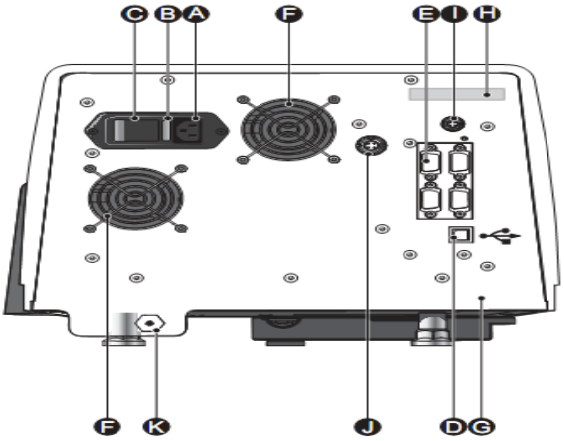
SOP for DLS and Zeta potential using Malvern Zetasizer Nano ZS

1. Turn on the zetasizer 30 minutes prior to measurement using the power switch at the back (C).



2. Sample preparation:

For size measurement:

-Prepare 0.5-2ml depending on the quivette used

-Test several sample concentration to make sure the range is correct

-In case organic buffers are used do not use plastic quivette

- Do not use polystyrene quivette above 50C !

- Fill the cell slowly to avoid air bubbles being created

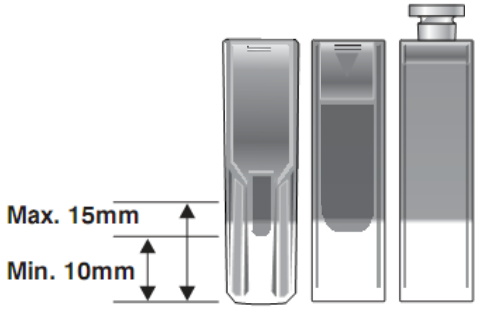
Appropriate cells:

From right to left:

-Quartz quivette

- polystyrene quivette

-Low volume polystyrene quivette (400μl)

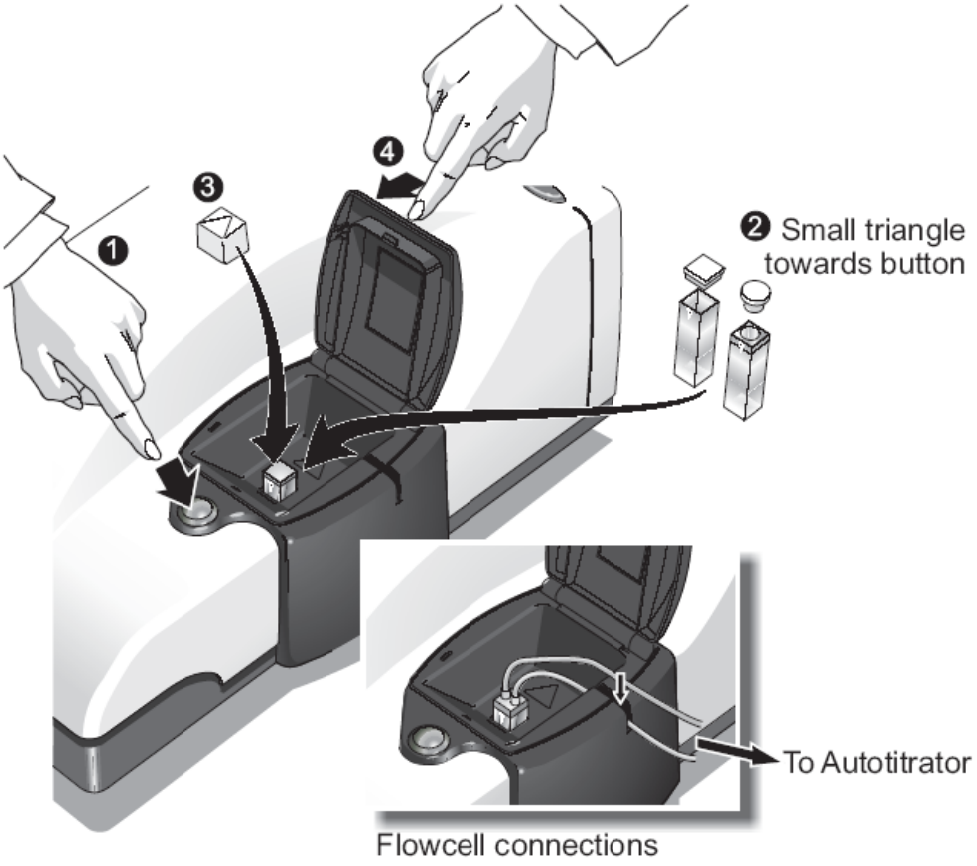


Inserting the cell:

-Press on button 1: the door will open

-Insert cell with the small triangle towards the button

-Close the lid



For zeta measurement:

-Prepare 1ml of sample

-Salt concentration should be low (Maximum sample conductivity 200mS/cm)

-No organic solvents can be used

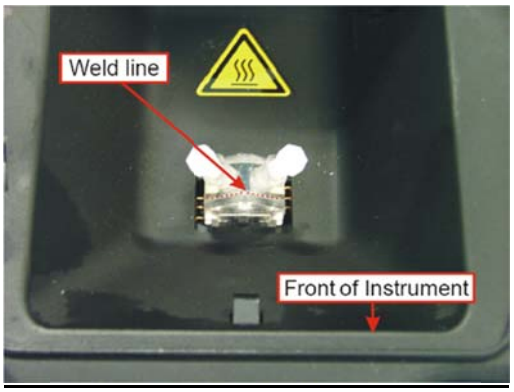
-Rinse the folded capillary cell thoroughly with ethanol

-Rinse thoroughly with RO water

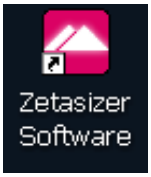
-Rinse with your sample dispersant (optional)

-Fill the cell with the sample for measurement using a syringe and tilting the cell sideways

