

# University of Minnesota Nano Center Standard Operating Procedure

**Equipment Name:** Zeta Potential Analyzer

**Model:** Stabino

**Location:** PAN 185

**Badger Name:** Not on Badger

**Revision Number:** 1

**Revisionist:** J. Marti

**Last Revision Date:** 5/6/2019

## A. Introduction

- 1. Tool Description.** The Stabino is a particle charge characterization tool that measures the zeta potential of particles suspended in aqueous or organic liquids. Zeta potential can be measured for particles in the size range 1 nanometer to 300 micrometers. The Stabino can also titrate a suspension with acid or base while measuring zeta potential at pH values from 2 to 10, enabling the determination of isoelectric point.
- 2. Tool Components.** Figure 1 shows a front view of the Stabino. Key components visible in this view are listed below.

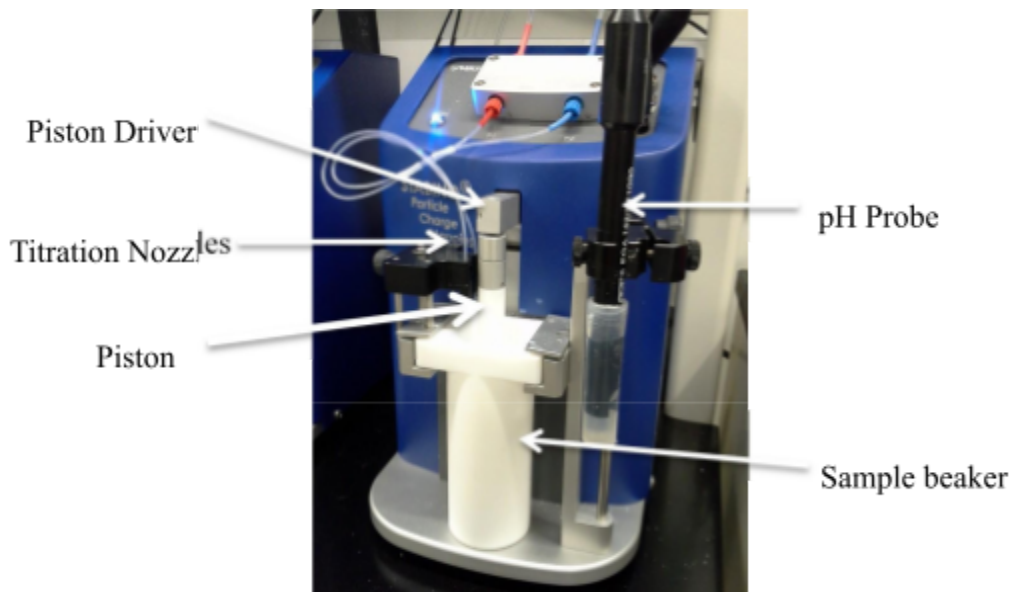


Figure 1. The Stabino zeta tool.

3. **Safety.** Safety glasses should be worn around the Stabino. The system uses reservoirs that contain solutions of hydrochloric acid (HCl) and sodium hydroxide (NaOH). Chemical safety gloves should be worn when directly handling these reservoirs.
4. **Restrictions/requirements.** Samples should be in the concentration range 0.001 to 40 wt%.  
The ionic content of the sample is limited to 0.1M NaCl; at higher salt levels, the zeta signal becomes too weak for a reliable measurement. The minimum sample sizes are:
  - a. Zeta potential only: 5 ml
  - b. Titration for isoelectric point determination: 10 ml.
5. **Required Facilities.** 120V AC power; connection to a Windows-based computer; a source of deionized lab-grade water; 1.0 and 0.1 M solutions of HCl and NaOH.
6. **Definitions**
  - a. *Zeta potential.* When small particles are placed in a polar medium like water, their surfaces will acquire an electrostatic charge via several mechanisms. This surface charge will attract oppositely charged counterions from the liquid. In water, there are always H<sup>+</sup> and OH<sup>-</sup> ions present, so if the surface has been charged +, it will become - + surrounded by OH ions from the water; if charged -, it will attract H ions from the liquid. This leads to a structure called the double layer, and sets up an electric field around each particle. When expressed as a voltage, this is the zeta potential of the particles, often represented as the Greek letter  $\zeta$ . Zeta potential depends on the material, the medium, and the ionic environment of the particle. Values of  $\zeta$  typically range from near zero to  $\pm 200$  millivolts (mV).
  - b. *Isoelectric point* is the pH value at which the zeta potential is zero. At this point there is no electrostatic stabilization of the suspended particles, and agglomeration occurs.

