HORIBA Scientific

Application Note

LA-300 to LA-350 Method Transfer and Data Correlation

AN239

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Introduction

Nearly three decades since its initial release, the LA-300 Laser Diffraction Particle Size Analyzer has entered the legacy product category*. Its unique attributes such as portability, affordability, ease of use, and excellent system-to-system agreement made the LA-300 the choice of analyzer in commercial sectors such as dairy, soil and sediments, and paper chemicals.

In developing the next-generation Partica mini LA-350, HORIBA has strategically retained these key qualities while extending the dynamic size range and enhancing automation. This note will demonstrate the robustness of the LA-350 and describe key points for successful method transfer and data correlation.

Background

Before diving into the inherent differences between instruments and resulting variance, it is vital to first discuss possible error(s) from historic measurements. This step is crucial to understanding resulting reliability and setting specifications.

Fundamentally, laser diffraction is a robust technique. When the same scattering pattern is recorded, the same particle size distribution (PSD) will be converted and attained using the same measurement conditions. The job of the analyst is to provide the system the best possible opportunity to collect data in a consistent manner.

Factors that may hinder the LA-300 and the LA-350 data correlation, otherwise referred to as error sources and diagnosis [1], are listed below:

- Poor sampling for polydisperse test samples [2].
- Dirty window cell.
- Measurements acquired with excessive levels of background or background fluctuation.
- Measurements acquired with insufficient measurement time.
- Misaligned optical system.
- Improper particle concentration (too low or too high laser transmittance (T%)).
- Improper stirring and pumping speed.

When possible errors are removed, excellent agreement can be achieved.

Instrument Qualification

There are six experimental factors described in ISO guidance [1] that help users think about instrument qualification and the trustworthiness of the data generation process. All these aspects funnel down to two main points: result accuracy relative to a certified reference material (CRM), and result repeatability.

This section will demonstrate both by using parameters Dv10, Dv50, and Dv90. Further system-to-system variability study can be found in Tech Note 175 [3].

Accuracy – CRM

Concerning accuracy, ISO recommends measuring at least three separate test samples and at least three repeats each to assess if the average for its perspective cumulative volume distribution falls within the acceptance limit. The acceptance limit is calculated by taking both instrument and CRM uncertainties into account (Formula 1 [1]).

For simplicity purposes, the expanded tolerance limit is calculated and illustrated in Table 1. Table 1 shows the average of 3-6 repeats for 100 µm, 10 µm, 300 nm NIST traceable microsphere standards**, and PS313 polydisperse glass beads. Each CoV (Dv10, Dv50, and Dv90 respectively) calculated from measured data did not deviate from the values set out by ISO standard.

***Note that NIST traceable CRM is a series of polymer beads with calibrated **mean** diameter; certified values of Dv10, Dv50, and Dv90 are not reported. In Table 1, we compare mean to Dv50 assuming for a monodisperse microsphere sample, mean and Dv50 should theoretically be equivalent.*

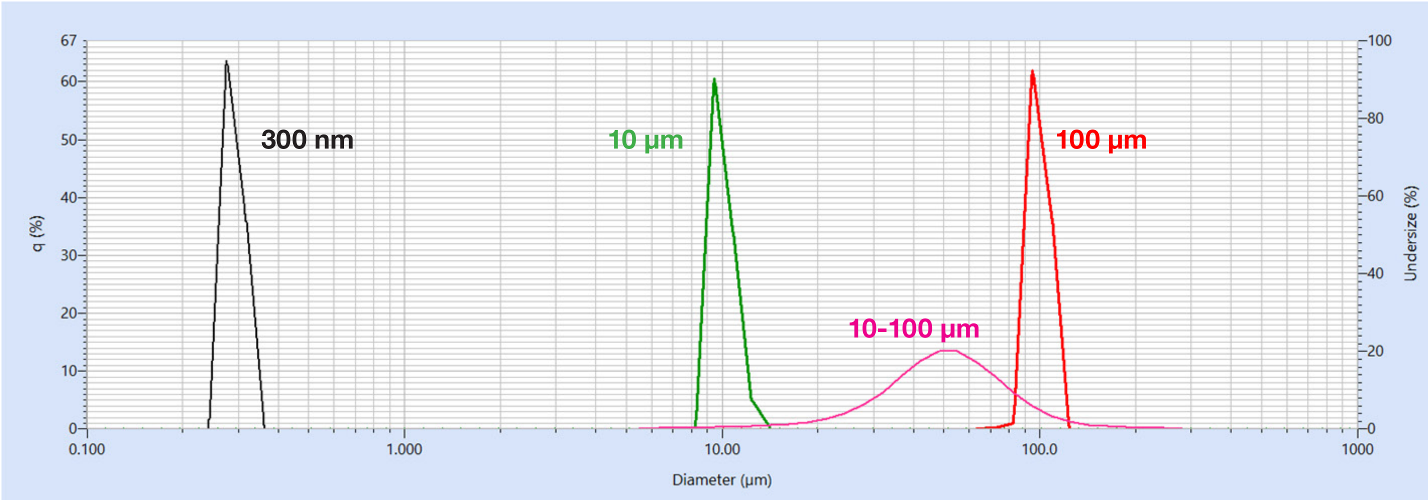

Figure 1. Measurements of 4 CRM demonstrate the exceptional accuracy of the LA-350.

