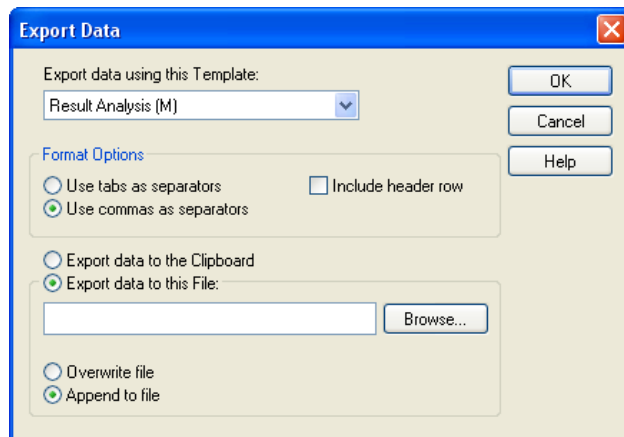
3. Select a template from the **Export data using this Template** list box.

4. Use the **Format Options** section to specify the separator character used between fields. Different spreadsheet packages require different field separation characters to correctly parse the data output. Commas are the most usual but these should not be used if they are numerical separators in the country where the instrument is to be used. (e.g. France).

The data can be exported to a file or directly to the clipboard by selecting the appropriate radio button. To use a file, **Browse** for it.

An alternative way to export data is to select one or more records and simply drag the records to another application and then drop them onto a page. This method uses the template specified in the **Default Template** list box in the **Data Export Templates** dialogue. This dialogue can be accessed by selecting **Configure-Data export templates**.

Creating an export template

New export templates can be created (or existing ones modified) by selecting **Configure-Data export templates** to open this dialogue:
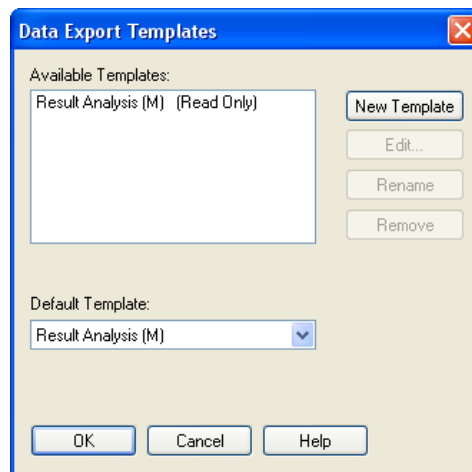
Selecting **New Template** adds a new template name in the **Available Templates** list and then gives the user the option of changing the template's name. (The template name can be changed later by pressing the **Rename** button.)

Having created it, set the template's parameters by pressing the **Edit..** button to open this dialogue:
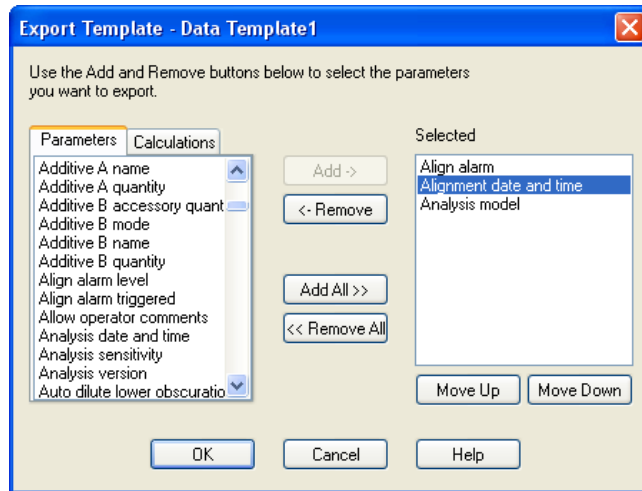
The **Parameters** list box displays all parameters available in a measurement record. The **Selected** list box displays all parameters that are to be included in the template. Add or remove parameters from this list by using the **Add** and **Remove** buttons.

To change the order in which the parameters are displayed in the **Selected** list, select a parameter then press the **Move up** or **Move Down** buttons as appropriate.

A standard template **Result Analysis(M)** is supplied that exports all fields of the standard report page **Result Analysis(M)**.

Extracting an SOP

The SOP settings used to produce a measurement can be extracted for reuse.

▶ **To extract an SOP:**

   1. Select a record in the measurement file.

   2. Either right-click on the record and select **Extract** in the menu this displays, or select **Edit-Extract SOP**.

   3. The **Extracted SOP editor** dialogue opens, showing the SOP or manual measurement parameters used to make the original measurement.

   4. Make any required changes to the settings and then click **OK**.

   5. Specify the SOP file to save the changes to as a new SOP.

Sample preparation

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 bad 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.

Sample preparation flow chart

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

SAMPLE

```
Take Representative Sample
(mix well or riffle if dry powder)
```

Yes Does it disperse in water? **No**

```
Ultrasound if
necessary
```

Analyse

Yes Does it float? **No**

```
Try Surfactant
```

No Does Ultrasound work? **Yes**

Analyse

Yes Does this disperse it? **No**

```
Ultrasound if
necessary
```

Analyse

```
Try Solvent

e.g. Ethanol
Propan-2-ol (IPA)
Methanol
Acetone
Butanone (Methyl Ethyl
Ketone)
Hexane
Toluene
Dimethyl Digol
```

```
Ultrasound if
necessary
```

Analyse

ill 7825

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.

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.

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**.

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.

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 **Sample Dispersion and Refractive Index guide** gives refractive index information for many more dispersants.

The cost of some of the organic dispersants may limit their use to the Hydro 2000S, which typically only uses 150ml 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.

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 by removing the surface charge effects on the sample that cause it to float on the surface or clump together.

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. Anti-foaming 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 aid dispersion by modifying the properties of the dispersant that are responsible for the problem. 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

- 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.

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 switching off the unit briefly to allow trapped air to escape the system. 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.

With the Hydro 2000G/S the software can remove bubbles and dissolved gases from the system using the degas facility. This pulses the ultrasonics for a period of time after the tank is cleaned and filled with fresh dispersant. To use it, press the **De-Gas** button in the **Configure-Accessory** dialogue or select the **Dispersant degassing** check box in the **SOP Wizard**'s **Cycles** tab.

> **Note**
> Software degassing is a last resort in removing bubbles and dissolved gases. 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 readings.	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/stirrer speed.
Sample swells in dispersant	Obscuration decreases.	Try another dispersant.
Particles sticks to the windows	Rapidly increasing obscuration.	Use an admixture.
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.

9

Advanced features

Introduction

This chapter covers features experienced users can use to improve their analyses:

- Obscuration settings – limits can be set, enabling the system to detect poor conditions and take action if a limit is exceeded.

- Advanced result processing – the result can be modified by performing a result transformation or reducing the result range before displaying it in a graph or table.

- Editing results – data can be re-analysed using different parameters and the result saved as a new record.

- Defining user size bands – customising the particles size ranges used for columns in histograms and tables.

- Alarms – users can set three alarms that monitor various measurement states. These are the cleanliness alarm, alignment alarm and diameter alarms.

- Averaging records – creates an average record from several records (usually repeats of the same sample).

- Importing results from older instruments.

- Result emulation – a technique that allows a Mastersizer result to be modified to emulate a result from an alternative particle sizing technique.