Orientation and Insertion of Capillary Cell



3. Start the Zetasizer software

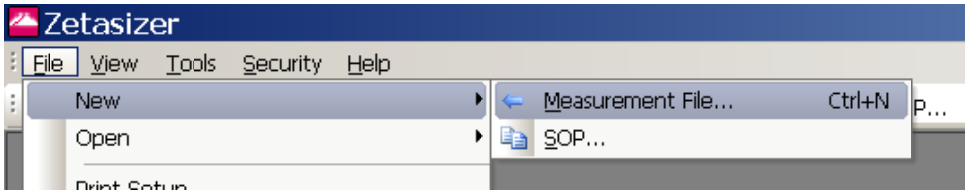


4. Create a measurement file

This is the file in which the results would be saved

Select

File->New->Measurement file



Save as: file_name.dts

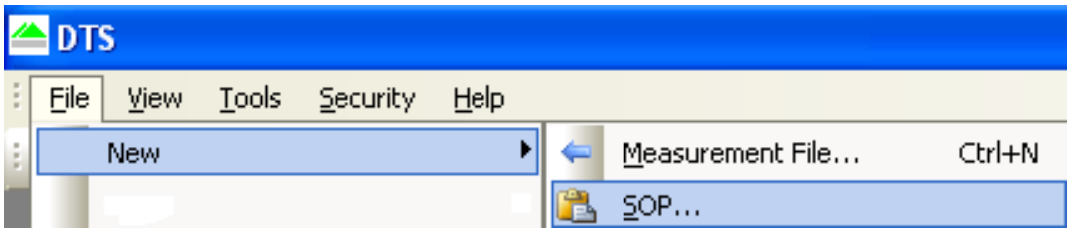
5. Create an SOP file.

SOP file can be used for all samples that share sample materials and dispersant ensuring similar measurement conditions

To create a new measurement file:

Select

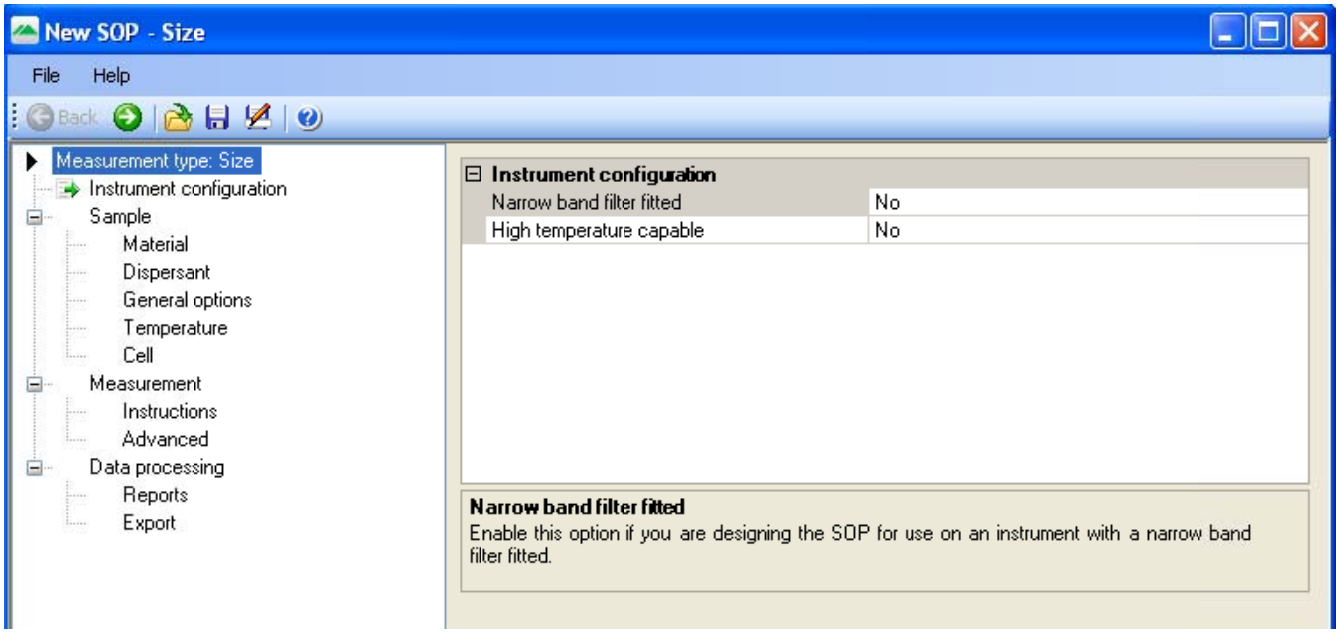
File->New.



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

SOP Editor Tree Menu View:

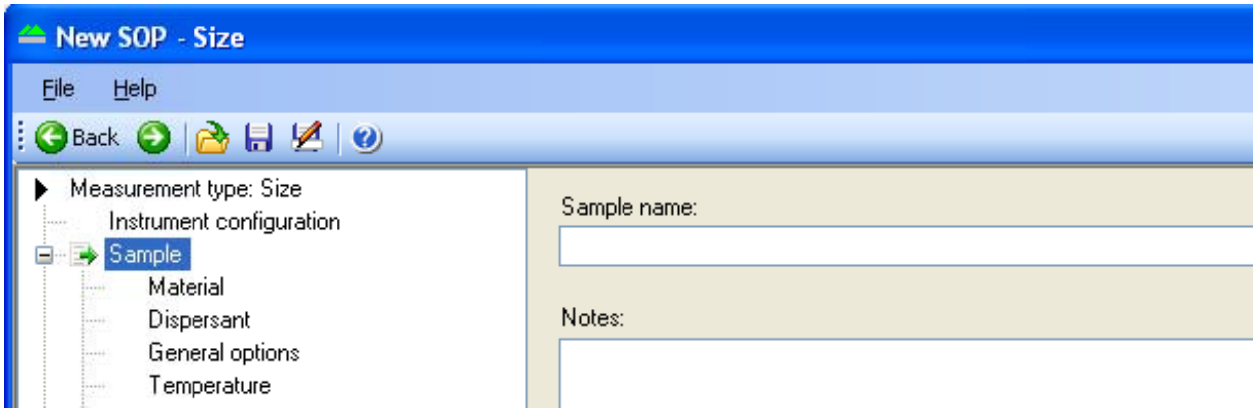
Measurement Type



Sample

Allows the measurement record to be named

All measurements made using this SOP are given the sample name entered in the SOP by default, but there is the option of changing this when the measurement is started