## B. Using the Stabino

### 1. Sample Preparation

- a. See notes above regarding limits on sample concentration and ionic content. Particles to be measured can be in any liquid.
- b. Although organic, nonpolar liquids can be used, better results will be obtained for particles suspended in water or other polar liquids.

### 2. Stabino Setup

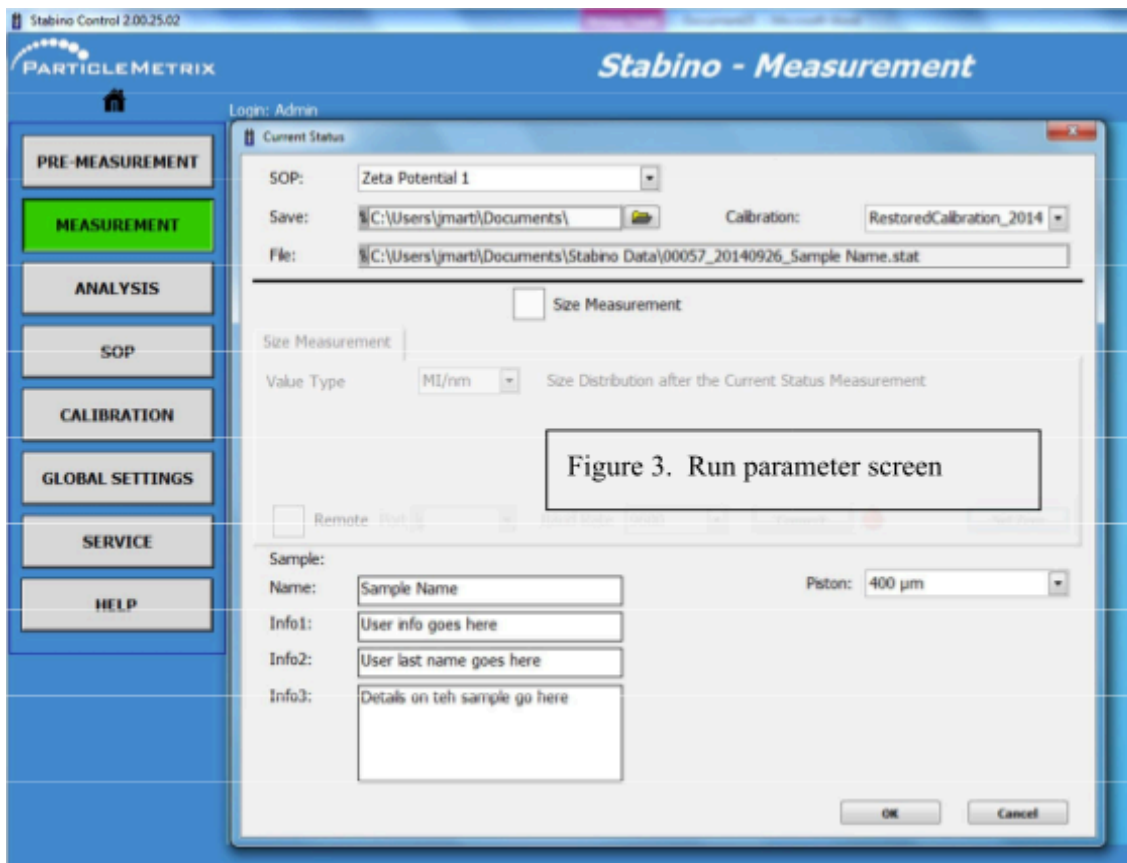
- a. Begin a new entry on the log sheet. List your name, the date, and the materials you are working with (particle material(s) and suspending liquid), If not already on, turn on the Stabino, then the computer. Log into the computer using your own X500 user name and password. **NOTE: the Stabino must be on before the computer is turned on to establish communication with the device.**

