Malvern Panalytical
a spectris company

MASTERSIZER 2000
SCIROCCO 2000/2000M

QUALITY AUDIT STANDARD

CCM0061-01-EN

QAS4002 MEASUREMENT PROTOCOLS

2.5g One-shot polydisperse glass bead transfer standard.

02-2021

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

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 Mastersizer 2000 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 Mastersizer 2000 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	5.07%	3.76%	6.26%
Upper Specification Limit	43.229	76.838	111.041
Target Value	41.145	74.055	104.500
Lower Specification Limit	39.061	71.272	97.959

SCIROCCO 2000/2000M

With a ceramic or stainless steel venturi

Fill in SOP settings using the table on the right.

Instructions during measurement:

1. On the General Purpose sample tray, set the sample feed gate gap to 10 mm.
2. Ensure that the sample area is clean and dry. Enter the serial number of the dispersion unit into the sample details along with the bottle number for the standard.
3. Empty the entire contents of the bottle onto the feed tray.
   - For the 2000 fill the front half of the feed tray, closest to the feed slit.
   - For the 2000M fill the back half of the feed tray, furthest from the feed slit.

If the Scirocco 2000 with a cement sample feed hopper is being used with a horizontal feed slit, set the feed slit aperture to 2.5 mm.

If using the Scirocco 2000M

Set the Air Pressure and Feed Rate values referenced above using the adjustment knobs on the front of the dispersion unit. Then, set the Mode Selector knob to 'Airflow'.

Run the SOP and follow the on-screen instructions.

If using the Scirocco 2000M

Once the background has finished and the Measure Sample screen is displayed, switch the Mode Selector knob on the dispersion unit to 'Feed'. The measurement will start automatically. Adjust the feed rate if necessary in order to ensure that the sample is fed gradually through the feed slit into the feeder basket, and that the correct measurement obscuration is achieved.

Close the SOP after measurement.

Stop

Materials	
Particle	Glass beads (typical)
RI	1.52
Absorption	0.00
Model	Single mode, Fine powder
Measurement	
Background measurement time (s)	12
Measurement time (s)	30
Lower Obscuration Limit (Advance Option)	0.5%
Higher Obscuration Limit (Advance Option)	6.0%
Obscuration Filtering (Advanced Option)	Enabled
Obscuration Filtering - Time Out (Advanced Option)	0 min 45 sec
Sampler Settings	
Sampler Tray	General purpose
Air Pressure	(2000) 1 bar (2000M) 1.2 bar
Feed rate	(2000) 50% (2000M) 35%

Note:
The suggested feed rate is an average setting which should ensure that the obscuration falls within the specified limits and that all the sample is consumed within the measurement time specified for the sample. This may be adjusted to allow for local conditions.

Cycles
Select a single aliquot and 1 measurement cycle

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