DLSC Measurement Quick Start Guide

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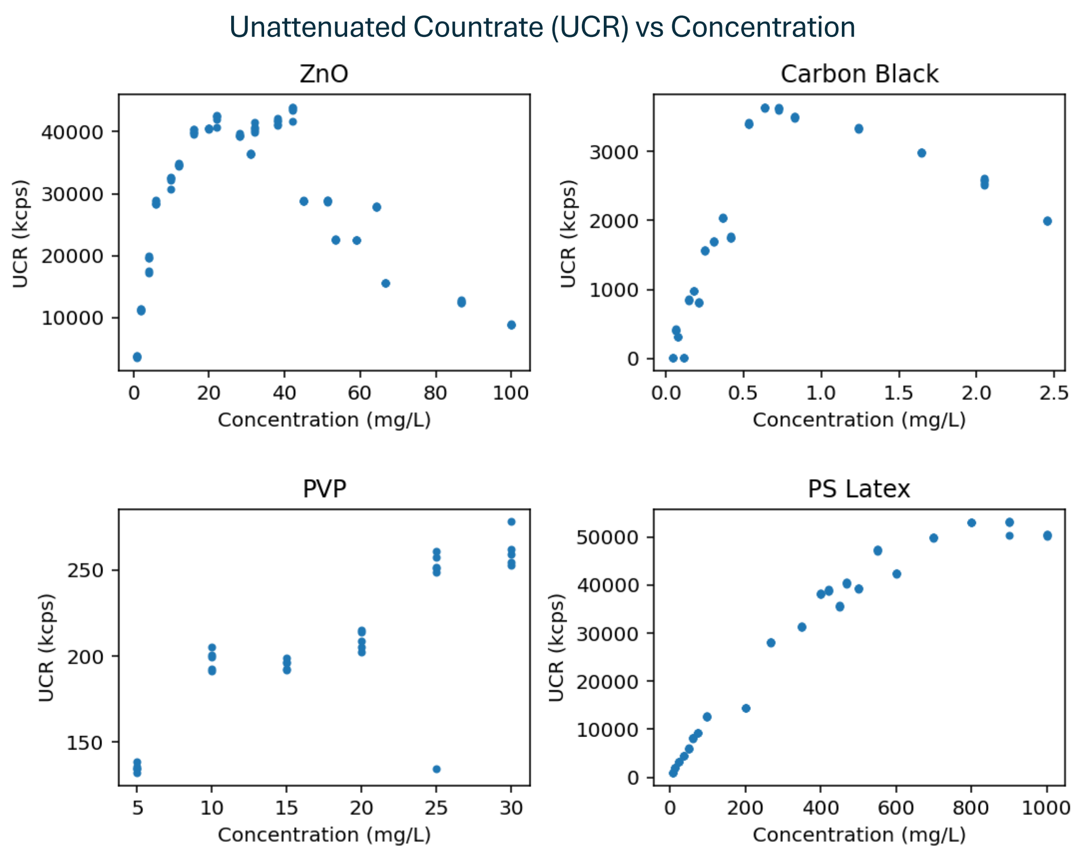
Introduction

DLSC, or the DLS Concentration Tool, is a measurement type for the NanoBrook using precise, digital laser power control and DLS data collection. The DLSC tool can collate measurements of different concentrations and use them to investigate the concentration of a sample of an undetermined concentration.

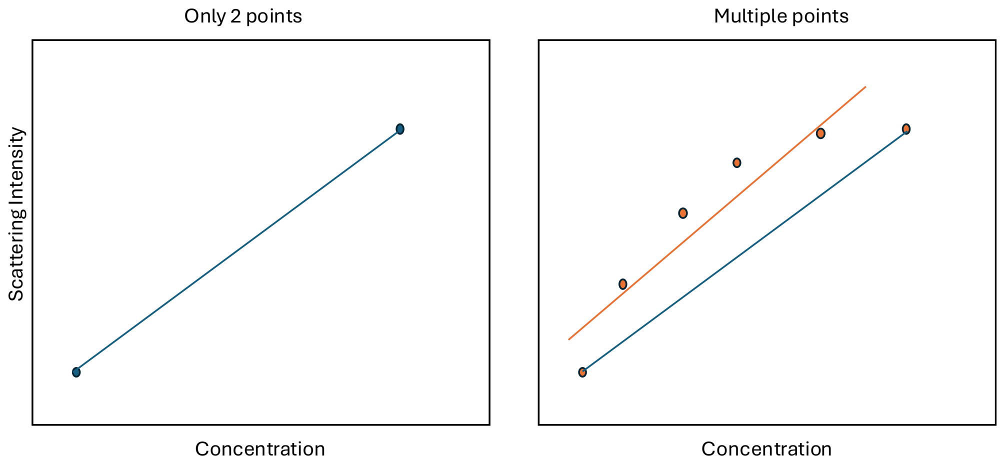
A new set of calibration measurements should be obtained for every sample type. However, once those calibration measurements are obtained, the calibration will remain valid for as long as the efficiency of the detector and the laser remain unchanged. The DLSC measurement type will give information about the particle size, as with typical DLS measurements, as well as concentration.

