

VERIFYING LASER DIFFRACTION SYSTEMS

Every laser diffraction system should be verified on a regular basis. The frequency of verification depends on usage and risk assessment but most industries agree that once a year is the minimum requirement. Many laboratories struggle with choosing which standard to use to test their systems. This document will discuss the current recommendations on options for verifying LA-series laser diffraction systems.

Monodisperse vs. Polydisperse

Monodisperse standards are typically spherical particles with an extremely narrow size distribution made of polystyrene latex. They are available through several sources including Duke Scientific (now owned by Thermo Fisher) - the manufacturer for the standards used for LA-series Installation Qualification/Operation Qualification (IQ/OQ) procedures. Monodisperse standards are available in many size ranges from 21 nm to 1000 μm .

Polydisperse standards are typically spherical glass beads with a known distribution "across one decade of size range." Depending on definition and interpretation, commercially available polydisperse standards do not actually span an entire decade of size range. The certificate of analysis for NIST 1003c lists a D5 of 18.9 μm and D95 of 43.3 μm . The size ranges of standards available from Whitehouse (the source used by HORIBA for IQ/OQ procedures) are listed below:

Nominal Size	D10	D90
0.1 - 1.0 μm	0.36	1.11
1 - 10 μm	2.89	6.21
3 -30 μm^*	9.14	20.34
10 - 100 μm^*	25.37	62.93
50 - 350 μm	67.6	274
150 - 650 μm	244	527
500 - 2000 μm	691	1534

* Samples used in HORIBA OQ procedures

Both ISO13320¹ and USP<429>² suggest the use of polydisperse standards to verify the performance of laser diffraction analyzers. Although neither standard recommends using monodisperse standard materials, these are still used by HORIBA and other laser diffraction

suppliers for IQ/OQ procedures, quick verification, and to test optical benches. Monodisperse latex standard materials provide excellent NIST traceability and adequately test system performance when used properly. HORIBA IQ/OQ procedures use a mixture of monodisperse and polydisperse standards, thus providing complete system verification.

Choosing Size Ranges

The LA-960 IQ/OQ procedure* uses the following (nominal) sizes of monodisperse standards; 0.1, 1.0, and 100 μm , along with the Whitehouse 3 – 30 μm polydisperse standard for wet samplers and the 10 – 100 μm polydisperse standard for dry samplers. This range of sizes stimulates all of the system detectors and is satisfactory to prove that the system is working properly. No additional sizes of standards are required or prove any additional capability of the system. Some customers hold the opinion that if their samples lie in a certain size range (such as 5 μm), then they should use a standard near that size range (5 μm) to test their system. This is unnecessary for several reasons:

- No standard documents suggest that systems be tested with standard materials near the size range of the products to be analyzed. Both ISO 13320 and USP<429> only mention the use of a single standard, without any mention of size range.
- Customers may end up testing variables other than system performance depending on the choice of standard materials. Using the Whitehouse 0.1 – 1.0 μm standard would test the skill of the operator to disperse the sample properly as much as the state of the system. For monodisperse standards, the customer may choose a difficult analysis point where the pass/fail criteria would need adjustment from the suggested +/-5% in the IQ/OQ procedure.
- IQ/OQ procedures can not be modified to meet individual customer requests. The standards in the IQ/OQ procedure are the same ones used to test the system at the factory. This provides proof of system performance and stability. The IQ/OQ procedure is fixed and service engineers are not authorized to change the procedure in the field to meet unique requests.

Conclusions

The LA-series of laser diffraction analyzers should be challenged by system verification tests on a regular basis. Using some combination of the standards used for factory release and field IQ/OQ procedures is recommended. Additional standards may be analyzed by the customer, but not as part of the approved IQ/OQ procedure. Additional standards will not provide improved or more detailed information regarding system performance.

References

1. ISO 13320, Particle size analysis -- Laser diffraction methods -- Part 1: General principles
2. USP <429> Light Diffraction Measurement of Particle Size, USP30, NF25

*Procedures may change prior to this document being updated.