		Dv10 (µm)	Dv50 (µm)	Dv90 (µm)
Test Sample 1 (100 µm NIST CRM, Thermo Scientific, Catalog Number: 4310A)	**Average**	90.33733	98.62867	112.03567
	CoV (%)	0.0036%	**0.0015%**	0.0005%
Maximum acceptable instrument uncertainty [1]		2.0%	1.5%	2.5%
Standard uncertainty of the certified value			0.76%	
Expanded tolerance limit defined by calculation			**5.0%**	
Test Sample 2 (10 µm NIST CRM, Thermo Scientific, Catalog Number: 4210A)	**Average**	9.01600	9.86471	11.33029
	CoV (%)	0.0479%	**0.2431%**	0.2690%
Maximum acceptable instrument uncertainty		2.0%	1.5%	2.5%
Standard uncertainty of the certified value			0.45%	
Expanded tolerance limit defined by calculation			**4.70%**	
Test Sample 3 (300 nm NIST CRM, Thermo Scientific, Catalog number: 3300A)	**Average**	0.26405	0.28853	0.32750
	CoV (%)	0.0667%	**0.1398%**	0.2269%
Maximum acceptable instrument uncertainty		2.0%	1.5%	2.5%
Standard uncertainty of the certified value			0.99%	
Expanded tolerance limit defined by calculation			**5.4%**	
Test Sample 4 (10-100 µm Whitehouse Standard, Part Number PS313)	**Average**	28.25933	50.85867	86.30650
	CoV (%)	**1.0114%**	**0.5235%**	**0.9915%**
Maximum acceptable instrument uncertainty		2.0%	1.5%	2.5%
Standard uncertainty of the certified value		7.16%	5.08%	7.28%
Expanded tolerance limit defined by calculation		**22.3%**	**15.9%**	**23.1%**

Table 1. Four CRM are measured in repeats using the LA-350. Individual result is then compared with the ISO calculated tolerance. Note the excellent instrument performance.

Instrument Repeatability – Sand

When testing real-world samples, ISO section 6.3.2 recommends that users perform at least six consecutive measurements to adequately evaluate instrument repeatability. Sand was chosen as a test sample to highlight the performance of the LA-350 because soil and sediment granules are highly heterogeneous, dense, and challenging, as it pushes the upper size limit of the analyzer. The sample was measured using the following conditions:

Refractive index: 1.53-0.001i
Circulation speed: 5
Data acquisition time: 5000
Transmission %: between 75-85%

The obtained percent CoV for Dv10, Dv50, and Dv90 using the LA-350 are 1.64%, 0.95%, and 0.93% respectively. Note that again, the instrument complies with ISO recommendations (2%, 1.5%, and 3% for Dv10, Dv50, and Dv90 respectively).

