

The HORIBA LA-960

Simple, Powerful, Reliable Particle Size Measurement



LA-960 Laser Particle Size Analyzer



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What we'll talk about



Ease of use
 Software, automation, speed
 Performance

Sample handlers

Reliability & support

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LA-960: Laser Diffraction



- Simple, powerful, reliable particle size
- Tenth generation design
- Lowest total cost of ownership
- Ultra durable
- Suspension, emulsion, powder, paste, gel
- 10 nanometer 5 mm



Watch the 3 minute overview video

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History of the LA-Series





Software ease of use



- Real-time particle size distribution tells you when to make the measurement
- All controls on one screen



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Software ease of use



Quickly review measurement results
 All information is on one screen



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LA-960 Method Expert



- Unique guided method development
- Easily choose measurement and calculation conditions
- Choose the best refractive index
- Create "one button" Sequence SOPs





LA-960 Method Expert





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Method Expert guides user to prepare the LA-960 for each test

Results displayed in multiple formats: PSD, D50, R-parameter



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LA-960 Method Expert



 Built-in *Expert Advice* guides your decisions
 Quickly create Sequence files which fully automate the LA-960

	+
1.5 Typical lower limit for most organic white powders (Default Value)	
pical real values used by the HORIBA Particle Experts include:	
suit will meet your material specification (if required).	
nally, examine the D90,D50,D10 graph to check if the size	
e c Palameter is orten a renable and justinable choice.	
the <i>R Parameter</i> graph. The imaginary value which minimizes	
nce you have concluded that the PSD is reasonable, then you should look	
raph to see how the size distribution changes with imaginary component value.	
o matter what other factors may weigh in the favor of a certain value re particle size distribution must be realistic. Look at the Distributions	
e common sense: Does my distribution make sense?	
e first factor to consider when changing refractive index should always	
a "best statistical guess" tool to evaluate reasonable values.	
however, you cannot or choose not to determine the real RI this test can be used	
arying degrees of suitability and accuracy (i.e. Becke line test, Abbe refractometer). he HORIBA Particle Experts will be glad to recommend the services of professional sting laboratories.	
e measured. Several techniques exist that can provide a quantitative value with	=
st. let's be clear that the real part of the refractive index is a value that can often	-
pert Advice for Real Refractive Index Test Results	-
xpert advice	1.5



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Automated review

Verification Setti	ng	×
Parameter	Median Size 💌	[
Specification	ISO 13320-1 💌	[
Standard Value	50	(μm)
Tolerance ±	5 (µr	n)
Certified range of value		
D(v,0.5) >= 10μm	± 0	%
D(v,0.5) < 10µm	± 0	%
Result Display Setting		
Lolor:		
Text:	JUK	
Fail:		
Color:		
l ext:	ING	
01	Cancel	

Distribution Graph Data Table	Result Data		
Mean Size Variance Median Size Std.Dev. Chi Square R Parameter Diameter on Cumulativ Cumulative % on Diam	 : 0.18408(μm) : 1.8988E-3(μm²) : 0.17730(μm) : 0.1649(μm) : 0.0436(μm) : 4.162519 : 3.7379E-1 /e % : (2)10.00 (%)- 0.1345(μm) : (9)90.00 (%)- 0.2450(μm) : (9)90.00 (%)- 100.000(%) : (2)600.0 (μm)- 100.000(%) : (3)425.0 (μm)- 100.000(%) : (4)300.0 (μm)- 100.000(%) : (5)212.0 (μm)- 100.000(%) : (6)150.0 (μm)- 100.000(%) : (7)106.0 (μm)- 100.000(%) : (8)75.00 (μm)- 100.000(%) : (10)38.00 (μm)- 100.000(%) 		
Verification	: 1.0K 4.3% [D(v,0.5) 0.170 : 2.0K 3.5% [D(v,0.1) 0.130 : 3.0K 6.5% [D(v,0.9) 0.230	(μm)(± 6.000%)] (μm)(± 10.00%)] (μm)(± 10.00%)]	
Data Name	Graph Type Transmittance(R)	Median Size	R Paramete
andy1'	88(3(%)	0.17730(µm)	0.373795
200801181026014	81.1(%)	9.35329(µm)	0.069234
andy1	88.3(%)	0.17730(µm)	0.373795

Automated review



Export Summary	Print Sum	mary B	Best Fit Columns			
	D/ OIL					
Sample name	D(V,U.1)	D(V,0.5)	D(V,0.9)			
19A	34.448	102.355	456.759			
19A	32.105	96.580	544.156			
19A	30.371	86.317	382.367			
Average	32.308	95.084	461.094			
Std. Dev.	2.046	8.123	80.982			
CV (%)	6.333	8.543	17.563			
Custom (5.0, 3.0, 5.0)	FAILED	FAILED	FAILED			

After a quick sampling adjustment, the user makes high precision measurements which easily pass

- Calculates COV and compares to ISO, USP, or custom standard
- Simple PASS/FAIL indicator notifies the user to check sampling and measurement conditions

Summary Report					
Export Summary	Print Sum	mary B	est Fit Columns		
Sample name	D(v.0.1)	D(v.0.5)	D(v.0.9)		
19A	18.315	61.758	337.027		
19A	17.995	61.016	335.442		
19A	17.946	61.326	364.517		
Average	18.085	61.367	345.662		
Std. Dev.	0.200	0.373	16.348		
CV (%)	1.108	0.607	4.730		
Custom (5.0, 3.0, 5.0)	PASSED	PASSED	PASSED		

What we'll talk about



Ease of use

Performance

• Sensitivity, Accuracy, Precision, Resolution

Sample handlers

Reliability & support

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Dynamic range 0.01 – 5,000 μm

Widest wet <u>and</u> dry measurement rangeOnly system to measure 30 nm!



