

Laser Diffraction Data Correlation

Particle size analysis may be slightly different than other analytical techniques in so far as result correlation from one system to another is concerned. This technical note investigates the sources of result differences and makes recommendations to analysts attempting to improve data correlation.

Comparing Results from Different Techniques: SEM vs. SSA, DLS and Laser Diffraction

Consider a “nanoparticle” sample like that shown in Figure 1. From the SEM image one could conclude the typical particle size to be near 50 nm by using the scale shown and comparing it to several particles. But like most nano scale samples the particles are aggregated. If the particles only touch each other at the surface, then the specific surface area (SSA) could be on the order of magnitude of individual 50 nm particles. If the SSA were measured using the BET gas adsorption technique then SSA can be converted to an average particle using the equation:



Figure 1: SEM image

$$SSA=6/\rho D$$

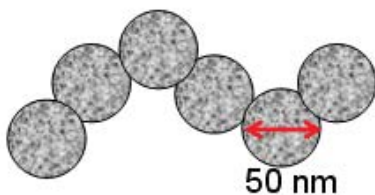


Figure 2: Aggregated particles

This diameter might be close to 60 nm. But if the sample were analyzed using dynamic light scattering (DLS) this technique measures the hydrodynamic diameter of the aggregated particles. DLS will report

the diameter of a sphere that diffuses at the rate of the observed particles. The most common result used from DLS is the z-average, which is based on the intensity distribution of the measured sample. If the sample were aggregated as shown in Figure 2, then DLS may report a result around 250 nm.

If the same aggregated sample were analyzed using laser diffraction the result could be near 220 nm, probably smaller than the DLS result since this technique reports results based on a volume distribution. Next consider what would happen if the aggregated particles were first exposed to several minutes of ultrasonic energy from a probe prior to analysis by DLS and laser diffraction. These results could be reported anywhere from 60-200 nm depending on the energy level the particles were exposed to. Sample preparation will have an enormous influence when measuring particles such as these.

So now we ask the question: Why doesn't one technique correlate well with another? Because different techniques measure different physical properties and report results using different basis. Results from SEM, DLS and laser diffraction should NOT all match. If they did, then the analyst should be more skeptical of the results than if they varied by technique.

Sieves vs. Laser Diffraction Correlation

Sieves are still widely used to analyze particle size – especially larger powders. Many analysts upgrade from using sieves to laser diffraction to save both time and effort, but the historic sieve results do not typically match the newer laser diffraction results. This often prompts calls to HORIBA technical support for help with data correlation. Our discussion with these analysts begin with the explanation of the effect of particle shape on reported results, and thus data correlations.

Consider the cylinder shown in Figure 3 that is 100 μm long by 50 μm diameter. Since the smallest 2 dimensional projected area is 50, it could theoretically pass through a sieve with a 50 μm opening. If you calculate the volume of this cylinder and then calculate the diameter of a sphere with the same volume, you get a diameter of around 72 μm. Measure this cylinder with laser diffraction and report an equivalent spherical diameter and we expect to get

around 72 μm , 44% larger than the sieve result. In the real world laser diffraction results can be anywhere from 10 to 40% larger than sieve results due to this particle shape effect.

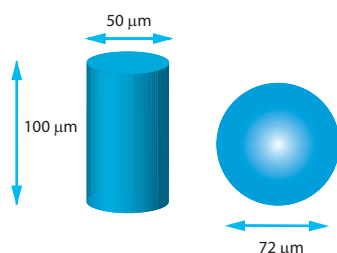


Figure 3: A cylinder $D = 50 \mu\text{m}$, $h = 100 \mu\text{m}$ and a sphere $D = 72 \mu\text{m}$

What does an analyst do who has switched from sieving to laser diffraction and sees the data shifted to larger sizes? One option is to change the product specifications to match the new results – an approach we prefer, but realize is not always possible. Another option is to accept and work with the size shift. If 50% of the sample passed through a 325 mesh sieve (44 μm), but the median size by diffraction is 53 μm (270 mesh), then just report the value at 53 μm as the pass 325 mesh result. This approach could be duplicated at other sizes.

Correlating Laser Diffraction to Laser Diffraction Data

Another frequently asked question is why one laser diffraction result does not match another. Without additional information such questions are often impossible to answer. Major sources of variation include sample preparation and method used, vendor to vendor differences, and generation (model number) to generation differences.

Sample Prep and Method

It is hard to overestimate how much sample prep and method affects laser diffraction results. A change in surfactant used or amount of ultrasound applied can greatly alter results. Does the method being used measure the sample as a dry powder or dispersed in liquid? What pump speed is used, which refractive index, at what concentration, which air pressure if analyzed dry? These questions must be asked and understood if data correlation is expected. As an example of the magnitude of differences in results Figure 4 shows various results from the LA-950 on the same sample measured different ways. The reported D50 varied by several decades depending on the dispersion and test method.