Theory of Operation

DLSC depends on making calibration measurements of known sample concentrations. It assumes that the intensity of light scattered by the particles is dependent on the concentration of particles in the solution. Specifically, the DLSC analysis module assumes a linear relationship between particle concentration and scattering intensity. For dilute solution conditions, this is true. As concentrations get higher, other effects such as multiple scattering begin to dominate, detector saturation can also become a problem at very high countrates.



It is recommended to make a standard curve with multiple data points at concentrations near that of the unknown sample. Five to seven data points would give a good indication of any non-linearity.



The scattering intensity of the unknown sample can then be measured. When combined with the best fit to the calibration curve, the software automatically fits the scattering intensity to the best fit line and reports the corresponding concentration. Note that while an extrapolation to concentrations outside the range of the calibration data points is possible, it is less likely to be accurate than an interpolation between data points.

Instructions

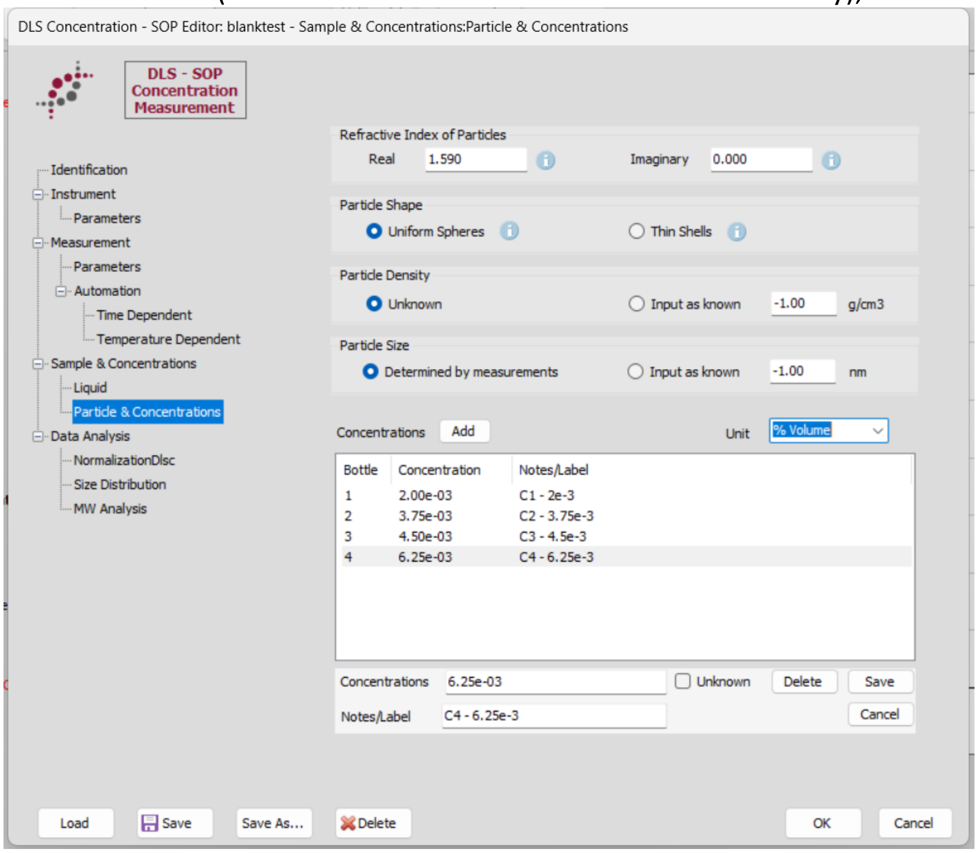
When making measurements, it is important to have a proper calibration curve. They should be of the same material, chemistry, solvent, and particle size as the unknown. Calibration curves can be reused with new unknown samples provided that these conditions are met.

