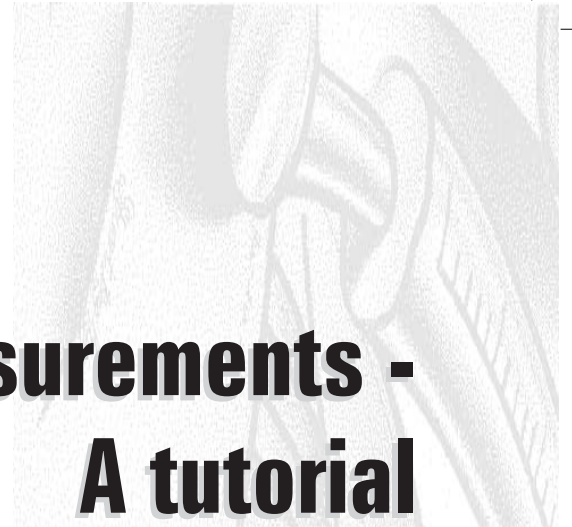
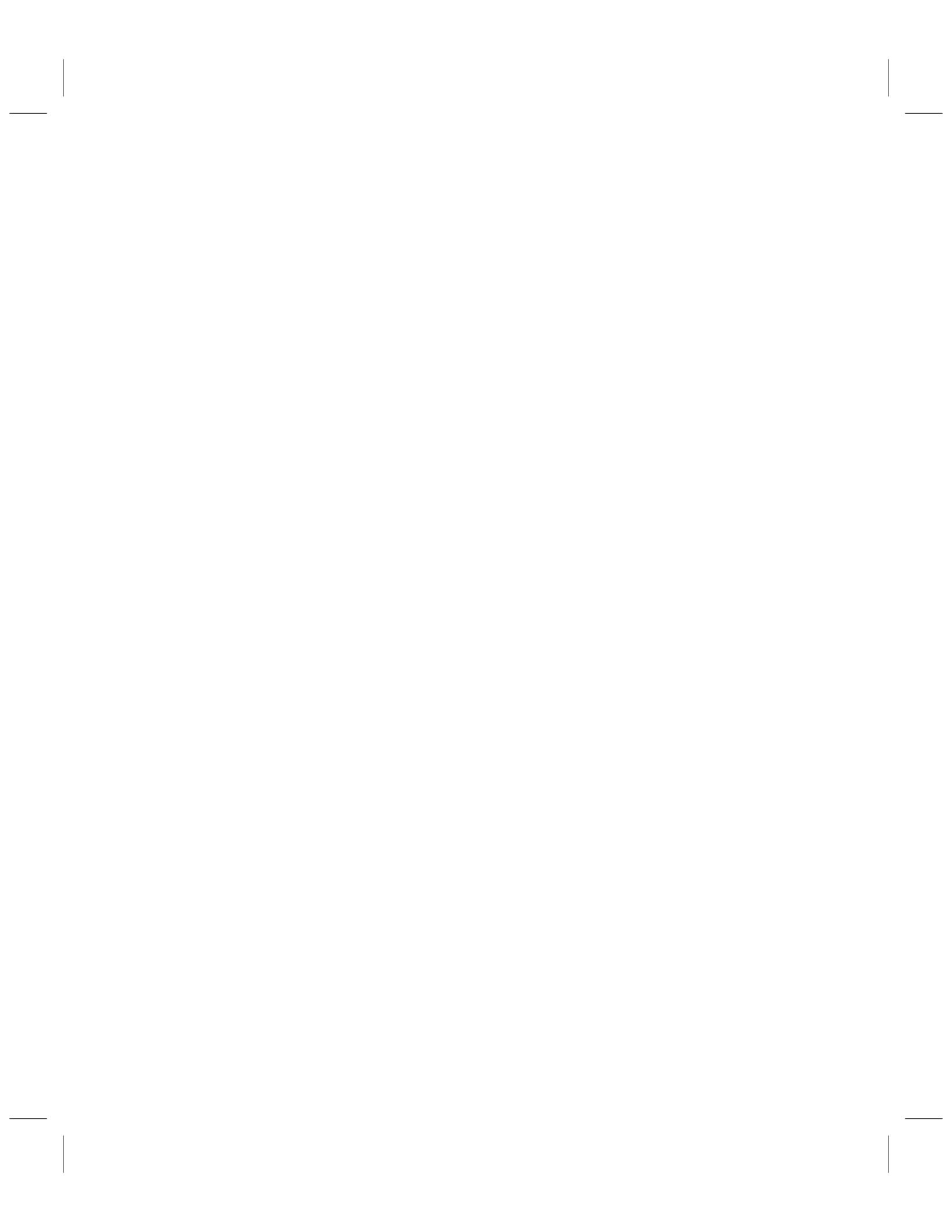


Making measurements - A tutorial

C H A P T E R 4







Introduction

After reading this chapter a user should be able to make simple measurements. The chapter goes through the basics from turning on the system to displaying the results of a measurement.

The first section, “**Quick guide to making a measurement**” will run through the basic steps, giving an overview of the measurement process. The rest of the chapter will go through the same steps but in more detail.

Once a measurement has been completed, the result can be edited to check the effects if one of the measurement parameters was altered - refer to **Editing the result** at the end of the chapter.

Manual and SOP measurements

It was mentioned in Chapter 3 that there are two basic measurement methods: **Manual** measurements and **Standard Operating Procedure (SOP)** measurements. It is important to understand and consider these methods before proceeding.

- A **Manual** measurement is basically a one-off measurement where all the measurement parameters are set up immediately prior to the measurement. This is ideal if measuring many different types of sample, or experimenting with the measurement parameters.
- An **SOP** measurement uses pre-set parameters (that have previously been defined) to ensure that measurements made on the same type of sample are made in a consistent way; this is useful in quality control environments. SOPs are also ideal if measuring the same sample in slightly different ways; having to type a majority of identical parameters each time a measurement is made is tedious and runs the risk of making errors in the settings. Instead, alter an existing SOP and just change the required parameters.

