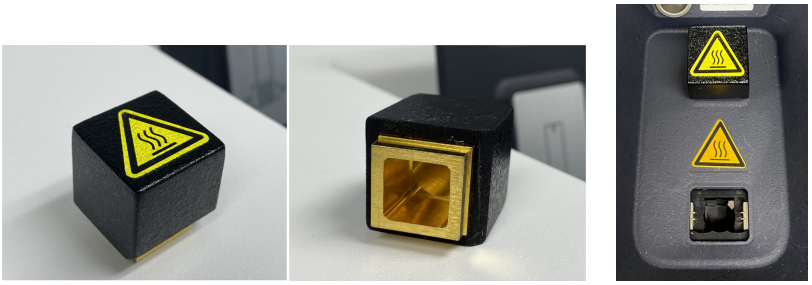
Dynamic Light Scattering Instrument (DLS) Quick Start Guide

**Since DLS purpose is characterize small particles (0.3 nm to 10 µm), please wear gloves during all the measurement process to avoid contamination get into the sample or the instrument*

1. Turn ON the DLS from the button at the back of the instrument
2. Check that the yellow light on top turns green. Wait 10 to 15 minutes for the laser to warm up [1]
3. Push the green button on the DLS to lift the cover
4. Notice that every cell/cuvette have a mark in the upper edge(for DTS0012 is an arrow, for the DTS1070 look for the MALVERN logo). Place the cell/cuvette so that the mark is facing you
5. If using temperature, place the Temperature cap over the cuvette.



(a) Top and inside view

(b) Not used position

Figure 1: Temperature Cap views

6. Close the DLS lid.

[1] You can use this time to prepare your sample. Check ***Sample Preparation*** in Apendix for a quick guide

7. Launch ZSXPLORER software

Figure 2: ZSXPLORER deskpot icon

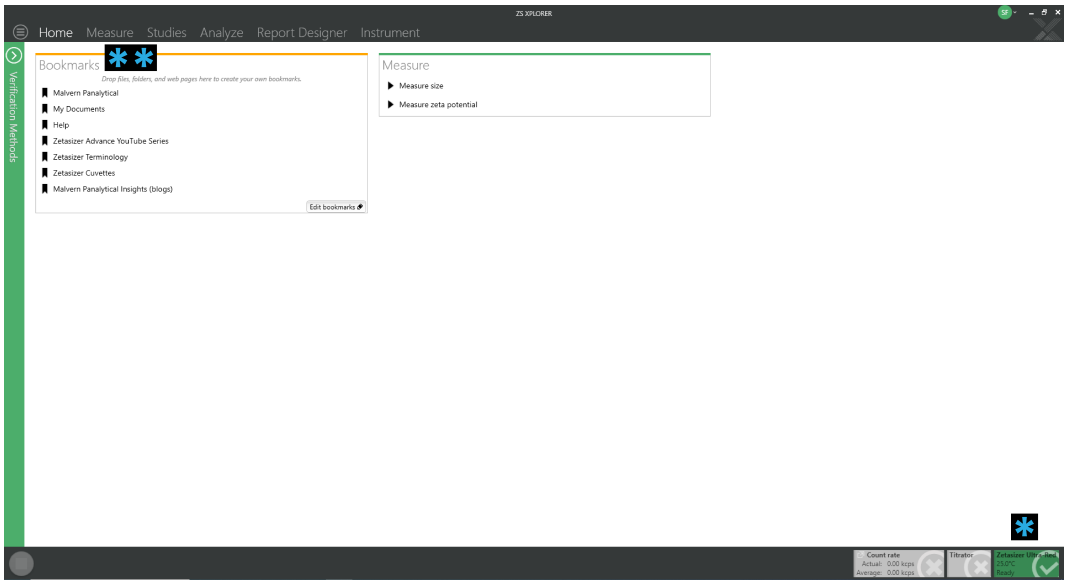


Figure 3: ZSXPLORER software main page

8. Go to Instrument Status icons (*) to check accessory connected, temperature[2] and status of the instrument

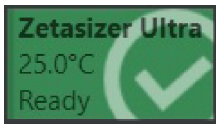


Figure 4: This icon indicates that instrument is ready for use

9. Go to Menu bar (**) and click on **Measurement** or **ZetaPotential**, depending of what you're measuring.

[2]If you are not using temperature for the measurement, the icon should indicate 25°C

