Malvern Zetasizer Nano Basic Operating Instructions
08/14/2015 S.V.

Location: **1238 Hach Hall**
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Safety

All researchers using the Zetasizer Nano instrument in 1238 Hach Hall must complete the EH&S course: *"Fire Safety and Extinguisher Training"*. When preparing samples in this room, please wear all appropriate personal protective equipment. Aprons, safety glasses, and rubber gloves are available in 1238A Hach Hall. If solvents are involved, consider preparing your sample in room 1238A.

Properly dispose of glass pipettes in the container provided. Waste solvents can be disposed of in the waste containers provided in 1238A. All of the computers in this lab have direct links from the desktop to MSDS sheets, the EH&S Laboratory Safety Manual and to the CIF Safety Manual.

Laboratory Safety Manual

Malvern Zetasizer Nano ZS

**Malvern Zetasizer Nano Dynamic Light Scattering Instrument**

This instrument is capable of measuring both particle size and zeta-potential for solids in suspension.

To begin…

Turn the monitor on. To save energy, the monitor is normally powered off when not in use. However, the computer should always be left on. The Zetasizer instrument is also always left on.

Log into the PC using your username and password. The operating system is Windows 7.   Your account has been established with User privileges. If you encounter limits anywhere in the application software, please consult with me.   Billing is based upon your logon and logoff times, so be sure to log out when you are done working. Your data is also stored automatically in our research cloud in the same location as your NMR, MS and X-ray data.
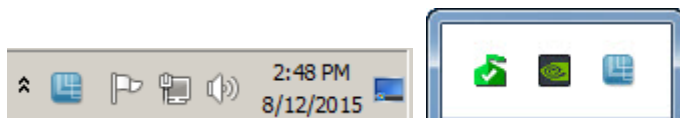
Desktop Organization.   Your desktop should contain several program shortcuts, PDF and MPEG training guides, and an Errors folder.
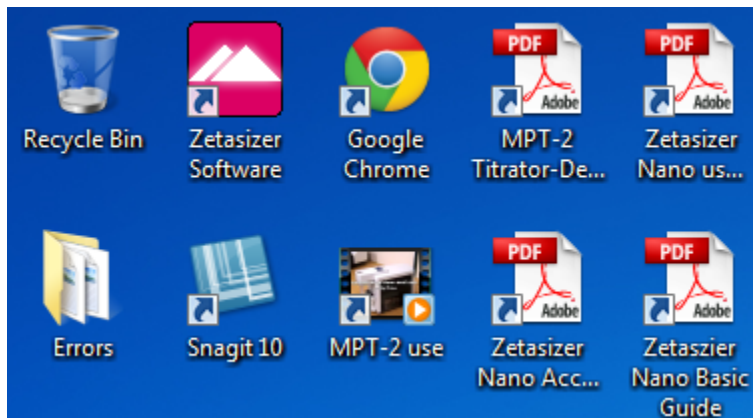
The taskbar should have the following program links:

The Taskbar notification area should show the following:

Capturing errors.   **Word** and **Snag-It** are present so that you can efficiently capture any errors or software quirks that you encounter. After you logon to the PC, start a Word document and save it in the Errors folder on the desktop; name it with the current date using the format *yyyy-mm-dd* (e.g. 2015-08-

13). Use Snag-It or Snipping Tool to screen capture the error or software anomaly; paste into the Word document. You MUST do this. Maintaining an accurate record of errors and anomalies is critical to obtaining proper support from the instrument company.

Software Organization. The Zetasizer software is well designed for multi-user environments and includes number of security and access level features that we may implement in the future. For the moment, all users will have all privileges.

The first time the program is started by a user, a standardized directory structure is established. All data files, macros, scripts, SOP's (standard operating procedures), reports, log files, et cetera will accumulate here. The path created is:
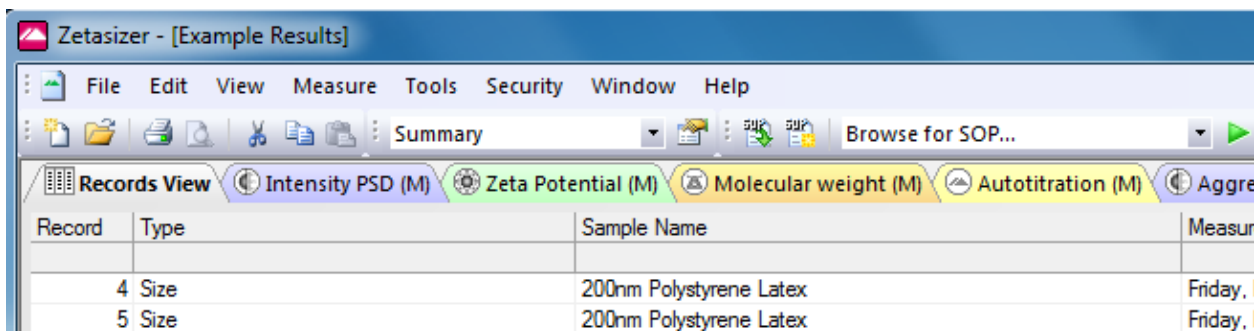
C:\Users\<username>\Documents\Malvern Instruments\Zetasizer\...

Without security settings enabled, the program will usually allow you to browse to see other users' data files and SOPs.

