A Guide to Proper Sample Preparation: Electrostatically-Stabilized Nanoparticles in Water©

By Eric Farrell, July 2010

Introduction: Sample preparation is the most important step in obtaining the best results for both dynamic and static light scattering. While the instrument or software is the easiest to blame for poor results, improper sample preparation is usually the culprit. This guide will outline in detail the steps necessary to acquire relatively dust-free samples.

For this guide, a 92 nm (± 3 nm) polystyrene latex standard was used. These steps will be relevant for many other samples but proper dilution will need to be determined by the user.

WEAR GLOVES AND PROPER EYE PROTECTION DURING THIS ENTIRE PROCESS

A. Preparation of glassware, syringe and syringe filter:

1. Attach a 0.1 µm aqueous syringe filter to the end of a 20 mL or 30 mL disposable syringe. Pass about 20 mL of de-ionized water through it to remove any particles left on the filter during manufacture.
2. Take a clean 20 mL to 30 mL bottle with cap and rinse 3 times with filtered (0.1 µm), de-ionized water.
3. Fill bottle with fresh 10 mM KNO_3 filtered through 0.1µm syringe filter.
4. Vortex or place in ultrasonic bath for approximately 10 seconds. This step helps to dislodge any particles stuck on the bottle.*
5. Empty contents of bottle to waste.

B. Preparation of diluent:

6. Fill bottle with at least 20 mL of fresh 10 mM KNO_3 filtered through 0.1 µm syringe filter. Gently, to avoid generating bubbles, roll capped bottle so any dust in top of cap is washed back into bottle for later filtering.
7. Detach syringe filter from syringe and remove plunger.
8. Re-attach syringe filter and pour contents of bottle into top of syringe, then replace plunger.
9. Pass contents of syringe through syringe filter back into bottle.
10. Repeat steps 6 through 9 so the 10 mM KNO_3 has been filtered 3 times through the 0.1 µm aqueous syringe filter, each

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time making sure dust is washed out of the cap. On the third filtration, use black lines on the syringe to ensure the final volume of diluent filtered into bottle is 20 mL.

C. Preparation of latex standard for analysis:

11. Take bottle of 92nm latex standard (confirm latex is not expired) hold in hand and rock back and forth gently to avoid bubble generation.
12. Place bottle in ultrasonic bath* for 10 seconds, then dry with a soft tissue.
13. Take cap off of latex standard bottle. Inspect both the inside of the cap and top of bottle for any dried latex. If dried latex is present, clean off carefully with a soft tissue.
14. Waste two drops of latex and place the third drop into the 20 mL of filtered 10 mM KNO_3 (prepared in step 10). Make sure to recap bottle immediately after latex drop is introduced to the diluent.
15. Gently rock the suspension back and forth, ensuring it becomes homogeneous (solution will become slightly turbid).
16. Place into ultrasonic bath for approximately 10 seconds.*
17. Rinse exterior of bottle with de-ionized water and dry with a soft tissue.

D. Preparing and filling sample cell with latex suspension:

18. Fill cell with 0.1 μm filtered, 10 mM KNO_3 solution.
19. Place capped cell into ultrasonic bath for approximately 10 seconds.* Again, this is to help dislodge any dust stuck to cell walls and cap.
20. Rinse exterior of cell with either de-ionized water or the filtered salt solution and wipe dry with soft tissue.
21. Empty contents of cell into waste.
22. Fill cell with the 92 nm latex suspension and cap. Pour but do not use a syringe or pipette for that only adds yet another, potentially, dust-laden surface.
23. Place in ultrasonic bath for approximately 10 seconds.*
24. Rinse exterior of cell with de-ionized water and wipe dry with soft tissue.
25. Empty contents of cell into waste.
26. Fill sample cell (by pouring) once more with 92nm latex suspension and quickly cap.
27. Place in ultrasonic bath for approximately 10 seconds.*
28. Wipe all sides of the cell with a soft tissue to ensure the outside of the cuvette is dry and clean of smudges and free of surface-contamination.

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29. After placing sample cell with 92 nm latex suspension into instrument for analysis, make sure to wait at least 5 minutes prior to beginning analysis so sample may reach the temperature of the cell holder. Be patient.

Summary: If you are unfamiliar with light scattering, using all the steps listed here may seem like unnecessary work. However, without proper sample preparation one's measurements could easily suffer: The Effective Diameter and, more significantly, the Polydispersity Index may be too high. Proper sample preparation is the user's responsibility. Poor samples will yield poor results.

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* If a vortexer or ultrasonic bath is not readily available, this step can be skipped, and accurate results can still be obtained.

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