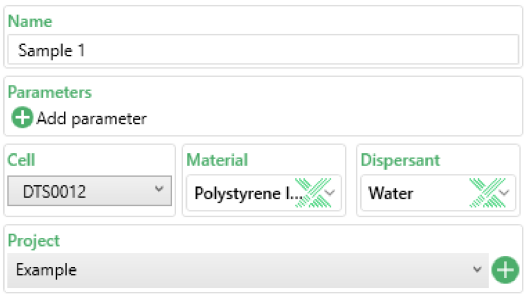


Figure 5: Section of basic sample information. Upper left part of the screen

10. In **Name** give your sample a name[3]
11. Click on **Cell** and select the kind of cell/cuvette you are using[4][5]
12. Click on **Material** and select the material your sample is made of[6]
13. Click on **Dispersant** and select the environment that your sample is in.
14. If this is your first time using DLS, in **Project** section create a new Project, save in your folder. If you already created one, you can open it from the folder icon at the top part of the window.
15. Click or drag to **Method Builder** section the desired type(s) of measurement(s). Once you add a method, a **Properties** section will appear

[3] Optional: in **Parameters** can be added other custom parameters to filter your results later

[4] The cell choice will affect which procedures are available

[5] If using organic solvent use a quartz or a glass cuvette. If using water based solve, use a plastic cuvette (DTS0012 and DTS1070 are made of polystyrene)

[6] Materials and dispersant can be edited or added to this list if necessary. In case of adding a material you'll need the refraction index and absorption

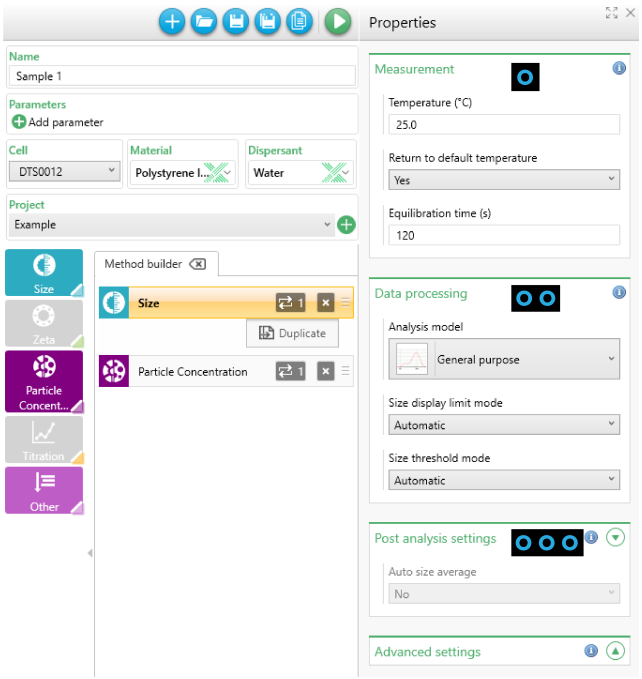


Figure 6: Properties of measurement and methods added for exemplification

16. *(Follow if temperature is required)* In Measurement properties box (o) define

    - **Temperature** to perform measurement[7]
    - **Equilibration Time** in which the sample will thermally stabilize once it has reached the specified temperature

17. In Data Processing box (oo) define

    - **Analysis Model**[8] based on your sample characteristics[9]. If you don't need a specific measurement use the default models **General Purpose** (for Size measurement) or **Auto-mode** (for Zeta potential measurement).
    - **Size Display Limit Mode** determines the range of possible measured sizes. **Automatic** is suitable for most samples[10]

[7] The upper limit is affected by the cell type and the dispersant used
[8] Analysis models for Size and Zeta potential measurements are different
[9] Check ***Analysis Models*** in Appendix if you want to choose a more specific model
[10] To avoid second-order scattering from adjacent particles, select the **Manual** option and indicate the lower and upper limits as appropriate

- **Size Threshold Mode** determines the range where the peak is going to be found. **Automatic** is suitable for most samples. However, you can click on **Manual** and indicate the lower and upper limits