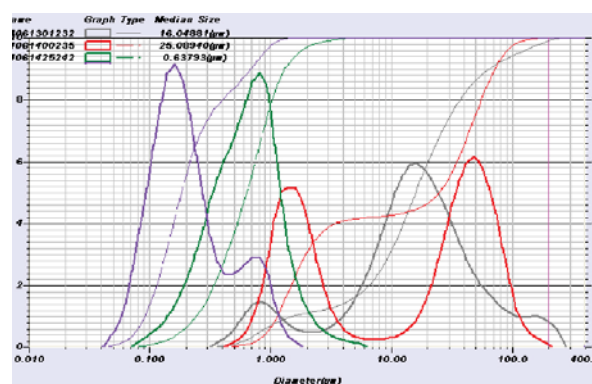


Figure 4: LA-950 results from the same sample analyzed different ways

Vendor to Vendor Variations

Another source of potential challenges with data correlation arises if more than one manufacturer of laser diffraction analyzer is involved. Choices made during the design process can influence results as well as many other factors including:

- Dynamic range, one system may be more sensitive to small or large particles
- Pump power and reservoir design – especially for large particles
- Power rating of an internal ultrasound source
- Blending of two optical techniques such as PIDS
- Fraunhofer vs. Mie modeling
- Scattered light to particle size conversion algorithm
- One vs. multiple lenses to cover a given dynamic range

Any one or a combination of the above design styles can influence results, making the data from one supplier different than another.

Generation to Generation

When the HORIBA LA-950 was introduced the data for some samples varied when compared to older models (LA-920 and 930). All suppliers who upgrade their technology experience this issue. Some of the reasons for the data correlation differences are listed in the table in the previous section of this document. An optional correlation tool exists to help facilitate data correlation between the LA-920 and LA-950 was introduced in mid 2012 to help analysts improve data comparability. Please contact the HORIBA technical support team for questions about this feature. It is only available for existing HORIBA LA-920/930 analysts analyzing their samples in wet mode.

Summary

A wide range of factors influencing data correlation challenges have been addressed in this technical note. Many of these concepts are incorporated into the flow chart shown in Figure 5. We hope this chart helps analysts

think through the issues involved before initiating any effort to investigate data correlation. Please remember to always run a standard sample to assure system integrity before questioning why results from one system vary significantly from another.

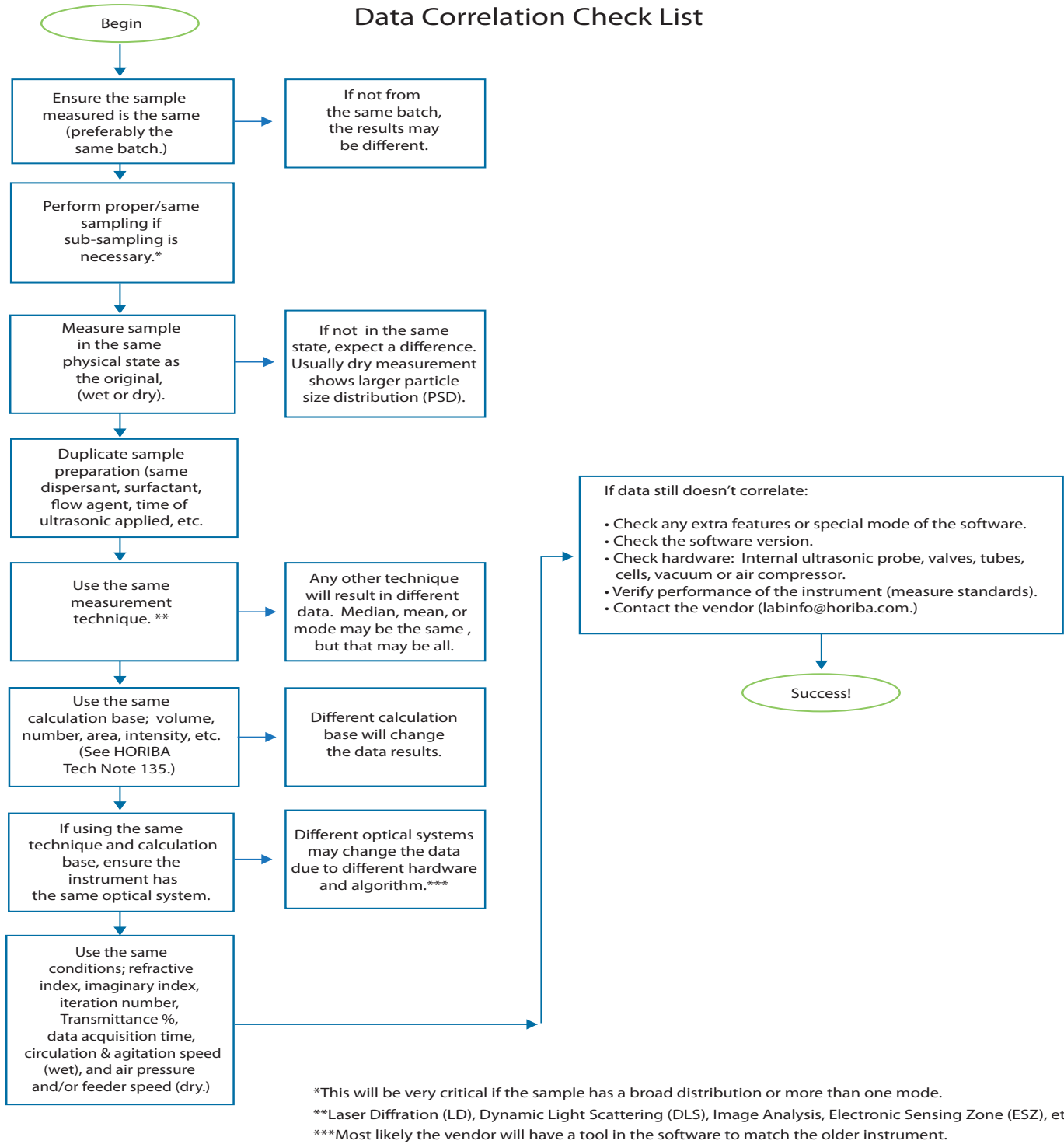


Figure 5: Data Correlation flow chart