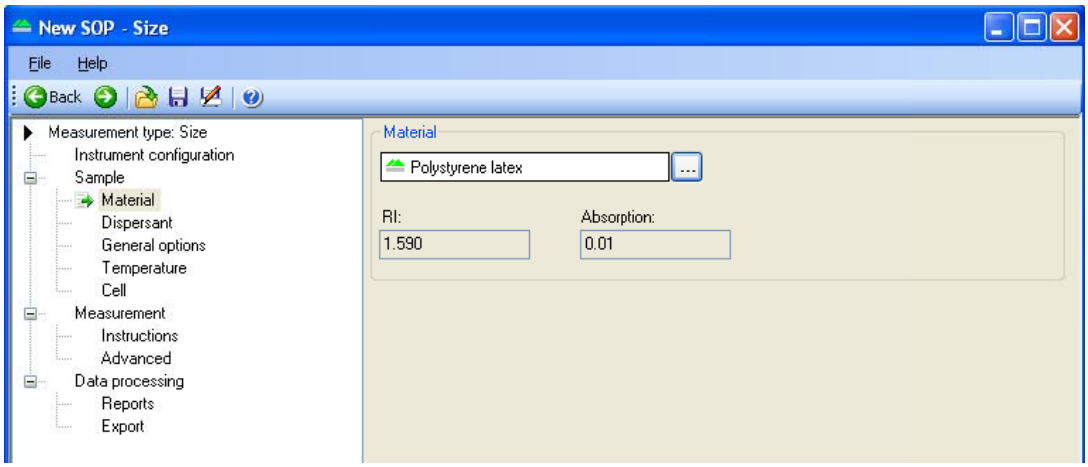


Sample – Material

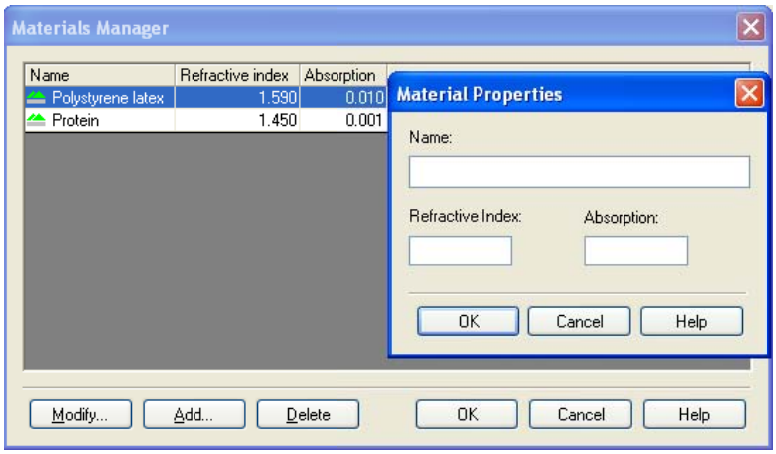
-Allows the optical properties of the sample material to be set

- Refractive Index (RI)
- Absorption

-Clicking on the … button accesses the Material Properties Manager



-You can add sample materials that are not listed in the database but you should supply their RI and absorption

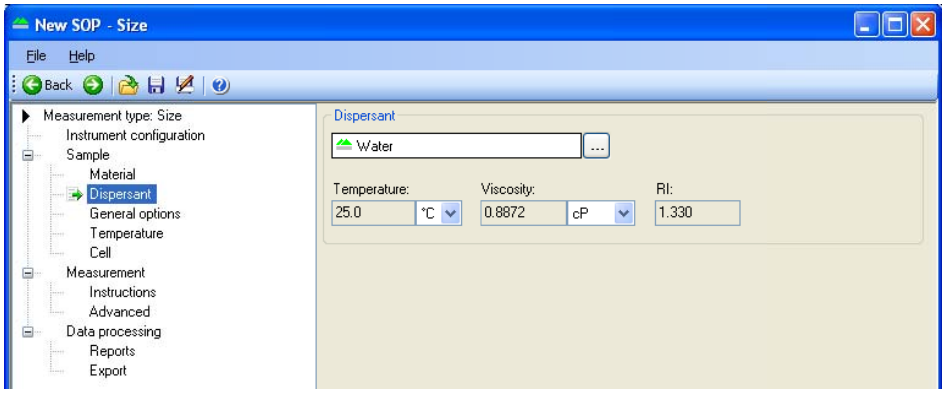


Sample – Dispersant

-Allows the physical properties of the dispersant to be entered

- Refractive index (RI)
- Viscosity

-Clicking on the ... button accesses the Dispersant Properties Manager



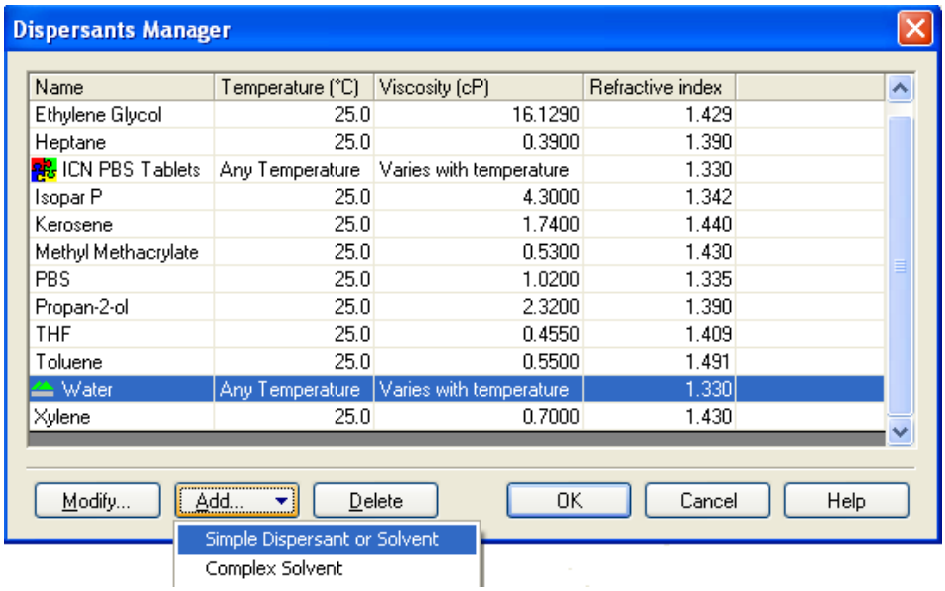
Sample - Dispersant - Dispersants Manager (1)

-From the list displayed, an available dispersant can be selected for inclusion into the SOP

-Alternatively, a dispersant can be added, modified or deleted from the list

-To add a new dispersant, press the Add button and choose either

- Simple Dispersant or Solvent
- Complex Solvent

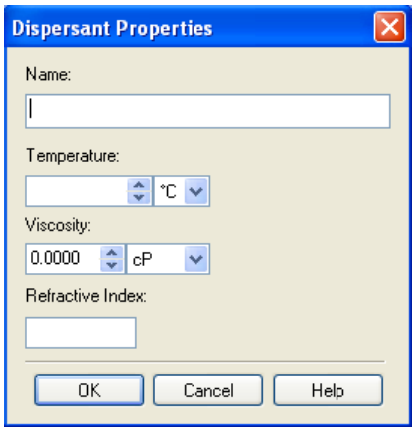


Sample - Dispersant - Dispersants Manager (2)