Obscuration settings

The obscuration is a measure of how much sample is in the beam at any one time. If this is too high multiple scattering may occur, if it's too low insufficient signal will be detected and precision lost. Setting limits enables the system to detect poor conditions and take action either to ignore data, raise alarms or trigger the measurement process once a limit has been exceeded. Values can be set manually or default values for the selected sample handler can be enabled.

Obscuration settings can be used when measuring wet or dry samples. However, obscuration settings are the **preferred** method for making dry measurements.

If using the **SOP Wizard**, click on the **Measurement…** button on the **Measurement** dialogue, as described in the appropriate **dispersion unit manual**.

▶ **To change the obscuration settings:**

1. For a manual measurement, click the **Options** button then click on the **Measurement…** button from the **Measurement** tab. This dialogue will appear:

2. Set obscuration limits by typing the desired limits into the **Lower limit** and **Upper limit** boxes. Selecting the **Default** check box will return the limits to the system defaults.

The lower limit is used mainly as a means of triggering measurements, particularly dry powder measurements. The practical upper limit for wet dispersions varies depending on the particle size of the sample.

3. Click on the **Raise alarm if the final obscuration is out of range** check box to display an alarm if the measurement's final obscuration is out of range.

Guidelines

Use a maximum obscuration of about 5% for dry measurements (or even lower for fine dry material).

For other samples, typically add enough sample to give the following obscurations:

■ 10% obscuration – for very fine material (up to 3 or 4 microns). Because the material is fine, there will be little problem in obtaining good sample representation but there is some risk of multiple scattering.

■ 15% obscuration – if the material has a median size of 50 microns or greater.

■ 20% obscuration – if the material has a broad distribution or is coarse. Here achieving good sample representation is the most important consideration.

The required obscuration can be determined experimentally by measuring the particle size at different obscurations. At low obscurations the result reproducibility may be poor due to poor sampling or signal-to-noise issues. At very high obscurations the calculated result may shift to smaller particle sizes due to the effects of multiple scattering. Obtain the ideal obscuration by finding the range where the result reproducibility is good and the results are independent of the obscuration chosen.

In each case, the amount of sample which was required to achieve the recommended levels of obscuration should be noted and specified in the SOP.

Autostart

Sometimes it is necessary to wait for the sample flow to stabilise before making a measurement, particularly in dry measurements, to avoid errors due to incomplete dispersion. Obscuration limits can be set up manually or using default values for the chosen sampler. Once the sample obscuration has stabilised within these limits for the specified time, the measurement is started.

Obscuration filtering

Obscuration filtering rejects data that does not fall within the set limits. To enable it click on the **Only include measurements….** check box and enter a **Time-out period**. Measurement will stop if the required number of snaps has not been made in this time-out period. The software records the number of snaps measured and this figure can be used in reports to indicate the completeness of a measurement. For example, a report can be configured to say "1750 of 2000 snaps measured".

This facility is particularly useful where the sample concentration is liable to some degree of fluctuation, as with some dry powder measurements. For dry powder measurements practical limits would be between 0.5% and 6%.

In case a totally inappropriate feed rate is set, so that the obscuration seldom (if ever) falls within the desired limits, a time-out is provided to allow the user to exit the measurement. This should not be set so long as to give the measurement the appearance of having "hung up", but sufficiently long to allow a fair chance of sample of the required concentration passing through the measurement cell.

Clicking on the **Raise alarm if time out period expires** check box will advise how many good snaps were collected in the event of a time-out, and offers the option of using them as the basis for the measurement

Autodilution

Autodilution allows the sample concentration to be automatically adjusted by adding more dispersant. This feature is not available in manual measurements. Good Laboratory Practice dictates that autodilution is an inferior procedure to the addition of a correctly predetermined sample quantity.

If operational requirements make the use of autodilution necessary, run a test to ensure that no modification of the particle size distribution is caused by the procedure.

A typical test is to perform a manual measurement of the sample at a higher obscuration than required, say 20%, and then run the sample at the same starting obscuration using autodilution with the target obscuration threshold set to a lower value than the ideal, say 5%.

If the two sets of results show a high level of comparability, autodilution is an acceptable procedure. Users who are required to conform to Good Laboratory Practice should keep the results of this test on file.

As a general rule, emulsions and fine suspensions (0.1 to 10 microns) will be suitable candidates for autodilution. Extreme caution must be exercised with materials having particle size distributions broader than this as fines could be lost and results biased.

Advanced result processing

The result of an analysis can be modified by performing a result transformation or reducing the result range before it is displayed in a graph or table.

Reducing the result range

The standard result range is:

- For the Mastersizer 2000 – 0.02 to 2000 microns.

- For the Mastersizer 2000E – 0.1 to 1000 microns.

By reducing the result range it may be possible to remove the effects of contaminants and bubbles. Contaminants form extra modes or peaks in the result after the analysis. By reducing the result range, the characteristics of the expected result distribution are isolated.

An alternative use for reducing the result range is to isolate a mode of the distribution so that the characteristics of that mode can be obtained separately from the complete distribution. This is particularly important for users who are also transforming the result, where small modes in the volume distribution, caused by errors, are magnified and can become dominant when transformed to a number distribution.

▶ To reduce the result range in an SOP:

1. In the **Material** tab select the **Advanced** button in the **Result calculation** section.

2. Change the size range by altering the entries in the **Include result in size range** box.

▶ To reduce the result range in a manual measurement:

1. Select **Measure-Manual**.

2. Once the background measurements are complete press the **Options** button.

3. Select the **Material** tab.

4. Change the size range by altering the entries in the **Include result in size range** box.

5. To remove any result range reduction press the **Default** button. This will restore the box to the full range.

Transforming results

The basic result produced by the analysis is a volume distribution which is the volume proportion within each size class of the total volume of the particles. It is possible to convert this volume distribution to a number, volume or length distribution. After volume the next most useful distribution type is probably number, which reports the number proportion of particles in each size class.

The transformation can be expressed mathematically as:

$$X_i \frac{100\,V_i / d_i^{\,n}}{\Sigma V_i / d_i^{\,n}}$$

where X_i is the transformed percentage distribution, V_i is the volume distribution result, d_i is the mean size of class i and n is the required distribution type (1 for surface, 2 for length and 3 for number).

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i

Note

A small volume of particles of small sizes can be transformed to a significant part of a number distribution. If this small volume is just due to noise or other errors in the measurement, it may swamp the actual desired distribution. These small errors in the measurement are, in effect, magnified by the transformation. Consider using the **Reduce result range** facility to isolate the significant part of the volume distribution before performing the transformation.

