ALV-5000 Instrument Instructions

These notes are intended for trained users only; if you require training, please contact us.

Static Light Scattering Software:
- Open "Multi-detector" software
- Dialog box will pop-up asking "do you want to se this configuration?"; click YES
- Software should say "ALV-LSE found and initialized" [*if not, see Usage Notes, below]

Navigating the Software Window:
> Lower Status Bar:
>> Sample Angle Wave Index Visc Temp Diode
>>> **Sample** opens a dialog box for solvent selection
>>> **Angle** is a user control; default value is 90
>>> **Index**, **Visc** are user inputs, required for accurate size measurements
>>>> -If solvent selected in **Sample**, both **Index** and **Visc** autopopulate.
>>>> -Default values are for water.
>>> **Wave**, **Temp** & **Diode** are measured values and cannot be modified
> Upper Control Bar:
>> File Edit Window Sample QuickSet Run Setup Fit Display Help

Before Beginning Measurement:
- Insert sample into sample chamber and replace cover
- Especially for dilute samples, sample should be placed in chamber and allowed to sit for a few minutes, to allow mechanical vibrations in the toluene bath to dissipate, as these can cause noise in the measurement
- Check diode level in lower right hand corner of window: for strongly scattering samples, diode intensity should be kept < ~5,000; for very weakly scattering samples (i.e. proteins, polymers, surfactants) diode can be set as high as 20,000 or higher, but with caution.
- Remove safety goggles case in front of detectors 1-7
- Open shutter on laser line to allow light into sample chamber; it is recommended to set up experimental parameters before opening final shutter.
- Visually inspect scattered light from sample; strongly scattering samples will appear very bright, suggesting the diode should be lowered.
- Diode control: clockwise lowers diode intensity; counterclockwise raises diode intensity

To configure SLS measurements:
- File → Set AutoSave
    - Check the box "Use AutoSave"
    - Use the pull-down menu to navigate to Drive E:/ and your data folder
    - Choose Filename; must include 4 characters followed by 4 numbers (default '0000' to start).
    - These will be "data-run files," with one file saved per run

- QuickSet → Dynamic / Static Light Scattering → General DLS and SLS
    - Dialog box opens, revealing Step 1, Step 2, and Step3
        - **Step 1 Measurement Control**
            - Min. Angle = 0; Max. Angle = 17; Angular Step = 1

Notes prepared by Sara Hashmi (sara . hashmi @ yale . edu)

- Runs = 1-3; Duration = 10-90 sec [see usage notes below]
- Click "Accept this Set" to populate table at right
  - To modify instructions, double-click row in table to delete, then correct angle parameters and "Accept this Set" again
- Click button "Setup" under table
  - In "Scaling" tab: use drop-down menu to select "Off"
- **Step 2 Static & Dynamic LS Options**
  - Leave as-is unless you are collecting a new "standard file"
- **Step 3 Measurement Type**
  - Radio button: select "Solution" (unless you are collecting a new standard)
  - Standard File: navigate to folder E:/slssta~
    - Choose "stnd05.tol"
  - Solvent File: navigate to folder E:/slssta~
    - Available solvent files are for DI water and methanol
    - If you have a different solvent, check the box "Measure without solvent information/file"
    - Even if your solvent file is available, if sample is dilute, consider using "Measure without solvent information/file"
  - Solution File: navigate to your folder; choose filename
  - Clicking "Start Measurement" will start measurement immediately, so be sure AutoSave is enabled, diode level is adjusted, and room lights are on/off as desired for measurement

After Beginning Measurement:
- Monitor scattered light intensity at 8 detectors in upper right hand corner: "C0" – "C7"
  - If any lights blink RED
    - Close shutter
    - "Stop Measurement"
    - Lower diode, Open shutter, return to 'QuickSet' instructions above
      - Choose new file name if desired, or
      - De-select "Append solution data to solution file" to overwrite truncated measurement
    - Iterate diode adjustment if necessary
    - Note that the Auto-Saved "data-run files" will continue to increment unless counter is reset to previous starting value

Adjusting Detector Sensitivities:
- Detector sensitivities are in 'Setup → Mutli-angle Setup'
- Open sample.xls in folder E:/sls standards, or start with previous SLS spreadsheet from your own samples, and "Save As" into your current folder
- Input current detector sensitivity values into column N; detector 1 has sensitivity = 1
- Upon completion of a measurement run, right click on data in pop-up window to "copy to clipboard" and paste data into cell A1
- Column O calculates ratio of intensities at angles measured by 2 adjacent detectors: values > ~1.08 or < ~0.92 mean data is not 'smooth' and sensitivities require adjustment
- Spreadsheet calculates suggested sensitivity values in column P; input these into 'Setup → Mutli-angle Setup' and start another run in 'Quickset,' iterating Solution File name

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Usage Notes:
   Instrument/Diode
- If instrument is not recognized, go to "Setup → Instrument Setup" and choose "ALV-LSE/II" is present on Port "COM1" in the upper left "ALV-LSE Support" box. Click "OK," and "found & initialized" dialogue should appear. If not, software may require restart.
- Diode control: clockwise lowers diode intensity; counterclockwise raises diode intensity
- When adjusting diode, if intensity is too high, close final laser line shutter (immediately before the sample), adjust diode intensity to a reasonable level, then reopen shutter and continue with experiments
   o Diode should be adjusted with caution to not over-illuminate the detectors
   o Diode level is indicated in lower right hand corner of software
   o Do not allow counts (in upper right) to blink RED

   Step 1 Measurement Control
- Note: SLS measurements are an iterative process, requiring adjustments to the detector sensitivities to obtain a smooth result for intensity as a function of angle
- Often, for initial steps in the iteration process: Runs = 1 and Duration = 20 is sufficient
- If dynamic data is sought at all angles, once detector sensitivities are properly adjusted, the Duration should be increased to ensure

Data Handling and Sample Organization:
- Data is saved in the following manner:
   o Summary file: Solution File chosen in "Step 3 Measurement Type." May be opened in ALV software (only).
   o Data-run files: Using "AutoSave" generates files containing 8 raw correlation data curves for each angular step, and the scattered light intensity vs. time over the duration of the run.
- For the purposes of subsequent data analysis, it is often convenient to create one folder per sample measured
- ***When switching from one sample to another, or iterating detector adjustments***
   o Choose a new Solution File name in Step 3 Measurement Type
   o AutoSave option continually increments the numeric suffix counter, so previous data will not be overwritten. If you wish to change the AutoSave Filename, for new samples, you should also reset the counter to "0000."
- Data can be transferred via Zip Disk (Drive F:)
- Zip Disks and a USB Zip Disk Drive are available in the ALV room; unless data is needed urgently please do not remove the USB Zip Disk Drive from the ALV room.

In Case of Emergency, Call 911, or 2-4400 from the phone on the wall in the ALV room.

In case of questions during measurements or instrument usage, contact Sara at 508-596-0683.

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