

Every laser diffraction system should be verified on a regular basis. Customers using the LA-960V2 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-960V2 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-960V2 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: $COV = (\text{standard deviation}/\text{mean}) * 100$

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-960V2 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-960V2 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

Figure 1: LA-960V2 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.

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.

File Name	Sample name	Material	Source	Lot number	Test or assay	Median Size	10.000%	90.000%	Mean Size	Mode
201809261109003	NGE					2521.316	2330.895	2902.131	2578.591	2514.245
201809261110004	NGE					2525.537	2332.690	2911.329	2502.734	2516.691
UNSAVED-201809261						2516.529	2328.576	2898.877	2571.049	2506.929
UNSAVED-201809261						2510.039	2323.935	2888.198	2560.724	2502.779
UNSAVED-201809261						2510.219	2326.780	2890.541	2561.975	2504.354

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-960V2 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 the change has occurred with the system.

References

1. ISO 13320, Particle size analysis – Laser diffraction methods, available at: <https://www.iso.org/store.html> or <https://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 from <https://www.edqm.eu/en>
4. For information on standards see: <https://www.whitehousescientific.com/>
5. TN156 Understanding and Interpreting Particle Size Distribution Calculations technical note, from the Download Center from: <https://www.horiba.com/particle>
6. ATM102 PS202 Glass Bead Standard analytical test method, from the Download Center from: <https://www.horiba.com/particle>
7. ATM103 Dry Glass Bead Standard analytical test method, from the Download Center from: <https://www.horiba.com/particle>
8. WP001 Introducing the Method Expert white paper, from the Download Center from: <http://www.horiba.com/particle>