Starting the Software. Click on the desktop Zetasizer icon. You will be presented with a login prompt.
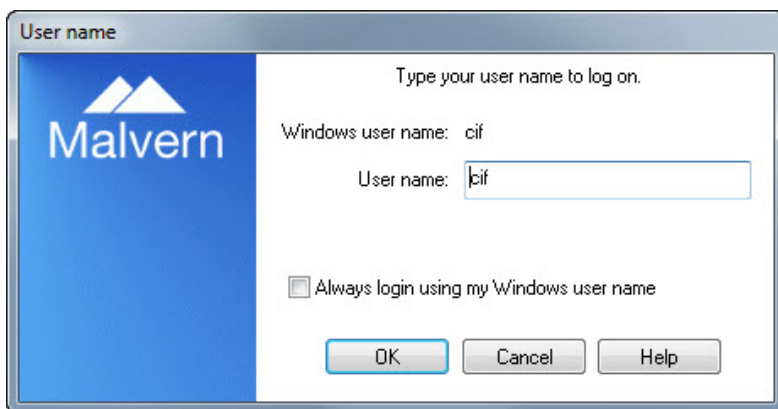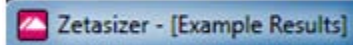

The software will open showing the last **workspace view** and **measurement file** you accessed.

You will also notice that within a few seconds the handshake with the Zetasizer instrument is established .

Measurement Files

In this example, the "measurement file" is <u>Example Results</u> as indicated at the top of the view. As expected, you can use the "*File->Open->Measurement File*" command (or the ribbon bar icon) to navigate to different measurement files. Measurement files usually contain multiple measurements (called records), each with a record number.   Similarly, you can use "*File->New->Measurement File*" (or the ribbon bar icon) to start a new set of measurements.   You should organize your work logically using the concept of measurement files, each containing multiple measurements (records) pertaining to a sample or project.

Workspaces

 In this example, the "workspace" is <u>Summary</u> as indicated in the workspace dropdown selection located towards the left in the ribbon bar.   A workspace is a configurable selection of tabs, usually containing reports, calculations, or result summaries appropriate to the type of record measurements performed.   The Summary workspace is currently configured to show all measurements (records) of all types in the measurement file. It contains sufficient tabs to show results for most types of measurements (e.g. size, zeta...) that might be in the measurement file. The workspaces currently configured are shown below. Note that invoking the workspace configuration editor allows you to completely customize each workspace. Do NOT edit the existing workspaces *[this advice may change]*. Create your own workspace if none of these suffice.
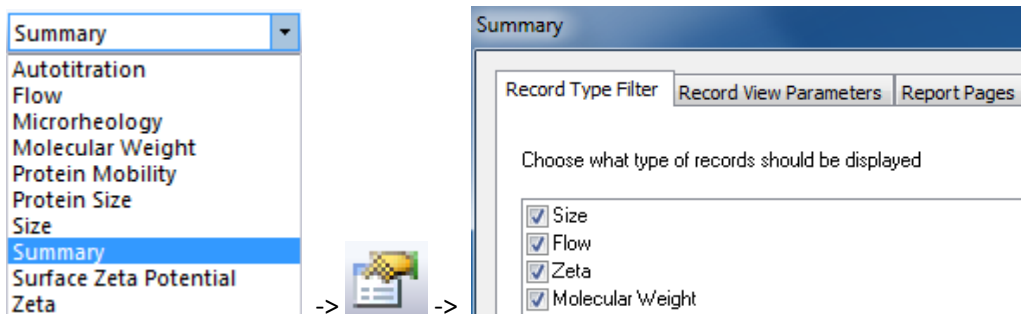
Note the extensive level of control you have in configuring the record view parameters and report pages for each workspace:
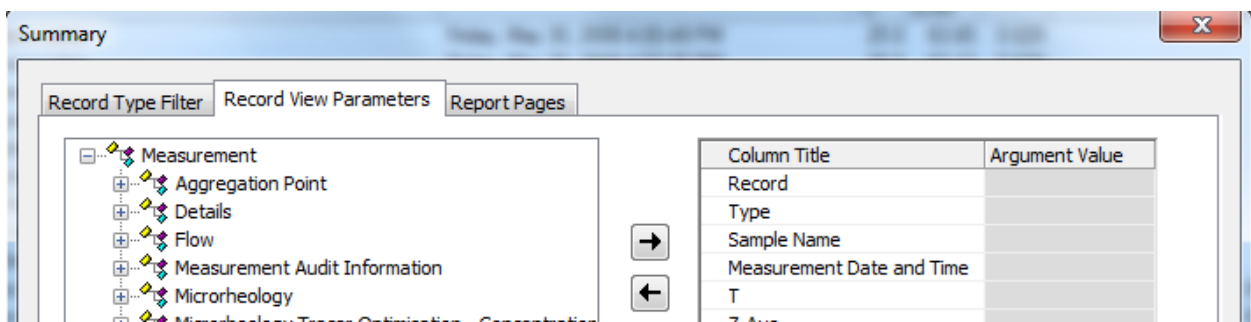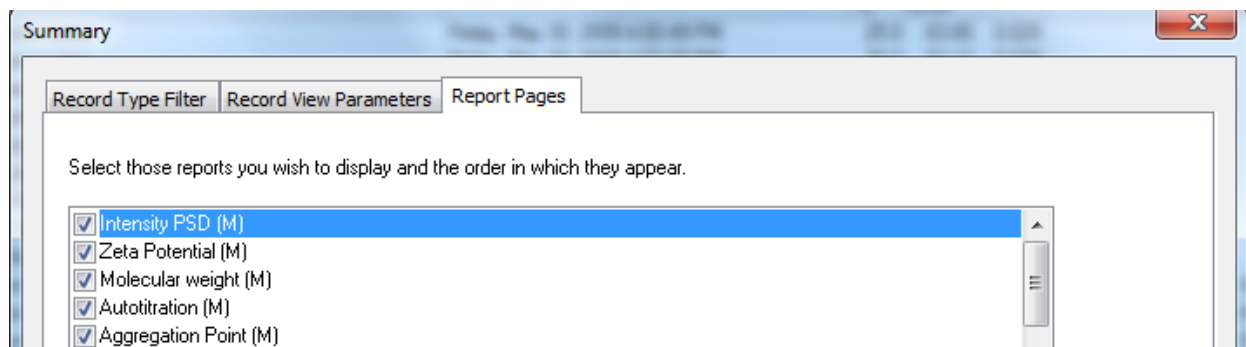
SOP's

 S.O.P.'s (standard-operating-procedures) are experiment files with various instrument parameters pre-selected from a range of choices. SOP's represent the heart of your measurement. Selecting, modifying, or creating new SOP's will be critical to your work. The drop-down will initially show two choices: (i) "browse for SOP's", and (ii)"manual measurement". However, as you use SOP's these are added to the dropdown list, making it convenient for you to find and reuse your favorite SOP's.