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Nanoparticle sensitivity



- Shorter wavelength = better pattern
- 12 Wide angle detectors (4 backscatter)







30, 40, 50, 70 nm latex standards overlay of individual runs

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Sensitivity: Nanoparticles



- Some concerns with particles < 100 nm in personal care products
- LA-960 superb at quantifying sub-100 nm
- Software can display % finer than any size
- Data shown is for skin cream containing TiO₂

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Sensitivity: Sample amount



Colloidal Silica (weak scatterer) Median (D50): **35 nm** Sample Amount: **132 mg**





Magnesium Stearate Median (D50): **9.33 µm** Sample Amount: **0.165 mg**

Bio-degradable Polymer Median (D50): **114 µm** Sample Amount: **1.29 mg**

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Large particle detection

LA-960 Measurement Result



- Need exceptionally stable optical bench
- Vertical design means no density limit for dry



CAMSIZER Measurement Result D(50)=3943.0um





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Concentration sensitivity





Concentration sensitivity





Concentration	Stable results for sample loading	Varying results based on
Concentration	between 70 -90% Transmission.	sample concentration.

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State of the art optical design

- 87 silicon photodiode detectors covering 0.010 – 166 degrees
- Entire optic system enclosed within LA-960, prevents dust contamination
- Optic system mounted on cast aluminum supports including 1" thick base → low maintenance, better large particle detection
- Ultra fast alignment in 1-10 seconds

Superior optical design





Decades of design improvements and a drive to be the best

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Tilted cell reduces stray light



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Enclosed optical system





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No blind spots





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Accuracy & precision specifications

Accuracy – Guaranteed!

+/- 0.6% on NIST-traceable polystyrene latex calibration standards
·3% on d50 (median) for broad-distribution glass bead standards
·5% on d10 and d90 for broad-distribution glass bead standards
Meets or exceeds all requirements of ISO 13320 and USP 429

Precision – 0.1%

The combination of a rigid optical bench, stable, high-intensity light sources, optimized detectors, and highly-refined electronics virtually eliminates variability in the background noise and fluctuations in the response of the instrument. The *Partica* LA-950 has a guaranteed precision of 0.1% on polystyrene latex calibration standards

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Accuracy

Comparison to referee technique

- Microscope (image analysis) is referee technique for particle characterization
- Challenged with particle size standards
 - Monodisperse latex spheres
 - Verifies optics
 - May pass even if problems with sampler
 - Polydisperse glass spheres
 - -Verifies complete system
 - Should find problems with samplers









Accuracy through the size range



PSL results at 0.03, 0.06, 0.1, 0.2, 0.5, 1.0, 10, 100, and 1,000 microns

Quality acceptance test for every LA-960



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Accuracy Test: PS202



PS202 (3-30μm)	D10	D50	D90
Standard Value (µm)	9.14	13.43	20.34
Uncertainty (µm)	0.86	0.86	1.44
ISO standard error	5%	3%	5%
Lower limit (µm)	7.866	12.193	17.955
Measured Result (µm)	9.721	13.916	18.959
Upper Limit (µm)	10.500	14.719	22.869



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Accuracy Test: PS 225



PS225 (50-350µm)	D10	D50	D90
Standard Value (µm)	93.7	150.5	238.8
Uncertainty (µm)	3.54	2.52	6.02
ISO standard error	5%	3%	5%
Lower limit (µm)	85.652	143.541	221.141
Measured Result (µm)	94.217	153.815	252.542
Upper Limit (µm)	102.102	157.611	257.061



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Accuracy Test: PS 181



PS181 (0.1-1μm)	D10	D50	D90
Standard Value (µm)	0.36	0.65	1.11
Uncertainty (µm)	0.06	0.06	0.13
ISO standard error	5%	3%	5%
Lower limit (µm)	0.285	0.5723	0.931
Measured Result (µm)	0.434	0.709	1.296
Upper Limit (µm)	0.441	0.7313	1.302



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Mix of 50/50 PS202 & 225





Excellent baseline resolution: 48/52 calculated proportions

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Mixed standards





100.0

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1000

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0.100

1.000

10.00

Diameter(µm)

Accuracy: Error calculations



$$\chi^{2} = \sum \left\{ \frac{1}{\sigma_{i}^{2}} [y_{i} - y(x_{i})]^{2} \right\} \qquad R = \frac{1}{N} \sum_{i=1}^{N} \left\{ \frac{1}{y_{(x_{i})}} |y_{i} - y(x_{i})| \right\}$$

yi The measured scattered light at each channel (i) of the detector.