Note that most of the settings and dialogues used for a manual measurement are the same as those used in an SOP measurement.

The sections that follow, “Quick guide to making a measurement” etc, will focus on SOP measurements. Chapter 9 will give details on creating and managing your own SOPs.



Quick guide to making a measurement

This section will give a brief overview of the measurement process using an SOP. More information on each stage can be found later in this chapter.

- Close the lid and **Turn on the instrument** and wait 30 minutes for the laser to stabilise.
- **Start the Zetasizer software.**
- **Prepare the sample** following the sample preparation guidelines.
- **Choose the cell(s)** appropriate for the sample and measurement type.
- **Fill the cell(s)** with the prepared sample.
- **Make an SOP measurement.**

If necessary **Open or create a new measurement file.**

Select **Measure-Start SOP** from the Zetasizer software.

Select the **SOP** required and select **Open.**

Follow any onscreen instructions that appear.

The **Measurement display** will now be shown.

- When requested, **insert the cell** into the instrument and wait for the temperature to stabilise.
- Click **Start** (▶). The measurement will be made and the results displayed and saved to the open measurement file.

Powering up the system


To power up the system, **Turn on the instrument** and then **Start the software.**

Turning on the instrument

On switch on an **initialisation routine** is performed that checks the instrument is functioning correctly.

Close the lid and **turn on** the optical unit, switch on the power at the power socket and turn the power switch at the rear of the unit on.

A “beep” will occur to indicate the instrument has been turned on and the initialisation routine will begin, followed by a second “beep” once the instrument has finished the routine. Two further beeps will be heard to indicate the instrument has reached the “default” temperature of 25°C.

 **Note** ■ **Important!** All laser based measuring instruments should be left powered up for approximately 30 minutes before measurements are made. This is to prevent any thermal equilibration problems affecting the measurement results.

Starting the Zetasizer Nano software

Double click on the icon to start the software.

If the desktop icon is not available, select **Start-Programs-Malvern Instruments-DTS-DTS** to start the program.



Sample preparation

The process of making a measurement is very simple - insert the sample into the instrument and then use the software to run either an SOP or manual measurement. However, the preparation of the sample before it is inserted into the instrument is paramount.

Please refer to Chapter 6 for sample preparation guidelines for the different measurement types.

Choosing the correct Cell

Malvern offers a range of cells for performing measurements with the Zetasizer system. Choice of cell is dependent upon the type of measurement being performed and the sample that will be measured.

The choices for each measurement type are outlined below with some discussion on their use.

General advice

Generally, for “easy to perform” measurements, such as with samples that scatter a reasonable amount of light (latex with 0.01% mass or higher, high scattering intensity etc.) the disposable polystyrene cuvettes can be used.

Disposable cuvettes are easily scratched though and should never be used more than once.



Disposable polystyrene cuvettes are not resistant to organic solvents, thus non-water based samples should generally be measured in glass or quartz type cuvettes.

The optical quality of the cells is vitally important when performing molecular weight and protein measurements, therefore glass or quartz type cuvettes should be used to ensure the optimum signal is achieved.

All the cells mentioned below are available from Malvern and should be used with the supplied cell caps. Using the caps will ensure greater thermal stability of the sample, as well as preventing dust introduction and possible spillage.



Caution!

Due to the risk of melting, polystyrene cuvettes must not be used for measurements above 50°C.

Size measurements

	<i>“Size & Zeta” Folded Capillary cell (DTS1060)</i>	<i>Disposable polystyrene (DTS0012)</i>
<i>Typical solvent</i>	<i>Water, Water/alcohol</i>	<i>Water, Water/ethanol</i>
<i>Optical quality</i>	<i>Good to very good</i>	<i>Good to very good</i>
<i>Minimum Sample volume</i>	<i>0.75ml</i>	<i>1ml</i>
<i>Advantages</i>	<i>Low cost.</i> <i>Single use disposable (no cleaning)</i> <i>Use with MPT-2 Autotitrator</i> <i>No sample cross-contamination</i> <i>Fast sample change over</i>	<i>Low cost</i> <i>Single use disposable (no cleaning)</i>
<i>Disadvantages</i>	<i>Not resistant to organic solvents</i> <i>Unsuitable for use at high temperatures (above 70°C)</i>	<i>Not resistant to organic solvents</i> <i>Unsuitable for use at high temperatures (above 50°C)</i>



	Disposable low volume polystyrene (ZEN0112)	Glass - round aperture (PCS8501)
Typical solvent	Water, Water/alcohol	Water, most organic and inorganic solvents
Optical quality	Good to very good	Excellent
Minimum Sample volume	375µl (100µl when using 'black cell spacer')	1ml
Advantages	Low cost Low volume Single use disposable (no cleaning)	Highest optical quality Can use nearly any dispersant
Disadvantages	Requires careful filling to avoid bubbles Not resistant to organic solvents Unsuitable for use at high temperatures. (above 50°C)	Requires cleaning after measurement
	Glass - square aperture (PCS1115)	Low volume quartz (ZEN2112)
Typical solvent	Water, most organic and inorganic solvents	Water, most organic and inorganic solvents
Optical quality	Excellent	Excellent
Minimum Sample volume	1ml	12µl
Advantages	Highest optical quality Can use nearly any dispersant Reusable	Highest optical quality Can use nearly any dispersant Low sample volume
Disadvantages	Requires cleaning after measurement	Requires cleaning after measurement Requires careful filling to avoid bubbles

**Low volume Glass flow
cuvette (ZEN0023)**

Typical solvent	Water, most organic and inorganic solvents
Optical quality	Excellent
Minimum Sample volume	75 μ l plus tubing
Advantages	Highest optical quality Can use nearly any solvent (tubing dependent) Use with Autotitrator
Disadvantages	Requires cleaning after measurement With manual use requires careful filling to avoid bubbles

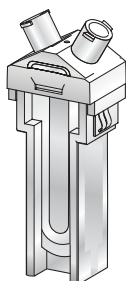
Molecular weight measurements

	Glass - round aperture (PCS8501)	Glass - square aperture (PCS1115)
Typical solvent	Water, most organic and inorganic solvents	Water, most organic and inorganic solvents
Optical quality	Excellent	Excellent
Minimum Sample volume	1ml	1ml
Advantages	Highest optical quality Can use nearly any dispersant Reusable	Highest optical quality Can use nearly any dispersant Reusable
Disadvantages	Requires cleaning after measurement	Requires cleaning after measurement

Zeta potential measurements

"Size & Zeta potential" Folded Capillary cell (DTS1060)

Description



ILL 6723

This is a maintenance-free capillary cell primarily designed for zeta potential measurements.

