Dynamic Light Scattering (DLS) Manual

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1. Turn **ON laser**, water bath and filter. **Wear safety goggles**. 20 min warm-up laser. Block with cardboard when warming up or changing sample. Block for safety reasons and because it prevents a flash of very intense light from damaging the photo-cathode whenever the cell is changed.
2. Dusty liquids limit the reproducibility of results. Dust distorts the baseline, artificially broadens the distribution, may act as a local oscillator, and give rise to number fluctuations. These effects change completely the model used to fit the correlation function. Dust is primarily a problem in highly polar liquids, especially **water**.
3. **Sample preparation** consists of three parts: solvent purification, cell cleaning, and solution/suspension preparation. The last part depends on the particular type of sample. Only general guidelines can be given. The solvent has to be free of trace organic impurities, and filtered. Residual metal ions may affect the chemistry, motion, or the shape of the molecules or particles of interest. In addition to the above effects, trace organics may significantly alter the surface tension of the primary solvent. Trace organics are removed by distillation or absorption. Finally, removal of dust by filtration greatly increases the repeatability of DLS measurements.
4. **Cells**: Order from Brookhaven Instruments: N.Y., fax: 631-758-3255 (phone: 631-758-3200). Round glass sample cell, 12mm O.D., min vol. 1.5mL, PE cap # BI-RC12 (unit price ~ $6.00).
5. **Cell cleaning**: The degree of cell cleaning depends on the application.
   - Dust removal: using compressed gas to blow off dust on the inside and outside of the cells.
   - Avoid fingerprints on the cell in the region where light will enter or exit. Most rigorous cleaning procedures follow. Choose the one most suitable for your work.
   - Rinse cells thoroughly in tap water. Use a nonabrasive soap to clean the inside and outside of cells. Do not use a brush. Instead, shake the cells vigorously.
   - For a more thorough cleaning place cells in an ultrasonic bath. Sonicate for several minutes. Rinse cells thoroughly in tap water, followed by a several rinses with filtered water. Let dry upside down to prevent dust from reentering the cell.
6. **If grease spots remain consider the following more drastic cleaning:**
   a. Soak cell for 30 min in concentrated H_2SO_4. Rinse thoroughly in tap water.
   b. Soak for 10 min in **Solution A**. Rinse in tap water.
   c. Dip cells in **Solution B** two or three times. Empty cell after each dipping. Rinse in tap water.
   d. Steam the cell.

   Dry cell: Place cell in a smooth piece to loosely fitting aluminum foil. Dry in an oven at 120 ºC overnight. Keep cells covered until ready for use.

   Solution A: Dissolve 160g of NaOH pellets in 2L of water. Add 7g of $KMnO_4$. Swirl until uniformly purple. **Solution B:** Dilute 1/3L of concentrated HCl to 2L. Add 0.7g hydroxyl amine hydrochloride. If the cell has been cleaned well then outside will dry without spotting. And water will evaporate in a continuous sheet. Water will not condense on the inside of the cell, and the steam will roll off in a continuous sheet.
7. **Preparation of the polystyrene standard solution:** Order polystyrene, standard latex ~ 90nm from Duke Scientific Corporation http://www.dukescientific.com. Make 200mL of a 10mM NaCl solution. Use a disposable 0.2µm filter to clean the liquid. Rinse thoroughly a 26mL vial and its screw-on cap with the filtered solution. The latex is packed into plastic squeeze bottles. They are usually full of surfactant. No more need to be added. The concentration varies from 2 to 10% solids by weight. Shake the bottle and squeeze about 4 drops into the vial. Add about 20mL of filtered water. Use a previously cleaned pipette to deliver about 2mL of the suspension into previously cleaned cell. Cap the cell. Ultrasonicate the cell for about one minute. Place the vial in the path of the laser beam. If the line is thick and fuzzy, the sample is too concentrated. Dilute it. If the lie is extremely weak, not much more visible than filtered water, the sample is too dilute. Add more latex. If random bursts of light are readily apparent, prepare a new solution paying more attention to cleaning the dilution vial and water. Turn the goniometer to 90º. This angle will minimize any stray light, since the detector likely to see residual interface flare.

8. **Sample:** Concentration ~ 5 mg/mL. All liquids should be filtered with a 0.2µm filter to eliminate dust. If you can't filter your final solution because your aggregates are too big, it is best to filter all the component solutions before you mix them together. Do not overuse surfactants (typically around 10^{-4} to 10^{-5} vol. %). If particles clump together, and obviously do not go into suspension, try wetting the particles with a few drops of ethanol first, followed by water.
9. **Decaline bath**: Matches refractive index of glass to reduce beam reflection/refraction. Clean with pump/filter. If the level of decaline is too low please contact Krystyna (kbrzez@mrl.ucsb.edu).
10. **Pinhole selection**: Select 200µm pinhole. For weak signals you can use 400 or 800µm pinholes. For strong signals choose the 100µm.
11. **Moving arm**. Click on the Motor in the Main Menu. Set the current angle (60º) and the destination angle (90º). When you click OK button, the detector arm will move to the destination angle.
12. **Parameter settings**- Enter: sample name, operator ID and temperature. If the Liquid is not listed select "Unspecified" and enter the refractive index and viscosity manually. Enter the wavelength of the laser – **633 nm**. The refractive index will vary slightly with wavelength and temperature for each of the liquids listed. Enter the real and imaginary parts of the particles refractive index, if they are known. **Measured baseline** is normally preferred to suppress the effect to any residual dust.
13. Do not click in the box Homodyne. Do click in the box Self Beating. **Dust filter**- it is better to clean liquids and samples, either by filtration or by centrifuge, than to rely solely on hardware and software.
14. **Measurements**: Click the Green/Start icon to start or the Red/Stop icon to stop the accumulation of the function. Click the Clear button to delete data from memory.
15. Setting Up multiple windows and using Window- **smart tile**. Click on Graphs in the main window. Click on Count Rate History. The count rate is displayed in the middle of the screen in Kcps (kilo-counts per second). It should be between 10^4 and $2x10^5$ counts per second. If your count is below 1000 cps, check that the beam is not blocked. You can increase the count rate by increasing the pinhole to 400 or 800µm or increasing concentration. If the count rate is greater than $2x10^5$, lower it. Decrease the pinhole to 100µm, or decrease concentration.
16. Note the **Effective diameter** and **POLY** in the control window. These two values often all one needs to characterize the distribution. POLY is a measure of the size distribution width. When the Poly equals zero, the sample is mono-disperse, and as Poly increases so does the width of the distribution.
17. **Save results**- File- In the database create your folder. Files can be exported in ASCII format. File-Save Correlation Function As.
18. To calculate diameter by Intensity, by Volume, by Number- select the Double Exponential window accessed from ISDA menu, new window and smart tile. You can select weighting by intensity, particle volume, surface area, or number (see Layout). Using ISDA data can be analyzed in Γ or τ space as well. This may be useful for polymer solutions, gels, glass transitions, or other complex fluid measurements. A Williams-Watts fitting function is also available. Click Cumulant Analysis in ISDA to open up a window with four individuals fits to the correlation function- linear, quadratic, cubic, and quartic. Correlation Function- Scale- Show Fit. Showing the fit is just convenience that is only useful when there are gross differences between measured and fitted functions.
19. Leave DLS at 60°. Put cardblock back. **Turn OFF**: Laser- Water Bath- Filter for decaline's bath.