-On selection of a Simple dispersant or solvent, the Dispersant properties dialogue will appear allowing new dispersants to be defined

-The dispersant name, refractive index and viscosity must all be specified

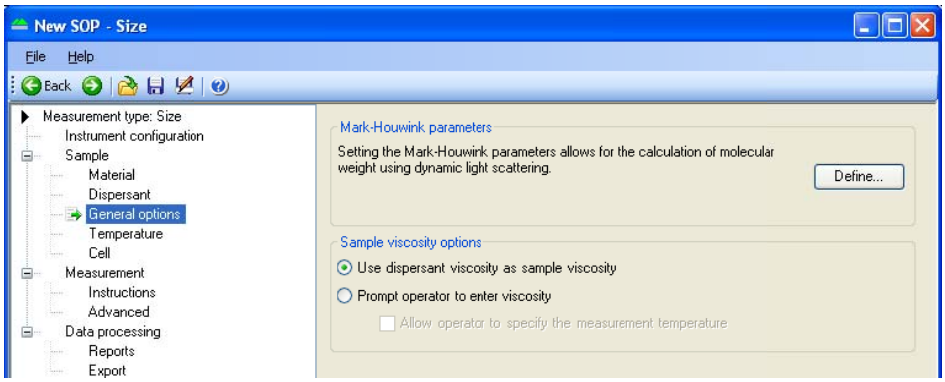
-Malvern defined dispersants include a built in viscosity calculation that determines the correct viscosity of the dispersant at any sample temperature



Sample - General Options

-Allows the Mark-Houwink parameters to be entered enabling a molecular weight to be calculated based on the DLS data

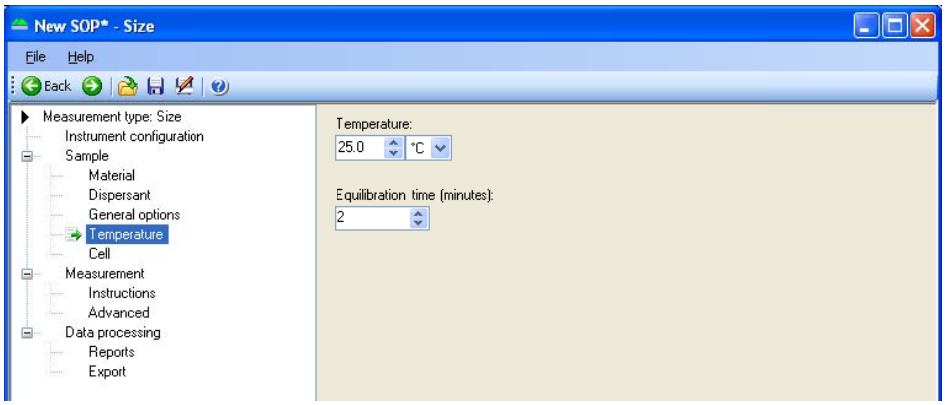
-The sample viscosity options are useful for customising the viscosity parameters



Sample – Temperature

-Allows the measurement temperature and any equilibration time to be defined

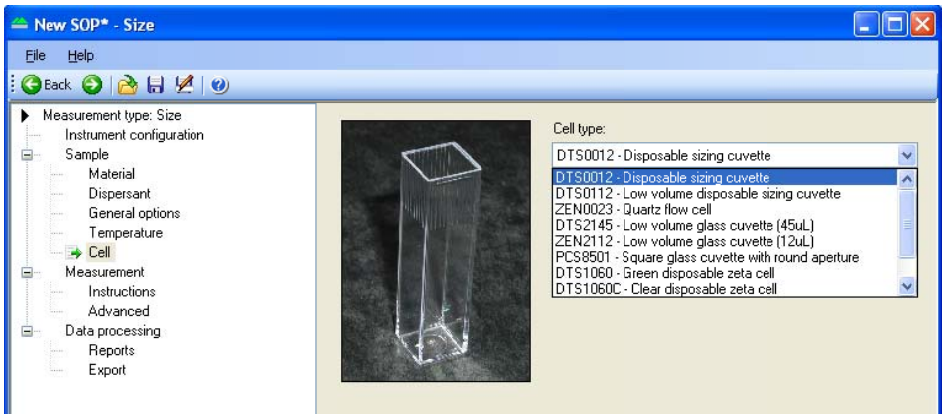
-The equilibration time adds a delay before the start of each measurement to ensure that the sample temperature is equal to the cell area temperature



Sample - Cell

-Allows selection of the appropriate cell from the list

-The software will automatically determine the optimum measurement position for the cell type selected in a back scatter system