It has been designed to be used for a single measurement or series of measurements, then discarded rather than cleaned. This removes the chances of cross-contamination. The cell can be inserted either way round.

The cell provides a low-cost alternative to previous reusable quartz capillary cells.

The stoppers can be replaced with 'Luer' connectors to provide leak-free connection to the optional MPT-2 Autotitrator.

Size measurements can also be performed without having to remove and reposition the cell.

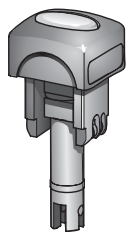
Sample details can be written on the textured area on the side of the cell with a permanent pen.

Application	<i>The cell is used for measurements of aqueous based samples</i>
Typical solvent	<i>Water, Water/alcohol</i>
Optical quality	<i>Good to very good</i>
Minimum Sample volume	<i>0.75ml</i>
Advantages	<i>Low cost</i> <i>Single use disposable (no cleaning)</i> <i>Use with Autotitrator</i> <i>No sample cross-contamination</i> <i>Fast sample change over</i>
Disadvantages	<i>Not resistant to organic solvents</i> <i>Unsuitable for use at high temperatures (above 70°C)</i>



Universal 'Dip' cell (ZEN1002)

Description



ILL6764

The Universal 'Dip' cell is used to provide a method to measure the zeta potential of both aqueous and non-aqueous samples - A number of samples can be prepared and the Dip cell inserted to measure each one in turn.

For aqueous samples the dip cell can be used in conjunction with the disposable polystyrene (DTS0012), and for non-aqueous samples use the reusable Glass - square aperture (PCS1115). These cells are described above.

Application

The 'Dip' cell can be used for measurements of aqueous and non-aqueous based samples.

The dip cell is supplied with three coloured labels, that can be fitted to the dip cell cap to identify the type of sample the cell will be used with. This is to avoid cross-contamination between aqueous and non-aqueous samples. It is suggested that the 'blue' label is fitted when the cell is used for aqueous samples, the 'green' label when the cell is used for non-aqueous samples, and the 'amber' label when the cell is used for both.

Filling the Cell

When filling the cell there are several actions to consider; some that applies to all cells and other actions that are only applicable to the measurement type and the cell chosen.

General advice

- Only clean cells should be used.
All size and zeta potential cells should be rinsed/cleaned with filtered dispersant before use - see **Cleaning the cells** section in chapter 7.
All molecular weight cells should be rinsed/cleaned with the filtered standard (i.e. Toluene) or solvent before use.
- The cell should be filled slowly to avoid air bubbles from being created.
Ultrasonication can be used to remove air bubbles - but only if the sample is suitable for use with ultrasonics.
- If using syringe filters for the dispersant, never use the first few drops from the syringe, in case there are any residual dust particles in the filter that may contaminate the dispersant.

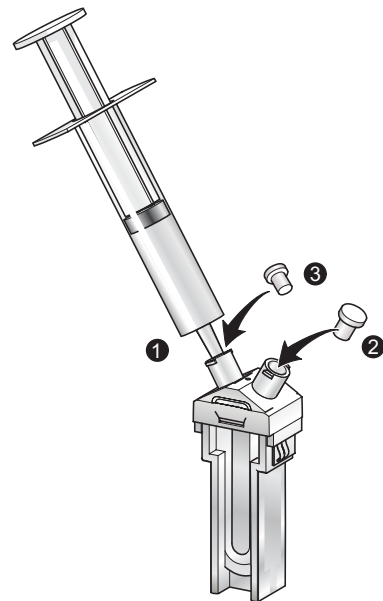
Zeta potential measurements

The two cells used for zeta measurements are the folded capillary cell and the dip cell; the dip cell will use square cuvettes to hold the sample. Though filling either cell is a simple task, there are a number of precautions to be aware of.

Folded capillary cell

Fill the cell as described below

- Prepare the sample in a syringe of at least 1ml capacity.
- Place the sample syringe into one of the sample ports.
- **Slowly** inject the sample through the cell ①, checking that all **air bubbles** are **removed**.
If a bubble forms under the sample port, pull the syringe plunger back to draw the bubble into the syringe body and then reinject.
- Once sample starts to emerge from the second sample port, insert a **stopper** ②.
- Remove the syringe and replace with a second **stopper** ③.
- **No** bubbles should be seen within the clear capillary area of the cell. If necessary tap the cell lightly to dislodge them. Check that the cell electrodes are still completely covered.
- Remove any liquid that may have spilt onto the electrodes.



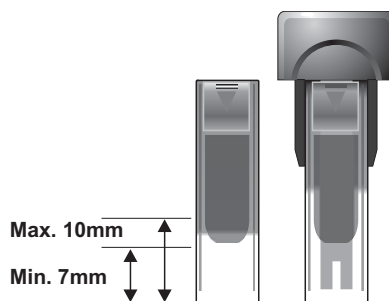
ILL6734

Note ■ The stoppers **must** be fitted before a measurement is performed.



Universal 'Dip' cell

With the insertion of the dip cell the sample will be displaced upwards within the cuvette. If too much sample is placed into the cuvette prior to insertion of the dip cell there is a risk that the cuvette will overflow.



To ensure a minimum sample volume is provided for the sample to be measured, but protect against overfilling we recommend the cuvette is filled to a depth of between **7mm** and **10mm** (**before** the dip cell is inserted). The minimum level relates to approximately **0.7ml** of sample.

