SOP for zeta potential measurement by Zetasizer Nano-ZS

Sample preparation guideline:

1. A concentration should be chosen such that the result is independent of the concentration chosen. The maximum concentration should be less than 1% by volume to avoid particle interaction. The minimum concentration should generate a minimum count rate of 20,000 counts per second (20kcps) for water as the dispersant. The recommended concentration is in the range of 0.1% (for particles as small as a few nm) to 0.0001% (for particles as large as 1 um) by volume.
2. In many cases dilution is needed. However, in this process the existing state of the surface needs to be preserved. One method is to filter or centrifuge some clear liquid from the original sample and use this to dilute the original concentrated sample. Another method is to let a sample naturally sediment and use the fine particles left in the supernatant. Both of these methods try to imitate the original medium as closely as possible. Remember zeta potential is not a size dependent parameter.
3. Zeta potential of particles can be influent by the total ionic concentration, pH and concentration of any surfactants or polymers in the system.

Disposible cuvette cleanup guideline:

The disposible cuvette (DTS1060C) can be reused after flushing by milliQ water for a few times. However, this work should be done carefully to avoid involving any possible contaminations.

Measurement guideline:

1. The instrument should be on all the time. In case it is off, use the following procedure to turn it on. Make sure the lid is close and then turn the power switch at the rear of the unit on (nearby the red label "Nano-ZS"). The button on the top will turn to green/red strip after the initialization routine is finished. Wait 30 minutes for the laser to stabilize.
2. Double click on the icon to start the software DTS (NANO) on the desktop. The button will turn to green. A cuvette holder under the instrument base can be taken out for your convenience. It is also a storage place for the thermal cap and the two cell thermal contact plates.
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3. The two cells used for zeta potential measurements are the folded capillary cell (DTS 1060) and the dip cell. In the case of using a folded capillary cell, the sample transfer procedure is described below:

Prepare the sample in a syringe of at least 1 ml capacity;
Place the sample syringe into one of the sample ports;
Slowly inject the sample through the cell, checking that all the air bubbles are removed;
Once sample starts to emerge from the second sample port, insert a stopper;
Remove the syringe and replace with a second stopper;
No bubbles should be seen within the clear capillary area of the cell;
Remove any liquid that may have spilt onto the electrode;
For dip cell, please check the user manual page 4.10 for details.

4. Place a thermal contact plate (stored at the cuvette holder sitting at the left front corner of the instrument) into the recess on either side of the folded capillary cell. The plates provide