Calibration curve measurement

To measure a calibration curve:

1. Create a series of concentrations of the target material, in the correct particle size and solvent.
    a. These concentrations should span the range of concentrations you expect to measure.
    b. A minimum of 5 concentrations are recommended.
    c. Make sure to both control and record the concentration of each sample as precisely as possible.
2. In order to set the initial position of the neutral density filter be sure to have a strongly scattering sample available. This does not need to be the same material as the unknown or the concentration standards.

3. Create a new DLSC SOP, saving it with a name easily traceable to the sample type you are making a calibration curve for.
    a. Set the DLS measurement parameters. For more information, see the DLS measurement Quick Start Guide.
    b. Navigate to the “Particles & Concentrations” menu.
    c. Enter the units you wish to use for your concentrations in the “Unit” box. If the drop-down menu does not list your preferred units, you can type in your own.
    d. Click the Add button to add a concentration. Double click on the concentration to edit it. A set of menu options will appear below the Concentrations list.
    e. Enter the concentration (in your selected units) in the “Concentrations” box. Enter any notes or labels in the “Notes/Labels” box. Click “Save” to save your inputs, or “Delete” to remove the concentration.
    f. When finished (make sure all concentrations saved correctly), save the SOP.



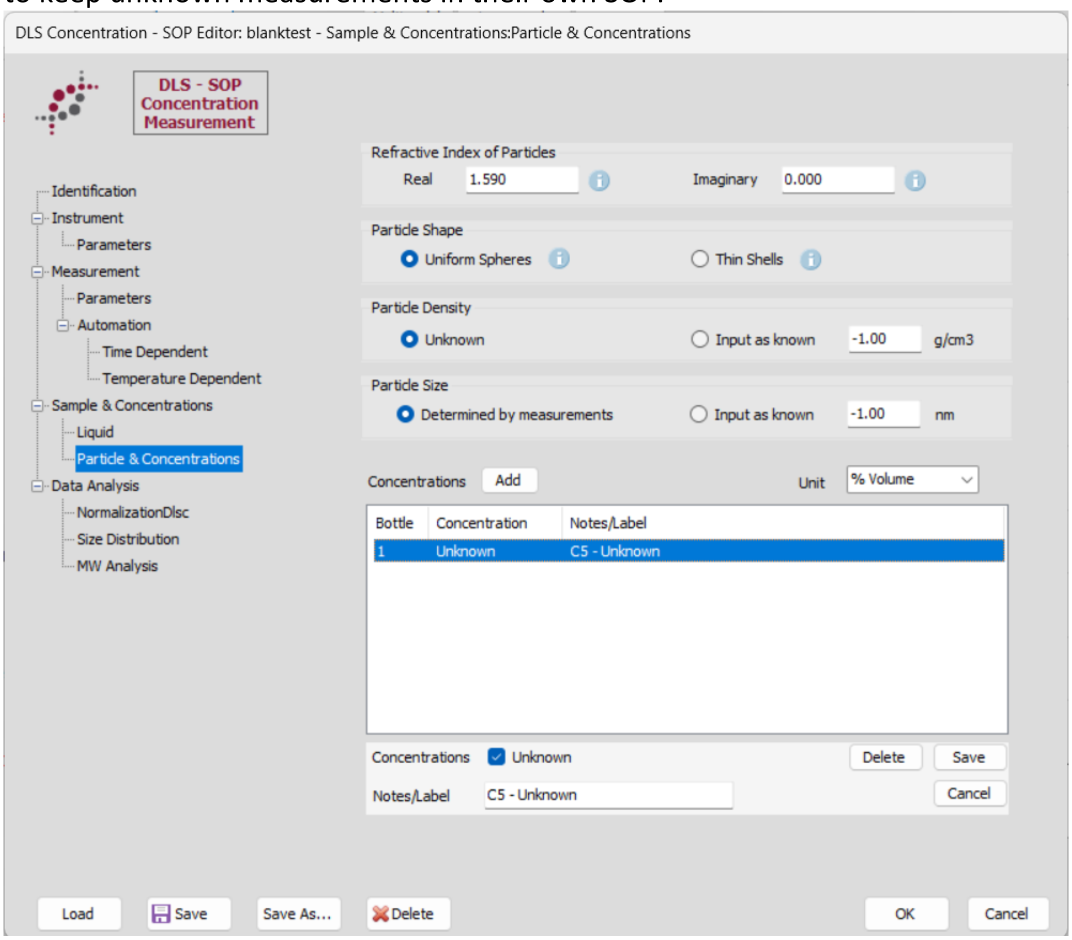
4. Start the measurement. The software will issue a series of prompts requesting specific samples. When it does so, remove the previous sample (if any) and insert the new sample.
    a. The software will initially request a strongly scattering, or optically dense (OD) sample to set the initial position of the neutral density filer (NDF). Insert the OD sample when prompted.
    b. The software will then request the first concentration to measure. Insert the concentration and allow it to make measurements.
    c. The software will continue to request concentrations until all samples in the SOP are measured.