When you select an SOP from the dropdown, you must then click the green arrow . Mouse-over identifies this as "Start Measurement", but that's not quite true. If "Browse for SOP" is selected, it opens the file browser in the SOP folder so that you can choose an SOP. If an actual SOP is selected, it opens the SOP and allows you to add text related to the sample, et cetera.

If "Browse for SOP" is selected:

When an actual SOP is selected:

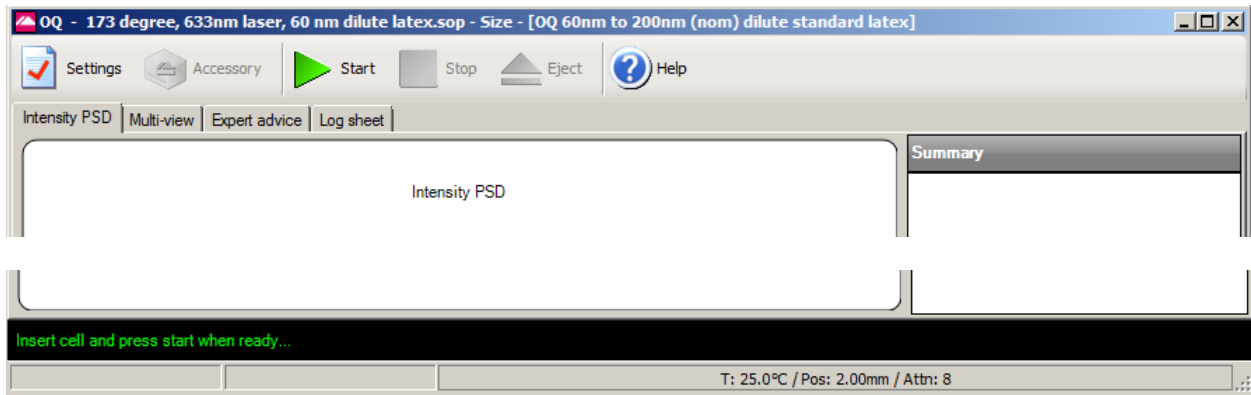After entering the appropriate text about your sample, select OK. This will open the actual "Start Measurement" view.

Note the instruction at the bottom of the view to "Insert cell and press start when ready".

If you are acquiring size data using one of the disposable plastic cuvettes, be sure that the "V" indicator at the top of the cell is facing forward. This "V" can be a bit hard to see; one suggestion is to place a dot in the triangle with a permanent marker before loading your sample.

If you are acquiring zeta-potential data with one of the DTS1070 plastic cells, you will notice that the fill ports at the top are off-center when viewed from the top. When you insert the cell, just remember that the rabbit ears need to be at the back!

When you press "start" you will notice status messages at the bottom of the view, including a bargraph progress indicator:

