

SPRAYTEC

SPRAYTEC WET UNIT

QUALITY AUDIT STANDARD

CCM0066-02-EN

QAS4001 MEASUREMENT PROTOCOLS

0.4g One-shot polydisperse glass bead transfer standard.

03-2021



Introduction

Malvern Panalytical's QAS4001 Quality Audit Standard has been produced to provide users of Malvern Panalytical laser diffraction particle size analysers with a single-shot, polydisperse transfer standard that enables users to check the performance of their systems on a regular basis.

Compliance with international standards

QAS4001 complies with the laser diffraction system validation guidance provided in ISO13320, USP <429> and EP 2.9.31.

Each single-shot sample consists of spherical particles of known refractive index which have a particle size distribution which extends over greater than one decade in size. In addition, a clear measurement procedure for use of the standard is provided in this datasheet. QAS4001 therefore provides a means of checking and documenting the performance of a laser diffraction system as part of laboratory accreditation schemes (e.g. ISO, NAMAS, and IAF) or in-line with regulatory (e.g. FDA, EMA or MHRA) requirements.

Sample variability

Each Quality Audit Standard bottle is filled using a riffle-splitting process which ensures each sample is representative of the entire 5,200 kg master batch. The sample variability (95% tolerance limit) following riffle-splitting has been measured for the QAS4001 Quality Audit Standard via testing using a single reference Mastersizer system and has been confirmed as:

	Dv10 / μm	Dv50 / μm	Dv90 / μm
QAS4001 Sample variability	+/- 0.801	+/- 0.443	+/- 0.427

Shelf life and batch numbering

Malvern Panalytical's Quality Audit Standards are inert and are stored in sealed containers. They have a shelf life of 5 years. They are produced from a single, large 5,200 kg master batch. As a result, the only batch number for QAS4001 is 03.

Traceability

The Quality Audit Standard Pass/Fail specifications have been defined via a documented test procedure using reference laser diffraction systems. These systems have been verified using NIST-

traceable polystyrene latex standards. As such, although these standards are transfer standards, they are indirectly traceable to NIST.

Establishing Pass/Fail criteria and measurement procedures

Malvern Panalytical carried out a programme of dispersion unit testing to characterize this Quality Audit Standard and establish the target specification. The allowable variation of this target specification is then set taking into account both the sample variability and the expected system measurement variability referenced by ISO13320.

Malvern Panalytical reserve the right to make adjustments to these target specifications to ensure they accurately reflect the expected performance of all Spraytec wet dispersion units. The measurement procedure may also be adjusted to improve the measurement robustness.

Given the above, it is important that the latest version of this datasheet is used. To confirm this is the latest datasheet, visit the Malvern Panalytical website or contact your local Malvern Panalytical representative. If there is any disagreement between the datasheet and the latest OQ procedure for your system, the OQ certificate and specification should take precedence over the datasheet.

Expected results

The specifications for the Spraytec dispersion units are based on guidance from ISO13320 (2020). This standard allows for a maximum instrument uncertainty (u_p) of $\pm 1.5\%$ for the Dv50, $\pm 2\%$ for the Dv10 and $\pm 2.5\%$ for the Dv90. The instrument uncertainty is combined with the sample uncertainty (u_{crm}) according to equation (1) where CF is the coverage factor. As defined in the ISO standard the coverage factor is usually set between 2 and 3 depending on the desired level of confidence. A coverage factor of 2.5 has been selected to provide a confidence level of 99% and to maintain a level of consistency with specifications set under the guidance of the previous edition of ISO 13320.

$$U_{lim} = \pm CF \cdot \sqrt{u_{\text{crm}}^2 + u_p^2} \quad (1)$$

Taking into account the instrument, sample variability, and coverage factor the target specification for this sample is as follows:

	Dv10 / μm	Dv50 / μm	Dv90 / μm
Combined sample variability and measurement tolerance	5.55%	3.83%	6.27%
Upper Specification Limit	43.877	72.303	120.327
Target Value	41.570	69.634	113.230
Lower Specification Limit	39.263	66.965	106.133

Spraytec

If mains water has been used to rinse the unit before measurement, ensure that the final 3 rinses (two rinses and one top-up) are performed using deionised water.

With the sample tank filled, turn the pump/stirrer on to full speed and then turn it off for about 10 sec to allow air to dissipate. Then, set the pump speed to 3,000 rpm.

Fill in SOP settings using the table on the right.

Run the SOP and follow the on-screen instructions.

Instructions during measurement:

1. Add 2 drops of 5% Igepal CA-630 surfactant to the tank and allow them to disperse before measuring the background.
2. Enter the sample details along with the bottle number for the standard.
3. Make sure that the entire contents of the sample bottle is emptied into the tank at the 'add sample' stage.
4. Half fill the empty bottle with deionised water, replace the cap, shake well and empty the contents into the tank.
5. Wait 30 to 50 seconds for the sample to disperse before starting the measurement.

If due to water quality issues, glass beads are observed on the surface of the liquid following sample addition, it may be necessary to add 4-5 drops of 5% Igepal to the tank to aid dispersion.

With the measurement size history file open, average all the data collected during the sample measurement. Select Average scatter data, Concentration-weighted average and Selection mode: All.

Stop

SOP Welcome Tab

Use Template Default Wet Measurement

SOP Type Tab

Hardware configuration Wet measurement cell

Measurement type Timed

Sampling Period (s) 1

Time (min) 1

Lens Either 300 mm or 750 mm, depending on the system configuration

Measurement

Background measurement time (s) 10

Inspection Enabled

Analysis

Particle	RI	1.52
	Absorption	0.00

Dispersant	RI	1.33
-------------------	----	------

Multiple scattering analysis Enabled

Derived Parameters: Select Dx(10), Dx(50), Dx(90) and Obs (%)

© 2021 Malvern Panalytical. Although diligent care has been used to ensure that the information in this material is accurate, nothing herein can be construed to imply any representation or warranty as to the accuracy, correctness or completeness of this information and we shall not be liable for errors contained herein or for damages in connection with the use of this material. Malvern Panalytical reserves the right to change the content in this material at any time without notice.