QUALITY AUDIT STANDARD FOR HYDRO LV ON MASTERSIZER 3000

QAS4002 MEASUREMENT PROTOCOLS
2.5g one-shot bottles of polydisperse glass-bead transfer standard.
Suitable for: Hydro LV

www.malvernpanalytical.com

QUALITY AUDIT STANDARD FOR HYDRO LV ON MASTERSIZER 3000

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 them 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 5200kg 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:

<table>
<thead>
<tr>
<th>Dv10 / μm</th>
<th>Dv50 / μm</th>
<th>Dv90 / μm</th>
</tr>
</thead>
<tbody>
<tr>
<td>QAS4002 Sample variability</td>
<td>+/-0.267</td>
<td>+/-0.140</td>
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</tbody>
</table>

Shelf Life and Batch Numbering
Malvern Panalytical's Quality Audit Standards are inert and are stored in sealed containers. They therefore have a shelf life of 5 years. They are also produced from a single, large 5200kg 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 ongoing programme of dispersion unit testing is carried out by Malvern Panalytical in order to characterize each Quality Audit Standard and establish the target specification. The allowable variation about 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 dispersion units. As the population increases, adjustments to the target specification may be required to ensure 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. In case of doubt, the latest version number (e.g. MRK2342-nn) can be confirmed by visiting the Malvern Panalytical website or by contacting 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 be considered to take precedence over the datasheet.

Expected Results
The specifications for the Mastersizer 3000 dispersion units are set relative to the target specification based on the guidance in ISO13320, which allows for ±2.5% for the Dv50, ±3% for the Dv10 and ±4% Dv90. Taking into account the sample variability, the target specification for this sample is as follows:

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<tr>
<td>Combined sample variability and measurement tolerance</td>
<td>3.67%</td>
<td>2.69%</td>
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<tr>
<td>Upper Specification Limit</td>
<td>40.245</td>
<td>74.934</td>
</tr>
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<td>Target</td>
<td>38.820</td>
<td>72.971</td>
</tr>
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With the sample tank filled, turn the pump/stirrer on to full speed and then turn it off for about 3 sec to allow air to dissipate. Then, set the pump speed to 3000rpm.

**Hydro LV**

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 de-ionised water.

**Sample**:

- **Particle Type**: Spherical
- **Material**: Material Name: glass beads (typical)
  - Refractive index: 1.52
  - Absorption index: 0.00
  - Density: 2.45
  - Different blue-light properties: unchecked
- **Dispersant**: Refractive index: 1.33

**Sample: Instructions Before Measurement**:

Check that the part No. of the standard being measured is QAS4002. Enter bottle number for the standard into the sample details. Ensure that the entire contents of the sample bottle is emptied into the tank at the ‘add sample’ stage. Half fill the empty bottle with deionised water, replace the cap, shake well and empty the contents into the tank. 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.

**Measurement**:

- **Duration**:
  - Background measurement duration (seconds): 15
  - Sample measurement duration (seconds): 15
- **Sequence**:
  - Number of measurements: 3
  - Delay between measurement (s): 0
  - Pre-measurement delay (s): 0
- **Obscuration**:
  - Obscuration lower limit (%) 10
  - Obscuration higher limit (%) 30

**Sample Dispersion**:

- **Accessory**: Stirrer Speed: 3000 rpm
- **Ultrasound Mode**: None
- **Cleaning**:
  - Clean type: Normal
  - Clean cycles: 3
  - Ultrasonication: Enable 'No ultrasound during clean'

**Data Processing**:

- **Analysis Mode**: Analysis Model: Narrow Modes
  - Advanced...: Single Mode: checked
    - Number of inner detectors to kill: 0
    - Remove blue light from analysis: unchecked
    - Sensitivity: Enhanced
- **Results**:
  - Limit the result size range: unchecked
  - Result Type: Volume Distribution (recommended)
  - Extend the Result: All options unchecked
- **User Sizes**: Use default sizes

Run the SOP and follow the on-screen instructions. Average the results to obtain the final result.

Stop