Do not overfill the cell; as well as overflowing the cuvette once the dip cell is inserted, this can also produce thermal gradients within the sample that will reduce the accuracy of the temperature control.

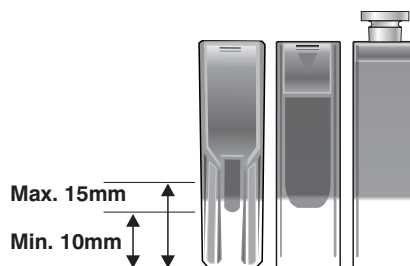
If necessary tap the cell lightly to dislodge any bubbles that may be caught between the electrodes.

Size and Molecular weight measurements

Standard cells

A minimum sample volume must be provided. However, this minimum volume depends on the actual cell type and it is easier to ensure a certain **depth** of the sample in the cell.

This **minimum** is **10mm** from the bottom of the cell (the measurement is made 8mm from the bottom of the cell).



Do not overfill the cell, about **15mm maximum**, as this can produce thermal gradients within the sample that will reduce the accuracy of the temperature control.

Low volume cell

This cell is designed to use the minimum volume of sample possible for a size or molecular weight measurement. The sample must be pipetted carefully into the bottom of the cuvette, so it is filled from the bottom up.

The minimum volume that can be used is 12 microlitres. This will only partly fill the visible cell volume. After filling, carefully inspect the cell for trapped bubbles.

Inserting the Cell

In the status bar, the software will prompt when the cell needs to be inserted. This will always be after the SOP has been started - see next section. When and how the cell is inserted will depend on the application, and the measurement choices selected.

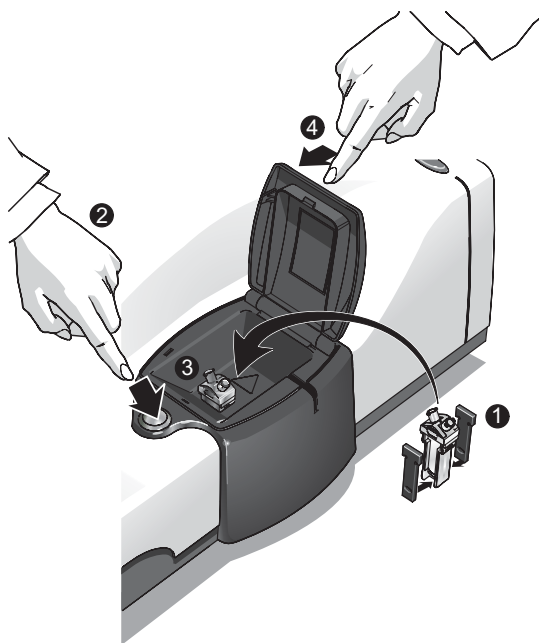
Zeta potential measurements

The two cells used for zeta potential measurements are the folded capillary cell and the dip cell; the dip cell needs to be used with a cuvette with a square aperture. Both cell types involve slightly different insertion routines.

Note ■ The electrode contacts on each cell, as well as applying the measurement voltage, provide identification to the software that a zeta potential cell is fitted.

Inserting the Folded capillary cell

① Place a **thermal contact plate** into the recess on either side of the folded capillary cell. The plates provide increased temperature stability.



ILL-4022

② Open the cell area lid by pushing the button in front of the lid.

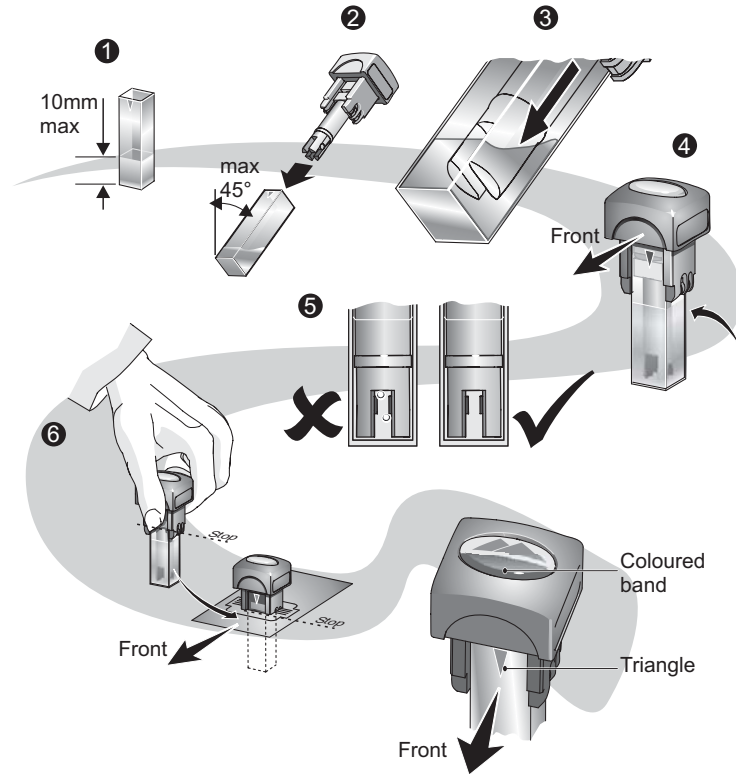
③ Hold the cell near the top, away from the lower measurement area, and push into the cell holder until it stops. The cell can be inserted either way round.

④ Close the cell area lid.

Inserting the Universal 'Dip' cell

Insertion of the dip cell, follows the same procedure as above, but first the dip cell must be placed into the sample cuvette, this must be done at an angle to avoid any bubbles being caught between the sample electrodes.

Note ■ With the procedure complete, the measurement face of the cuvette (some have a small **triangle** at the top of the cell), and the coloured band on the Dip cell label must face in the same direction, this is to ensure the orientation is correct when inserted into the cell holder.