The example below shows the result of transforming a skewed volume distribution ① to number ②.
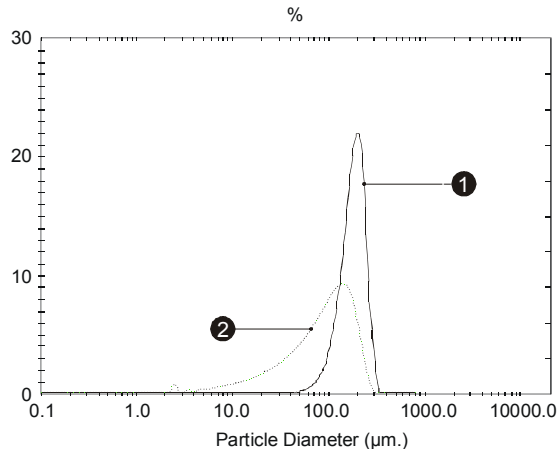
ill 1874

▶ **To transform a result in a manual measurement:**

1. Select **Measure-Manual**.

2. Once the background measurements are complete, press the **Options** button.

3. Select the **Material** tab.

4. Select the **Advanced** button in the **Result calculation** section.

5. Select the **Enable transformation** check box and select the relevant **Transform Volume to** radio button.

▶ **To transform a result in an SOP measurement:**

1. Select the **Material** tab.

2. Select the **Advanced** button in the **Result calculation** section.

3. Select the **Enable transformation** check box and select the relevant **Transform Volume to** radio button.

▶ **To transform a result in an existing record:**

1. Open the relevant measurement file.

2. Select the record to transform.

3. Select **Edit-Result** and then click **OK** in the displayed dialogue.

4. Select the **Material** tab.

5. Select the **Advanced** button in the **Specify new result calculation** section.

6. Select the **Enable transformation** check box and select the relevant **Transform Volume to radio** button. Click **OK** twice.

Editing results

What happens if the user makes a measurement then at a later date wants to change one of the parameters of the result? For example, suppose that days after a measurement the Refractive Index specified as an optical property is found to be incorrect. A user can change the properties of the record by selecting it then using **Edit-Result editor**.

The **Result Editor** dialogue contains tabs similar to the **SOP Wizard**'s **Material**, **Labels** and **Report/Saving** dialogues:
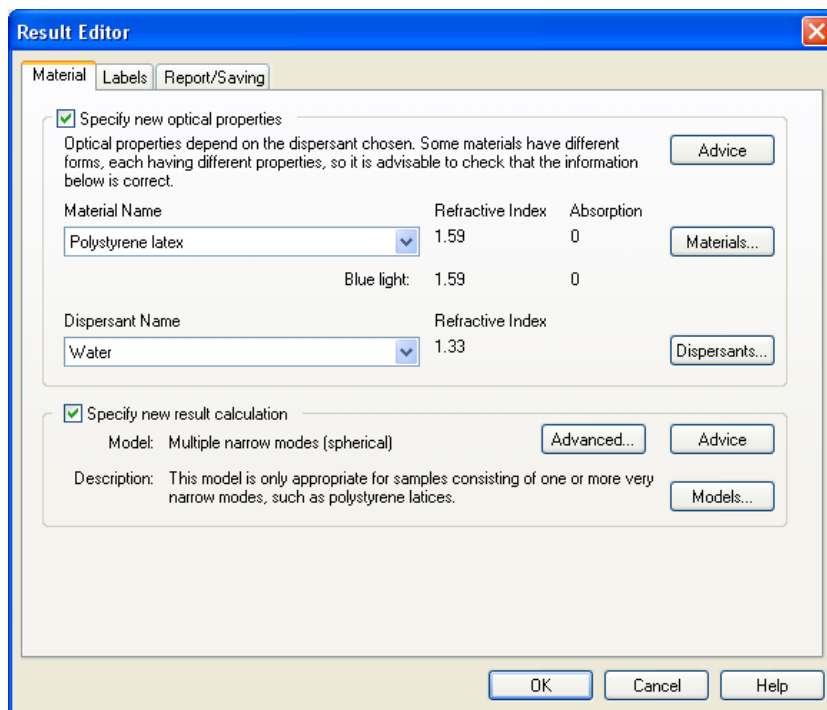
Each section has a **Specify new result calculation** check box that, when selected, lets the user change the settings. The **Measurement Options** section in **Chapter 4** has details of these dialogues.

Remember that the original record cannot be changed. When a user alters a record using the **Result Editor** the changes are saved as a **new** record. A modified record can always be identified as its **Source** parameter shows whether the record has come from a measurement or been recalculated using the **Result Editor**.

When the **Result Editor** is closed down, any changes specified are remembered so that the next time **Result Editor** is opened the changes can be assigned to a new record.

Defining user size bands

Within a histogram graph or result table, each column of the histogram represents a size band of particles, for example from 0.25 to 0.7 microns. The default settings for these are set by the fundamental geometry of the optics within the system. However, these size bands are fully customisable by the user. A practical use of this is shown in the sieve reports. These reports use customised size bands so that they match those required by the relevant standard for sieves.

Size bands are defined by selecting **Edit-User sizes**. The dialogue below will appear. This dialogue allows the user to either manually set the size bands using the **Edit sizes** tab or automatically generating the size classes between user defined limits, using the **Generate Sizes** tab.
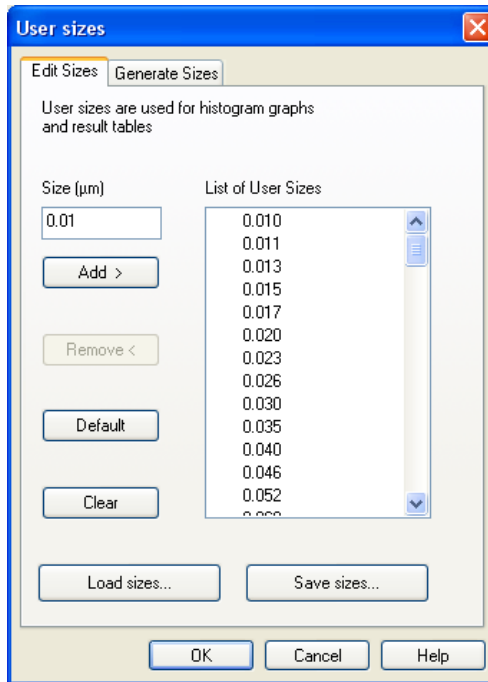
▶ **To define a size band manually:**

1. Select the **Edit Sizes** tab in the **Edit-User sizes** dialogue (shown above).

2. Type the particle size that will form a new size band boundary in the **Size (μm)** box.

3. Select **ADD**. The new size boundary will be added to the **List of User Sizes** list box, creating a new size band.

A size band can be removed using this dialogue by selecting a size from the **List of User Sizes** list box and pressing the **Remove** button.

Pressing the **Clear** button removes all entries in the **List of User Sizes** list box.

Note

The default size bands can be loaded by pressing the **Clear** button then **OK**. The software asks if the user wants to load the default size bands.

User-defined size bands can be saved and loaded using this dialogue.

Generating size bands

Using the **Generate** facility ensures that the size classes are as evenly distributed as possible. This approach is quicker than creating a range of sizes manually.

