HORIBA
Scientific

Technical Note

LA-950 Precision
TN172

LA-950 Repeatability & Reproducibility Studies

Repeatability, reproducibility, and instrument to instrument agreement are important performance characteristics of any analytical instrument. This technical note presents multiple data sets that prove the excellent performance analysts should expect from the HORIBA LA-950 laser diffraction analyzer.

Introduction

Two studies are presented in this technical note; one performed at multiple HORIBA Application Labs using polydisperse standards, and one performed by a customer analyzing their own sample. Both studies tested the repeatability, reproducibility and/or instrument to instrument variation of the LA-950.

Definitions

The word *precision* is often used as a catch-all to describe the results from any kind of repeated test. Understanding the different types of precision is important because some tests are more difficult (and meaningful) than others.

- *Repeatability* - Measurement variation with a single operator and single instrument on the same sample, over a short amount of time with all other variables held constant (i.e. location). Think of this as taking a sampling, loading it into the LA-950, and taking three consecutive measurements without draining.

- *Reproducibility* - Measurement variation with either multiple operators on multiple instruments with the same sample (but possibly multiple lots) in multiple locations. Not all of these conditions must be satisfied. This is a much more taxing test than repeatability and is the test performed for this study. When a manufacturer makes a claim about precision, make sure to know which type.

- *Intermediate Precision* - Measurement variation with multiple operators on either single or multiple instruments, in the same location across multiple days.

The table on the next page summarizes how these three tests differ. This information appears courtesy of ASTM and can be found in ASTM E177, Practice for Use of the Terms Precision and Bias in ASTM Test Methods (1), and E456, Terminology Relating to Quality and Statistics (2).

	Repeatability Condition	Intermediate Precision Condition	Reproducibility Condition
Laboratory	Same	Same	Different
Operator	Same	Different	Different
Apparatus	Same	Same*	Different
Time between Tests	Short**	Multiple Days	Not Specified

*This situation can be different instruments meeting the same design requirement.

** Standard test method dependent, typically does not exceed one day.

HORIBA Study

A reproducibility study was performed on 40 unique, randomly selected LA-950 systems; 20 for wet measurements, 20 for dry measurements. Two NIST-traceable polydisperse (range of sizes) glass bead reference samples were used in this study. The challenge samples were PS-202 (3-30 µm) and PS-215 (10-100 µm) from Whitehouse Scientific. The PS-202 sample was measured as an aqueous wet dispersion according to the method outlined in Analytical Test Method 102 (3). The PS-215 sample was measured as a dry powder using the PowderJet accessory according to the method outlined in Analytical test method 103 (4). The instrument settings used are shown below.

PS-202:

Circulation: 3;
Agitation: 2;
Liquid level: LOW;
Refractive index: STD-GLASS BEADS (1.51-0.00i);
Distribution base: VOLUME;
Form of distribution: Manual (15 iterations);
Data acquisition time LD=5000, LED=5000

*PS-215***:**

Refractive index STD-GLASSBEADS (1.51-0.0i);
Distribution Base VOLUME;
Form of distribution Manual (15 iterations);
Data sampling times: LD=50000;
T% for Sampling ; Max T%:= 99%, Min T%= 95%;
Air pressure; 0.3 MPa (3 bar)

Figures 1 and 2 and Tables 1 and 2 show the results for the PS-202 wet measurements and PS-215 dry measurements.

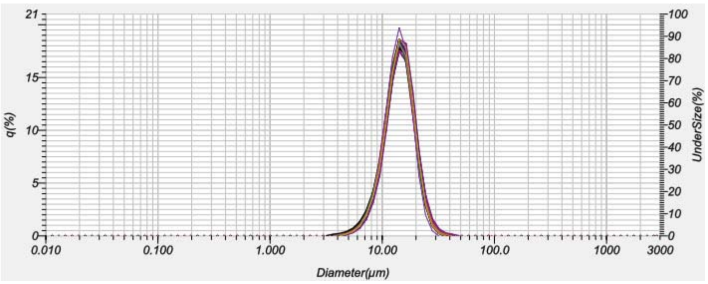

Figure 1: Overlay of 20 wet results from 20 systems.

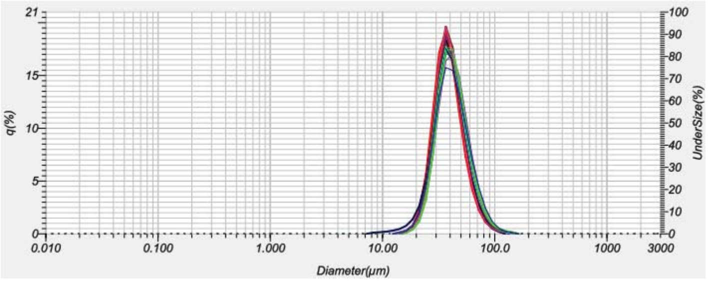

Figure 2: Overlay of 20 dry results from 20 systems.