d. If the DLS measurement shows errors, see DLS measurement quick start guide for tips on troubleshooting.

Unknown sample measurement

To measure an unknown sample:

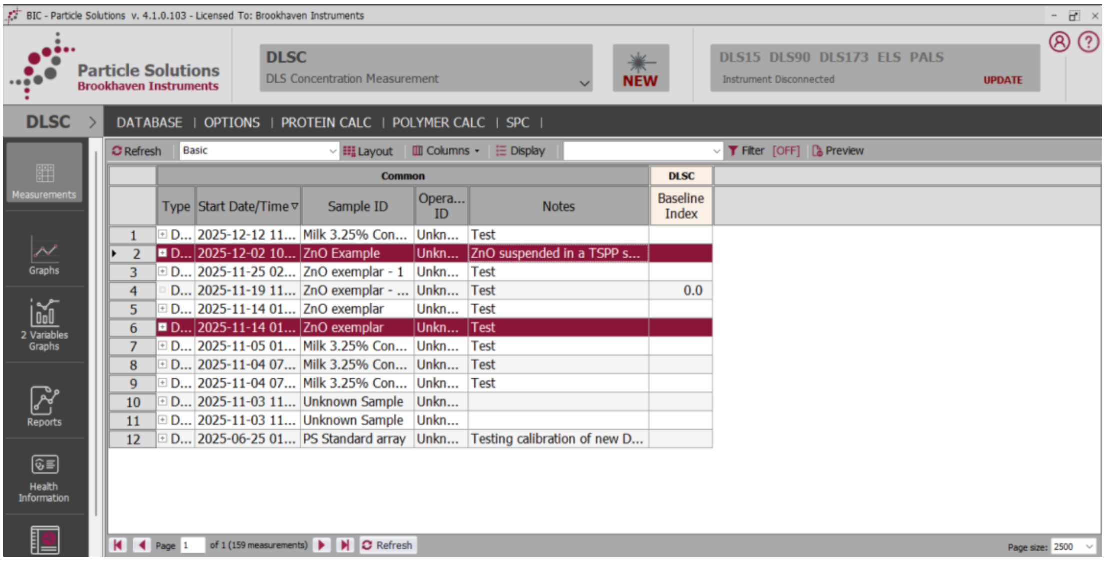
1. If it is necessary to dilute the sample, make careful note of all dilutions.
2. Create or modify a DLSC SOP, using the same parameters as the calibration curve.
    a. The “Set Duration”, “Equilibration Time”, and “Total Measurements” fields can be set as desired, as well as any field in the “Identification” menu.
    b. Navigate to the “Particles & Concentrations” menu
    c. Remove all particle concentrations. Add a new concentration.
    d. Input any notes or label required. Check the “Unknown” box beside the “Concentrations” header.
    e. Additional samples can be added, known or unknown, however, it is best practice to keep unknown measurements in their own SOP.



3. Start the measurement.
4. Follow the prompts for which sample to insert at which time.

Data Analysis

To analyze results, choose the DLSC measurement analysis window. Select 1) the relevant calibration curves, then 2) the unknown sample measurements.



When all measurements are selected, click on "2 Variables Graphs." Make sure you are in Concentration Mode. This will display the calibration measurements, a dashed best-fit line, and a red dot for the unknown concentration. The display will also show an equation for the best fit line and the fit for the unknown sample's concentration value.