increased temperature stability.
5. Open the cell area lid by pushing the button in front of the lid.
6. Hold the cell near the top, away from the lower measurement area, and push the cell into the cell holder until it stops.
7. Close the cell area lid. Wait a few minutes for the temperature equilibrium.
8. Click **File-New** to create the measurement/record file or open an existing record file. Create a size SOP if it is necessary or go to step 22 for measurement.
9. Select **Configure-New SOP** to create a new one (or **Configure-Existing SOP** to modify an existing one) and click **Next**. **Zeta standard.sop** is recommended for general applications.
10. In the **Measurement type** step, choose "zeta potential". Do not check any options in Instrument settings. Click **Next**.
11. In **Sample identification** step, insert Sample name, General notes, and Custom Parameters if it is necessary, then click **Next**.
12. In the **Operator instructions** step, insert instructions and click **Next**.
13. In the **Cell type** step, choose DTS1060C, clear disposable zeta cuvette and click Next.
14. In the Sample Settings step, for F(ka) selection, the model will be used is Smoluchowski: 1.5 and Huckel: 1.0 in the aqueous media and non-aqueous media, respectively. The detailed description can be found in the user manual chapter 16. Choose the material and the dispersant. The properties (reflective index, absorption, viscosity) of new materials can be added. Click **Next**.
15. In the **Temperature** step, choose the Temperature and the Equilibrium time, then click **Next**.
16. In the **Measurement settings** step, choose the Measurement duration and the number of measurements base on your application. For advanced, please check the user manual page 9.32 for details. Click **Next**.
17. In the **Result calculation** step, choose the General Purpose for most of applications (more than 10 runs). The monomodal should be used if only the mean value of zeta potential is required (5 to 10 runs). For **Advanced option**, please check the user manual page 9.34 for details.
18. Skip the **Reports** step if no printer is hooked up with the computer.
19. In the **Result export** step, the Export template, the name and the path of the output file can be specified. Click **Next**.
20. Click **Finish** to accomplish SOP generation.
21. Save the SOP in the folder ..\DTS\SOP\
22. Click **Measure-Start SOP** to select the SOP file. Click OK if the cuvette you are using is DTS1060C.
23. Input Sample name and General notes.
24. The **SOP Measurement** window will pop up. Click **Start** to initialize the measurement.
25. The quality of the measurement can be monitored by Click Count Rate. The profile should be random without a clear time dependent trend. Click Correction to check the correction function, which should be a quite smooth curve used to calculate the particle size. Click Result to see the mean size. The portions of the multiple sizes is also available if the sample has mulitple particle sizes. The Attenuator 1 means the maximum attenuation and attenuator 11 means no attenuation. By using 60 nm PSL in 1 mN NaCl, the zeta potential is in the range of -62 ~ -66 mV with mean -64 mV. By using 5.05 um PSL particle in 1 mN NaCl, the zeta pootential is in the range of -81 ~ -89 mV with mean 86 mV. The conductivity value is also available.
26. The measurement result is shown in the measurement file window. Units such as temperature T, zeta potential ZP can be modified by clicking the unit. The detailed measurement results, such

as intensity and zeta potential can be checked as well. The graphs can be copied and pasted to a word file.

27. Change another cuvette and click **Start** to initialize the next measurement.

28. The measurement results can be exported by **File-Export** to multiple destinations. It also can be edited by selecting the record, right clicking the mouse and selecting Edit Results. The SOP measurement has to be closed before doing any edit work.

29. After finish the measurement, take the cuvette out and close the lip. Close the software and shut down the power of the Zetasizer. Put the thermal contact plates into the cuvette holder and swing it back. The data can be transferred by a USB extension cable to your flash drive. Please clean up the operation bench before you leave.

30. For any unexpected error, please exit the program and reopen it.

SOP for hydrodynamic size measurement by Zetasizer Nano-ZS

Sample preparation guideline:

1. A concentration should be chosen such that the result is independent of the concentration chosen. The maximum concentration should be less than 1% by volume to avoid particle interaction. The minimum concentration should generate a minimum count rate of 10,000 counts per second (10kcps) for water as the dispersant. The recommended concentration is in the range of 0.1% (for particles as small as a few nm) to 0.0001% (for particles as large as 1 um) by volume.
2. Ultrasonication can be used to remove air bubbles or to breakup agglomerates. Keep in mind that in some cases the primary particles may be damaged.
3. The most common dispersant is water. Others like alcohol are also acceptable. The disposible cuvette (DTS0012) can be reused after rinsing by milliQ water for a few times. However, this work should be done carefully to avoid involving any possible contaminations.
4. The size range is from 1 nm up to 6 um, based on 1 g/cm3 density.

Measurement guidelines:

