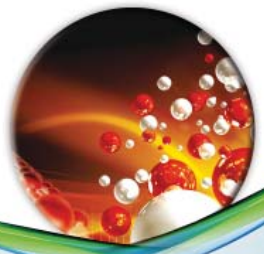
HORIBA Scientific

Technical Note



LA-960 Precision
TN174

LA-960 Repeatability & Reproducibility Studies

Repeatability, reproducibility, and instrument to instrument agreement are important performance characteristics of any analytical instrument. This technical note present multiple data sets that prove the excellent performance customers can expect from the HORIBA LA-960 particle size analyzer.

Introduction

The study presented in this technical note uses data gathered at the manufacturing and QC site in Kyoto, Japan. The purpose is to test the reproducibility and/or instrument-to-instrument variation of the LA-960.

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-960, and taking three consecutive measurements without draining.
- Reproducibility - Measurement variation with either multiple operators on multiple instruments on 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-960 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.

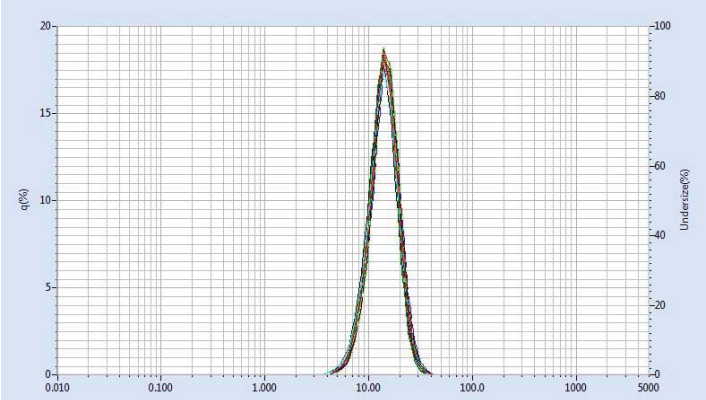


Figure 1: Overlay of 20 wet results

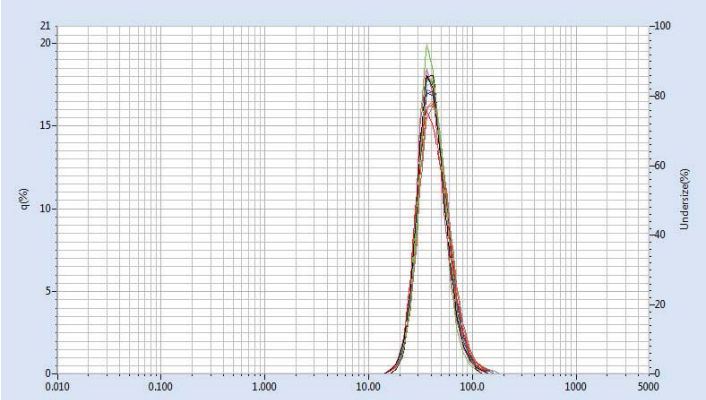


Figure 2: Overlay of 20 dry results from 20 systems

PS-202 (μm)			
	D10	**D50**	**D90**
PS202 (U2A).NGB	8.08	13.29	20.50
PS202 (W03).NGB	8.86	14.16	22.12
PS202 (CA2).NGB	8.39	13.75	21.39
PS202 (SY9).NGB	8.26	13.54	20.75
PS202 (U19).NGB	8.46	13.68	20.87
PS202 (DEE).NGB	9.09	13.82	21.83
PS202 (X4T).NGB	8.45	13.76	21.06
PS202 (V5T).NGB	8.77	14.08	21.30
PS202 (TVV).NGB	8.94	14.12	21.23
PS202 (RPR.NGB	8.91	14.14	21.48
PS202 (U9U).NGB	8.79	13.86	21.07
PS202 (XYN).NGB	8.98	14.07	21.48
PS202 (ABG).NGB	8.80	14.06	21.21
PS202 (67S).NGB	9.42	14.48	21.57
PS202 (YET).NGB	8.78	13.80	21.35
PS202 (SS7).NGB	9.04	14.41	21.34
PS202 (UDH).NGB	8.26	13.64	20.88
PS202 (WRT).NGB	9.05	14.15	21.48
PS202 (PLS).NGB	8.24	13.62	20.71
PS202 (NKU).NGB	8.72	13.24	19.25
Average	8.71	13.88	21.14
Std. Dev.	0.351	0.333	0.591
CV (%)	4.03	2.40	2.79

