

MASTERSIZER 3000 AERO S/M

QUALITY AUDIT STANDARD

CCM0060-01-EN

QAS4002 MEASUREMENT PROTOCOLS

2.5g One-shot polydisperse glass bead transfer standard.

01-2021



Introduction

Malvern Panalytical's QAS4002 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

QAS4002 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. QAS4002 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 QAS4002 Quality Audit Standard via testing using a single reference Mastersizer system and has been confirmed as:

	Dv10 / μm	Dv50 / μm	Dv90 / μm
QAS4002 Sample variability	+/- 0.267	+/- 0.140	+/- 0.280

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 QAS4002 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 $\pm 1.5\%$ 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.

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

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

Aero (All std Venturi)	Dv10 / μm	Dv50 / μm	Dv90 / μm
Combined sample variability and measurement tolerance	5.07%	3.76%	6.26%
Upper Specification Limit	41.101	76.475	111.894
Target Value	39.118	73.704	105.302
Lower Specification Limit	37.135	70.933	98.710

Aero S (HE)	Dv10 / μm	Dv50 / μm	Dv90 / μm
Combined sample variability and measurement tolerance	5.07%	3.76%	6.26%
Upper Specification Limit	43.166	77.562	113.049
Target Value	41.083	74.751	106.389
Lower Specification Limit	39.000	71.940	99.729

AERO S/M

All venturi types

Fill in SOP settings using the table on the right.

Run the SOP and follow the on-screen instructions. If you are working to compendial procedures, repeat the measurement process until 3 QAS samples have been successfully measured.

Instructions during measurement:

1. Make sure the sample area is clean and dry, that the tray and venturi are correctly fitted.
2. Enter the sample details along with the bottle number for the standard.
3. For Aero S set the hopper gap to 1 mm. Empty the entire contents of the bottle into the sample hopper and close the lid of the dispersion unit firmly.
4. For Aero M set the gate gap to 1 mm. Empty the entire contents of the bottle into the centre of the circular end of the tray and close the lid of the dispersion unit firmly.

Close the SOP after measurement. If multiple QAS samples have been measured, average the results to obtain the final result.

Stop

Sample		
Particle type	Spherical	
Material	Name	Glass beads (typical)
	Refractive index	1.52
	Absorption index	0.00
	Density	2.45
Measurement		
Duration	Background measurement duration (s)	10
	Sample measurement duration (s)	30
Sequence	Number of measurements	1
	Obscuration lower limit (%)	0.1
Obscuration	Obscuration higher limit (%)	10
	Auto start measurement, when obscuration is in range	<input checked="" type="checkbox"/>
	Stabilisation time (s)	0
	Enable filtering	<input checked="" type="checkbox"/>
	Time out (s)	5
Sample Dispersion		
Accessory	Air pressure	1 bar g
	Feed rate (%)	40
	Venturi type	Select as appropriate
	Tray type	General purpose tray
	Hopper gap (Aero S) (mm)	1
	Gate gap (Aero M) (mm)	1
Cleaning	Clean type	Normal
Data Processing		
Analysis mode	Narrow modes	
Advanced...	Single mode	<input checked="" type="checkbox"/>
	Number of inner light detectors to exclude	0
	Sensitivity	Enhanced
Results	Limit the result size range	<input type="checkbox"/>
	Result type	Volume Distribution (recommended)
	Extend the result	<input type="checkbox"/> All options
User sizes	Use default sizes	

© 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.