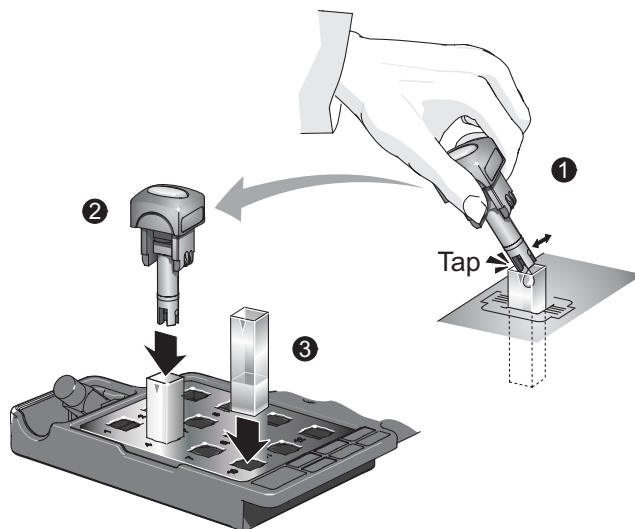


ILL 6907

- The cuvette **must not** be filled more than the recommended **maximum** depth of **10mm** ①.
- Tilt the cuvette to a maximum angle of 45° ②.
- Slowly insert the cell into the cuvette until the metal electrodes are covered ③. As the cell is inserted it displaces the sample so any bubbles will be pushed out from the top of the electrode gap.
- Once the electrodes are covered bring the cuvette up to the vertical ④.
- Inspect the combined cell and cuvette and check for any bubbles ⑤. If bubbles are present gently tap the bottom of the cuvette to dislodge or repeat the above sequence.
- The cell can only be inserted one way round. Hold the base of the dip cell cap and the top of the cuvette simultaneously ⑥. Ensure the coloured band on the label (and cuvette triangle) is facing the front of the instrument and push the cell into the cell holder until it stops - a '**stop**' on the dip cell **must** rest on the top of the cell holder. Check that the cell sits flat on the cell holder.

Removing the Universal 'Dip' cell

With care, by simultaneously holding the base of the dip cell cap and the top of the cuvette, both the dip cell and cuvette can be removed together. If adequate purchase cannot be obtained on both parts, then the following procedure is recommended.



ILL 6906

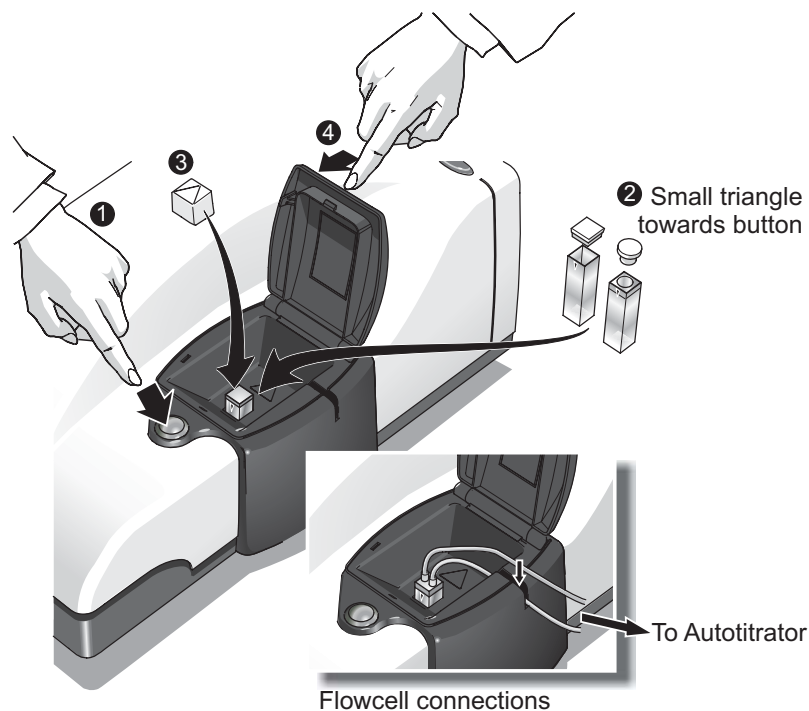
- Lift the dip cell up out of the cuvette, but before completely removing, gently tap the bottom of the dip cell on the top of the cuvette ①. This will dislodge any remaining drops of sample from the cell into the cuvette.

If the dip cell is simply lifted out of the cuvette there is a risk of drops of sample falling from the bottom of the dip cell onto the instrument and surrounding area. This is especially important when using solvent based samples.

- Place the dip cell immediately into an empty cuvette for storage ②. This will prevent any potential damage occurring either to the cell electrodes or the workspace.
- Remove the sample cuvette afterwards and place in the cuvette holder ③.

Note ■ Storage is also provided in the dip cell case if the cell will not be used for a while.

Size and Molecular weight measurements



ILL 4022

- ① Open the cell area lid by pushing the button in front of the lid.
 - ② Push the cell into the cell holder until it stops. Some cells have opaque surfaces as well as polished optical surfaces. A polished optical surface must be facing the front of the instrument (towards the button). Most cells have a small **triangle** at the top to indicate the side that faces the front. This is especially critical for molecular weight measurements.
- If a **flowcell** is used, insert the sample tubes into the threaded inserts and screw into the top of the flowcell. The tubing is then inserted into the channel on the side of the cell area. One tube will be held by the pinch valve with the other resting **above** it.
- ③ Place the thermal cap over the cell; Do not fit if using the flowcell.
 - ④ Close the cell area lid.



Making an SOP measurement


If a measurement is being made using an SOP, then all the hard work has already been done. The instrument has been turned on and the software started; the sample has been prepared and added to the cuvette. Now all that remains is to open or create a measurement file, open the required SOP, place the filled cuvette into the instrument and finally to press the **Start** (▶) button.

This process is outlined below. Chapter 9 gives all the detail required to create new SOPs.

Opening or creating a Measurement File.