When data begins to acquire the view will show the spectrum as it accumulates:
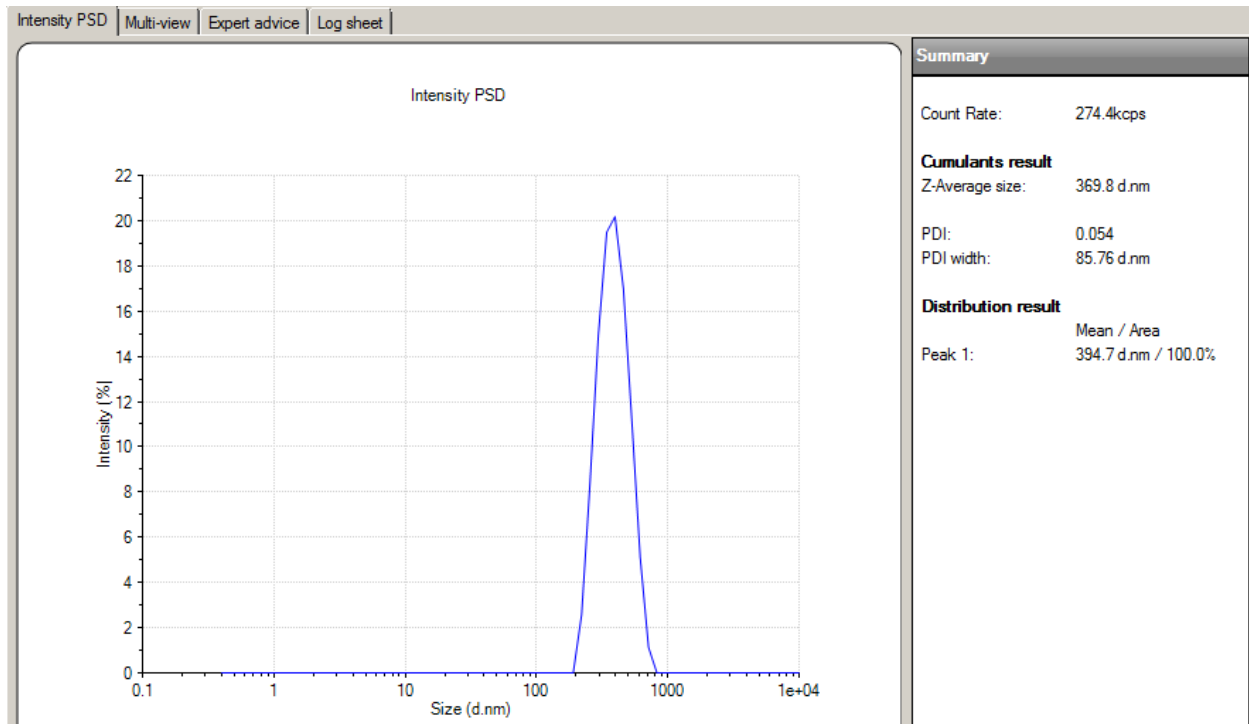
Note that this SOP measures the sample three times. In this SOP each measurement is the sum of up to 100 scans; however scan accumulation stops when certain S/N or other quality criteria are met. For this test sample, the measurement completes after about 12 scans. As each measurement completes, you will notice that it becomes a new record in the measurement file, and can be viewed even as the remaining records of the triplicate (in this case) are being acquired.
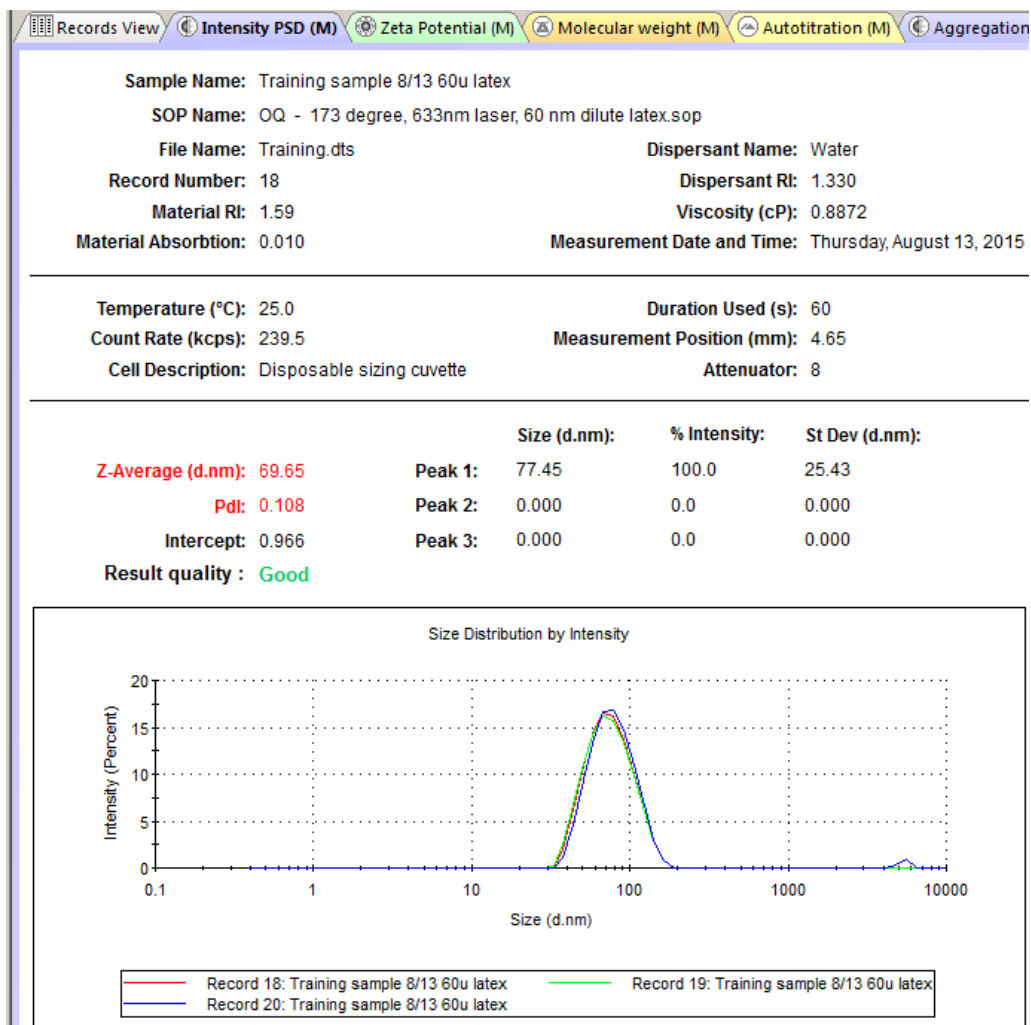
When the three measurements are completed, the "settings" icon again becomes active. This will take you back to the SOP text entry view so that you can identify a new sample if you wish and then start another set of measurements under exactly the same conditions. Once you've completed your group of measurements, exit from the measurement view by closing the view .

Viewing and Printing Results

Select the appropriate workspace view (summary, size, zeta…) from the dropdown. Then click to select the record you wish to view. Results for that record will be presented on each workspace tab. You can also click and drag to select multiple records. These will be presented simultaneously on the workspace tabs with cumulative or averaged numerical results.

17	Size	OQ 60nm nom dilute latex	Friday, August 07, 2015 2:50:37 PM	25.0	64.68	0.002
18	Size	Training sample 8/13 60u latex	Thursday, August 13, 2015 2:07:10 PM	25.0	69.65	0.108
19	Size	Training sample 8/13 60u latex	Thursday, August 13, 2015 2:09:15 PM	25.0	70.03	0.122
20	Size	Training sample 8/13 60u latex	Thursday, August 13, 2015 2:11:19 PM	25.0	77.38	0.208
21	Size	OQ 60nm to 200nm (nom) dilute standard latex	Friday, August 14, 2015 10:59:27 AM	25.0	363.7	0.109

Records View	Intensity PSD (M)	Zeta Potential (M)	Molecular weight (M)	Autotitration (M)	Aggregation

