Sub 1 nm Sizing

Nicomp® DLS System

The specifications for dynamic light scattering, DLS systems are typically sample dependent. But many DLS systems specify the lower size limit as either 1 nm or smaller (0.3 nm). This technical note documents proof that the Nicomp® DLS system is capable of repeatedly measuring particle size below 1 nm and provides details on how the measurements were performed.

INTRODUCTION

Dynamic light scattering is the preferred measurement technique for particle size analysis below 1 µm. The basic dynamic range for this technique is roughly 1 nm – 1 µm, but the performance at both ends is sample and system dependent.

The upper size limit of DLS depends on the size and density of the dispersed phase. Emulsion samples may be possible at ~10 µm since the density of the dispersed and continuous phase are fairly similar. But for a high density solid particle such as gold (19.29 g/cc), the upper limit will be much smaller. Once the particle motion is dominated by sedimentation, rather than Brownian Motion, the result is no longer reliable. The upper size limit of DLS is also hindered by number fluctuations as the few large particle enter and leave the inspection zone.

On the small size, the specification is dependent on the construction and optical quality of the instrument, as well as the scattering properties and concentration of the sample. Please see Entegris Technical Note, 0.1 mg/mL Lysozyme[1] for proof of superior Nicomp performance of this difficult sample at very low concentration. Measurements near 1 nm are challenging and require careful sample preparation.[2] This technical note describes measurements made at/below 1 nm using sucrose and the sample. Note: Optimum Nicomp performance near 1 nm typically requires the 35 mW laser and APD detector.

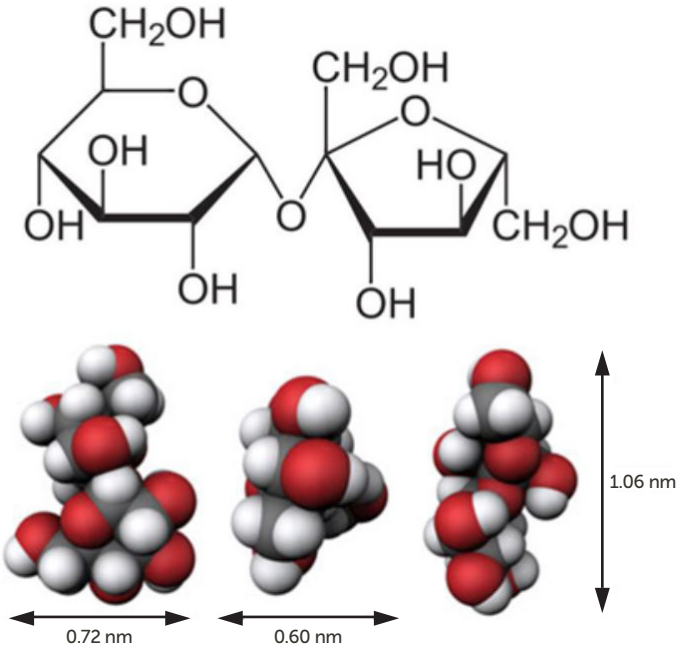


Figure 1. Sucrose molecule.

MATERIALS

Sucrose (Figure 1) was purchased from Fisher Chemical, certified ACS crystalline, S5-500, lot 173322, chemical formula $C_{12}H_{22}O_{11}$, molecular weight = 342.2965 g/mol. Sucrose solution was prepared at 10 volume percent using filtered DI water.

All measurements were made using the Nicomp Z3000 DLS particle size analyzer (Figure 2) configured with a 35 mW laser diode and APD detector.

Figure 2. Nicomp Z3000 DLS system.

INSTRUMENT SETTINGS

The typical measurement settings used in this study is shown in Figure 3.

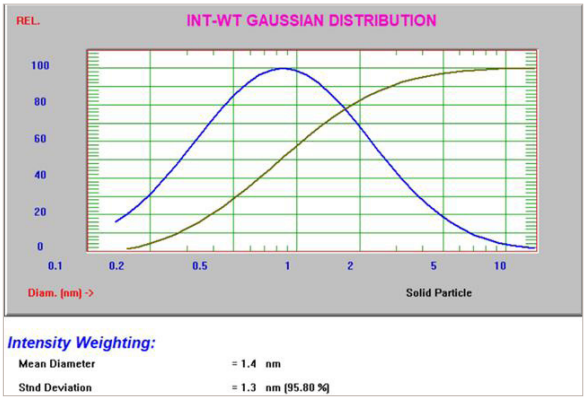
Refractive index	1.333
Viscosity	1.172 cP
Auto sensitivity	Checked
Auto baseline adjustment	Checked
Channel width	5 µsec
Temperature	23° C
Intensity set point	300 kHz
First channel used	2
Scattering angle	90°
Cell type	Round
Run time	7 minutes

Figure 3. Nicomp measurement settings.

Note: The viscosity data used was 1.172 cP for 10% sucrose. Accurate viscosity values are required for accurate DLS results for non-dilute based measurements.

RESULTS: GAUSSIAN

The Gaussian intensity and volume results[3] for 10% sucrose are shown in Figure 4.



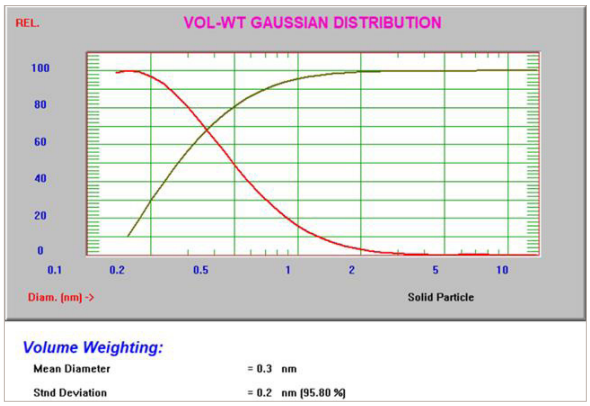


Figure 4. Intensity and volume Gaussian results for 10% sucrose.

RESULTS: NICOMP

The Nicomp results are based on a non-negative least squares algorithm optimized to resolve multi-modal distributions. All results in this study generated high Chi-square[3] values, indicating the presence of multiple modes. The Nicomp intensity result for the 10% sucrose is shown in Figure 5.

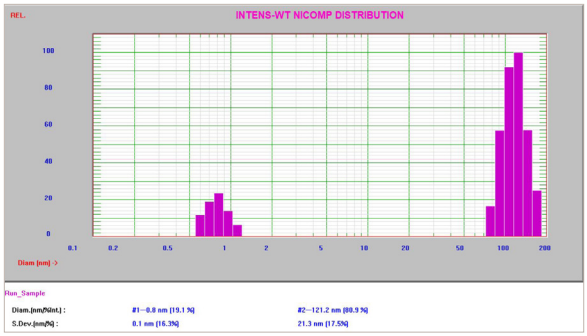


Figure 5. Intensity Nicomp result for 10% sucrose.

CONCLUSIONS

The Nicomp DLS system is quite capable of measuring down to, and below, 1 nm, supporting the lower size specification of 0.3 nm. The larger second peak at around 100 – 200 nm has been reported in other published literature[4] and is believed to be impurities from the raw material and processing.

References

[1] Entegris Technical Note, 0.1 mg/mL Lysozyme

[2] Entegris Technical Note, DLS Sample Preparation

[3] Entegris Technical Note, DLS Data Interpretation

[4] Weinbuch et al., *nanoparticulate impurities in pharmaceutical-grade sugars and their interference with light scattering-based analysis of protein formulations,* pharm res (2015) 32:2419–2427

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