Malvern Panalytical
a spectris company

# MASTERSIZER 2000
# HYDRO 2000S

# QUALITY AUDIT STANDARD

**CCM0064-01-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 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 | 5.64% | 3.83% | 6.27% |
| Upper Specification Limit | 40.572 | 74.613 | 110.809 |
| Target Value | 38.406 | 71.862 | 104.270 |
| Lower Specification Limit | 36.240 | 69.111 | 97.731 |

# HYDRO 2000S and 2000S+

with the Mastersizer 2000

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 dispersion unit filled, adjust the pump/stirrer speed to 3,000 rpm and then turn the pump off for about 3 sec to allow air to dissipate. Then, slowly adjust the pump/stirrer speed back up to 3,000 rpm. Top up the water in the dispersion unit to replace the volume of air displaced.

Ensure the liquid level is 5-10 mm lower than the tank overflow, back top right side of the tank, to prevent dispersant and material loss.

Fill in SOP settings as below, then run and follow on-screen instructions.

| **Materials** | | |
|---|---|---|
| **Particle** | Name | Glass beads (typical) |
| | Refractive index: 1.52 | Absorption: 0.00 |
| **Dispersant** | Water (RI: 1.33) | IPA (RI: 1.39) |
| **Model** | Single mode, Spherical | |
| **Measurement** | | |
| Background measurement duration (s) | | 15 |
| Measurement time (s) | | 15 |
| **Sample Settings** | | |
| Pump (rpm) | | 3,000 |
| Ultrasound | | None |
| Tank fill | | Manual |
| **Cycles** | | |
| Select a single aliquot and 3 measurement cycles | | |
| Select 'Create Average result' | | |

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

# HYDRO 2000S

with Autosampler 2000 using deionised water

Add the contents of two bottles of QAS4001 to an autosampler pot, and load the pot onto the Autosampler tray.

If mains water has been used to rinse the Hydro 2000S unit before measurement, ensure that the final 3 rinses (two rinses and one top-up) are performed using deionised water.

With the dispersion unit filled, turn the pump off and turn on the ultrasonics for 30 sec. Turn off the ultrasonics and allow any air bubbles to dissipate. Next, adjust the pump and stirrer speed to maximum and then turn the pump and stirrer off for about 3 sec to allow air to dissipate. After 10 sec set the pump/stirrer speed to 3,000 rpm.

Fill in SOP settings as below, then run and follow on-screen instructions.

| **Materials** | | |
|---|---|---|
| **Particle** | Name | Glass beads (typical) |
| | Refractive index: 1.52 | Absorption: 0.00 |
| **Model** | Single mode, Spherical | |
| **Measurement** | | |
| Background measurement duration (s) | | 15 |
| Measurement time (s) | | 15 |
| **Autosampler Settings** | | |
| Sample Quantity (ml) | | 3 |
| Sample Transfer | | By volume |
| Sub-sample volume (ml) | | 15 |
| In-pot mixingtime (s) | | 15 |
| Dispersal Delay (s) | | 15 |
| Dispersant equilibration delay (s) | | 30 |
| **Sample Settings** | | |
| Pump (rpm) | | 3,000 |
| Ultrasound | | None |
| **Cycles** | | |
| Select a single aliquot and 3 measurement cycles | | |
| Select a full wash with one clean cycle for the autosampler and HYDRO 2000S | | |
| Select 'Create Average result' | | |

Instructions during measurement:

Enter the serial number of the dispersion unit into the sample details along with the bottle number for the standard.

Associate the sample on the autosampler tray with the SOP using the autosampler schedule. Run the measurement in accordance with the instructions in the autosampler manual.

Stop



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