18. In Post Analysis Settings (ooo) select whether to create an average after the measurement[11]

19. Click **Start Method** (green play button)

Figure 7: Monitor display for method in process. Graphs will begin to update with live data

20. The measurement(s) will show as Complete in the Method list (right side of the screen) when done

21. Check the **Count Rate** or **Correlogram** graph information to corroborate that the procedure ran correctly[12] a photon count in the range of 200-500 kcps and a correlogram with intercept 1 (or above 0.8) and with a smooth exponential decay are indicators of a good measurement

22. To extract measurements information result go to Menu bar (**) and click on **Analyze**

[11]It is recommended to select this option when creating a Size, Zeta, MADLS or Particle Concentration measurement with multiple repeats. PArticle concentration works for sizes up to 500nm

[12]Check ***Data Quality*** in Appendix to read about irregularities in these graphs

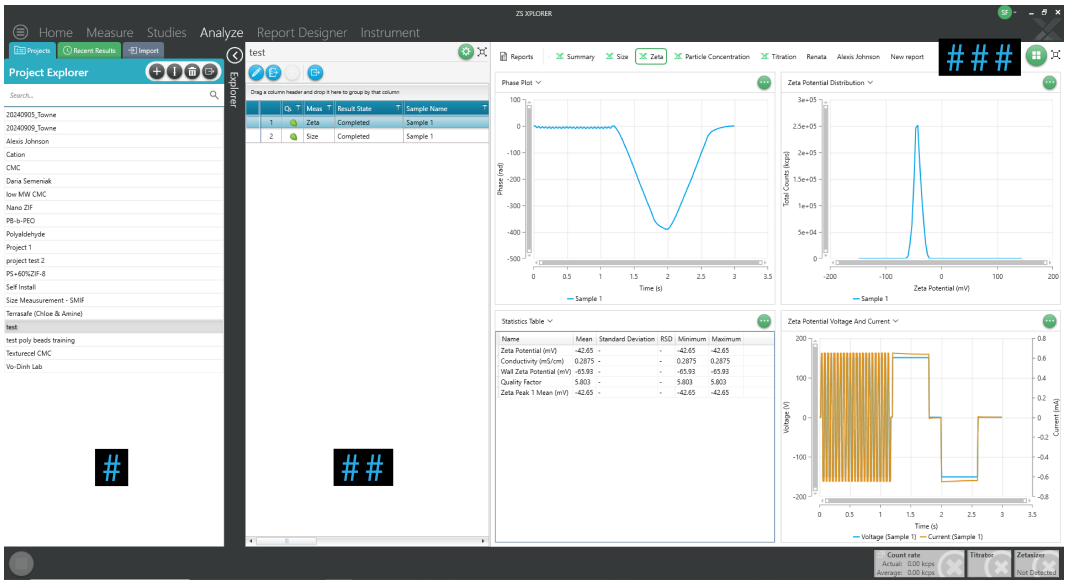


Figure 8: Monitor display for saved measurement

23. In the left size of your screen click on Explorer (#). Here you can find your project, click on it

24. A new window will display (##) with a list of your performed measurements. Click on the one for which you want to save the information. Choose size, if you did a SIZE measurements and ZETA POTENTIAL if you did a zetapotential measurement.

25. A window with all the information provided by the selected measurement will display. In the upper bar (###) click on the measurement performed to see graphs and statistics table

26. To save data from a graph, click the green button in the right upper edge of the figure and click on **Copy Data**. Then go to an Excel spreadsheet, paste the information and save the file[13]