Sample Name: Training sample 8/13 60u latex
SOP Name: OQ - 173 degree, 633nm laser, 60 nm dilute latex.sop
File Name: Training.dts
Record Number: 18
Material RI: 1.59
Material Absorbtion: 0.010

Dispersant Name: Water
Dispersant RI: 1.330
Viscosity (cP): 0.8872
Measurement Date and Time: Thursday, August 13, 2015

Temperature (°C): 25.0
Count Rate (kcps): 239.5
Cell Description: Disposable sizing cuvette

Duration Used (s): 60
Measurement Position (mm): 4.65
Attenuator: 8

			Size (d.nm):	% Intensity:	St Dev (d.nm):
Z-Average (d.nm): 69.65	**Peak 1:**		77.45	100.0	25.43
PdI: 0.108	**Peak 2:**		0.000	0.0	0.000
Intercept: 0.966	**Peak 3:**		0.000	0.0	0.000
Result quality : Good					

Size Distribution by Intensity

Record 18: Training sample 8/13 60u latex
Record 19: Training sample 8/13 60u latex
Record 20: Training sample 8/13 60u latex

Use the "Print Preview" and/or "Print" icons to manually print the views . At the moment, direct printing to the LJ400 B/W network printer in the lab.
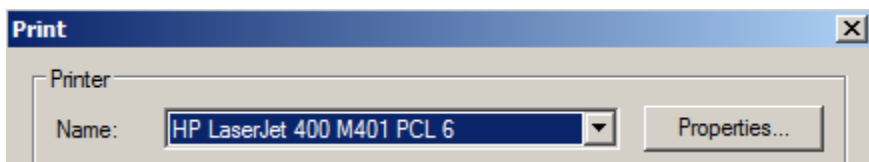
Note that you can enable automatic printing of selected reports as part of the SOP if you so choose.

Using the MPT-2 Titrator

This section will be included in the next version of the training guide.

APPENDIX A. Creating and Modifying SOP's

Editing existing SOPs or creating new ones involves the same process. There are about 50 parameters that must be set to define a measurement, woith multiple choices for each parameter. In this example we will show views from an existing SOP, one of the "operational quality" (OQ) SOPs used to test the instrument during installation. Browse to the SOP and then double-click to open it for viewing or editing.
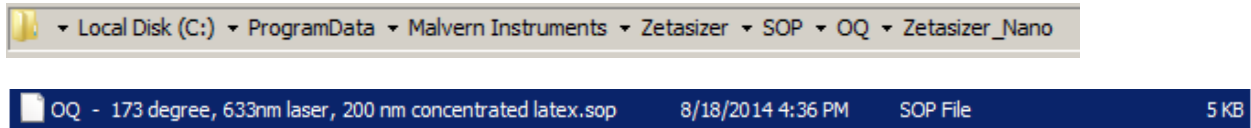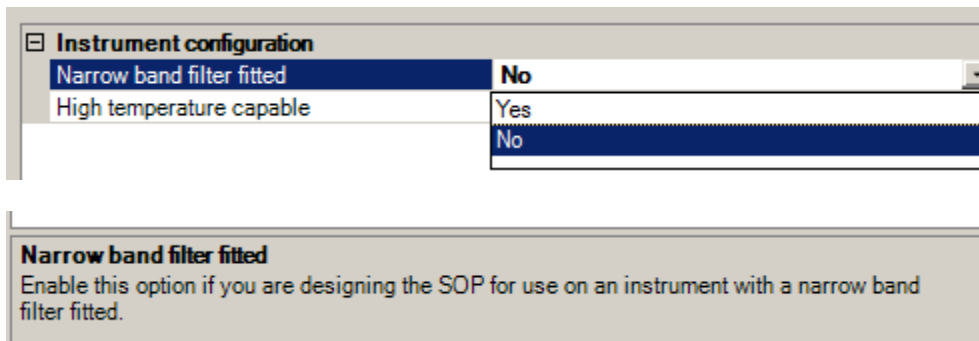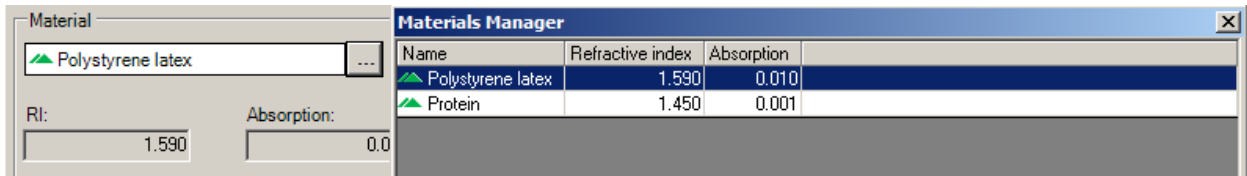