▶ **To generate size bands:**

1. Select the **Generate Sizes** tab in the **User sizes** dialogue (shown below).
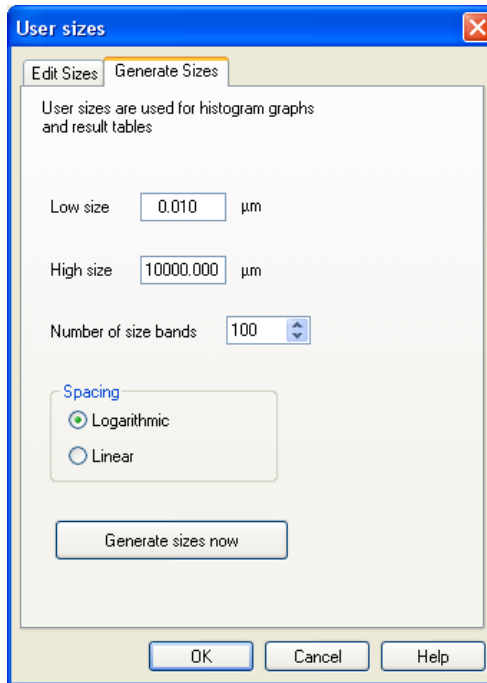
2. Choose the upper and lower limits for the whole distribution using the **Low size** and **High size** boxes.

3. Define the number of size bands by changing the **Number of size bands** value.

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4. Decide if the size classes are to be spaced logarithmically or linearly by checking the relevant radio button under **Spacing**.

5. Press the **Generate sizes now** button.

The size classes generated can be edited or saved by selecting the **Edit Sizes** tab.

Alarms

The user can set three alarms that monitor various states of the measurements. These are the cleanliness alarm, alignment alarm and diameter alarms.

Cleanliness alarms

The software can monitor the cleanliness of the optics by measuring the background signal while clean dispersant is circulating. The measurement procedure can be set up so that the user sets a threshold and an alarm is automatically triggered if this threshold is passed. As an alternative, the background screen can be displayed during the measurement for the user to make a judgment on the cleanliness of the system.

▶ To set a Cleanliness alarm in a manual measurement:

1. Select **Measure-Manual**.

2. Once the background measurements are complete, press the **Options** button.

3. Select the **Measurement** tab.

4. Select **Alarms**.

5. Select the **Enable** check box to enable the cleanliness alarm.

6. Move the slider on the **Cleanliness level** to set the sensitivity of the alarm.

▶ To set a Cleanliness alarm in an SOP measurement:

1. Select the **Measurement** dialogue.

2. Select the **Alarms** button in the **Advanced options** section. There are four options for the actual alarm on the **Cleanliness Alarms** tab:

   ■ **No cleanliness alarm** – the alarm is disabled.

   ■ **Check cleanliness level** – the threshold is automatically monitored and an alarm is triggered if the threshold is exceeded. Select this and use the **Cleanliness level** slider to set the threshold for the alarm trigger.

   ■ **Display background (exited by key press)** – a display appears, showing the background signal. This display disappears when any key is pressed.

■   **Display background (exited by time-out)** – a display appears, showing the background signal. This display disappears when the time-out set in the **Time-out** box is exceeded.

Alignment alarms

In normal operation the alignment of the system is monitored by the software and an auto-alignment procedure is automatically performed if the system is out of alignment. This will add a few seconds to the measurement time. Setting an alignment alarm will warn that the alignment threshold has been triggered but will not stop the measurement.

▶ **To set an Alignment alarm in a manual measurement:**

1.   Select **Measure-Manual**.

2.   Once the background measurements are complete, press the **Options** button.

3.   Select the **Measurement** tab.

4.   Select **Alarms**.

5.   Select the **Enable (warns when alignment is poor)** check box.

6.   Adjust the **Sensitivity** slider if necessary.

Diameter alarms

It is possible to set alarm limits for the three percentiles (D[v,0.1], D[v,0.5], D[v,0.9]) that will trigger if the reported value of a percentile falls outside a predefined size range. An upper and lower size limit can be set for each of the three percentiles.

The alarm can be set to stop the measurement if the alarm is triggered to allow investigation of the source of the alarm.

▶ **To set a Diameter alarm in a manual measurement:**

1.   Select **Measure-Manual**.

2.   Once the background measurements are complete, press the **Options** button.

3.   Select the **Measurement** tab.

4.   Select **Alarms**.

5.   From the **Diameter alarms** section, enable a percentile alarm by selecting the relevant **Enable** check box.

6.   Set the upper and lower size limits for the alarm by changing the values within the **Lower limit** and **Upper limit** boxes.

7.   Click **OK**.

▶ **To set a Diameter alarm in an SOP measurement:**

1. Select the **Measurement** dialogue.

2. Select the **Alarms** button in the **Advanced options** section.

3. On the **Diameter alarms** tab, enable a percentile alarm by selecting the relevant **Enable** check box.

4. Set the upper and lower size limits for the alarm by changing the values within the **Lower limit** and **Upper limit** boxes.

5. Click **OK**.

Averaging records

It is possible to create an average record from several records (usually repeats of the same sample). Select multiple records by using the **CTRL** or **Shift** keys, then select **Edit-Create average result**.

A dialogue will appear that will give the user the opportunity to enter a sample name and the record numbers that were used to create the average. The average will be created as a separate new measurement record capable of being saved and recalled.

Importing results from older instruments

Records created on Mastersizer S, Mastersizer X and Mastersizer Micro/Microplus instruments running v2.15 or later software can be viewed using the current software. This provides an ongoing means of viewing records without the need to retain the original instrument.

To do this select **File-Import S/X**, choose the required data file and press **Open**.
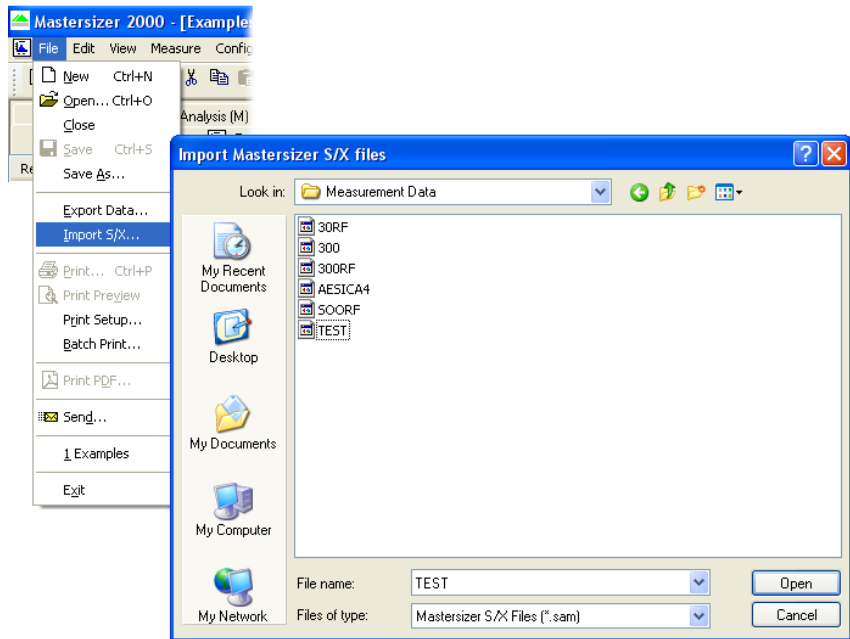
The measurement records imported will now be displayed. The field view should be changed to display the software version.

> **i** **Note**
> Only the complete data file can be imported. It is not possible to import individual data records.

Result emulation

Result emulation is a technique that allows a Mastersizer result to be modified to emulate a result from an alternative particle sizing technique. As an example, a user may have previously used sieve measurements to control a process and may now want to replace the sieves with the Mastersizer. The Mastersizer results may well differ from the sieve results owing to the particles being non-spherical but the user may want the opportunity to produce a sieve-like measurement for comparison purposes. Result emulation satisfies this requirement.

