

BI-ZR5 Zeta Potential Reference Material Preparation Sheet

Brookhaven BI-ZR5 Zeta Potential Reference Material is a dark, blue, inorganic powder that is insoluble in water. Its mean particle size is approximately 300 nm. It is intended for use in the Brookhaven ZetaPlus and ZetaPALS zeta potential analyzers as a reference material. It is useful in monitoring instrument performance and factors affecting the zeta potential determination process. It is not, however, a zeta potential *standard*.

I. Preparation

Photon count rates depend upon instrument specifications (concentration, laser power, detector sensitivity, neutral density filter wheel position, etc.). Therefore, using the count rate to monitor concentration is not sufficient. A dilute, pale-purple suspension is required. There are two ways to achieve this:

- A) A single, average flake, $\sim 500 \mu\text{g}$, in $\sim 20 \text{ mL}$ of filtered, 1 mM KCl. Do not filter the suspension. The concentration is $\sim 2.5 \times 10^{-2} \text{ mg/mL}$. The BI-RC27 screw cap vials hold approximately 20 mL and are convenient to use in the preparation. Bath sonicate for 30 seconds to ensure wetted, de-agglomerated, stable particles.
- B) A two-step dilution to achieve $\sim 2.5 \times 10^{-2} \text{ mg/mL}$. Use $\sim 5 \text{ mg}$ of powder in 20 mL of filtered, 1 mM KCl. Do not filter the suspension. Bath sonicate for 30 seconds to ensure wetted, de-agglomerated, stable particles. Add 1 mL of suspension to 9 mL of filtered, 1 mM KCl.

Exceedingly small quantities of impurities can contaminate the particle surface since there is such a small surface area when dealing with a single flake or even 10 flakes ($\sim 5 \text{ mg}$). These impurities include finger oils, dirt and oils on a spatula used to isolate a flake, weighing containers, and even the towel used for wiping a spatula's tip. Use a chemical wipe. When wipes, spatulas and rubber gloves are left on a lab bench, they will pick up minute quantities of impurities that may ruin the BI-ZR5 preparation. **Discard the diluted sample after 1 day.**

II. Making Measurements

Zeta potential measurements should be carried out at 25 °C. These and other parameters are specified in the PARAMETERS window. Make sure that water has been selected as the liquid. Click on OK to enable your selections and return to the main menu. With the ZetaPlus, click Mode/High Precision in the main menubar. Click START. Thirty, single-cycle measurements are made and automatic statistical analyses are applied. The result is an average, standard error, and percent retained. If the percent retained is less than 90%, rinse thoroughly the cell and

electrodes* with filtered, 1 mM KCl, refill with suspension, and try again. If after three tries, the results are still not satisfactory, make a new BI-ZR5 preparation as described in I. A. or I. B.

With the ZetaPALS, in the PARAMETERS window, select 10 runs. In the Run Time box, click Manual and enter 30 cycles. Select Smoluchowski for the zeta potential model. Make sure water and 25 °C have been entered. Click on OK to enable your selections and return to the main menu. Click START. When the measurement is over, examine the results. Double click to discard any single run if it differs by more than the mean plus or minus three times the standard error. If you have to discard more than three runs, rinse thoroughly the cell and electrodes* with filtered, 1 mM KCl, refill with suspension, and try again. If after three tries, the results are still not satisfactory, make a new BI-ZR5 preparation as described in I. A. or I. B.

Keep the electronic portion of the electrode assembly dry at all times and never touch with bare hands.

III. Results

Under the prescribed conditions, a mean zeta potential value of $-(44 \pm 8)$ mV is acceptable. The conductance should be $320 \mu\text{S} \pm 10\%$.

The single leading cause of errors is sample preparation. The second leading cause is electrode contamination. Discoloration of the electrodes is only a problem if the measured conductance using an accurate, 1 mM, filtered KCl solution is significantly outside the range of 270-370 μS range. Electrodes slowly discolor in the presence of dilute chloride solutions like the 1 mM KCl solution used for the BI-ZR5 measurement. Do not worry about discoloration if the KCl conductance value is not changed.

Thoroughly rinse electrodes between measurements. Never let them dry with sample. If you suspect a contaminate buildup other than discoloration, rub the two inner electrode surfaces with an ethanol-soaked cotton swab or paper towel and rinse with the next sample to be measured. It is also recommended that the electrodes are cleaned regularly between measurements using brief (few seconds) ultrasonication in de-ionized water. (*KEEP THE ELECTRONIC PORTION DRY AT ALL TIMES. Suspending the electrodes in a BI-RC27 filled with liquid protects the electronic portion.*)

If rubbing with alcohol is not sufficient, order the **BI-ELECCK** electrode cleaning kit. Use it sparingly as excessive scratching or removal of electrode metal may lead to poor results.

Electrode conditioning—a procedure used to darken electrodes in high concentration chloride solutions—is required if routine measurements are made in >50 mM electrolyte solutions. Such conditioning is not normally required to obtain good BI-ZR5 results; yet, conditioning is something to try if you cannot achieve the desired results. Contact the factory for an application note explaining how to condition electrodes. Once conditioned, they can be used in low or high salt measurements.