[13]The PC have internet connection, you can send your file by email

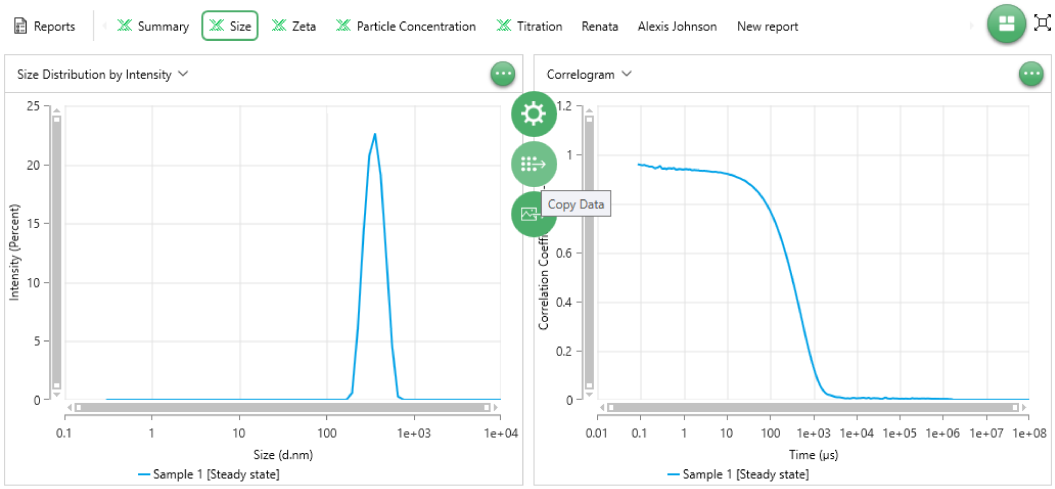


Figure 9: Save data from graph

27. Take off your sample from the DLS and leave the cover closed
28. If no user is signed up in *CoreResearch* to use the DLS on the same day or early the next day, switch OFF the instrument

Appendix

0.1 Sample Preparation

To prepare the sample you can bring your own equipment. Otherwise, at the right where the DLS is placed you can find a drawer labeled *DLS* with some supplies. Please register your name and what you used from the drawer in the spreadsheet file at desktop named *DLS1_Consumables*.
To prepare your sample

1. Select the right cell/cuvette according to the following information that indicates the uses for each cell/cuvette available in the laboratory

Cell	**Size**	**Zeta**	**MADLS**	**Particle Concen-tration**	**pH**	**Titra-tion**	**Molecular weight and k_D**
Plastic cell (DTS0012)	✓		✓	✓			✓
Low volume sizing cell (ZSU1002)	✓						
Folded capil-lary cell (DTS1070)	✓	✓			✓		
ZEN1002 Dip cell	✓	✓					

Table 1: Cell types available in the *Soft Materials Facility* and measurement compatibility

If temperature is required for measurement, only the DTS0012 should be used.

2. Select the proper dispersant. These are dived in two categories

- **Polar dispersants** - those with dielectric constant > 20, e.g. ethanol or water. This kind of dispersant is used when a Zeta measurement is performed[14]
- **Non-polar or low polarity dispersants** - those with dielectric constant < 20, e.g. hydrocarbons or higher alcohols. To measure samples in insulating media the ZEN1002 Dip Cell should be used[15]

3. Choose the right concentration of your material in the dispersant according to the following recommendations based on sample size[16]

Particle Size	Min. Concentration	Max. Concentration
$<$ 10 nm	0.5 mg/mL or at least 10,000 kcps	Only limited by the sample material interaction, gelation, etc.
10 nm to 100 nm	0.1 mg/mL	5% mass
100 nm to 1 µm	0.01 mg/mL (10 – 3% mass or at least 1,000 particles present)	1% mass
$>$ 1 µm	0.1 mg/mL (10 – 2% mass or at least 1,000 particles present)	1% mass

Table 2: Particle size and the appropriate sample concentrations

4. Filter the liquids used to dilute the sample. The size of the filter will be determined by the estimated size of the sample[17]
5. Prepare the solution by mixing the material and dispersant with the accurate concentration into the chosen cell/cuvette

[14]Zeta potential uses voltage to measure the electrophoretic mobility of a sample. This includes pH Titration measurements

[15]The material is required for its chemical compatibility and ability to generate strong electric fields with low voltages due to the close electrode spacing

[16]Sample concentration can have an impact on scattering levels and diffusion

[17]Filters down to 20 nm should consider chemical compatibility of the filter material when using non-aqueous solvents

6. At first observation, the concentration selected should cause the sample develops a slightly milky appearance - i.e. becomes slightly turbid[18]

Others consideration depending on type of measurement are

Particle Size

This type of measurement includes MADLS, Particle concentration, Molecular weight and pH Titration measurements.
For a reliable MADLS measurement, the concentration should be optimized in order to give good quality data in all angles. Prepare a ramp of different concentrations and measure them until the data stabilized.