Result emulation requires a "result emulation factors file". This file describes how to translate the Mastersizer result into an external result (the particle size distribution measured by another instrument or technique) and is made up of a set of multiplying factors with its corresponding particle size.

The result emulation generator wizard calculates the factors from a Mastersizer result and an external result. The factors may also be individually edited using the factor editor dialogue box, but this should rarely be necessary.

> **i** **Note**
> The success of this technique depends on having as large a number of measurement points in the external result as possible. It should also be remembered that a set of factors produced for one particular variant of a material will not necessarily be suitable for all variants - changes in production processes will often have a significant effect on the morphology of a material.

When results to be emulated are variable as is frequently the case with sieve analysis, it is good practice to use an average result as the basis of result emulation.

Consult the online help for details on how to use result emulation.

Part 2 - Supervisor's Guide
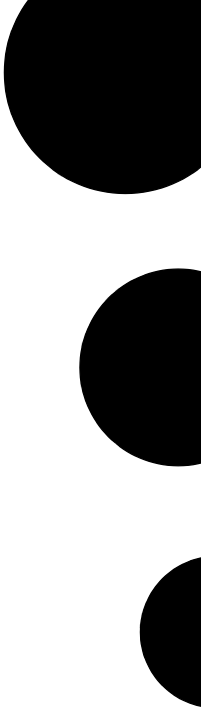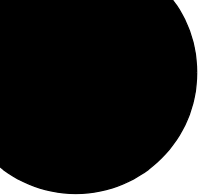
Security

Introduction

This chapter describes:

- Security principles.
- Setting up the Administrator.
- Security guidelines.
- Setting up user groups.
- Setting up individual users.

Security principles

As instrument users may have varying skill levels, user access can be limited so that certain functions such as editing SOPs, deleting records and editing results are reserved for specific users only.

Administrators, users and permissions

One or more persons are set up as **administrator**. The administrator then controls access to the instrument by defining **User groups** and **Permissions**:

■   A **User group** consists of one or more users that have the same access rights.

■   **Permissions** are the access rights that are allowed for each user group; these range from the right to edit SOPs to the right to disable view selection.

The administrator:

■   Creates users – normally they assign each user a password, although this is not mandatory. If passwords are not set up, the user just has to type their username to log in, otherwise they must provide the password too.

■   Creates user groups.

■   Adds users to one or more user groups.

After installation

The first time the software is run the security system will be disabled and an administrator user and administrators group will be created by the system. This is so at least one user will have permission to configure the security system.

21 CFR Part 11

The security system can be upgraded to provide technical compliance with 21 CFR Part 11 by installing a **feature key**. Once this is installed, 21 CFR Part 11 security settings can be applied and **audit trails** can be viewed. If the feature key is installed the grey **21 CFR** icon on the status bar becomes coloured.

This manual does not detail the 21 CFR Part 11 option but concentrates on the standard security software.

Setting up the Administrator

The first time the system is run just one user (named **Administrator**) and one user group (named **Administrators**) are created automatically. The **Administrators** group is originally set only to allow configuration of the security system and to deny access to all other features of the system.

The first task is to set up the **Administrator** account.

▶ **To set up the administrator:**

1. Select **Security-Configure security** to display this dialogue:
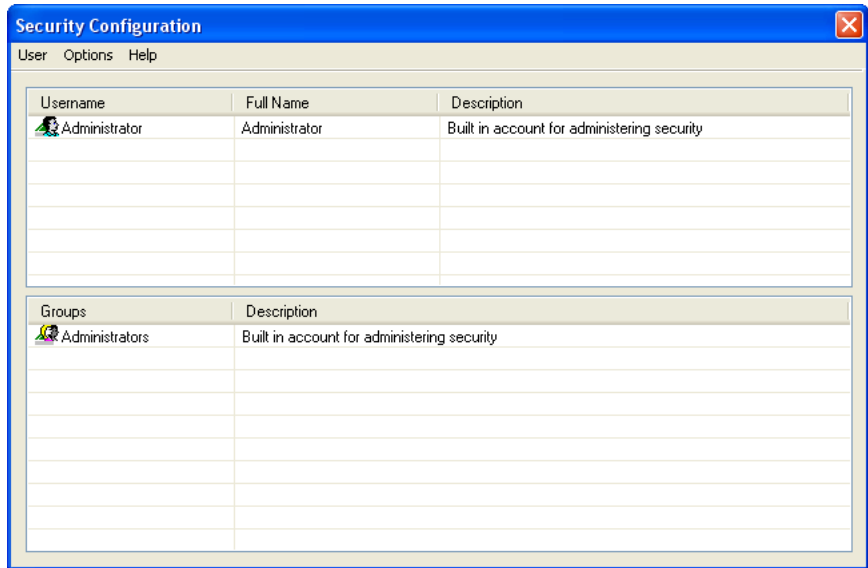
When the software is run for the first time, the security system defaults to a member of the **Administrators** group with no password.

2. Select the **Administrator** name in the first row and then **User-Properties.** Confirm the blank password for the **Administrator** account (that is, do not type anything in) and click **OK**.

3. Click **Options-Security settings...** and in the **Security Settings** dialogue shown below select the **Enable security** check box and click **OK**.
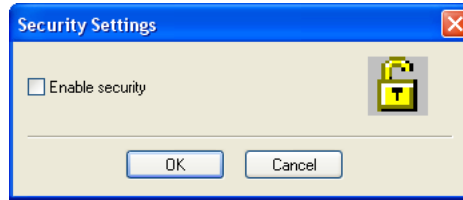
Security is now enabled on the system. This forces subsequent users to log in to access the software.

Note
If the **21 CFR part 11** feature key is installed a different dialogue is displayed. Once **21 CFR part 11** security is enabled, it cannot be disabled.

Security guidelines

Once security is enabled each user must log in when the software starts. Once the user is logged in, only functions matching their relevant permissions will be accessible; all other functions will be greyed out.

To change from one user to another without closing down the software, the first user must select **Security-Logout** and then the second user must select **Security-Login** and enter the appropriate username/password.

Note
It is preferable to assign at least two users to the **Administrators** group. These users' user names and passwords should be stored in a secure location. This is to safeguard against accidental lockout or deletion of permissions that may prevent the security settings being available.

Remember that after any changes to the security system – adding users, changing permissions, etc. – the new changes must be saved (by selecting **User-Save**). A dialogue will appear on exit reminding the user to save changes.

Setting up user groups

All users must be a member of at least one user group.

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i **Note**
Only users assigned to the **Administrators** group can add or edit the user group properties. We recommend setting up at least two administrator users, so the system can still be accessed if one user's password is lost.

▶ **To add a new group:**

1. In the **Security Configuration** dialogue select **User-New Group...** or double-click on an empty **Groups** row to display the **Group Properties** dialogue shown below:

2. Enter a **Group name** and a **Description** of the group's purpose. Example names might be:

   ■ **Operators** – general users of the system.

   ■ **Supervisors** – skilled users who can perform configuration and create SOPs.

3. The **Members** list shows all the users currently assigned to the group. To add a user click **Add**; a list of all users not currently allocated to that group is

shown. Select one or more users (hold down the Ctrl key to select multiple users) and press **OK** to add those users to the group.

