



AUTOMATED VERIFICATION FUNCTIONS IN THE LA-950 SOFTWARE

Every laser diffraction system should be verified on a regular basis. Customers using the LA-950 laser diffraction analyzer can easily and automatically verify performance following the accepted standards ISO13320 and USP<429>. This document provides a description of the automated verification features and how to use them.

Introduction

There are two internationally accepted standards that describe a procedure for verifying the performance of laser diffraction particle size analyzers; ISO 13320 (1) and USP<429> (2) or the equivalent EP 2.9.31 (3). The USP/EP procedure is based on the previously published ISO document with slightly different pass/fail criteria. Both procedures explain an approach to verify that a laser diffraction particle size analyzer is working properly as defined by passing both accuracy and reproducibility tests. The LA-950 software has automated these verification tests so that any user can easily perform the procedures and document that the system is operating within accepted guidelines.

Samples Used

Both the ISO and USP tests require that the operator use polydisperse standards for the verification tests. This implies that monodisperse latex standards are not acceptable when following these procedures. The polydisperse standards recommended by HORIBA for LA-950 verification testing come from Whitehouse Scientific (4). For wet analysis we recommend using Whitehouse PS202 (0.1g/bottle) 3-30 μm standard. For dry analysis we recommend using the Whitehouse PS215 (1 g/bottle) 10-100 μm standard.

Procedure

Both the ISO and USP tests require making three separate measurements (measure, clean, measure - three times) and recording the d10, d50, and d90 values. The mean of the three results for the d10, d50, and d90 (5) must be within a specified range of the certified values in order to pass the accuracy test. The reproducibility test requires calculating the coefficient of variation (COV) values for the d10, d50, and d90 for the three measurements. The COV is defined as

$$\text{COV} = (\text{standard deviation}/\text{mean}) * 100$$

The COV values must be below a specified percentage in order to pass the reproducibility test.

The pass/fail criteria for the ISO and USP tests are shown below.

Accuracy

- d50 within 3% of the certified range of values
- d10 & d90 within 5% of the certified range of values

Reproducibility

- COV d50 < 3%
- COV d10 & d90 < 5%



System Configuration

The LA-950 must be optimized for analyzing the spherical glass standards in order to generate the most accurate results. The software settings required for configuring the system for the verification tests are described below. To select the proper refractive index click on Conditions, Set Conditions for Next Measurement, Refractive Index, Select. Choose "STD – GLASSBEADS" (1.51-0.00i). Selecting this refractive index also sets the iteration number for the algorithm to the recommended number of 15. For more information on how to correctly set conditions for wet or dry glass bead measurement refer to ATM102 (6) and ATM103 (7).

Automation

The LA-950 software automates the measurement calculations for the ISO and USP standards and also allows for the creation of a user defined specification. The dialog box for entering the information required for the test is shown in Figure 1 below. In order to reach this dialog box click on:

Conditions, Set Conditions for Next Measurement, Advanced, Calculation, Verification – Setting

Parameter	D(v,0.1)
Specification	USP-429
Standard Value	25.37 (µm)
Tolerance	± 2.08 (µm)
Certified range of values	
D(v,0.5) >= 10µm	± 5 %
D(v,0.5) < 10µm	± 10 %
Result Display Setting	
Pass:	
Color:	Green
Text:	OK
Fail:	
Color:	Red
Text:	NG

Figure 1: LA-950 software settings

For "Parameter" select either the D(v,0.1), D(v,0.5), or D(v,0.9).

For "Specification" select either ISO 13320, USP-429, or User Defined.

For "Standard Value" enter the Final Mean Size value from the Certificate of Analysis sheet for the standard being used. This must be repeated for the D(v,0.1), D(v,0.5), or D(v,0.9).

For "Tolerance" enter the Uncertainty value from the Certificate of Analysis sheet for the standard being used. This must be repeated for the D(v,0.1), D(v,0.5), or D(v,0.9).

The values shown in Figure 1 come from the Certificate of Analysis for the d10 of Whitehouse standard PS215.

It is convenient at this point to save the current Conditions (with Verification settings) to make the setup of future measurements much faster.

Measurement

After the Verification Setting screen is completed it is time to make the three measurements. These can be made either manually from the Measurement View screen, or with automation using either a Navigation sequence or through the Method Expert software wizard (8).