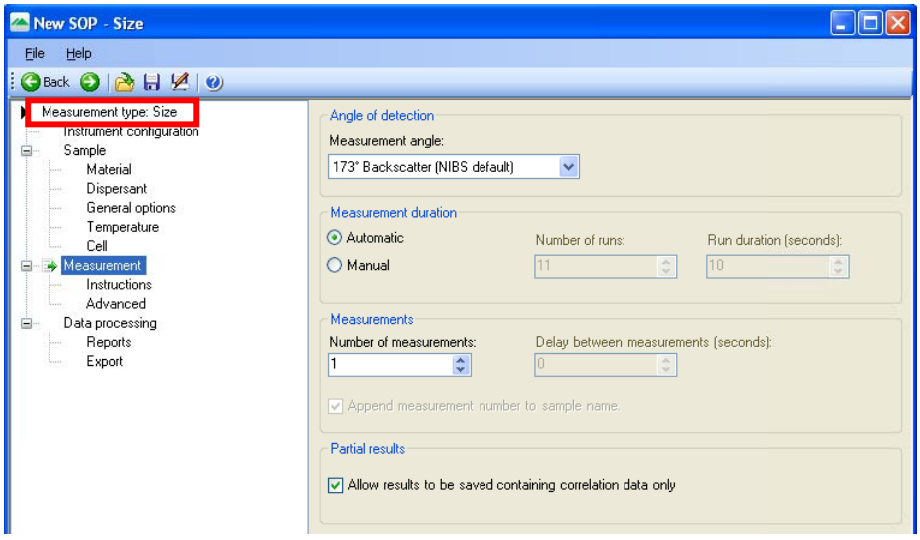


Measurement

-Measurement angle

-Measurement duration

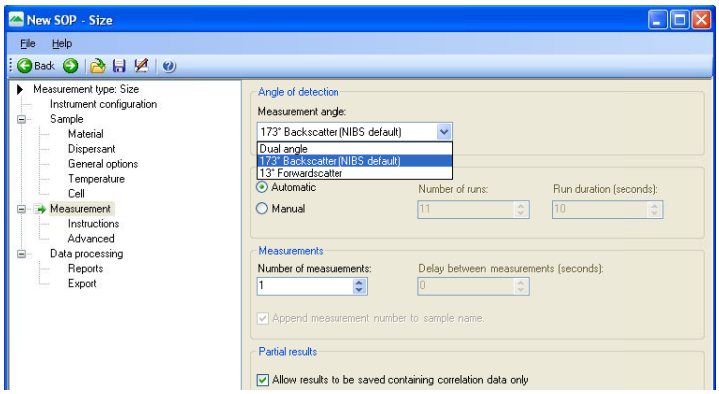
-Number of repeat measurements (I recommend 4)



-Dual angle (13 and 173o)

-173o Backscatter NIBS (default)

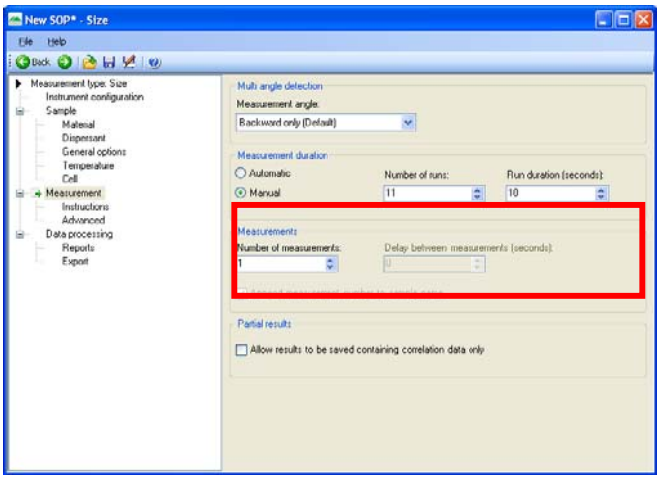
-13o Forwardscatter



Measurement - Measurements

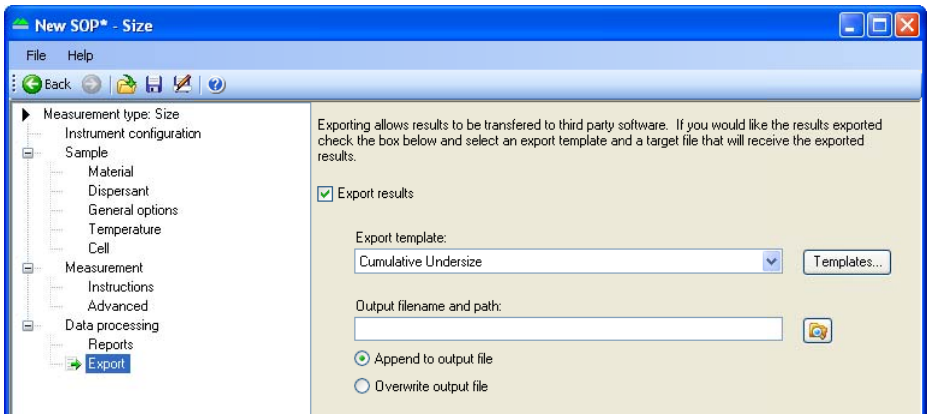
-The Measurements option allows the number of repeat measurements on a sample to be defined

-A delay between measurements can be specified by adding a time in the appropriate box

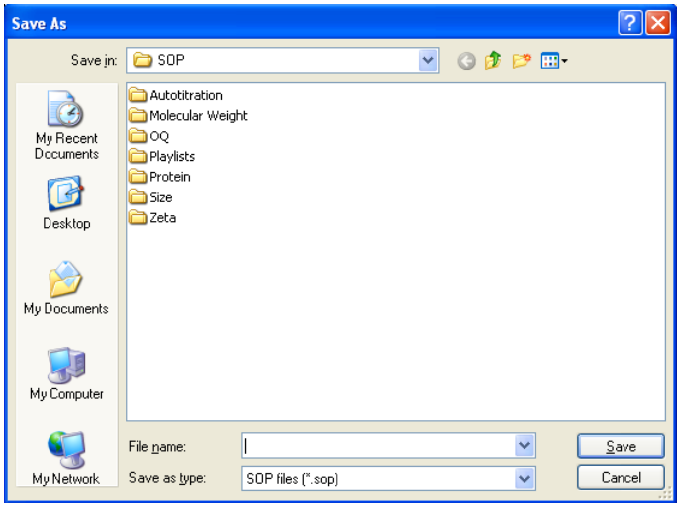


Saving the SOP

Once the SOP has been completed, it can be saved either by clicking on File –> Save or on the Disk  icon on the toolbar



This will bring up a window which will allow the SOP to be named and saved into an appropriate sub-directory, e.g. Size (but you can also save it with your measurement data)



Save as: SOP_name.sop

<u>To open an existing SOP file:</u>

Select

File->Open.

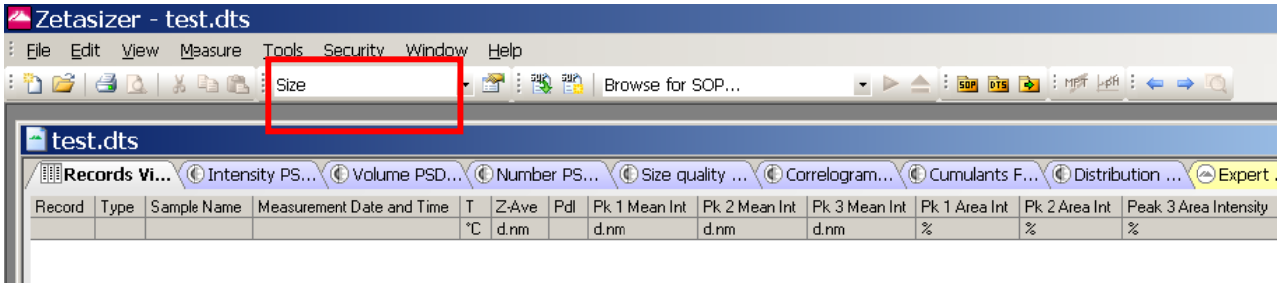
A dialogue will appear allowing selection of a measurement file.

