DLS Time of Measurement

Nicomp® DLS system

Dynamic light scattering (DLS) is the most common particle size analysis technique for submicron samples because it is relatively easy-to-use and has short analysis times. But users should be wary of defaulting to short analysis times (around two to three minutes) when measuring unknown, or complex samples. This technical note provides guidance for how to determine the proper analysis time when using the Nicomp® DLS system.

INTRODUCTION

How long should a DLS measurement last? That depends on the sample. DLS measurements can be fairly quick (a few minutes) for narrow, single-mode samples. Narrow distributions can be analyzed more quickly than broad distributions. Single-mode distributions can be analyzed more quickly than multi-modal samples. A good standard with DLS is "don't be in a hurry." Instrument manufacturers that tout quick measurement times in their marketing data are doing their customers a disservice. It's more important to generate good, reproducible results than to shave a minute or two from the analysis time. You get an answer every time you make a measurement but that doesn't mean every answer is good enough to base important decisions on.

NICOMP SOFTWARE

To select the analysis time open the Auto Print/Save Menu and enter the desired measurement sequence in the Auto Operations dialog box, see Figure 1.

In this example, the total analysis time will be 10 minutes. Two results will be saved during the measurement, one at 5 minutes and one at 10 minutes. This is typically better practice than choosing No. Print/Save Cycles 1 and Using Run Time 10 minutes, which would also have a total analysis time of 10 minutes but only one result would be saved.

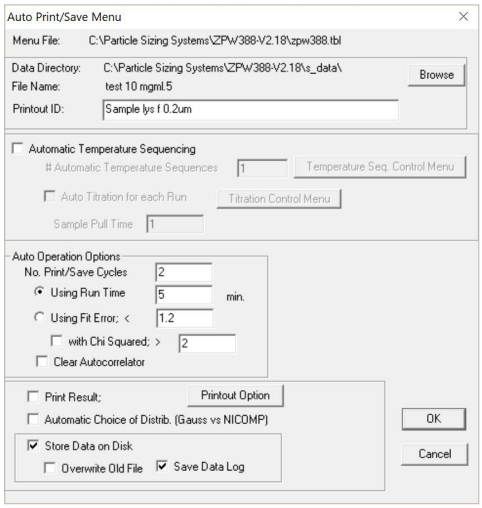

Figure 1. Auto Print/Save Menu

When not clicking on Clear Autocorrelator the system continuously accumulates the data over the entire time, possibly improving the final answer. This is the typical suggested mode of operation with the run time and No. Print/Save Cycles adjusted for the specific sample.

The analysis can also be programmed to run until a specified fit error, or Chi squared is achieved, but this is not the typical mode of operation.

EXAMPLE RESULT 1: NARROW DISTRIBUTION

A 92 nm polystyrene latex (PSL) sample is the easiest type of sample to analyze by DLS and can be measured fairly quickly. The result shown in Figure 2 is accurate, narrow and reproducible.

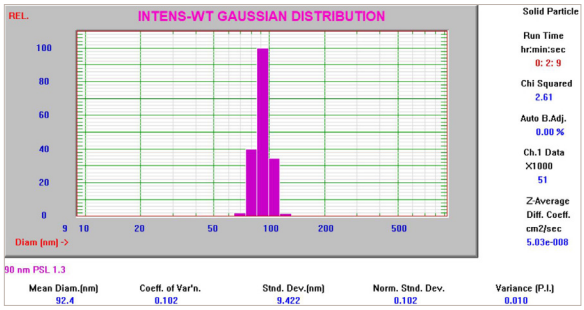

Figure 2. 92 nm Polystyrene latex (PSL) result

For any sample, the best way to check post measurement if the analysis time was sufficiently long is to open the Time History plot. To view this result click on Display, then Time History. The Time History plot for the result seen in Figure 2 is shown in Figure 3. The most important result is the intensity weighted (Wt) mean value (in red). The volume weighted mean is shown (in dark blue) and the number weighted mean (in aqua). The volume and number mean values are typically only used when comparing DLS to other techniques such as laser diffraction (volume) or microscopy (number). For this sample, the result reached a stable value after about 2 minutes. This is the shortest suggested measurement time, and is only appropriate for narrow, single peaks like PSL standards.

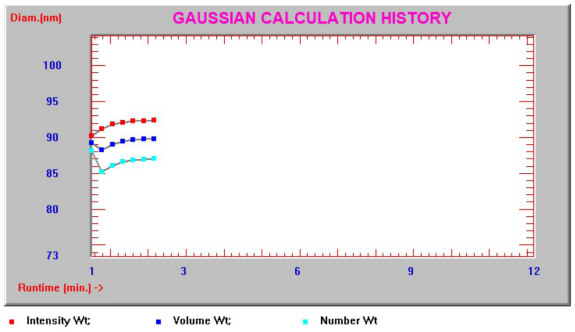

Figure 3. Time history plot for 92 nm PSL

EXAMPLE RESULT 2: BROAD DISTRIBUTION

A broad distribution requires a longer analysis time. The result shown in Figure 4 is a poorly dispersed mineral oil emulsion. This sample was analyzed for 3 minutes. As seen in Figure 5 this was clearly not a long enough analysis time since all three mean values are still changing with time.