- b. Open the operating software, located on the computer desktop or found in the Start menu under “Stabino”. The Stabino login page will load. Log in to the software by clicking “login” (no password necessary). This will bring you to the Stabino home page, shown in Figure 2.
- c. If you are planning to titrate your sample,
  - i. check to ensure that the HCl and NaOH reservoirs are filled. If not, refill them with the 0.1 M HCl and NaOH solutions stored in the lab’s corrosives storage locker.
  - ii. click on the “Premeasurement” box in the Stabino home page. Follow the prompts to enter the levels of the acid and base solutions, and to fill liquid lines 1 and 2. When done, close the Premeasurement screen.
- d. Select a piston. The 400 um piston is used for more viscous suspensions and/or large particles (100 um and above); the 200 um piston can be used for less viscous suspensions (water-thin or nearly so) and/or particles under 100 um in size.
- e. Select a beaker. The system has two beakers, labeled 2 and 3.
- f. Pre-clean both the beaker and piston to ensure the Teflon surfaces carry no trace of the last user’s materials. To do this,
  - i. rinse the sample beaker and piston with tap water
  - ii. fill the beaker  $\frac{1}{2}$  full with a 30% acetone-in-water solution.
  - iii. work the piston up and down in this cleaning solution.
  - iv. use the soft-end brush to clean the interior of the beaker and the surface of the piston.
  - v. discard the cleaning solution in the sink.
  - vi. repeat steps ii to iv three times.
  - vii. rinse the beaker and piston with DI water and shake off excess.
- g. Place the piston into a sample beaker. Note the beaker number written on the front—this will be needed to select the correct calibration.
- h. Slide the beaker into place on the Stabino. Raise the piston slightly so that it magnetically attaches to the piston driver.
- i. Remove the rubber boot from the end of the pH probe and rinse it in a beaker of tap water (do not use DI water). Then place the probe in the probe holder and lower it into the sample holder so that will be immersed in your liquid sample.
- j. Pour your sample into the sample holder. The liquid should be a few mm above the bottom of the wide part of the beaker. To use the pH probe, the liquid must cover the lower 5 mm of the probe bulb.



3. **Selecting an SOP.** Before running a measurement, you must select or create a Standard Operating Procedure (SOP), which is a run recipe.
  - a. From the software home page, select “SOP”. The SOP page provides a dropdown menu of previously defined SOPs. These are of several types.
    - i. Current Status: zeta potential measurements at a fixed pH over a specified time.
    - ii. Kinetics: a series of zeta potential measurements at a fixed pH. The user sets run time, number of runs, and interval between runs.
    - iii. pH Titration: zeta potential measurements over a specified range of pH. The user sets final pH value and acid/base dosing parameters.
    - iv. Polyelectrolyte titration and salt titration are for advanced users. Consult MNC staff on their use.
  - b. Select an appropriate SOP. Most users will use one of the Current Status or Kinetics zeta potential SOPs. The values to modify here are the run time in seconds (Current Status) or run time, number of runs, and interval between runs (Kinetics). Sixty seconds is a minimum run time value; use longer times for smaller potentials.
  - c. If you modify any parameters of an SOP, you must hit “Save” to update the SOP.
  - d. You may define and save a new SOP that fit your analysis needs. Please include your last name in the title. Hit Save to save it for future use.
4. **Zeta Potential Measurement.** Select “Measurement” on the Stabino home page. You will have several options: “Check Sample”, “Potential Measurement”, and “Titration”.
  - a. Select Check Sample to get a quick read of the sample zeta potential, pH values, and conductivity. Select Potential Measurement for a more complete measurement.