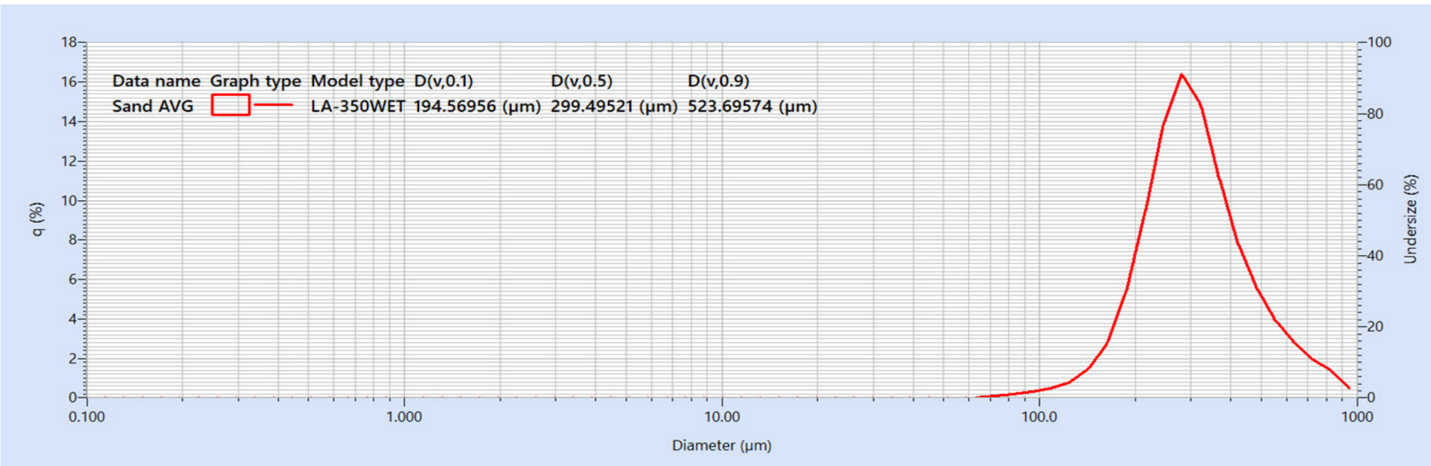

Figure 2. The average size distribution from 9 repeat sand measurements is calculated and presented in this graph.

File Name	Dv10 (µm)	Dv50 (µm)	Dv90 (µm)
SAND_1	194.674	299.783	522.861
SAND_2	196.798	301.035	527.463
SAND_3	197.937	301.198	526.333
SAND_4	196.508	301.962	527.201
SAND_5	196.298	301.006	525.919
SAND_6	197.961	303.12	529.916
SAND_7	189.487	296.604	519.212
SAND_8	191.199	295.317	516.461
SAND_9	191.473	295.932	517.391
Average	**194.704**	**299.551**	**523.640**
Std. Dev.	**3.186**	**2.858**	**4.873**
CoV (%)	**1.64%**	**0.95%**	**0.93%**
ISO 13320:2020 Section 6.3.2	**2%**	**1.50%**	**3 %**

Table 2. Repeat measurements of polydisperse sand sample using the LA-350.

Case Studies: LA-300 to LA-350 Correlation

After the LA-350 accuracy and repeatability are qualified as shown in the previous section, it is then appropriate to shift to method transfer and data correlation from the LA-300 to the LA-350.

The LA-300 and LA-350 are built fundamentally different. The LA-350 has more detectors, extended dynamic size range, improved circulation, and a more powerful 15-step centrifugal pump than its predecessor does. The LA-350 software is equipped with a data emulation function to mimic the scattering pattern of the LA-300.

However, in the following case studies where two different types of emulsions were analyzed, it is observed that the differences were insignificant. A comparable result can be readily obtained between the LA-300 and the LA-350 without any data manipulation.

To maintain a stable, undisrupted emulsion suspension, the lowest circulation speed was used in the LA-350. The LA-350 detail analytical test method can be found in Application Note 135, Homogenization of Milk [4].

White Emulsion (Non-Homogenized milk)

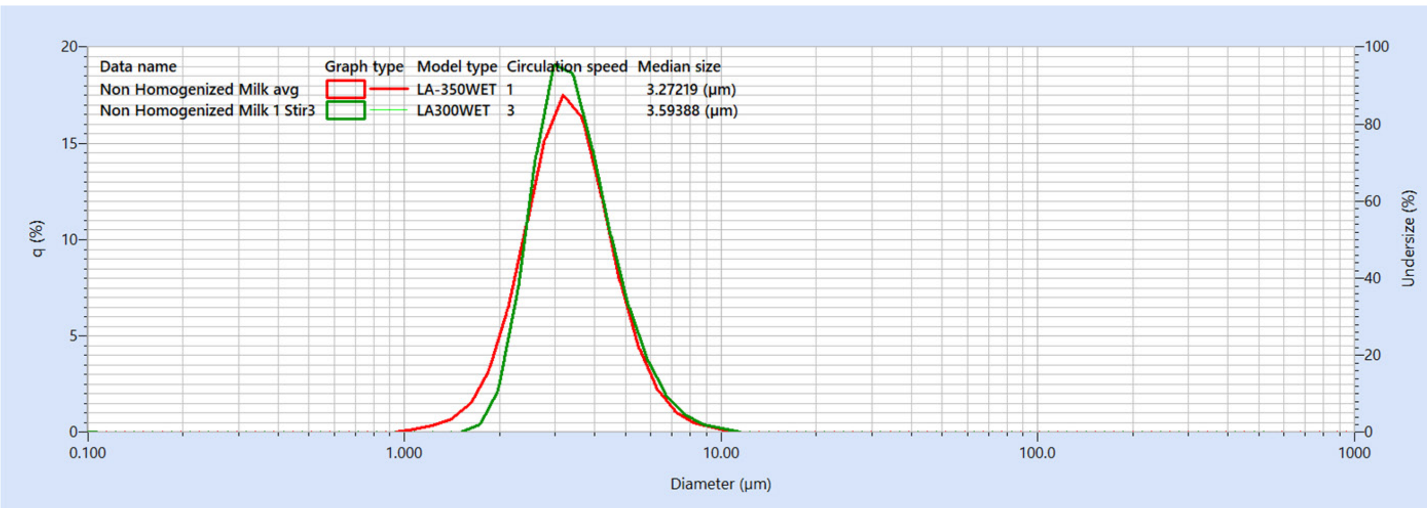

Figure 3. Overlay of two measurements using the LA-350 and LA-300.