PS-202 (μm)			
	D10	D50	D90
PS202 (5JW).NGB	9.291	14.066	20.312
PS202 (A6K).NGB	9.484	14.420	21.052
PS202 (D00).NGB	8.992	14.202	20.467
PS202 (E1W).NGB	9.712	14.610	20.925
PS202 (F00).NGB	9.327	14.373	21.348
PS202 (XD1).NGB	9.403	14.125	19.957
PS202 (H00).NGB	9.236	14.226	20.363
PS202 (HVY).NGB	9.417	14.271	20.429
PS202 (J31).NGB	9.199	13.976	20.164
PS202 (PWW).NGB	9.333	13.916	19.462
PS202 (R8X).NGB	9.366	14.241	20.712
PS202 (RP2).NGB	9.240	14.253	20.917
PS202 (S1N).NGB	9.426	14.360	20.431
PS202 (SBJ).NGB	9.717	14.545	20.704
PS202 (U12).NGB	9.086	13.875	20.164
PS202 (UB6).NGB	9.207	13.824	19.612
PS202 (USL).NGB	9.356	14.189	20.133
PS202 (VB1).NGB	9.103	13.844	19.768
PS202 (WFU).NGB	8.971	13.318	18.634
PS202 (WTF).NGB	9.580	14.382	20.541
Average	9.322	14.151	20.305
Std. Dev	0.21	0.30	0.62
CV (%)	2.21	2.11	3.05

Table 1: Results from 20 wet analyses on 20 systems.

PS-215 (μm)			
	D10	D50	D90
PS215 (2PN).NGB	27.002	38.445	60.784
PS215 (5JW).NGB	26.863	39.970	62.391
PS215 (5TT).NGB	26.986	38.913	59.778
PS215 (7P0).NGB	28.293	41.117	64.453
PS215 (A6D).NGB	27.912	39.891	60.961
PS215 (CX6).NGB	27.415	38.752	58.100
PS215 (E2W).NGB	25.324	38.490	58.739
PS215 (GB5).NGB	28.120	40.758	63.033
PS215 (M0F).NGB	27.337	40.909	64.624
PS215 (P7B).NGB	27.493	40.165	62.889
PS215 (P9G).NGB	27.326	39.725	61.375
PS215 (R8X).NGB	28.653	41.501	64.832
PS215 (RDC).NGB	28.474	41.411	64.420
PS215 (RP2).NGB	25.324	38.490	58.739
PS215 (SX6).NGB	27.147	38.466	57.976
PS215 (WFU).NGB	27.058	39.480	61.334
PS215 (WHK).NGB	26.967	39.014	60.804
PS215 (XHM).NGB	27.087	40.974	66.039
PS215 (EGX).NGB	27.898	41.059	63.956
PS215 (G00).NGB	28.434	41.771	65.232
Average	27.356	39.983	62,023
Std. Dev	0.90	1.14	2.53
CV (%)	3.28	2.84	4.08

Table 2: Results from 20 dry analyses on 20 systems.

Additional statistical information including graphs showing the 1 standard deviation errors bar are shown in Figures 3 and 4 and Tables 3 and 4.

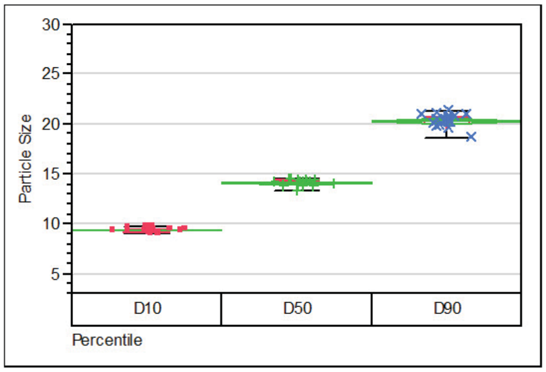

Figure 3: Statistical analysis of 20 wet results on 20 systems.

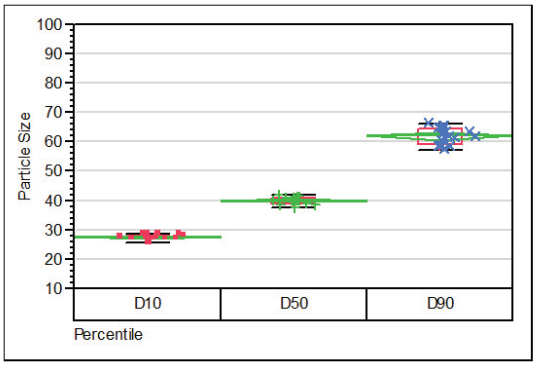

Figure 4: Statistical analysis of 20 dry results on 20 systems

Particle Size	Percentile [D10]	Percentile [D50]	Percentile [D90]
Mean	9.322	14.151	20.305
Std. Dev	0.206	0.296	0.620
COV	2.21%	2.11%	3.05%
Lower 95%	9.226	14.011	20.014
Upper 95%	9.419	14.291	20.595
Minimum	8.971	13.318	18.634
Maximum	9.717	14.610	21.348

Table 3: Statistical analysis of 20 wet results on 20 systems.