- b. Under Potential Measurement, you have the options of Current Status or Kinetics. Selecting either choice will open up a run parameter page, shown in Figure 3.
  - c. On the run parameter screen, your selected SOP should appear in the top parameter box. If not, select it from the drop-down menu.
  - d. In the second parameter box, specify where you want the data files to be stored. A browse button can be used to locate a file location.
  - e. Select the correct calibration file from the drop-down menu to the right. **It is important to select the calibration file that corresponds to the piston and beaker you are using.**
  - f. Complete the sample description boxes: Sample name and Info 1, 2, and 3. Ensure that you place your last name in the Info 2 box.
  - g. Select the piston you are using from the piston drop-down menu.
  - h. When complete, hit “OK”. The Stabino will run your SOP as you have set it up.
- 5. Zeta Potential Measurement with Titration.** To measure the ZP while varying the pH over a specified range, and get a plot of zeta potential vs pH, select Titration from the Measurement page. This will open up a run parameter page similar to that shown in Figure 3.
- a. On the run parameter screen, select an SOP for titration from the drop-down menu.
  - b. In the second parameter box, specify where you want the data files to be stored. A browse button can be used to locate a file location.
  - c. On the right side of the page, select the titrant (HCl for lowering pH, NaOH for raising it).
  - d. Select the correct calibration file from the drop-down menu to the right. **It is important to select the calibration file that corresponds to the piston and beaker you are using.**
  - e. If you'd like to back titrate after the end point pH has been reached, select the Back Titrate box.
  - f. Complete the sample description boxes: Sample name and Info 1, 2, and 3. Ensure that you place your last name in the Info 2 box.
  - g. Select the piston you are using from the piston drop-down menu.
  - h. When complete, hit “OK”. The Stabino will run your SOP as you have set it up.
- 6. Other Types of Titration.** In addition to acid/base titration, samples can be titrated with polyelectrolytes and salt solutions. Consult MNC staff for more details.



7. **Displaying Your Data.** After the measurement run is complete, select the “Analysis” button from the home page. You will see a graph of the results.
  - a. The X and Y axes may be changed to any of the measured parameters. Click on the axis titles to see the options.
  - b. The upper and lower values of the Y axis can be set to your preferred values. Highlight the value with the mouse and change or enter new numbers.
  
8. **Using the database.** You can also recall and display data from prior measurement runs using the Analysis screen.
  - a. In the Analysis screen, click on the “Open” button. This opens up the directory where you have specified that your data files be stored. You may also navigate to any other file location.
  - b. Double click on any data file to present it as a graph. The “Data” button provides a list of all files being displayed.
  - c. The “Table” button shows key measured data in tabular form.
  - d. The data graph, table, and sample descriptive info may be printed to the local printer in room 185, or to a PDF document.
  - e. All measurement data may be exported to a portable or network drive by using the “Export” function.

- 9. Tool Shutdown.** When your work is complete,
- a. Remove the pH probe from your sample and rinse it in a beaker of tap water.
  - b. Disconnect the piston from the driver. Remove the sample beaker with the piston from the Stabino.
  - c. Discard your sample and clean the beaker and piston as follows:
    - i. rinse the sample beaker and piston with tap water
    - ii. fill the beaker  $\frac{1}{2}$  full with a 30% acetone-in-water solution
    - iii. work the piston up and down in this cleaning solution
    - iv. use the soft-end brush to clean the interior of the beaker and the surface of the piston
    - v. discard the cleaning solution in the sink
    - vi. repeat steps ii to iv three times
    - vii. rinse the beaker and piston with DI water and shake off excess.
  - d. Lay both beaker and piston(s) on a clean Kimwipe to air dry.
  - e. Place the pH probe in its holder. Ensure that there is KCl solution in the rubber probe boot, and put it on the probe end.
  - f. Update the log sheet and note any issues you encountered during your run.
  - g. Clean the work area and place used beakers near the sink for cleaning.
  - h. Log out of the computer.