Zeta potential

This type of measurement includes pH Titration measurements.
The minimum count rate that is acceptable for a zeta potential measurement to proceed is set to 10 kcps. According to that, to prepare your sample consider

1. Particle size is proportional to the scattered light. The larger the particle size, the lower the concentration should be

2. Particle refractive index will also be proportional to the scattered light

[18] If the concentration can't be choosen easily, various concentrations of the sample should be measured in order to detect concentration dependent effects. Check ***Data Quality*** in Appendix for more information

0.2 Analysis Models

Analysis Model	Description
General purpose	The default processing type. General purpose should be suitable for most samples
General purpose extended range	This model is appropriate for samples where indicative information about larger particles is required
Multiple narrow modes	This analysis model should be used when you know you have more than one peak, and the peaks are narrow. This analysis method gives a higher resolution and will resolve the peaks more effectively
L-curve analysis	This analysis method optimizes the distribution result to give the highest possible resolution while maintaining minimal noise. This process is suitable for low scattering samples

Table 3: Models available for Size measurement

Analysis Model	Description
Auto-mode	The default setting for Zeta potential measurements. The software automatically selects the most appropriate model to use based on the cell type chosen, dispersant properties, and measured sample conductivity
General purpose	Applicable for most Zeta potential measurements where a distribution plot is needed
Monomodal	This should be used for samples in high conductivity dispersant, fast measurements, protein samples, diffusion barrier measurements, and when a distribution plot is not needed

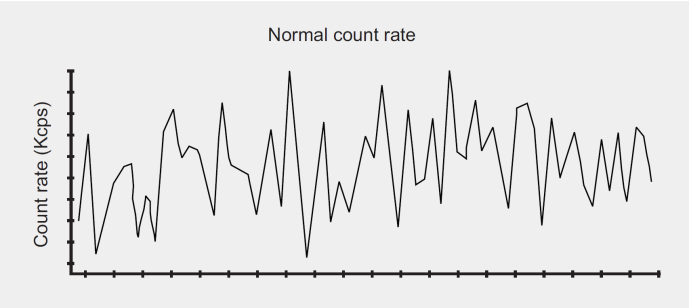
Table 4: Models available for Zeta potential measurement

0.3 Data Quality

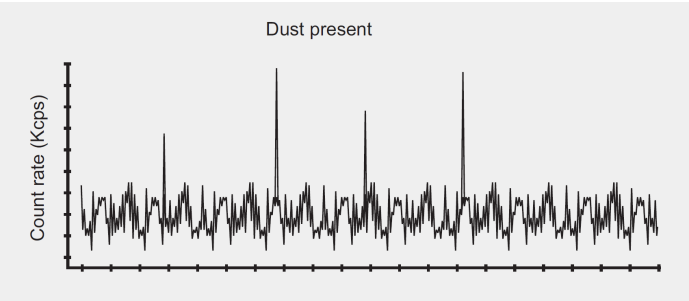
For a quick assessment of your data quality, the following information highlights potential irregularities to watch for and what they may indicate[19].

Count Rate Graph

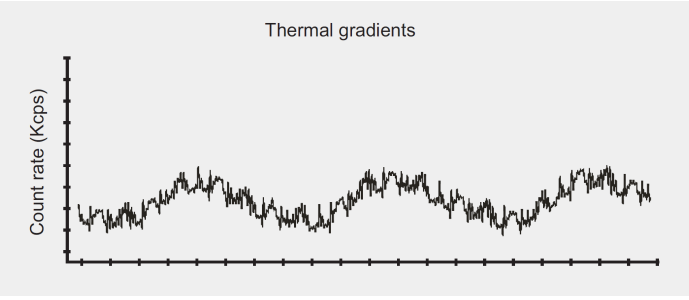
Normal count rate of photons detected per second



If dust, larger particles, or aggregates are present then sharp spikes will be observed



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



[19] If the issues persist even when you modified your sample, try to make a ramp of concentration and measure them until the data stabilized

Correlogram Graph

The upper images contain normal correlograms and the graphs must intersect the y-axis at the point $y = 1$. The lower images indicates inconvenience with the sample