Each time a measurement is made, the measurement data will be saved to a measurement file. How the measurement files are managed is down to preference. As an example:

- One measurement file may be used for all the measurement records (not recommended).
- Separate files are used for each type of sample i.e. one for titanium dioxide and one for carbon black.
- A separate measurement file is used for each week or month.
- A separate measurement file is used for each user.

 **Note** ■ If more than one measurement file window is open, the measurement record will be saved to the measurement file currently active. When the software starts it will automatically open the last measurement file used.

▶ **To open an existing measurement file:**

Select **File-Open**.

A dialogue will appear allowing selection of a measurement file.

Select **Open**.


▶ **To create a new measurement file:**

Select **File-New**.

A dialogue will appear allowing the new measurement file to be named and specify where it will be saved.



Select **Save**.

 **Note** ■ All measurement files have the extension **.DTS**. This is added automatically to all new files.

Starting an SOP measurement


Everything should now be ready to make the actual measurement.

To start an SOP measurement, select **Measure-Start SOP**. The **Open SOP** dialogue will appear. Select the SOP that will be used and select **Open**. If an SOP has not been specified for the sample, read chapter 9 for details on how to create one.

Pre-measurement instructions may appear to advise of any actions that need to be performed before the measurement can proceed.

This may be followed by a **Labels** dialogue, allowing the measurement to be named (displayed as the **Sample name** in the records view). This dialogue also allows any other information about the measurement to be entered in the **General notes** box, such as a batch number etc. Once the measurement record has been named and any comments added, select the **OK** button.

The **Measurement display**, discussed below, will now appear.

 **Note** ■ It may be that the SOP was not configured to automatically show the **Labels** dialogue. If the dialogue does not appear, but is required, select the **Settings** button in the measurement display.

Follow the instructions on the status line of the measurement display - i.e. **Insert the Cell** (described above) and press the **Start (▶)** button to start the measurement.

The progress of the measurement can be viewed in the measurement display. The measurement may take anything from 2 minutes to over an hour per measurement, depending on the settings within the SOP.

Once the **measurement sequence** (below) is complete the measurement display can be closed and the new record will be shown in the measurement file window. The results can now be viewed - see **Displaying the results** in chapter 5.




Making a manual measurement

Making a manual measurement is essentially the same as making an SOP measurement, except, where in an SOP measurement all the measurement options are pre-specified, it will be necessary to set them immediately. All the dialogues are available at once in a tabbed format.

Follow the measurement procedures described above. Instead of starting an SOP, select **Measure-Manual**. This will open the **Manual measurement settings** dialogue allowing any measurement types to be chosen and the settings to be configured.

The dialogues are virtually identical to those used to define a new SOP. To save repeating the same information here, please refer to Chapter 9 - Managing SOPs, for more details.

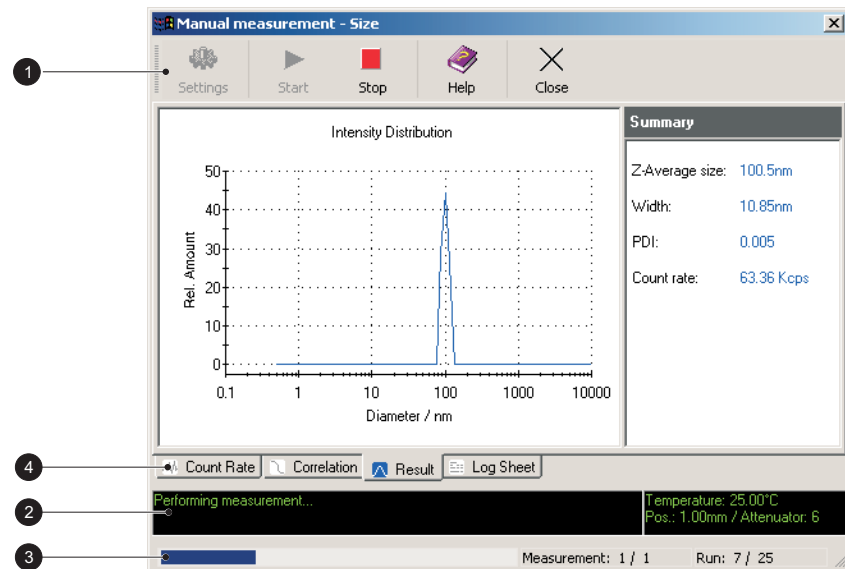
Once all settings have been made select the **Save as SOP...** button, if required, to store the settings. Click the **OK** button to close the manual measurement settings dialogue and return to the measurement display.

 **Note** ■ The manual measurement settings can be viewed and subsequently saved by selecting **Edit-Extract SOP**.

The Measurement display

When an SOP or manual measurement is started the measurement display will appear showing the progress of the measurement.

The measurement display for all measurement types is generally the same and shows a number of dialogues representing the progress of the **measurement sequence**. The dialogues displayed is dependent upon the measurement type selected. The diagram below shows the display for a size measurement.



The features of the measurement display are:

① Button bar

The button bar provides the control for the measurement operation.

Settings

Opens the measurement settings dialogue. Extra comments and changes to the measurement parameters can be added prior to the measurement being started.

Start (▶) and Stop (■)

Starts and stops the measurement. If **Stop** is pressed while performing a measurement then the measurement must be started again from the beginning. **Stop** does not act like a pause.

Help

Opens the Help file.

Close

Closes the measurement display and returns to the record view. If close is pressed while a measurement is in progress the screen will close and all measured data will be lost; a warning box will appear asking “**Are you sure you wish to abort the measurement**”.



② Status bar

The status bar shows instructions and the current operation in the measurement sequence, plus the temperature, measurement position and attenuator settings

③ Progress meter

The progress meter shows how far the measurement has progressed plus the number of measurements performed and the measurement runs completed.