Particle Size	Percentile [D10]	Percentile [D50]	Percentile [D90]
Mean	27.328	39.915	61.843
Std. Dev	0.917	1.246	2.743
COV	3.36%	3.12%	4.44%
Lower 95%	26.898	39.332	60.560
Upper 95%	27.757	40.498	63.127
Minimum	25.324	38.445	57.194
Maximum	28.653	41.771	66.039

Table 4: Statistical analysis of 20 dry results on 20 systems

ISO 13320:2009 (5) section 6.4 states that the coefficient of variation (CV %) should be less than 3% at the D50 and less than 5% at the D10 and D90 when testing reproducibility. In the context of the ISO document this pass/fail criteria refers to testing a single instrument. This study was performed across 20 different instruments and still exceeded the ISO guidelines.

Customer Case Study

An existing LA-910 user studied LA-950 performance when considering upgrading to the newer model. To begin the user used two LA-950 systems and one sample (Formulation 1) to test repeatability. The results for these studies are shown in Figures 6 and 7 with size expressed in nm. The CV% values are extremely low, partly due to the nature of the sample which was small, narrow, and easily dispersed, but also because of the high performance level shown by the LA-950 systems.

Formula-tion 1	Dmean	D5	D10	D50	D90	D95
1	156	113	120	154	195	209
2	155	112	119	153	194	208
3	155	112	119	153	194	208
4	156	113	119	154	195	209
5	154	111	119	152	193	207
6	155	112	119	152	194	208
Average	155	112	119	153	194	208
Std. Dev.	0.8	0.8	0.5	1.0	0.8	0.7
CV%	0.5	0.7	0.4	0.6	0.4	0.4

Figure 6: Repeatability of sample Formulation 1 on LA-950 Unit 1

Formula-tion 1	Dmean	D5	D10	D50	D90	D95
1	154	112	119	152	192	208
2	154	112	119	152	192	208
3	155	113	119	152	192	208
4	155	115	119	152	193	208
5	154	112	119	152	193	107
6	155	112	119	153	193	208
Average	155	112	119	152	192	208
Std. Dev.	0.5	0.5	0.0	0.6	0.3	0.5
CV%	0.3	0.5	0.0	0.4	0.1	0.3

Figure 7: Repeatability of sample Formulation 1 on LA-950 Unit 2

Impressed with this performance, the user purchased four LA-950 systems and investigated the instrument to instrument variation using two samples, Formulation 1 and Formulation 2. The results from these studies are shown in Figures 8 and 9. The user was satisfied with these results.

Formula-tion 1	Dmean	D5	D10	D50	D90	D95
Average (nm)	155	112	119	152	193	208
Std. Dev. (nm)	0.8	0.8	0.7	1.0	1.1	0.7
CV (%)	0.5	0.7	0.6	0.6	0.6	0.3

Figure 8: Instrument to instrument variation across four LA-950 systems for Formulation 1.

Formula-tion 1	Dmean	D5	D10	D50	D90	D95
Average (nm)	193	136	147	187	247	264
Std. Dev (nm)	1.5	0.5	0.4	0.6	0.4	1.1
CV (%)	0.8	0.4	0.3	0.3	0.2	0.4

Figure 9: Instrument to instrument variation across four LA-950 systems for Formulation 2.

Conclusions

This studies mimic real-world conditions for many users who must reconcile results from multiple operators, units, and locations. This is particularly important for users with units across the world where the challenge of supporting across multiple time zones and languages grows quickly.

In this context the LA-950 Particle Size Analyzer data proves an excellent solution with superb data correlation for realistic (polydisperse) samples. In the HORIBA study this was proven across:

- 40 randomly selected units
- 2 locations
- 6 operators
- and acquired over 6 years (i.e. no drift)

This is accomplished without any unit-matching technique and at normal performance (i.e. no low sensitivity data processing). Such performance is unmatched on the market today.

In the customer study instrument to instrument agreement was proven across four systems measuring their own real world samples.

References

1. ASTM E177-10, Standard Practice for Use of the Terms Precision and Bias in ASTM Test Methods, available at www.astm.org.
2. ASTM E456-12, Standard Terminology Relating to Quality and Statistics, available at www.astm.org.
3. Analytical Test Method 102, Test Method for PS-202 Polydisperse Glass Bead Standards on Partica LA-950 available in the Download center at www.horiba.com/particle.
4. Analytical Test Method 103, Setup of Automatic Dry Measurement Partica LA-950 with PowderJet available in the Download center at www.horiba.com/particle.
5. ISO13320 Particle size analysis – Laser diffraction methods, available at www.iso.org.

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