Result View

The pass/fail results for accuracy can be viewed after each measurement from the Result Data View screen as shown below in Figure 2.



Distribution Graph	Data Table	Result Data
Verification : 1.0K -5.5% [D(v,0.9) 109.0 (µm)(± 5.000%)]		
: 2.0K -6.2% [D(v,0.5) 32.00 (µm)(± 3.000%)]		
: 3.0K -5.8% [D(v,0.1) 10.00 (µm)(± 5.000%)]		
D(v,0.1)	: 9.41689(µm)	
D(v,0.5)	: 30.02489(µm)	
D(v,0.9)	: 103.04584(µm)	

Figure 2: Accuracy verification result

The reproducibility and accuracy results can both be viewed using the File List Utility function and Summary Report. In order to configure the software to see these results click on:

File List Utility, Open List View, delete unwanted results and only display results used for this calculation, Summary, Edit Layout, and choose the desired standard in the Validation – Specification box as shown in Figure 3.

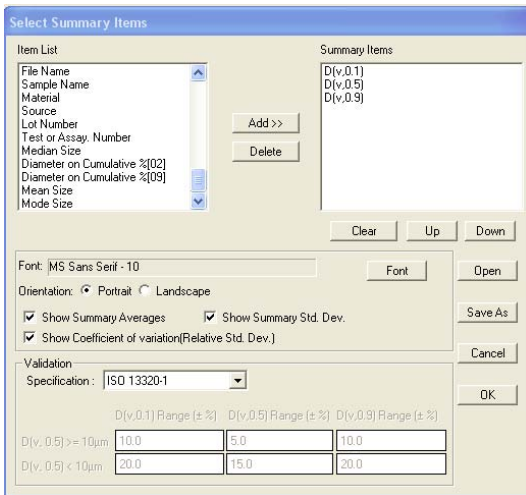


Figure 3: Configuring for accuracy and COV result view

When this has been configured properly the accuracy and reproducibility results will be shown as seen in Figure 4.

Summary Report					
Export Summary	Print Summary	Edit Layout	Best Fit Columns	Hide Selected	Exit
File Name	Sample	D(v,0.1)	D(v,0.5)	D(v,0.9)	
201112131416011.NGB	quartz	9.921	32.497	103.290	
201112131417012.NGB	quartz	9.740	31.490	101.827	
201112131418013.NGB	quartz	9.617	30.829	100.721	
201112131418014.NGB	quartz	9.616	31.008	101.159	
201112131420017.NGB	quartz	9.417	30.025	103.046	
Average		9.662	31.170	102.009	
Std. Dev.		0.185	0.911	1.133	
CV (%)		1.918	2.921	1.110	
ISO 13320-1 (10.0, 5.0, 10.0)		PASSED	PASSED	PASSED	

Figure 4: Accuracy and reproducibility results

This can then be printed and kept in the instrument log book as proof of verification following the selected standard procedure, in this case ISO 13320.

Comments

An in-house quartz reference material was used for the measurements shown in this study. This may actually be the approach followed by many LA-950 users. Many customers prefer to have a HORIBA trained service person perform the periodic test using a certified standard, and then perform the verification test described in this document on a more regular basis to assure to change has occurred with the system.



References

1. ISO 13320-1 Particle size analysis – Laser diffraction method available at either

<http://www.iso.org/iso/store.htm> or
<http://webstore.ansi.org/>

2. USP<429> Light Diffraction Measurement of Particle Size

<http://www.usp.org>

3. EP EP 2.9.31 Laser Diffraction Measurement of Particle Size

<http://www.edqm.eu/en/Homepage-628.html#>

4. For information on standards see
<http://www.whitehousescientific.com/>

5. See TN156 Understanding and Interpreting Particle Size Distribution Calculations technical note from the Download Center from the website
<http://www.horiba.com/scientific/products/particle-characterization/download-center/>

6. See ATM102 PS202 Glass Bead Standard analytical test method from the Download Center from the website
<http://www.horiba.com/scientific/products/particle-characterization/download-center/>

7. See ATM103 Dry Glass Bead Standard analytical test method from the Download Center from the website
<http://www.horiba.com/scientific/products/particle-characterization/download-center/>

8. See WP001 Introducing the Method Expert white paper from the Download Center from the website
<http://www.horiba.com/scientific/products/particle-characterization/download-center/>

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