④ Tab dialogues

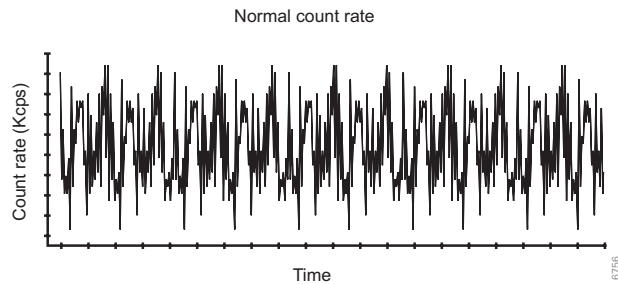
The **Tab** dialogues alter depending upon the measurement type selected. The dialogues available are:

Size measurements

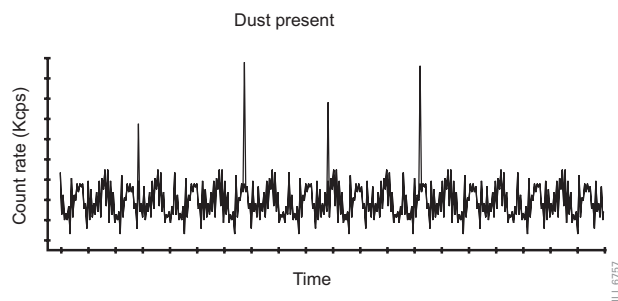
Count rate

Displays the number of photons detected per second. The count rate is useful for monitoring the sample quality.

Normal count rate display

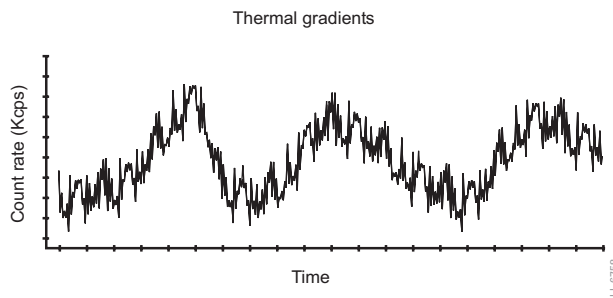


If dust is present then sharp spikes will be observed. Measurement runs with dust present will be removed from the final measurement calculation by a dust filtration algorithm.

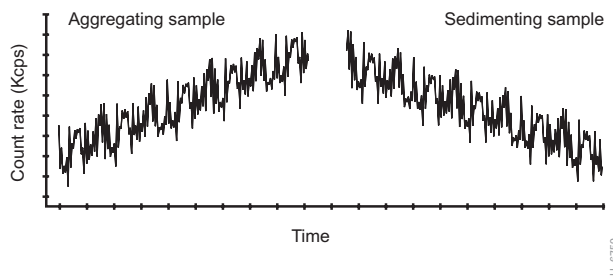




A wildly fluctuating count rate may indicate that thermal gradients are present in the sample, and further time is required for temperature equilibration.



A steadily increasing count rate will indicate an aggregating sample, while a decreasing count rate will indicate a sedimenting sample.

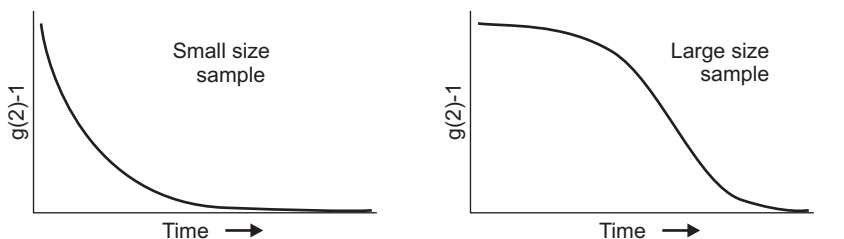


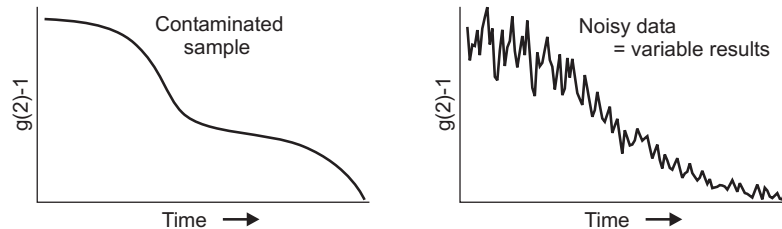
Log sheet

Displays the progress of the measurement. The log sheet can be saved by right-clicking the mouse on it, pressing the **Save to file...** button that appears and saving it as a **.txt** file.

Correlation function

The correlation function helps the experienced user to interpret any problems with the sample.



**Result**

The result view will be updated after every run of the measurement. The result shown will be the sum of the acceptable data collected. When a result is available to view the result tab will turn blue.

Molecular weight measurements**Count rate**

Please refer to size description above.

Correlation function

Please refer to size description above.

Result

The result view will be updated after each of the individual concentration measurements. The result shown will be an evolving value from the data collected so far.

Debye

Displays the current result as a Debye plot. The Debye plot displayed will show an evolving plot generated from the data collected so far.

Log sheet

Displays the progress of the measurement.

Zeta measurements**Count rate**

Displays the number of photons detected in kilo-counts per second.

Zeta potential

Displays the zeta potential result. The view will be updated after every individual measurement run, with the result being the sum of the completed measurement runs.

Log sheet

Displays the progress of the measurement.




Trend and protein melting point measurements

The trend and melting point dialogues are generally the same as those used when either a size or zeta measurement SOP is chosen. The difference is the inclusion of a **Trend** tab - this will show an evolving plot as the measurement progresses.

Titration measurements

As with trend and protein melting point measurements, the dialogues are generally the same as those used when either a size or zeta measurement SOP is chosen. The difference is the inclusion of a **Titration/Measurement type** tab (i.e. pH/Zeta) - this shows a plot of the titration against the measurement type selected. This is explained further in the Autotitrator accessory manual.

Measurement sequence

 **Note** ■ The status bar will prompt for certain actions during the course of the measurement.

Before the measurement sequence begins the cell temperature will change to the starting temperature requested in the SOP.

The measurement will then continue with an optimisation or initialisation stage, where the cell positions, compensation and attenuator settings for the cell, sample and measurement type will be determined.

