

Protocol

Zeta potential

1. Method

ZetaPALS measures zeta potential using Phase Analysis Light Scattering, to determine the electrophoretic mobility of charged, colloidal suspensions in an electric field. A few [mL] of suspension are required to carry out the measurements. The measurements are conducted in conductive liquids, such as KNO_3 , at different pH. Just a few minutes are required for the sample and cell to equilibrate with the actively controlled temperature environment inside the instrument. Please note that avoiding dust is essential to carry out these measurements. It is often necessary to filter¹ the solution of dispersant used for preparing the various dilutions and to wash all vessels in order to avoid the presence of dust.

2. Equipment

- Measurement device: Brookhaven BI-9000AT (more info from <http://www.bic.com>);
- A special electrode for zeta potential measurement;
- Disposable, acrylic square cells are used for aqueous and simple alcohol suspensions;
- A special glass cell can be used with aggressive solvents;
- Brand new polystyrene vessel of 50 [ml] volume with lid (external diameter 35 [mm], height 70 [mm], for instance Semadeni reference 2278);
- Spatula for powder samples, plastic pipette for liquid samples;
- Stirring rod (26×6 [mm]);
- Ultrasonication horn: Telsonic Ultrasonics, model DG-100, 5 or 15 [min] (see below), 150 [W];
- Ultrasonication bath: Wisag, 5 [minute], 150-300 [W];
- Magnetic stirrer
- Analytical scale (precision 0.1 [mg]);
- Micropipette of 100 [μL], 1000 [μL] and 10 [ml] for preparing dilutions of concentrated samples.

3. Preparations of the samples

- The ideal concentration range for the measurement is 0.1-10 [mg]/[g].
- A stock solution of the sample is prepared, with a concentration of 30 [mg]/[mL], in KNO_3 0.001 [M].
- Three solutions of KNO_3 0.001[M] at pH 3, 6, and 9 are prepared, by adjusting the pH with HNO_3 and KOH 0.01 [M].
- Suspensions, with a concentration of 1.5 [mg]/[mL], are prepared by mixing 1.0 [mL] of the stock suspension to 20 [mL] of pH 3, 6 or 9 KNO_3 solution. These suspensions are placed in an ultrasonic bath during 5 [minutes].
- A few [mL] of this suspension is then transferred to the cell of measurement.
- It is recommended to perform 2 measurements per sample, at each pH (3, 6 and 9).
- If the sample is prepared in an aggressive solvent, use a specific glass electrode.
- It can take some time to reach the equilibrium state. The time of measurement with respect to the time of first contact with H_2O must be noted. If there is an obvious trend in

¹ 20 nm filters, for example Whatmann Anopore

the measurement the sample should be left to equilibrate for a given time (1 hour to 24 hours), and the measurement repeated.

4. Operations

- Switch on the instrument (using the main switch or behind instrument).
- Type the username: ZetaPals. There is no password.
- Click twice the icon “Brookhaven Instruments Corp (Win32)”.
- Select by clicking twice (careful, sometimes slow to open the software)
 - o BIC PALS Zeta Potential Analyser
 - o One can hear a “clic” which indicates the laser has started
 - o Before starting a measurement a delay of minimum 5 minutes must be respected to stabilize the laser and the temperature in the cell
- Select the menu “File”, then “Database”, double click on the file name you want to start with, then select exit.
- Click on “Clear” before starting a new measurement
- In the bottom of the page, click on “Parameters”, and fill the table as described with the example of silica.

Sample ID	SiO ₂ 45nm
Operator ID	FJ
Notes	Klebosol 1508-35-mesure 2- dilué 100×, pH 10
Runs	10
Manual	Cycles 15
Temperature	25°C (it is advised to work at room temperature to improve the sample stability, but this can be changed if necessary)
Liquid	aqueous (⇒ viscosity, refractive index are automatic)
Batch	1
pH	6
Concentration	1.5 [mg]/[mL]
Particle size	45 [nm]
Zeta potential model	Smoluchowski
Auto save results	Select

- Click on “Runs”, and select the number of measurements: 10
- Add your sample in the specific cell of measurement, introduce the electrode of measurement inside, and close it. Connect it to the equipment. Close the door.
- Select “Setup/Incident power setting/optimize signal intensity”, then OK.
- Select “Setup/Diagnostics/temperature stability graph”, to check the temperature stability in the cell of measurement.
- Click on “Start”
- During the measurement:
 - o Check “Reference count rate”: must be between 1500 and 2500 [kcps], and stable
 - o The count rate must be around 500 [kcps]
 - o the blue curve (model) should fit the best and as early as possible the red dots (measurement)

- At the end of the measurement, it is possible to delete some runs which are far from the mean value of the zeta potential, by double clicking on them.
- A standard deviation of ± 5 [mV] is good. A standard deviation of ± 10 [mV] is acceptable.

5. Presentation of the results, data storage and data treatment

Print the results

- File/Print File/Report file settings: choose the options
- File/ Print report.

Export the results

- File/Printer setup: choose PdfCreator
- File/Print report. Save as [Powder-Lotn°-ZetaPals-Experimentn°-Operator.pdf](#).
- File/Create report from saved measurements. Save this file as [Powder-Lotn°-ZetaPals-Experimentn°-Operator.txt](#).

Data storage

- Copy the PDF report and the TXT file.
- Go to \\Ltpc40\powderfiles. Copy the folder *Powderfiles*. Paste it in your project folder, and change its name into [Powder-Lotn°](#).
- Paste the TXT and PDF files respectively in the folders [Project/Powder-Lotn°/ZetaPals/Data](#) and [PDF](#).

Data treatment

- Go to \\Ltpc40\powderfiles. In the folder [Project/Powder-Lotn°](#), open the Excel sheet "Powdersheet.xls"
- Click on the *ZetaPals* button, and follow the instructions given in the Excel sheet.