Select Open.

6. Measuring a sample

-Create a measurement file (detailed above)

-Make sure you choose the correct workspace: size/zeta



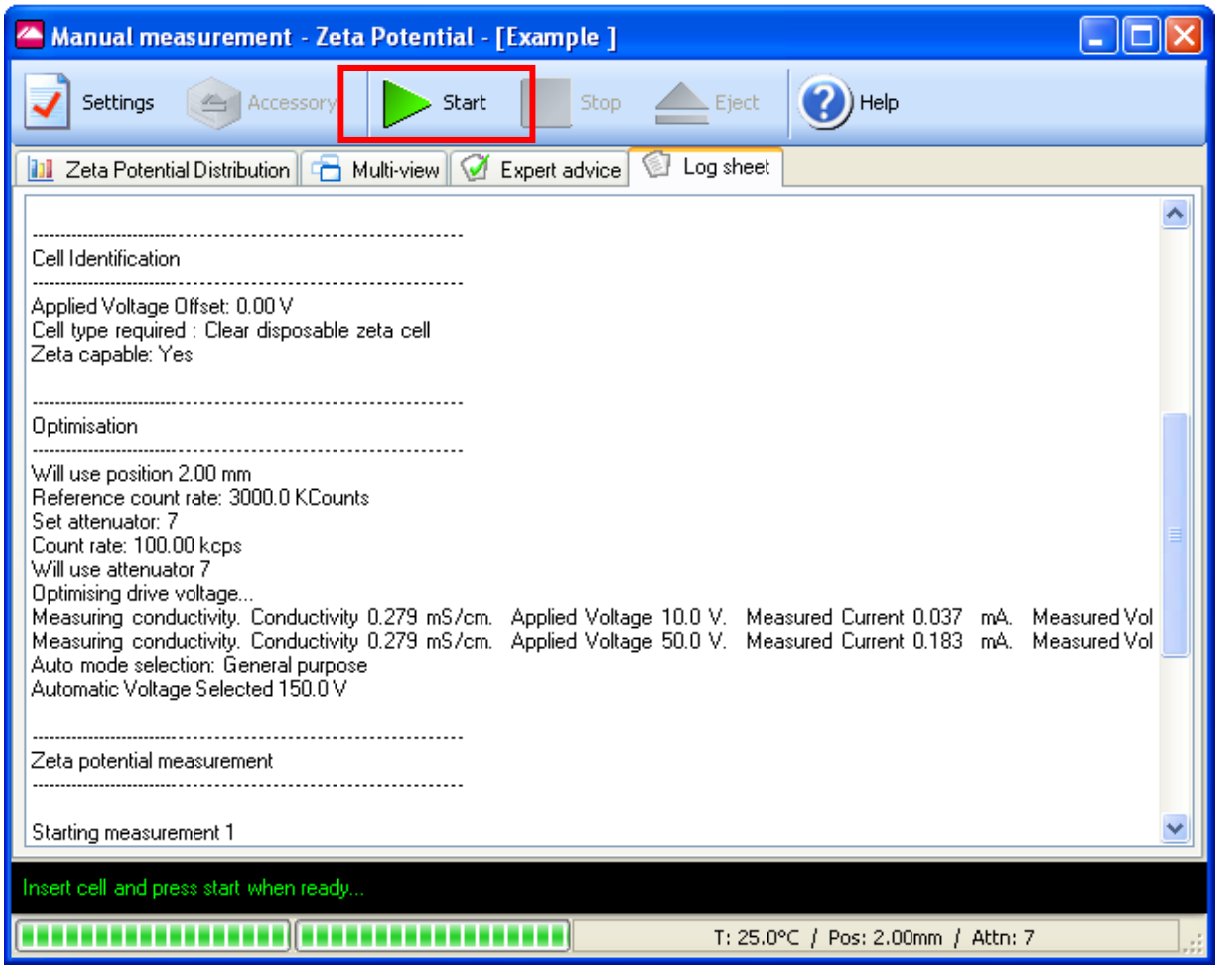
-Start the appropriate SOP by choosing:

Measure-> Start SOP

-You can add sample name and press OK

-A Measurement Display Window will open

-Insert the sample as detailed above close the lid and press start



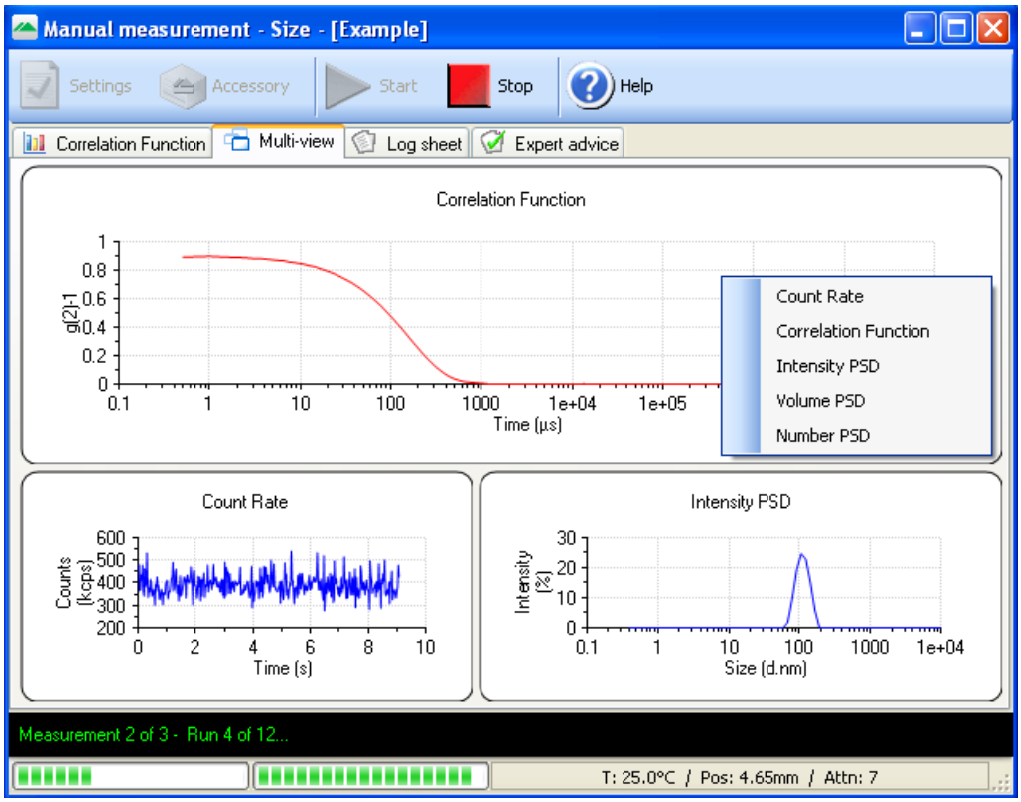
-After 2 min of equilibration there is adjustment of optics and the mesuarment will begin