You should see:
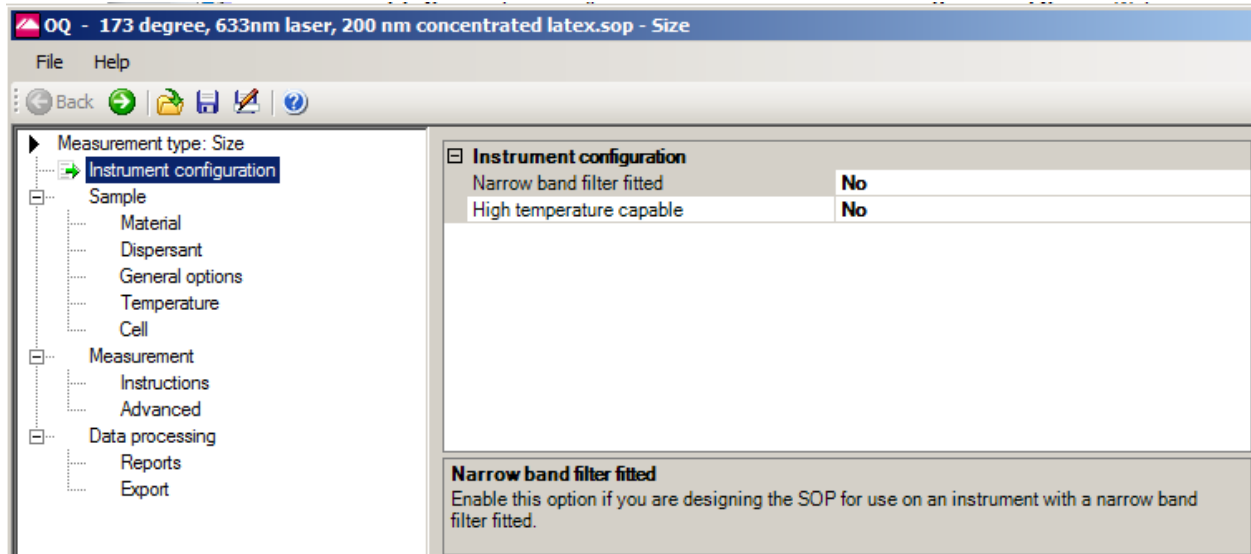
Click on each parameter (or parameter group) shown in the browser to view the settings and make choices. Note that many sections shown in the browser will need to be expanded to be sure that you access all parameters.

To make a change, click on the parameter shown in the right panel. You will be presented with choices or a dropdown of choices. You may also see an explanation of the parameter and the choices.
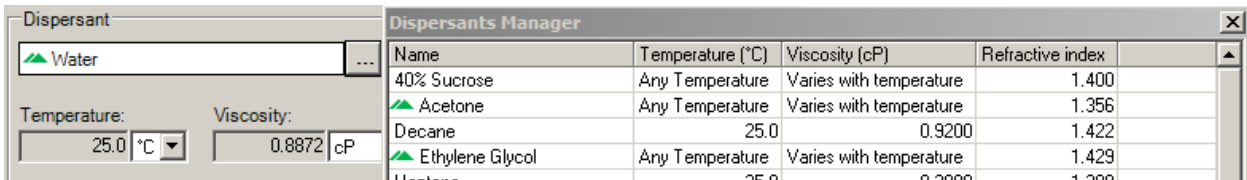
You should go through several SOPs completely in order to better understand the various parameters and choices before you actually create your own SOPs or modify existing SOPs. All of the parameter settings are important. Examples of a few are presented below.

Material. Select a material from the "Materials Manager". Some calculations (not all) need to know the refractive index of the material.

Material			Materials Manager			
▲ Polystyrene latex		...	Name	Refractive index	Absorption	
			▲ Polystyrene latex	1.590	0.010	
RI:	Absorption:		▲ Protein	1.450	0.001	
1.590	0.0					

Dispersant. Select a dispersant from the "Dispersants Manager". Many calculations need to know the viscosity of the mixture.

Dispersant			Dispersants Manager				
▲ Water		...	Name	Temperature (°C)	Viscosity (cP)	Refractive index	
			40% Sucrose	Any Temperature	Varies with temperature	1.400	
Temperature:	Viscosity:		▲ Acetone	Any Temperature	Varies with temperature	1.356	
			Decane	25.0	0.9200	1.422	
25.0 °C ▼	0.8872 cP		▲ Ethylene Glycol	Any Temperature	Varies with temperature	1.429	

Cell. Choose the cell type appropriate for your measurement, and then choose one of the choices presented. If you are using a cell not listed…. You're on your own!
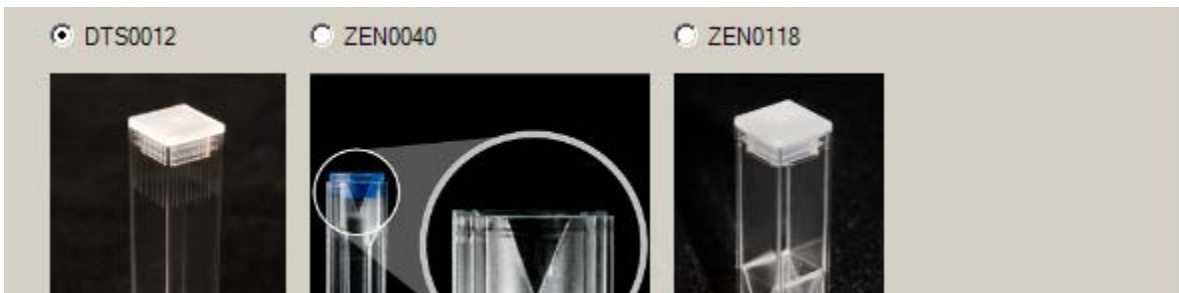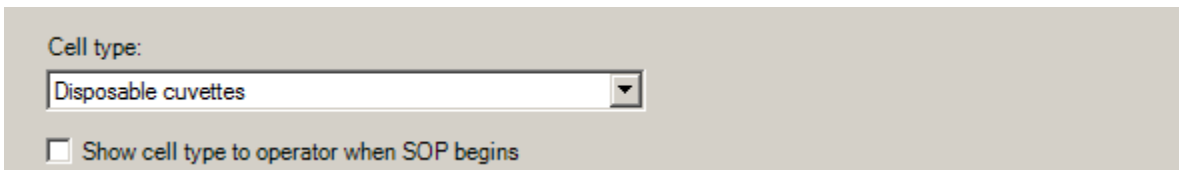
Measurement. Various parameters can be modified. Note that in this example three measurements (a triplicate) will be done on each sample, with no delay between measurements, and that measurement duration is "automatic". I'm not sure what this means.

Measurement – Advanced. Note the various values for finding the measurement position in the cell.
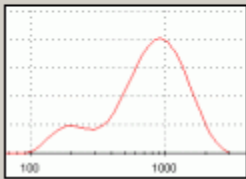
**Data Processing.** Choose the appropriate analysis model and configure the settings for the model. Note that different models are used for Size and Zeta measurements.


