

PHASE ANALYSIS LIGHT SCATTERING

Features of the PALS SOP:

From the bottom of the SOP Window, users have the option to “Load” a previous SOP. For PALS measurements, the list on the left hand side is as follows:

- Identification:
- Instrument Parameters:
- Measurement:
 - Parameters
 - Advanced Settings
 - Automation
 - Time Dependent
 - Temperature Dependent
- Titration
 - Setup
 - pH Titration Measurement
 - Additive Titration Measurement
- Sample Parameters
 - Liquid
 - Particle
- Data Analysis
 - Model

Of the items above, the *options which must always be specified* are:

- 1) **Identification**, in which the user sets the **Sample ID** and inputs any notes to accompany the saved data.
- 2) **Measurement → Parameters**, in which the user sets either a **Number of Cycles** per measurement (typically 30), or a **Target Residual** value for the raw data fit. The number of cycles accumulates data: the final cycle determines the zeta potential measurement for each particular data point. The number of data points is set in the next item. The Target Residual option is recommended for samples with low mobility.
- 3) **Measurement → Automation → Time Dependent**, in which the users sets the number of **Total Measurements** (typically 5, but can be increased for more statistics, as is recommended for low-mobility materials), as well as the **Time Interval Between Measurements**, which is typically 0 unless a time course is required. The interval is the number of seconds between the end of one measurement and the beginning of the next.
- 4) **Sample Parameters → Liquid**, in which the dispersing fluid is specified. The default solvent is **Water**, and the dropdown menu contains several common solvents. If the solvent information is missing, the user must select **Unspecified** in the dropdown menu and input the solvent Viscosity, Refractive Index, and Dielectric Constant in order to obtain meaningful data. pH may also be entered, but will not affect the measurement.

Other options, *some of which are important for non-polar solvent measurement*, include:

- 1) **Instrument Parameters** allows the user to select the cell type. The default **BI-SCP** refers to the Square Plastic Cuvettes used for aqueous samples. **BI-SCGO** must be selected when the Square Optical Glass Cuvettes are used for non-aqueous samples. Similarly, the electrode assembly defaults to the standard electrode **BI-ZEL (1.25 mL)**, but **BI-SREL (1.25 mL)** must be used and selected if the Solvent Resistant electrode is required for non-aqueous samples.
- 2) **Measurement → Automation → Temperature Dependent** allows the user to specify Starting and Final Temperatures, as well as the Temperature Increment. Note that the Total Measurements requested in Item 3 above will be made for each temperature in the automation series.
- 3) **Sample Parameters → Particle** does not affect the measurement, but is included in the full Data Summary, and may be helpful for future reference.
- 4) **Data Analysis → Model** defaults to **Smoluchowski**, for aqueous samples. The **Huckel** option should be selected for non-aqueous samples or samples with very low salt concentrations, and therefore long screening lengths. The third option, the **Henry** model, is identical to the **Huckel** model.

Note: Titration instructions are contained in a separate section below.

Upon completion of specifying your SOP, you may either “Save” to overwrite or “Save As,” and then click “OK,” which closes the SOP window and returns to the Measurement Window. ***It is recommended that you include your name or NetID in the name of your SOP.***

Preparation of PALS sample

Place 1.7 mL volume of sample into a square plastic cuvette, and insert the electrode into the cuvette. A sample volume of 1.7 mL should allow the electrode enough room in the cuvette with neither spillage nor air bubbles remaining. Place the sample into the sample chamber and plug in the electrode. Close the top cover of the chamber when the sample is in place. **IMPORTANT NOTE:** For non-aqueous samples, use the square glass cuvettes and the **BI-SREL** electrode. ***Glass cuvettes are not disposable;*** be careful using them and clean them for the next user.

Begin measurement in the PALS Measurement Window:

Once the sample is in the sample chamber, select “Start” in the upper left hand corner of the Measurement Window. The instrument will equilibrate the sample chamber temperature, adjust the incident laser intensity, and begin measurements. The plot displays the raw data with the fit overlaid. The Residual value displayed on the left shows root-mean-squared residual between the data and the fit.