If no users have been added yet, add these as described below, then add them to the group.

Note
To remove a member from the group, select them in the **Members** list and click **Remove**. This removes them from the group but not from the system.

4. Use the **Permissions** list to enable/disable functions of the software for the group. Simply check the boxes for the permissions required. Scroll through the **Permissions** list using its scroll bar. Click **OK** to save the group.

▶ **To edit an existing group:**

1. Double-click on an existing group in the **Security Configuration** dialogue to display the **Group Properties** dialogue.

2. Proceed as described above for adding a group.

Setting up individual users

Each user is defined by their:

■ Unique **Username** – this, along with the user **Password**, forms the unique key required to identify each individual using the system. The **Username** is commonly an abbreviated form of the individual name or a unique identifier such as an employee code.

■ **Full Name** – the full printed name of the individual. This can be used on reports to identify the user if an employee code is used as a **Username**.

■ **Description** field (optional) – this is used to add descriptive text for the user.

Note
Only **Administrators** group members can add or edit user properties. We recommend setting up at least two administrator users, so the system can still be accessed if one user's password is lost.

▶ **To add a user:**

1. In the **Security Configuration** dialogue, select **User-New User...** or double-click on an empty **Username** row to display the **User Properties** dialogue:
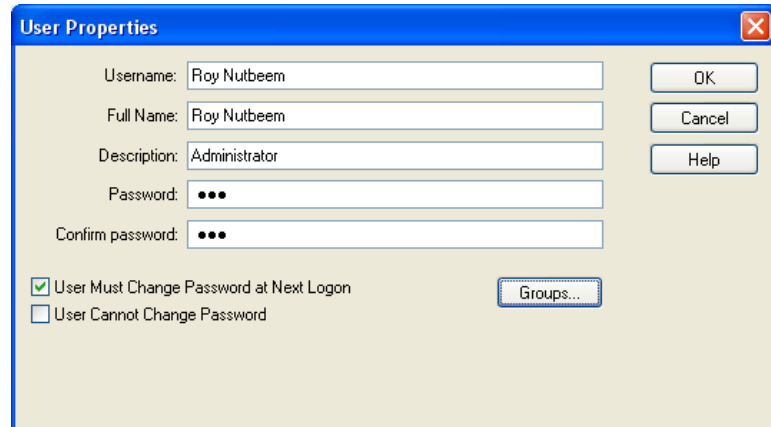
2. Type in the user information in the first three fields.

   To set passwords, the recommended approach is for the administrator to spec-
   ify a previously arranged password, such as the user's name, and force the user
   to change their password the next time they log on by selecting the **User Must
   Change Password at Next Logon** check box.

3. Press the **Groups** button to display this **Group Memberships** dialogue:
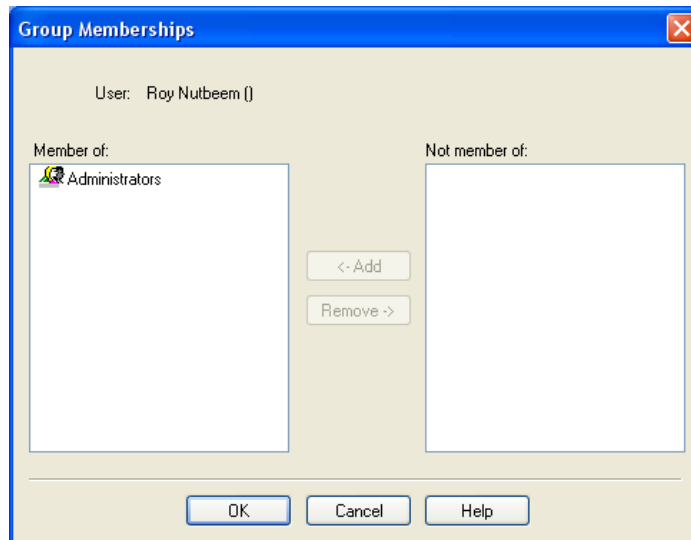
4. Use the **Add** button to allocate the user to appropriate group(s) by moving
   each group to the **Member of:** list.

5. After adding the user to all required groups, click **OK**.

▶ **To edit a user:**

1. To edit an existing user double-click their name in the **Security Configuration** dialogue to display the **User Properties** dialogue.

2. Proceed as described above for adding a user.

When a user logs on

If security is configured users are prompted for a password when they try to log in:
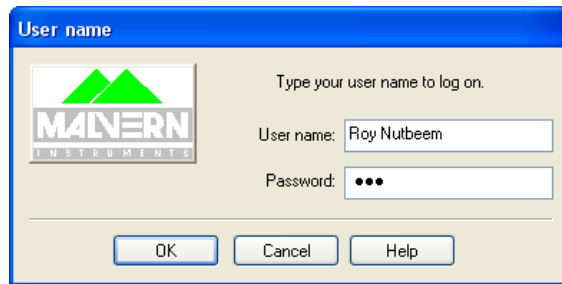
If the administrator selected the **User Must Change Password at Next Logon** check box (see above), the user sees this dialogue when they log in:

To change the user password, the user has to enter the current password and specify a new password with confirmation. Pressing **OK** changes the security settings.

If the administrator used the **User cannot change password** option, this prevents a user from changing their password. Otherwise, once they have logged in a user can change their own password by using **Security-Change password**.

To keep the system secure, users should change their passwords regularly to prevent unauthorised access. The only possible exception is the security administrator's account as forgetting the password to this account could prevent further configuration of the security system.

Part 3 - Appendices

A
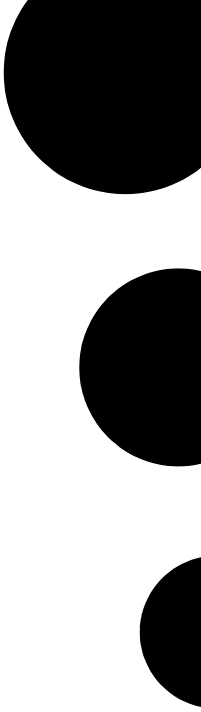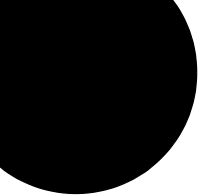
Specification

Optical bench

Size range (2000)	0.02 to 2000 microns, depending on material properties.
Size range (2000E)	0.1 to 1000 microns, depending on material properties.
Measurement principle	Mie Scattering
Detection systems	Red light: Forward scattering, side scattering and back scattering. Blue light: Wide angle forward and back scattering (Mastersizer 2000 only)
Light sources	Red light: Helium neon laser. Blue light: Solid state light source (Mastersizer 2000 only)
Optical alignment system	Automatic rapid align system with dark field optical reticle and multi-element alignment detector.
Sample dispersion unit interchange system.	Sample dispersion units automatically recognised, configured and enabled on insertion of measurement cell cassettes into optical bench.
Power	110/240V, 50/60Hz, 60VA
Dimensions	Length: 1293mm Depth: 255mm Height: 375mm
Weight	35kg

All specifications are subject to change without notice.

Chemical compatibility

Introduction

The Mastersizer and its dispersion units have been manufactured from materials that are considered to give the widest protection from chemical attack. However, it is important to check that any sample or dispersant you may use is chemically compatible with the materials that they will come in contact with within the system.