Print Reports. You can have the SOP automatically print reports as the measurements are acquired. This SOP does not have that feature enabled, but in this example I've checked the box so you can better see the view.
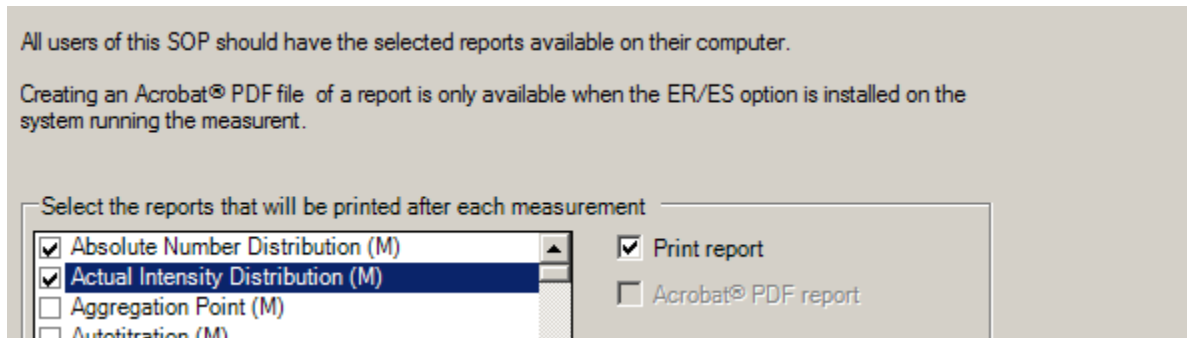
Export Results. If you check this box (as I have for this example) you can choose from two export templates. Note that the configuration of these templates is done elsewhere!
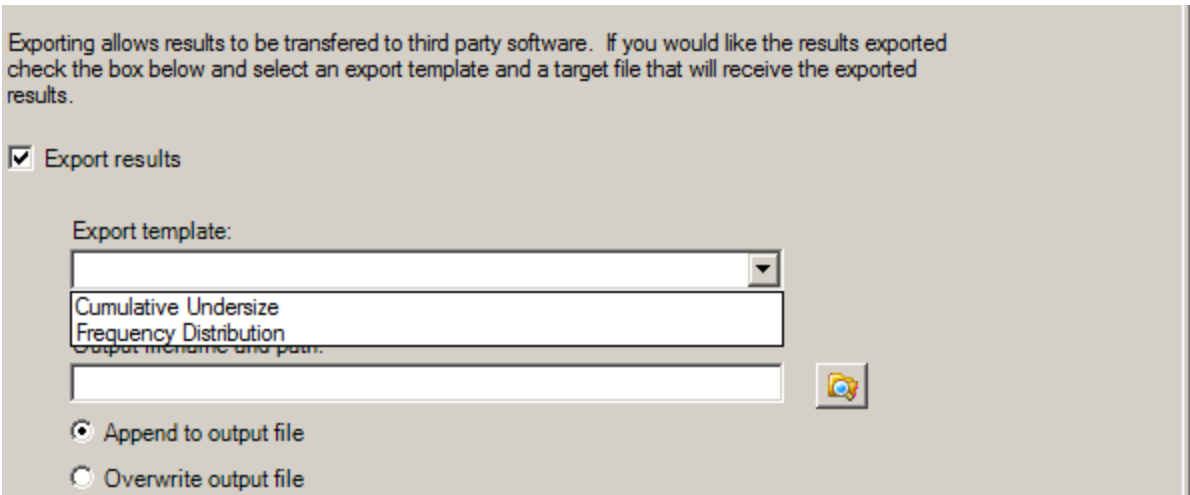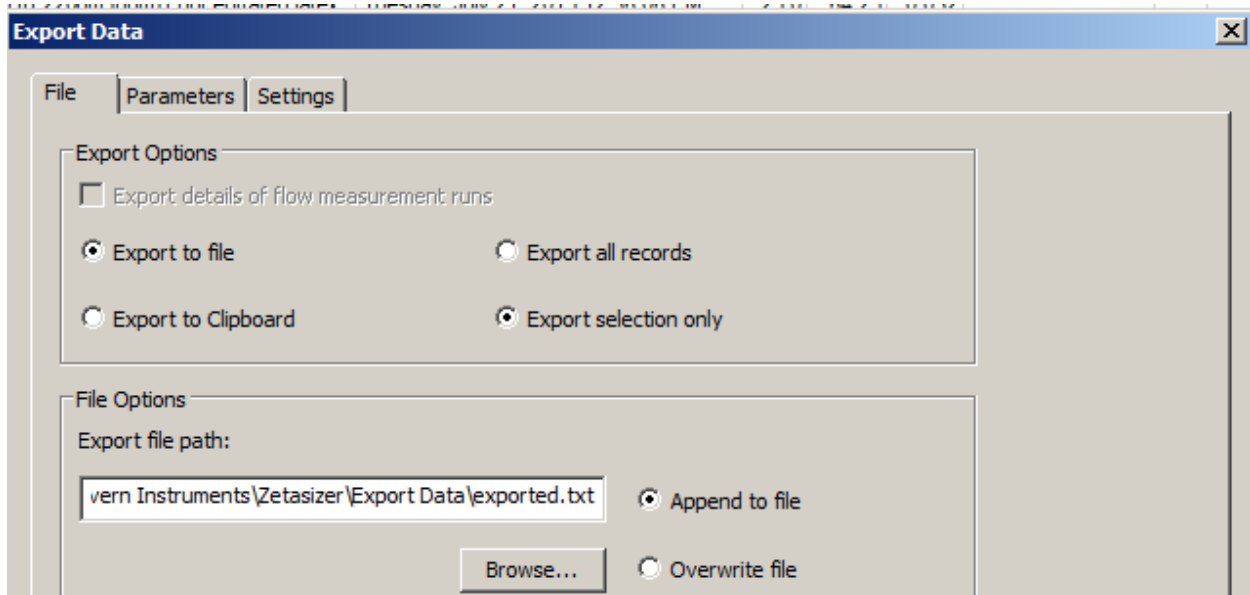