- y (xi) The calculated scattered light at each channel (i) of the detector based on the chosen refractive index kernel and reported particle size distribution.
- σi The standard deviation of the scattered light intensity at each channel (i) of the detector. A larger σi indicates lower reliability of the signal on a given detector.
- N The number of detectors used for the calculation



- Prepare sample, measure, drain, repeat
- What would be good reproducibility?
- Test COV according to ISO13320
 - •CV < 3% at D₅₀
 - CV < 5% at D₁₀ & D₉₀
 - Double values if D_{50} <10 μ m
- Test COV according to USP<429>
 - ●CV < 10% at D₅₀
 - CV < 15% at D_{10} & D_{90}
 - Double values if D_{50} <10 μ m

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Class leading precision



- 3-30 µm and 10-100 µm NIST-traceable glass beads
- 40 randomly selected units
- Manufactured over 2 years
- 6 different operators
- 2 different locations

	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.

Repeatability & reproducibility

20 wet results on the LA-960 flow system



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		

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Repeatability & reproducibility

20 dry results on the LA-960 PowderJet



D10

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	

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D90

D50

Percentile

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Reproducibility: Customer sample

Microcrystalline cellulose (MCC)



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Why reproducibility matters



Better reproducibility = Wider internal margins

What is resolution?

- Ability to measure small differences in particle size
- Small differences between successive samples (different production lots) are most important
- Detection limit of small amount of material outside of main size distribution
- Best defined by user's real-world requirements



PLA nanoparticles for drug delivery





Resolution: Outliers





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- •Black size distribution is internal standard
- •Red result is considered a passing batch
- •Blue result is a bad batch
- •Highlights problem in production
- •All accomplished with complex product formulation

What we'll talk about



Ease of use

Performance

Sample handlers

Powders, precious samples, throughput, pastes
 Reliability & support



Superior dry powder feeder



Direct flow of powder down to cell rather than turn 90°, then around plastic tube, no density restriction like horizontal units, zero impact surfaces means good dispersion w/o comminution

Superior dry powder feeder

Automatic control of sample feed rate

- LA-960 monitors amount of sample supplied by the vibratory feeder. Automatic feed back control keeps constant mass flow rate of powder during measurement
- This is CRITICAL
 - More reproducible, robust
 - No ghost peaks
 - No cutting off results
 - Fewer headaches!
- Unique to HORIBA

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Wet vs. dry results





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1 000

10.00

Diameter(um)

0.010

Solution for every application HORIBA



- 5 mL < 2 mL 35 mL 100 mL variable
- Widest range of wet sample handlers
- Excellent S/N minimizes sample quantity
- Unique FractionCell, PasteCell, MiniFlow, and Slurry Sampler
- Temperature Control available

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What we'll talk about



Ease of use

Performance

Sample handlers

Reliability & support

• Applications Labs, build quality, TCO

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Our expertise = Your solution

- HORIBA's experienced staff of technical and applications specialists support the LA-960 in 54 offices across 45 countries
- We are committed to the satisfaction of our users and to the education of the greater industry and provide many channels of support including:
 - Sample analysis via the many Applications Lab around the world
 - Free software updates, webinars, technical notes, and much more in the Download Center
 - Instant support via phone, e-mail, and online meeting
 - On-site and in-house user training courses
 - Service contracts, verifications, and validations to fit every requirement
 - Advanced software tools to correlate data from other particle size analyzers to maintain historic specifications



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Long lifetime parts



- 5mW 650nm laser diode (LD)
 - LD lasts much longer than gas lasers
 - LD put in low-power mode between measurements to slow aging
 - Gas lasers have no low-power option
 - Typical gas laser lasts 3-5 years
 - LD in LA-300, LA-950 lasts 11+ years
- Zero LD failures since LA-300 launched (12+ years)

Long lifetime parts



3mW 405nm blue LED

LED has effectively zero warm-up, can be turned on right before measurement, means <u>very</u> slow aging

Expected lifetime 10+ years

- Zero LED failures since LA-950 launched (9 years)
- Low wavelength is key to acquiring useful scattering pattern

Wavelength more critical than power

Ease of service



HORIBA LA-950S2

- Average repair cost, parts and labor, over first three years is 0.1% of initial purchase price
- Measurement cells can be replaced without needing calibration
- HORIBA offers either on-site service or return to Irvine, Chicago, Houston
- Expect the same with the LA-960



Gas lasers **fade** as they age

- Lower output \rightarrow lower scattering
- •Lower scattering \rightarrow changing size results
- Laser replacements are **expensive**



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Build quality





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Build quality





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Summary



- The LA-960 simplifies the challenging science of particle size analysis
- Refined software makes it easy to make and review actionable measurements
- Decades of experience have created the world's most advanced optical system
 - Highly accurate
 - Excellent reproducibility
 - Unmatched resolution
- Widest range of sample handlers to solve every application
- HORIBA's global support and build quality for the LA-960 mean users make better measurements with less effort





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- Receive news of updates
- View application & technical notes (170+), webinars (60+), white papers.