	LA-300	LA-350	% Difference
Refractive Index	1.46-0.01i	1.46-0.01i	
Circulation Speed	3	1	
Median Diameter (µm)	3.59388	3.27219	8.95%
Convergence Factor	Standard	15	

Table 3. Measurement conditions selected for milk emulsion led to strong LA-300 and LA-350 agreement.

Blue Emulsion

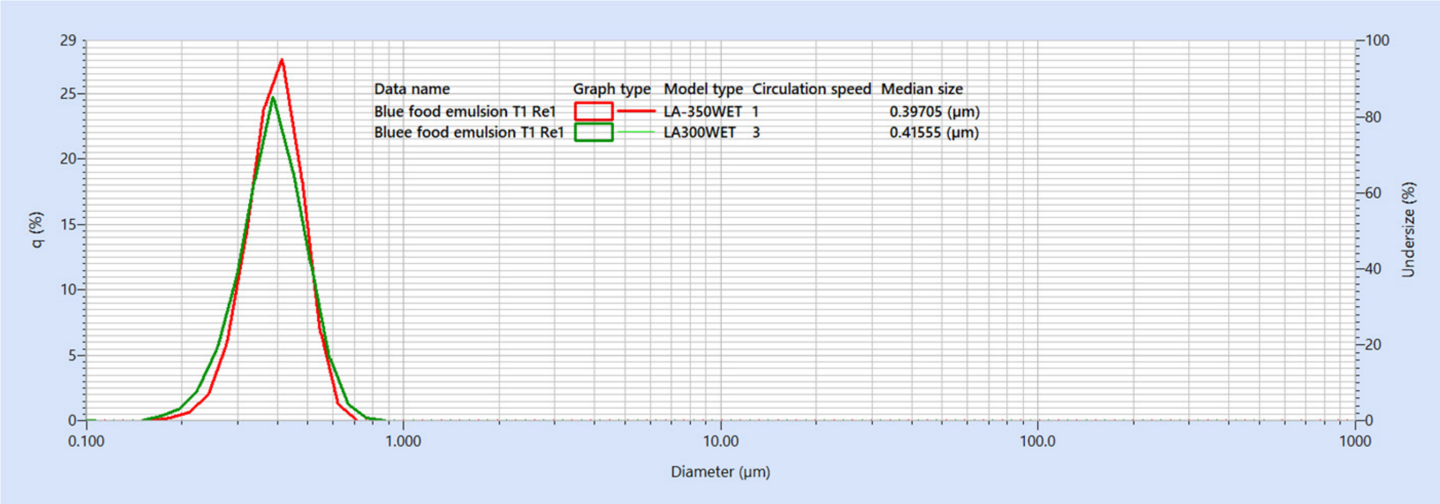

Figure 4. Overlay of two measurements using the LA-350 and LA-300.

	LA-300	LA-350	% Difference
Refractive Index	1.45-0.01i	1.45-0.01i	
Circulation Speed	3	1	
Median Diameter (µm)	0.41555	0.39705	4.45%
Convergence Factor	Standard	30	

Table 4. Measurement conditions selected for blue emulsion that led to good LA-300 and LA-350 agreement.

Conclusion

Similar to the LA-300, the LA-350 has proven to be a robust analyzer that offers high accuracy, good instrument repeatability, exceptional system-to-system variability [3], and easy method and data transfer. The data demonstrated here also infer similar outcomes for various other test samples.

References

1. ISO-13320:2020 Particle Characterization – Laser Diffraction Method

2. ISO 14488:2007 Particulate materials — Sampling and sample splitting for the determination of particulate properties

3. Tech Note 175 LA-350 Repeatability and Reproducibility https://static.horiba.com/fileadmin/Horiba/Products/Scientific/Particle_Characterization/Downloads/Technical_Notes/TN175_LA-350_Repeatability.pdf

4. Application Note 135 Homogenization of Milk https://www.horiba.com/int/scientific/applications/food-beverage/pages/milk-homogenization-evaluation-by-particle-analysis/

**The LA-300 Laser Diffraction Particle Size Analyzer has reached its obsolescence and end of support 26 years after its initial launch. To inquire for more info, email us at labinfo@horiba.com or visit: https://www.horiba.com/int/scientific/products/particle-characterization/particle-characterization-legacy-instruments/la-300-particle-size-analyzer-retired/*