In normal operation the sample and dispersant should not come into contact with any component of the optical bench (the sample path is contained within the dispersion unit and the sample cell). However, if a sample pipe or sealing ring should fail then sample and dispersant may drip into the cell area of the optical bench. A drain is fitted in the base of the cell area to limit the spread of any leak.

Should a small leak occur the sample/dispersant may come into contact with the following materials:

Component	Materials
Detector lens	Glass
Protection window	Glass
Cell area walls	Aluminium (hard anodised)
Cell area base	Aluminium (hard anodised)
Drain assembly	Stainless steel, Tygon™ tubing
Cell shroud	Polyurethane

Caution!
The paint finish of the external covers may be permanently damaged if samples or dispersants are spilt on them. Clean up any spillage immediately.

Remember to check the chemical compatibility of the sample dispersion units before using a new sample and dispersant.

Unpacking instructions

Déballez l'instrument

Auspacken des Gerates

Desempaquetado del aparato

Desempacotamento do instrumento

1.
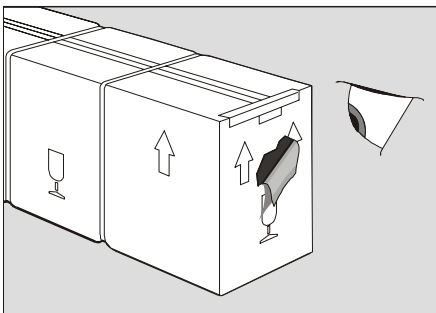
ill 1708

If there is any sign of damage contact the freight carrier immediately.

Si vous constatez des signes de dommages quelconques, contactez immédiatement le transporteur.

Bei irgendwelchen Zeichen von Transportschäden den Spediteur sofort benachrichtigen.

Si se advierte algún signo de deterioro, contactar inmediatamente con el transportista.

Contacte imediatamente a empresa transportadora se houver qualquer sinal de embalagem danificada.

2.
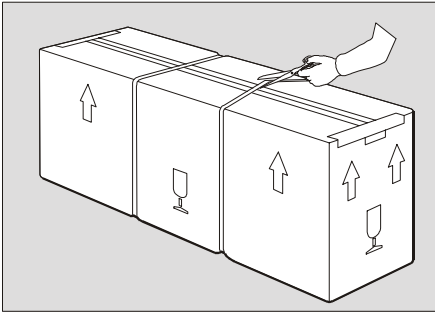
ill 1721

Cut through the plastic bonding straps.

Découpez les courroies d'attache en plastique.

Die Kunststoffbänder durchschneiden.

Cortar las tiras de sujeción de plástico del embalaje.

Corte as tiras plásticas que prendem o conjunto.

3.
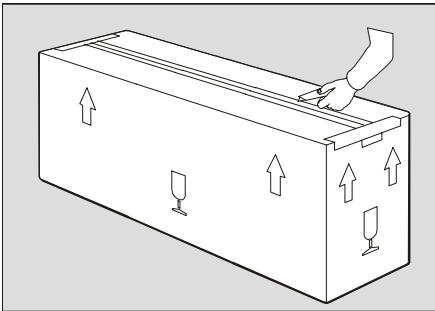
ill 1730

Carefully cut the plastic packing tape along the fold lines.

Découpez avec précaution le ruban d'emballage en plastique, le long des pliures.

Die Kunststoffverpackungsbänder vorsichtig entlang den Falzlinien durchschneiden.

Cortar con cuidado la cinta de embalaje de plástico por la líneas de doblado.

Com cuidado, corte a fita de embalagem plástica ao longo das dobras.

4.
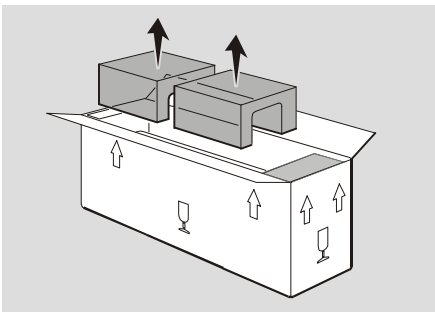
ill 4924

Open the wings of the carton and remove the top foam inserts.

Ouvrez les rabats du carton et retirez les blocs de mousse du haut.

Die Klappen des Kartons öffnen und die oberen Schaumeinlagen entfernen.

Abrir los costados de la caja y extraer las piezas intercaladas de espuma de la parte superior.

Abra as abas da caixa e retire o enchimento de espuma que está em cima.

5.
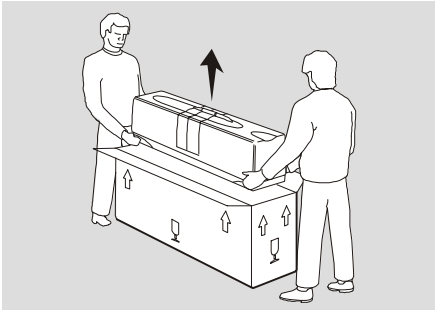
ill 4925

Lift the instrument out of the carton and place on the workbench.

Soulevez l'instrument pour le sortir du carton, et posez-le sur le banc.

Das Gerät aus dem Karton heben und auf der Werkbank aufstellen.

Levantar el aparato, sacarlo de la caja, y colocarlo sobre el banco de trabajo.

Tire o instrumento da caixa e coloque-o na bancada.

6.
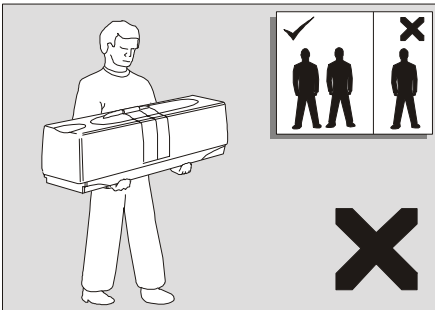
ill 4926

Never try to lift the instrument without the help of others.

Ne tentez jamais de soulever l'instrument tout(e) seul(e).

Bitte versuchen Sie niemals, das Gerät ohne Hilfe einer zweiten Person zu heben. Stets die korrekte Hubart verwenden, um eine Rückenverletzung zu vermeiden

No se debe intentar nunca levantar el aparato sin ayuda de otras personas.

Aplicar siempre las técnicas de elevación adecuadas para evitar daños en la espalda.

Não tente nunca levantar o instrumento da caixa sem o auxílio de outros - use sempre técnicas de levantamento adequadas, para evitar lesões nas costas.

7.
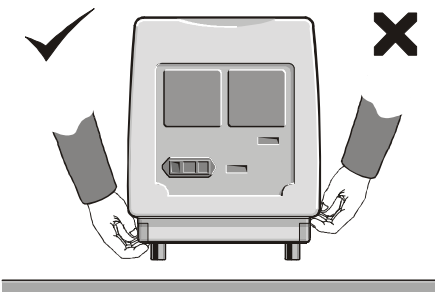
ill 4927

Never lift the instrument by its covers.

Pour soulever l'instrument, ne le tenez jamais par ses couvercles.

Niemals das Gerät an den Abdeckungen anheben.

No levantar nunca el aparato por sus cubiertas.

Nunca levante o instrumento pegando pelas suas tampas.

