Malvern Panalytical
a spectris company

MASTERSIZER 3000
HYDRO SM

QUALITY AUDIT STANDARD

CCM0056-01-EN

QAS4001 MEASUREMENT PROTOCOLS

0.4g One-shot polydisperse glass bead transfer standard.

01-2021

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

An on-going programme of dispersion unit testing is carried out by Malvern Panalytical to characterize each 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 constantly assesses the average measurement values obtained over the entire population of Mastersizer 3000 dispersion units. As the population increases, adjustments to the target specification may be required to make sure these accurately reflect the expected performance of all 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 Mastersizer 3000 dispersion units are based on guidance from ISO13320 (2020). This standard allows for a maximum instrument uncertainty (u_p) of ± 1.5% for the Dv50, ±2% for the Dv10 and ±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.

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

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	5.63%	3.83%	6.27%
Upper Specification Limit	40.708	74.604	110.910
Target Value	38.538	71.852	104.366
Lower Specification Limit	36.368	69.100	97.822

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

For IPA users, carry out sufficient rinses with Propan-2-ol in order to yield a good background with no thermal fluctuations. If the background is unstable, replace the lid on the unit to reduce thermal instability and wait until the background stabilizes. This should occur in under 5 minutes.

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. (IPA users, use propan-2-ol instead of water).
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.

Close the SOP after measurement and generate an average of the three measurements to obtain final result.

Stop

Sample		
Particle type	Spherical	
Material	Name	Glass beads (typical)
	Refractive index	1.52
	Absorption index	0.00
	Different blue-light properties	☐
Dispersant	Refractive index	(Water) 1.33 (IPA) 1.39
Measurement		
Duration	Background measurement duration (s)	15
	Sample measurement duration (s)	15
	Don't perform blue light measurement	☑
Sequence	Number of measurements	3
	Delay between measurement (s)	0
	Pre-measurement delays (s)	0
Obscuration	Obscuration lower limit (%)	10
	Obscuration higher limit (%)	30
	Auto start measurement, when obscuration is in range	☐
	Enable filtering	☐
Sample Dispersion		
Accessory	Stirrer speed (rpm)	3,000
	Tank fill behavior mode	Manual. Manual degas after fill (start stop the stirrer)
	Ultrasound mode	N/A
Cleaning	Clean type	Manual
	Clean cycles	N/A
	Ultrasonication	N/A
Data Processing		
Analysis mode	Analysis Model	Narrow modes
Advanced...	Single mode	☑
	Number of inner light detectors to exclude	0
	Remove blue light from analysis	☐
	Sensitivity	Enhanced
Results	Limit the result size range	☐
	Result type	Volume Distribution (recommended)
	Extend the result	☐ All options
User sizes	Use default sizes	

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