Table 1: Results from 20 wet analyses on 20 systems

PS215 (μm)			
	D10	D50	D90
PS215 (VGR).NGB	28.66	42.48	68.04
PS215 (XM3).NGB	27.20	40.34	65.68
PS215 (R8C).NGB	28.20	40.76	61.60
PS215 (U0A).NGB	28.66	41.53	65.52
PS215 (TGV).NGB	28.69	42.41	67.34
PS215 (TLB).NGB	27.98	40.70	62.85
PS215 (PGC).NGB	27.91	39.22	58.28
PS215 (W1X).NGB	27.15	39.12	60.51
PS215 (VRF).NGB	28.58	41.11	63.27
PS215 (PSA).NGB	28.79	41.80	65.29
PS215 (TBA).NGB	28.24	41.24	64.75
PS215 (RJC).NGB	28.51	41.15	63.72
PS215 (SV3).NGB	27.56	40.03	61.80
PS15 (AB6).NGB	26.45	37.99	57.87
PS215 (G07).NGB	27.81	40.80	6.296
POS215 (XES).NGB	28.23	40.82	63.64
PS215 (X1G).NGB	28.71	41.63	64.22
PS215 (CKS).NGB	27.61	40.21	61.45
PS215 (T9X).NGB	27.49	40.67	62.82
PS251 (Y4B).NGB	28.81	43.09	69.22
Average	28.06	40.85	63.54
Std. Dev.	0.658	1.21	2.92
CV (%)	2.35	2.95	4.60

Table 2: Results from 20 dry analyses on 20 systems

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

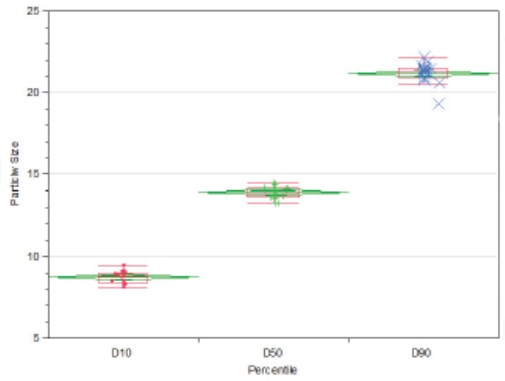


Figure 3: Statistical analysis of 20 wet results on 20 systems

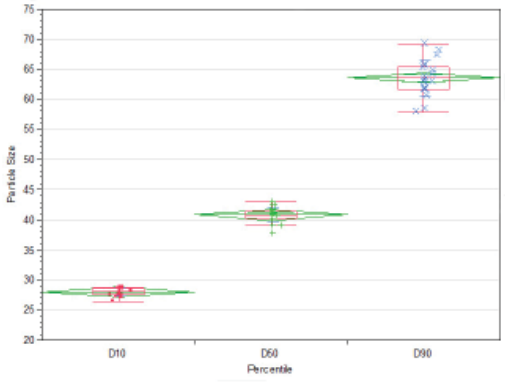


Figure 4: Statistical analysis of 20 dry results on 20 systems

Particle Size	Percentiel (D10)	Percentile (D50)	Percentile (D90)
Mean	8.71	13.88	21.14
Std. Dev.	0.351	0.333	0.591
COV	4.03	2.40	2.79
Lower 95%	8.52	16.69	20.05
Upper 95%	8.91	14.08	21.34
Minimum	8.08	13.24	19.25
Maximum	9.42	14.48	22.12

Table 3: Statistical anaylsis of 20 wet results on 20 systems

Particle Size	Percentiel (D10)	Percentile (D50)	Percentile (D90)
Mean	28.06	40.85	63.54
Std. Dev.	0.658	1.21	2.92
COV	2.35	2.95	4.60
Lower 95%	27.23	40.02	6.271
Upper 95%	28.90	41.69	64.38
Minimum	26.45	37.99	57.87
Maximum	28.81	43.09	69.22

Table 4: Statistical anaylsis 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.

Conclusions

Laser diffraction is an inherently repeatable technique, but the LA-960 system shows extremely impressive results when tested for repeatability, reproducibility, and instrument to instrument variation. The results reported will be somewhat sample dependent as seen in these studies where the samples with the broader distributions are not as repeatable as samples with a more narrow distribution. But potential customers should feel comfortable that the LA-960 provides industry leading performance both on the samples shown in this report and most likely on their own samples as well.

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-960 available in the Download center at www.horiba.com/particle
4. Analytical Test Method 103, Setup of Automatic Dry Measurement Partica LA-960 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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