

Zetasizer Pro Red Label

Sample Preparation Overview

A back scatter measurement can measure the size of any sample in which the particles are mobile. Therefore, high sample concentrations can be measured. However, each type of sample has its own ideal range of concentration where measurements should be made. This should be determined through a series of measurements to ensure that the size obtained is independent of concentration (ISO22412 (2017)).

Sample Dilution

Any sample dilution needs to be carefully performed to ensure that the equilibrium of any absorbed species between the particle surface and bulk solution is preserved. Diluent should be the same as the continuous phase of the original sample, making up a continuous phase as close as possible to that of the sample.

Diluent Filtration

Dust is one of the major problems in DLS measurements and may bias the results obtained. To avoid any possible dust contamination during dilution, the medium should be ideally filtered. Commercial syringe filters are available for use with pore sizes ranging from 1 μm down to 20 nm. If using syringe filters for the dispersant, discard the first few drops in case of any residual dust particles in the filter that may contaminate the dispersant.

A quick guide to making a measurement

1. **Close the lid and turn on the instrument by the power switch at the rear of the unit** (a “beep” will occur to indicate the instrument has been turned on and the initialization 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). **Wait 30 minutes for the laser to stabilize**, to prevent any thermal equilibration problems affecting the measurement results.
2. **Start the Zetasizer Nano software** by double click on the icon.
(Computer password: DLS).



3. **Choose the appropriate cell or cuvette** for the type of measurement being performed and the sample that will be measured. (The cells available for each measurement type, Size, and ζ -potential, are fully documented in the *Zetasizer Nano Accessories Manual* with some discussion on their use and application.

Disposable plastic cells (DTS0012) are usually used for Size analysis of water dispersed samples:



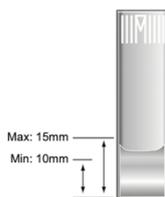
Disposable Folded Capillary Cells (DTS1070) are usually used for ζ -potential of water dispersed samples:



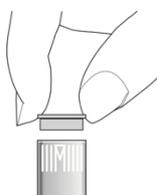
4. **Fill the cell or cuvette** with the sample prepared according to the Zetasizer Nano User Manual guidelines for each measurement type (Size and ζ -potential).

- **Size measurement:**

- Cells should be rinsed/cleaned with filtered dispersant before use.
- Fill the cell with between 1.0 mL and 1.5 mL of the sample by tilting the cell/cuvette and allowing the sample to flow slowly (this procedure avoids bubbles).
- Check to ensure the sample depth is between 10 mm and 15 mm by placing the cuvette against the diagram on the inside of the cell area lid, as shown:



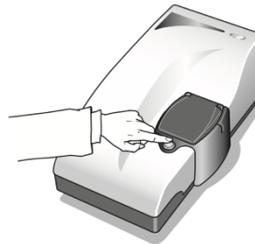
- Push the lid securely onto the cuvette as shown:



- Most cuvettes have a triangle or spot mark, as shown below. This mark must face towards the front of the Zetasizer Nano:



- Press the button to open the chamber lid:



- Push the cuvette down into the cell area on top of the instrument so that it is firmly located. Cover the cell with the thermal cap and then push the chamber lid down:

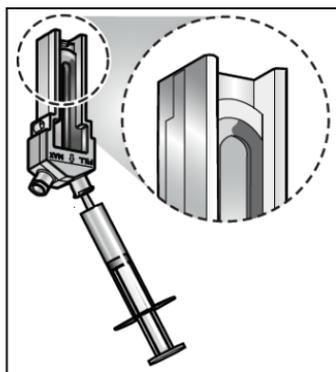


- **Z-potential measurement:**

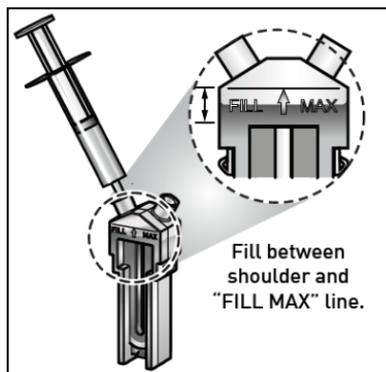
- It is recommended to flush the cell to ensure cleanliness and reduce the risk of bubble formation. The recommended procedure requires two syringes, filtered deionized (DI) water and ethanol or methanol:
 - Flush the cell with ethanol to facilitate wetting.
 - Fill one of the syringes with the DI water and place it in one of the sample ports on the cell, the empty syringe is placed into the other port.
 - Flush the contents of the full syringe, through the capillary, into the empty syringe, then flush back.
 - Repeat this process 4 more times before removing the syringes and performing a final flush with the dispersant being used for the measurement.

After this, the cell is ready for use. Never attempt to clean the optical surface of the folded capillary cell as this will cause small surface scratches that will give inaccurate results.

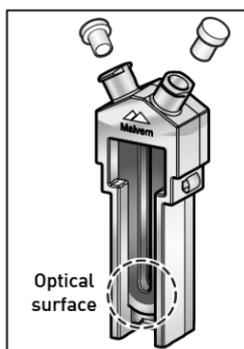
- Fill the cell according to the cell type below:



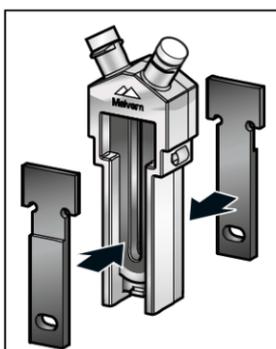
Invert the cell and slowly inject the sample from its syringe into the cell, filling the U tube to just over half way.



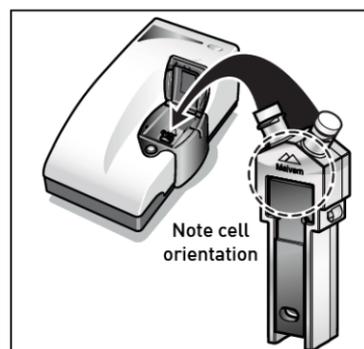
Check no air bubbles form in the cell - Tap the cell gently to dislodge any that form. Turn the cell upright and continue injecting slowly until the liquid reaches the fill area as shown. Remove syringe.



Fit one stopper firmly, the other one loosely, to avoid pressurisation of the cell. Remove any spillage from the electrodes.



Fit the thermal contact plates as shown. These are stored in the pull out cuvette holder, located under the instrument.



When inserting the cell, ensure that the Malvern logo faces towards the front of the instrument. Press down until the cell clicks into place. Before starting an experiment, ensure the software is set up to use the DTS1070 cell.

4. Making a manual measurement.

In the Zetasizer software, select Measure. This will open the manual measurement editor allowing any measurement types to be chosen and the settings to be configured: Name (sample name), Cell, Material (reference material for the viscosity), Dispersant, Project (operator's folder to save the data), Method builder (Size, ζ -potential, Titration, Other), Properties (Measurement, Data processing, Advanced settings).

5. Click start.

When a manual measurement is started the measurement display shows the progress of the measurement. The displayed tables depend upon the selected measurement type. Note the messages in the black status bar (near the base of the window) which shows the progress of the measurement.

If you want to interrupt the analysis, click the red button "stop method".

6. **Close the window** in the upper right corner of the measurement display when the measurement is finished.