1. The instrument should be on all the time. In case it is off, use the following procedure to turn it on. Make sure the lid is close and then turn the power switch at the rear of the unit on (nearby the red label "Nano-ZS"). The button on the top will turn to green/red strip after the initialization routine is finished. Wait 30 minutes for the laser to stabilize.
2. Double click on the icon to start the software DTS (NANO) on the desktop. The button will turn to green. A cuvette holder under the instrument base can be taken out for your convenience. It is also a storage place for the thermal cap and the two cell thermal contact plates.
3. The disposable polystyrene cuvette is stored in the consumable kit. This type of cuvette is easily scratched. However, it can be reused after it has been carefully cleaned up by milliQ water. It is also not resistant to organic solvents. Use glass or quartz cuvette for organic dispersants. Detailed information can be found in the user manual page 4.4.
4. Use plastic syringe or pipet to transfer about 1~1.5 ml liquid sample to the cuvette (1 ml is the minimum sample volume or 10 mm from the bottom of the cell). Do not overfill the cell (about 15 mm maximum) since it will reduce the accuracy of the temperature control.
5. Open the cell area lid by pushing the button in front of the lid.
6. Push the cell into the cell holder until it stops.
7. Close the cell area lid. Wait a few minutes for the temperature equilibrium.
8. Click **File-New** to create the measurement/record file or open an existing record file. Create a size SOP if it is necessary or go to step 22 for measurement.
9. Select **Configure-New SOP** to create a new one (or **Configure-Existing SOP** to modify an existing one) and click **Next**. **Generic size.sop** is recommended for general applications.
10. In **the Measurement type step**, choose "size". Do not check any options in Instrument settings. Click **Next**.
11. In **Sample identification step**, insert Sample name, General notes, and Custom Parameters if it is necessary, then click **Next**.
12. In the **Operator instructions step**, insert instructions and click **Next**.
13. In the **Cell type step**, choose DTS0012-Disposable sizing cuvette and click **Next**.

14. In the **Sample Settings step**, choose the material and the dispersant. The properties (reflective index, absorption, viscosity) of new materials can be added if you want to calculate the volume of the particle (Caution!). These parameters are not necessary for size distribution measurement. Click **Next**.
15. In the **Temperature step**, choose the Temperature and the Equilibrium time, then click **Next**.
16. In **the Measurement settings step**, choose the Measurement duration and the number of measurements base on your application. Click **Next**.
17. In the **Result calculation step**, choose the General Purpose for most of applications. If the profile of size distribution is known to have multiple peaks, choose Multiple Narrow Modes. Using **Advanced options** can further optimize the analysis results. Click **Next**.
18. Skip the **Reports step** if no printer is hooked up with the computer.
19. In the **Result export step**, the Export template, the name and the path of the output file can be specified. Click **Next**.
20. Click **Finish to accomplish SOP generation**.
21. Save the SOP in the folder ..\DTS\SOP\
22. Click **Measure-Start SOP** to select the SOP file. Click OK if the cuvette you are using is DTS0012.
23. Input Sample name and General notes.
24. The SOP Measurement window will pop up. Click Start to initialize the measurement.
25. The quality of the measurement can be monitored by Click **Count Rate**. The profile should be random without a clear time dependent trend. Click **Correction** to check the correction function, which should be a quite smooth curve used to calculate the particle size. Click **Result** to see the mean size. Portion of the multiple sizes is also available if the sample has mulitple particle sizes. The Attenuator 1 means the maximum attenuation and 11 means no attenuation. For 60 nm PSL particle in 1 mN NaCl with milliQ water, the hydrodynamic size is around 65 nm. For 5.05 um PSL particle in 1 mN NaCl with milliQ water, the hydrodynamic size is in the range of 4.0 to 4.8 um.
26. The measurement result is shown in the measurement file window. Units such as temperature T, zeta potential ZP can be modified by clicking the unit. The detailed measurement results, such as intensity and zeta potential can be checked as well. The graphs can be copied and pasted to a word file.
27. Change another cuvette and click **Start** to initialize the next measurement.
28. The measurement results can be exported by **File-Export** to multiple destinations. It also can be edited by selecting the record, right clicking the mouse and selecting Edit Results.
29. After finish the measurement, take the cuvette out and close the lip. Close the software and shut down the power of the Zetasizer. Put the thermal contact plates into the cuvette holder and swing it back. The data can be transferred by a USB extension cable to your flash drive. Please clean up the operation bench before you leave.
30. For any unexpected error, please exit the program and reopen it.