APPENDIX B.   Exporting Results

NOTE: I have not explored this completely yet, so more complete instructions will be in the next version of this training guide. However, you should feel free to explore, and share what you learn!
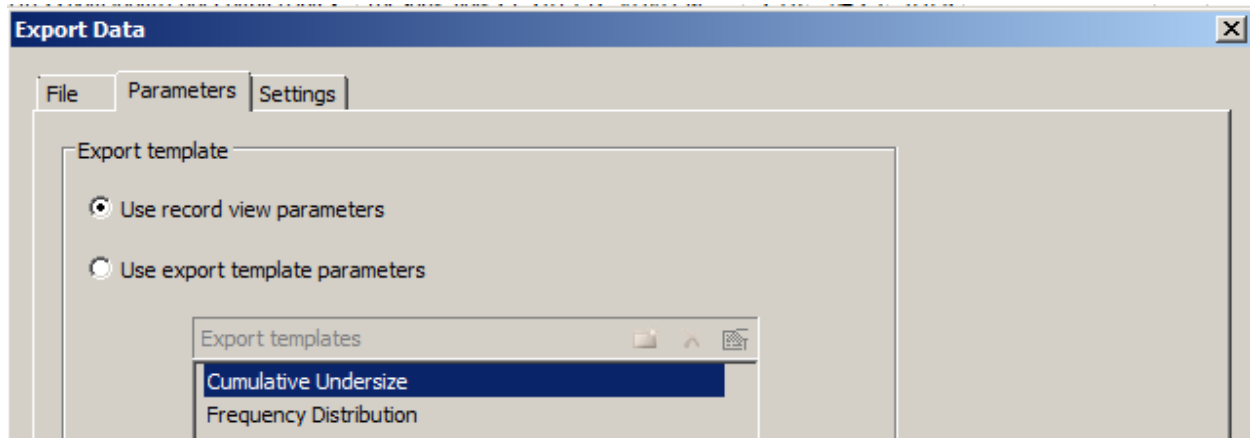
On the Records View tab of the workspace, select a record. From the File pulldown, select "Export".
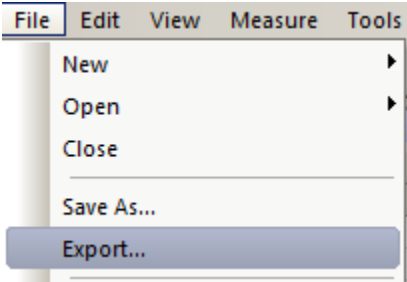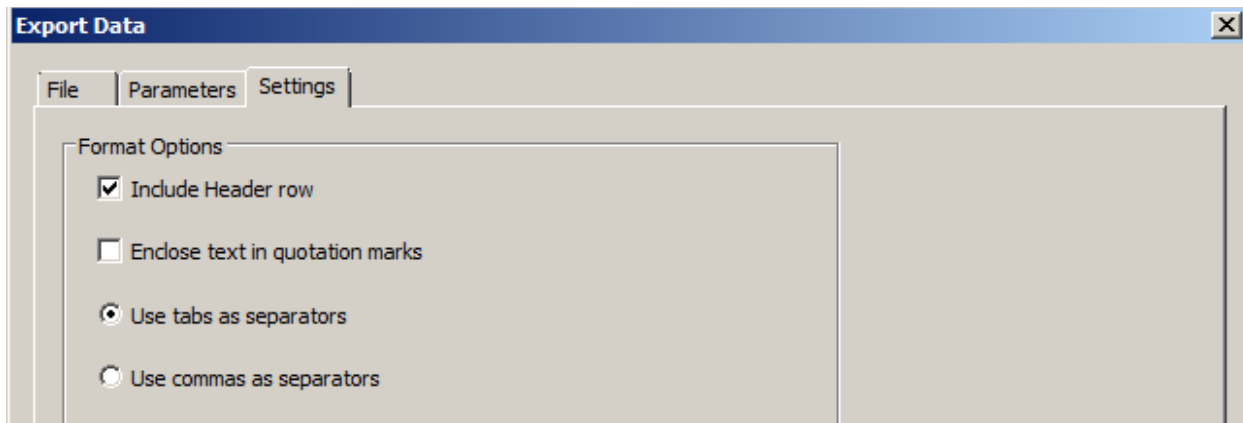

The Export Data view will open, with three tabs.

Note that you can choose to export all of the results in the measurement file, or just the record(s) selected.
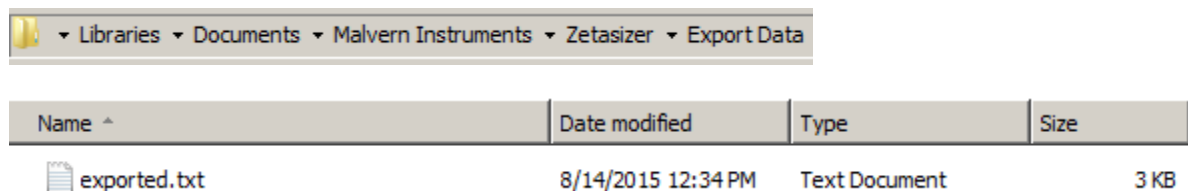

It appears you can export based upon the current workspace results-view parameters, or you can define a completely different set of result parameters to export.

Finally, you can choose tabs or commas as separators.

In this example, the file showed up in an "Exported Text" folder; it was named "exported.txt".

The file opened in Notepad to show (in part):