The multi view tab offers view of the following:

For size:

The Multi-View tab consists of 3 display areas, each of which can be manually defined by clicking the right mouse button and selecting from a list of plots

- Count Rate
- Correlation Function
- Intensity PSD
- Volume PSD
- Number PSD



For Zeta:

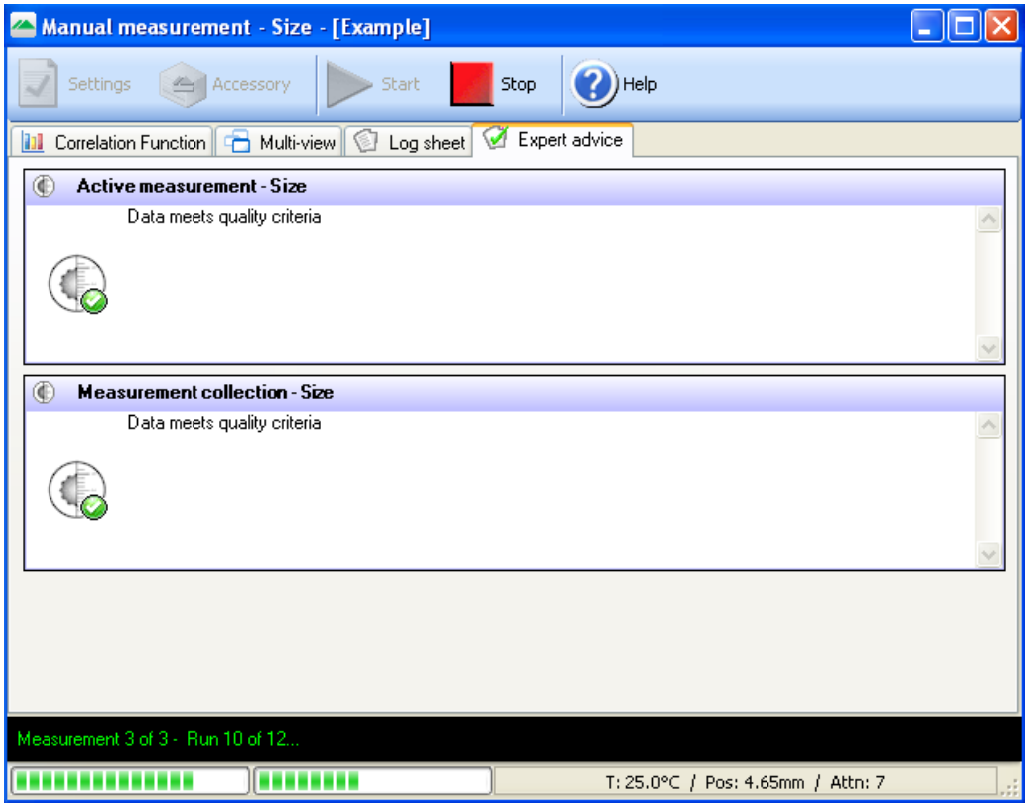
The Multi-View tab consists of 3 display areas, each of which can be manually defined by clicking the right mouse button and selecting from a list of plots

- Zeta Potential Distribution
- Electrophoretic Mobility Distribution
- Count Rate Trace
- Phase Plot

The Expert advice tab:

-The Expert Tab is a live report of the quality of the data being generated for both a single measurement and a series of repeat measurements

-Comments are given about variability in the results from changes in the sample as a result of, for example, aggregation or dissolution, or where the first measurement is different because the sample has not had time to thermally equilibrate.



7. Viewing your results

The following tabs can be viewed using the measurement file:

Record view- summarizes data obtained from all measurements in a table that can be exported to xls file (details below)

Intensity PSD

Section 1 gives details of parameters relating to the sample: sample and SOP name, the file name where the record is saved, the record number, sample and dispersant refractive indices and viscosities and the date and time when the measurement was made.

Section 2 gives details on instrument settings for this particular measurement. Specifically, these are the measured temperature at the start of the measurement. The average count rate for the measurement in kilo counts per second of k c p s for short. The cell type used to make the measurement. The duration of the measurement used in the analysis of the result in seconds. The measurement position in the cuvette used to make the measurement and the attenuator position used to make the measurement. The measurement position will be fixed for 90 degree.

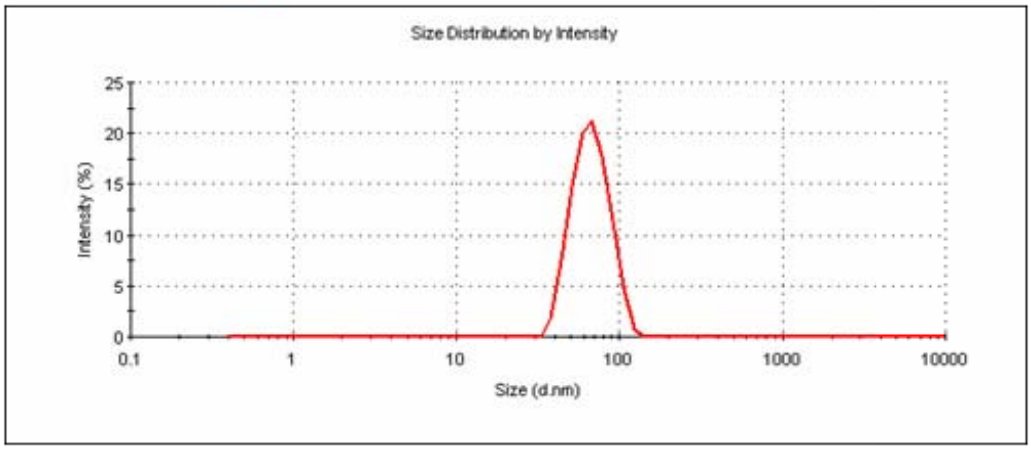
Section 3 gives the results of the measurement.