Monitoring the **status bar** or clicking on the **Log sheet** tab will give more detail about what is happening during this procedure. The **progress meter** indicates how far the system is through the optimisation stages.

Once these stages have been completed, the measurement proper will start; again the actual measurement sequence will depend upon the measurement being performed.

Size measurements

The cell is inserted, **Start** is pressed and data collection begins. The progress meter indicates the measurement progress, while **Measurement** and **Run** show the number of runs completed and measurements performed.

The measurement is divided into a number of 'runs', This is done to allow data filtering. At the end of data collection the data quality of each 'run' is assessed; the runs that contain the poorest data are rejected while the remaining runs are analysed and used in the final measurement calculation.



As soon as a run is completed the result tab changes to blue to indicate a preliminary size result is available to view (by clicking on the tab). As more runs are made and assessed the quality of the result will improve.

Molecular weight measurements

The molecular weight measurement sequence requires a series of intensity measurements to be made, first of a standard to establish the reference scattering intensity, and then of each of a number of prepared sample concentrations. At each part of the sequence the user will be prompted for the insertion of the next concentration. As this requires more interaction than for size and zeta potential measurements, the sequence has been described below

- Press **Start** to begin the dark count measurement.
The laser is turned off and a measurement is taken of the background light level.
- Insert the scattering standard cell (i.e. Toluene) and press Start when ready. The measurement will measure the scattering intensity of the scattering standard used.
- Once the standard has been measured a dialogue box will appear to prompt insertion of the first sample concentration (i.e. the pure solvent). Insert the first sample concentration and press **Start**.
- The software displays another dialogue where the sample concentration can be entered. Type in the concentration and press **Enter**.
The measurement continues.
- On completion of the first sample measurement, a dialogue is displayed - answer **Yes** to “Repeat measurement of concentration 1?” or **No** to continue with the second concentration.
- Continue as above until all sample concentrations have been measured.
- On completion of the last concentration the final result will be calculated.

The progress meter indicates the measurement progress during each stage.


Zeta potential measurements

The cell is inserted and **Start** is pressed. The cell is first checked to identify the cell type fitted, and that it agrees with that selected in the SOP. Once identified the measurement sequence continues automatically. The status bar will indicate the instrument is now “**Performing the measurement**”.

Each **complete measurement** is divided into a number of **measurement runs**. All the individual measurement runs are accumulated together and then summed to give a final **Zeta result**.



As soon as **one** measurement run is completed the zeta result tab changes to blue to indicate a preliminary zeta result is available to view (by clicking on the tab). As more measurements runs are made, the zeta result will change as more runs are accumulated and averaged until the final result is achieved.

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-  **Note** ■ To shorten the measurement sequence, select the '**Auto**' measurement duration in the SOP; the change in zeta potential will now be monitored as the measurement progresses. The default number of runs in 'auto' measurement duration is 30, but for a stable sample as few as 10 runs may be required. The measurement will then complete even though the displayed run total has not been achieved.
-

During a measurement sequence it is possible to select and view the information on any of the displayed tabs.


The final result is displayed and saved to the current open file. This report pages can be viewed when the measurement display has been closed.

Editing the result

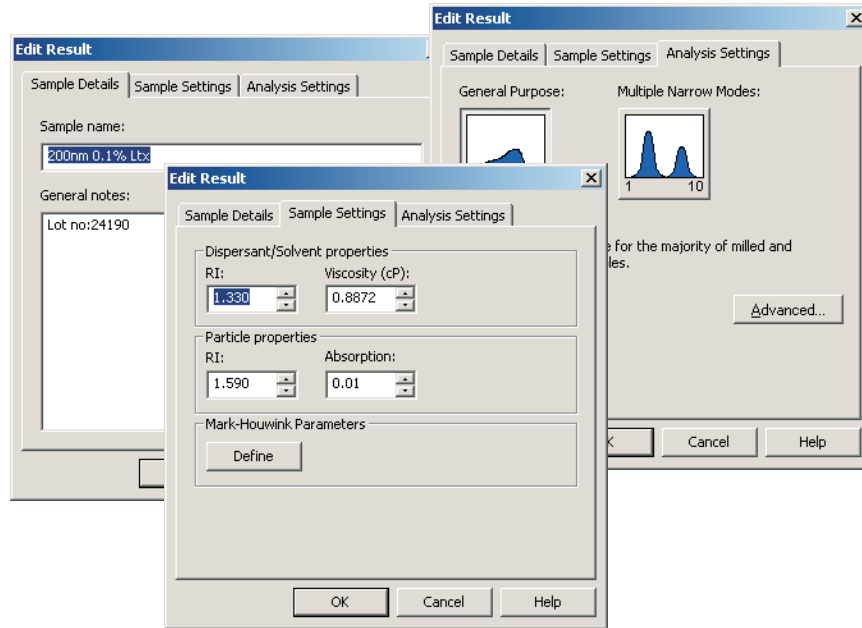
It is possible to re-analyse a measurement record using different measurement parameters. The re-analysed record will be added to end of the current file. Comments on the reasons for editing can be added and viewed in the report views.

This option allows measurements to be reanalysed without the need for the instrument to be connected.

Right-click on a measurement record and select **Edit Result**; the dialogue below will appear. The **Edit result** tabs will show choices similar to those within the SOP editor.

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-  **Note** ■ Each edit result dialogue will be slightly different depending upon the measurement type originally performed. The picture below shows the size view.
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Alter the appropriate parameter and press **OK**. It is advisable to add the modified parameter to the records list so the altered records can be instantly identified.



If the 21 CFR part 11 feature is installed, a **Reason for change** dialogue will appear so comments can be entered detailing what changes have been made.

The reasons for change, can be displayed in the **Records view** tab by selecting the **Measurement-Audit information-Reason** in the Workspace settings dialogue (Select **Configure-Workspaces-“.. workspace choice..”** and then the **Record view** parameters tab).

The result will instantly be reanalysed and the result added to the **Record view**.