8.

ill 4928
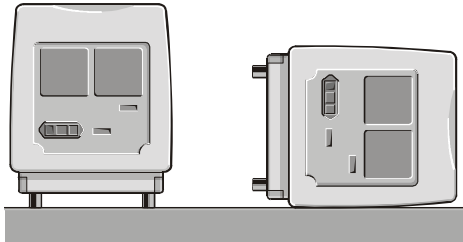
Always place the instrument on its feet. Placing it on its side will damage the covers.

Posez toujours l'instrument sur ses pieds. Si vous le posez sur le côté, vous endommagerez les couvercles.

Das Gerät stets auf seinen Füßen aufstellen. Ein seitliches Aufliegen beschädigt die Abdeckungen.

Se debe colocar siempre el aparato sobre sus patas. Depositarlo de costado dañaría las cubiertas.

Coloque sempre o instrumento na vertical, sobre os seus pés. Se o colocar de lado irá danificar as tampas.

9.

ill 4929
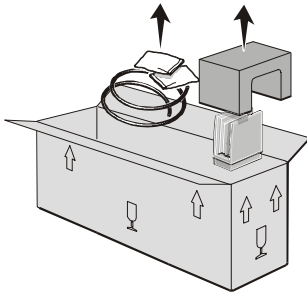
Remove all other packages from the carton and unwrap.

Sortez tous les autres paquets du carton et déballez-les.

Sämtliche andere Pakete aus dem Karton nehmen und auspacken.

Retirar todos los demás paquetes de la caja y desenvolverlos.

Retire todas as outras embalagens da caixa e abra-as.

10.

ill 1715
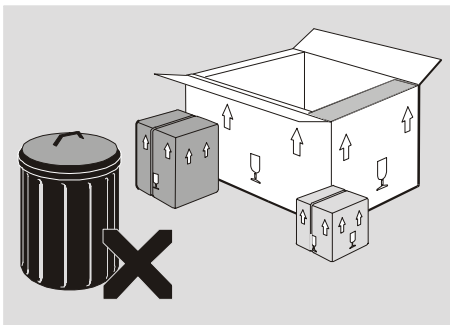
Retain all packaging in case the instrument requires transporting in the future.

Conservez tous les emballages, au cas où l'instrument devrait être transporté ultérieurement.

Das gesamte Verpackungsmaterial aufbewahren, falls in Zukunft ein weiterer Transport des Geräts erforderlich wird.

Conservar todo el material de embalaje por si el aparato debiera ser transportado en el futuro.

Guarde todo o material de embalagem, caso seja necessário no futuro transportar o instrumento.

Regulatory statements

This appendix shows the regulatory information for each product.

CE Declaration of Conformity (APA2000)

The CE badge on this product signifies conformance to European Commission Directives.

CE Declaration of Conformity

(in accordance with ISO/IEC 17050-1)   Form:CE declaration of conformity for APA2000 (2007)

Supplier details

Document (NAME / NUMBER)

CE declaration of conformity for APA2000 (2007)

Issuer's Name (NAME OF MANUFACTURER)

Malvern Instruments Limited

Issuer's Address

Enigma Business Park,
Grovewood Road,
Malvern,
Worcestershire, WR14 1XZ, UK

Product details

Product description – name and model number(s)

Name: Mastersizer 2000(LF)

Model(s): APA2000

Description: Particle Sizer

Directive(s) and Standard(s) to which conformity is declared

EMC directive 89/336/EEC as amended by 92/31/EEC and 93/68/EEC
Low Voltage Directive 2006/95/EC

BS EN 61326-1: 2006 and part 2 as required
BS EN 61010-1: 2001 and part 2 as required

Additional Information

None.

Declaration

I hereby declare that the product specified conforms
with the provisions of Council Directive(s) and
standard(s) listed above.

Signed for and the behalf of Malvern Instruments
Limited.

Signature   Date of issue

5 /FEB /2007

Place of issue   Name (Function)

Malvern Instruments Limited (UK)   Alan Bragginton (Production Director)

ill 7964

CE Declaration of Conformity (EPA2000)

The CE badge on this product signifies conformance to European Commission Directives.

CE Declaration of Conformity

MALVERN INSTRUMENTS

(in accordance with ISO/IEC 17050-1)   Form:CE declaration of conformity for EPA2000 (2007)

Supplier details

Document (NAME / NUMBER)

CE declaration of conformity for EPA2000 (2007)

Issuer's Name (NAME OF MANUFACTURER)

Malvern Instruments Limited

Issuer's Address

Enigma Business Park,
Grovewood Road,
Malvern,
Worcestershire, WR14 1XZ, UK

Product details

Product description – name and model number(s)

Name: Mastersizer 2000(LFE)

Model(s): EPA2000

Description: Particle Sizer

Directive(s) and Standard(s) to which conformity is declared

EMC directive 89/336/EEC as amended by 92/31/EEC and 93/68/EEC
Low Voltage Directive 2006/95/EC

BS EN 61326-1: 2006 and part 2 as required
BS EN 61010-1: 2001 and part 2 as required

Additional Information

None.

Declaration

I hereby declare that the product specified conforms with the provisions of Council Directive(s) and standard(s) listed above.

Signed for and the behalf of Malvern Instruments Limited.

Signature

Date of issue
5/FEB/2007

Place of issue

Malvern Instruments Limited (UK)

Name (Function)

Alan Bragginton (Production Director)

ill 7965

FCC Notice (US only)

The Federal Communications Commission (FCC) mark on this product signifies conformance to FCC regulations relating to Radio Frequency Devices. These have been satisfied by testing the product against, and being found to be compliant with:

FCC CFR 47 Part 15:March 2003.Class A digital device.

The device complies with part 15 of the FCC Rules. Operation is subject to the following two conditions:

1) This device may not cause harmful interference, and

2) this device must accept any interference received, including interference that may cause undesired operation.

Note
This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to part 15 of the FCC rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses, and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at his own expense.

Note
Changes or modifications not expressly approved by Malvern Instruments Limited could void the user's authority to operate the equipment.

Canadian Regulatory Information (Canada Only)

This digital apparatus does not exceed the Class A limits for radio noise emissions from digital apparatus set out in the Radio Interference Regulations of the Canadian Department of Communications.

Note that Canadian Department of Communications (DOC) regulations provide, that changes or modifications not expressly approved by Malvern Instruments Limited could void your authority to operate this equipment.

This Class A digital apparatus complies with Canadian ICES-003.

Cet appareil numérique de la classe A est conforme à la norme NMB-003 du Canada.

VCCI acceptance (Japan only)

The Voluntary Control Council for Interference (VCCI) mark on this product signifies compliance to Japanese EMC regulations as specified by VCCI.

> この装置は、情報処理装置等電波障害自主規制協議会（ＶＣＣＩ）の基準に基づくクラスＡ情報技術装置です。この装置を家庭環境で使用すると電波妨害を引き起こすことがあります。この場合には使用者が適切な対策を講ずるよう要求されることがあります。

ill 6793

Translation:
This is a Class A product based on the standard of the Voluntary Control Council for Interference by Information Technology Equipment (VCCI). If this equipment is used in a domestic environment, radio disturbance may occur, in which case the user may be required to take corrective actions.

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