Section 4 shows the result in graphical form. The result shown is a plot of, on the Y axis, the relative intensity of light scattered by particles in various size classes, on the X-axis. This plot is therefore known as an intensity particle size distribution. The z-average diameter is the mean hydrodynamic diameter based upon the intensity of scattered light. The P D I, the polydispersity index, is a dimensionless estimate of the width of the distribution. These will be discussed in more detail in a later training module. The intercept is the amplitude of the correlation function at time zero. For a good measurement this will between zero point eight five and zero point nine five.

Records View | Intensity PSD...

Sample Name:	60nm Latex		
SOP Name:	Manual measurement settings		
File Name:	Version 4 Example results.dts	**Dispersant Name:**	Water
Record Number:	28	**Dispersant RI:**	1.330
Material RI:	1.59	**Viscosity (cP):**	0.8872
Material Absorbtion:	0.01	**Measurement Date and Time:**	06 July 2004 09:55:06
Temperature (°C):	25.0	**Duration Used (s):**	70
Count Rate (kcps):	189.7	**Measurement Position (mm):**	4.65
Cell Description:	Disposable sizing cuvette	**Attenuator:**	6

			Diam. (nm)	% Intensity	Width (nm)
Z-Average (d.nm):	64.38	**Peak 1:**	68.11	100.0	17.16
PdI:	0.035	**Peak 2:**	0.000	0.0	0.000
Intercept:	0.912	**Peak 3:**	0.000	0.0	0.000
Result quality :	Good				

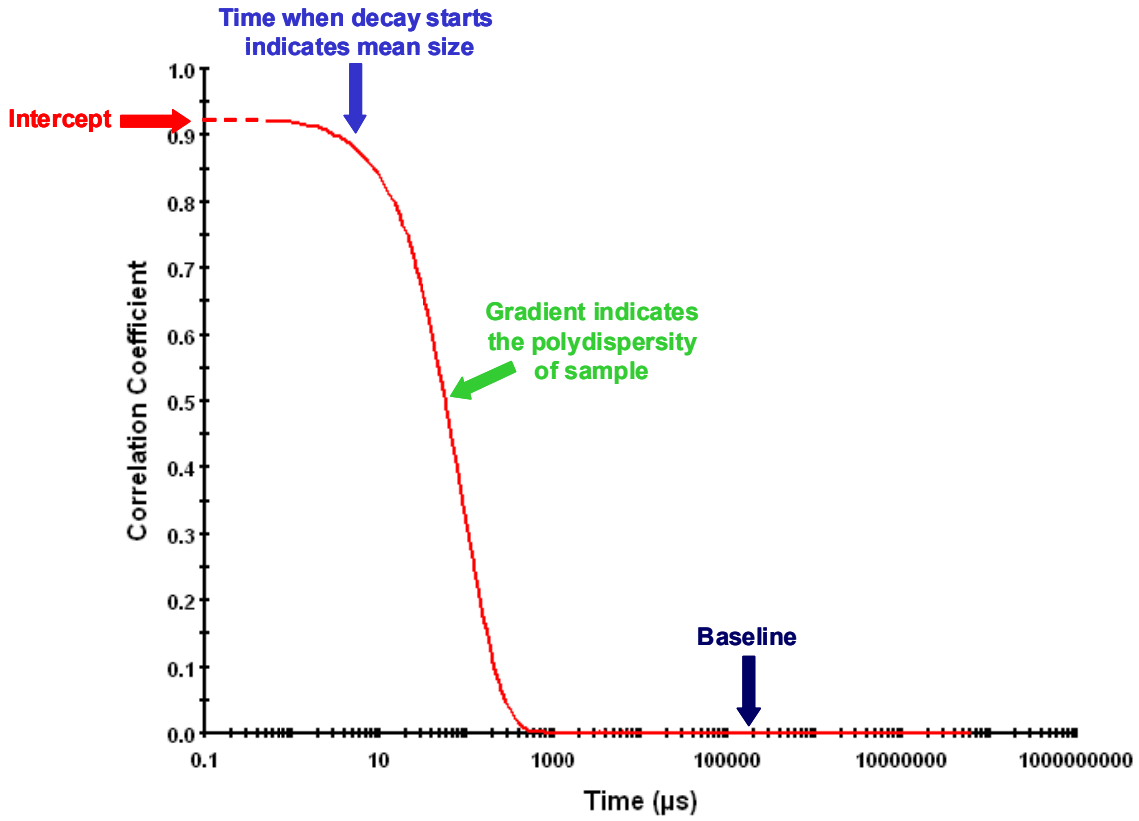


Correlation Functions:

-The correlogram shows the correlation coefficients displayed in each delay time channel and provides information about the sample

-The shape of the curve will show some obvious problems that may be present

-The correlogram ought to be checked for noise contained within the data Noisy data can result for various reasons; count rate too low, instability of the sample or external effects such as vibration or interference from another source



Cumulants and Distribution Fits

-The cumulants fit report shows the quality of the cumulants fit to the measured data indicating whether the z-average diameter and polydispersity obtained for a particular measurement are reliable

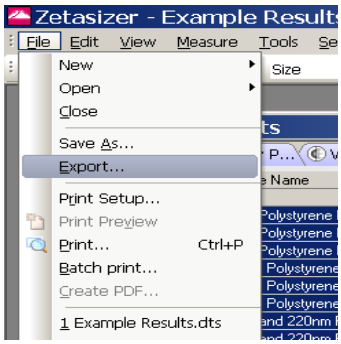
-The multimodal fit report shows the quality of the fit of the data with either the general purpose or multiple narrow modes analysis indicating whether the intensity size distribution obtained is reliable or not

-Fit errors of less than 0.005 are considered good

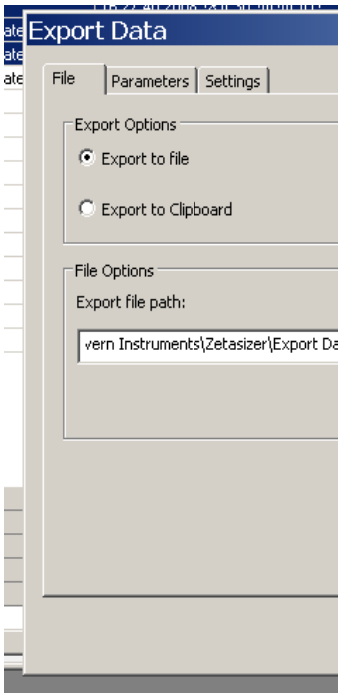
8. Exporting your data

-Mark requested lines in the records view

-File-> export



-The following dialogue



-Specify file name and r

-The format can be eith

-The file can be opened

<u>To export images:</u>

-Go to the tab with the r

-Edit-> copy graph