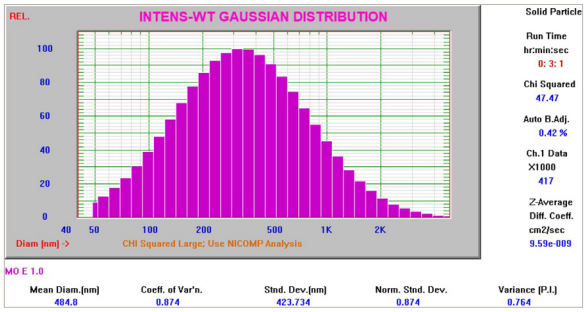

Figure 4. Broad distribution of mineral oil emulsion

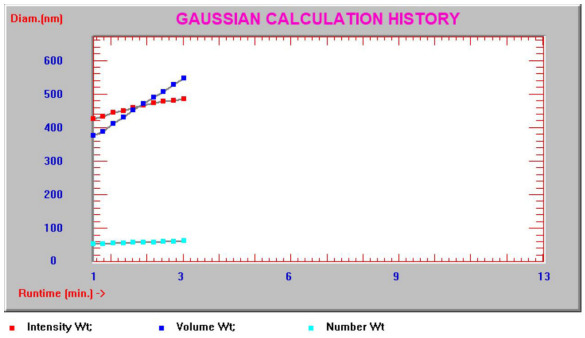

Figure 5. Time History plot of mineral oil emulsion

Note: Although there is a high Chi square value and the software prompts considering the multi-modal Nicomp result, there is no reason to believe this emulsion has more than one peak so the Gaussian result is a better option than the Nicomp result.

EXAMPLE RESULT 3: MULTI-MODAL DISTRIBUTION #1

A multi-modal result also typically requires a longer analysis time. A silica abrasive sample was analyzed using Run Time = 9 minutes, Save Cycles = 8. The final result and Time History results are shown in Figures 6 and 7. The Nicomp algorithm did not resolve the result into three peaks until after 7 minutes analysis time – when the third peak in aqua appears in the time history result. Eight to ten minutes is a good analysis duration for difficult, multi-modal samples.

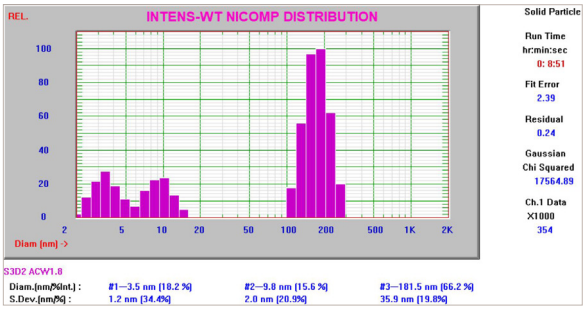

Figure 6. Multimodal size result #1

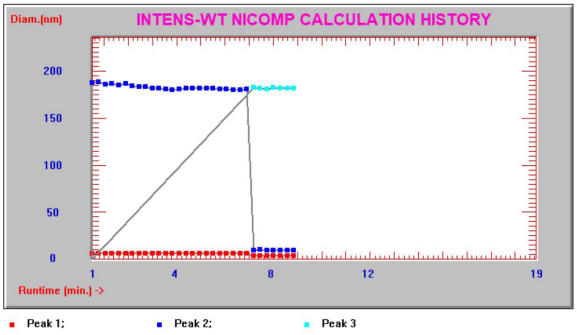

Figure 7. Time History plot of multimodal sample #1

EXAMPLE RESULT 4: MULTI-MODAL DISTRIBUTION #2

A multi-modal result can occasionally be properly analyzed using shorter analysis times. A silica abrasive sample was analyzed using Run Time = 5.5 minutes and Save Cycles = 5. The Nicomp size results and time history results are shown in Figures 8 and 9. This shows the rare example when shorter analysis times can be used for multi-modal samples. But note that the last two calculations are most likely the better results showing a slightly smaller first peak.

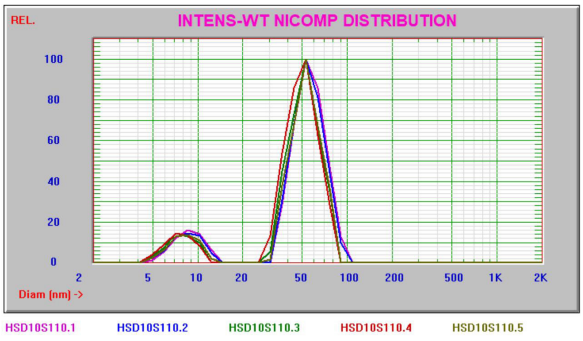

Figure 8. Size result for multimodal sample #2

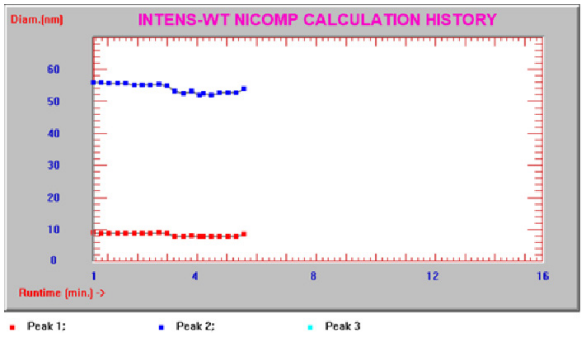

Figure 9. Time History plot for multimodal sample #2

CONCLUSIONS

It is tempting to make quick DLS experiments and just use the first results generated, but this is not the recommended best practice. Make sure to always make longer measurements when analyzing broad distributions or multi-modal samples. A two minute analysis time is the quickest recommended duration. Five minutes is a reasonable quick measurement time for broad (PI >0.2) distributions. Multi-modal samples should typically be measured for seven to ten minutes. Whenever analyzing an unknown sample check the Time History plot to assure the mean result has stabilized.

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