Malvern Validation Initiative

Quality Audit Standard for the Hydro 2000µP Small Volume Sample dispersion unit QAS3004

Pack of 10 100mg one-shot bottles of 15 to 150µm polydisperse glass-bead transfer standard, Part No.CRM0020.

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Malvern

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Introduction

Malvern's QAS3004 Quality Audit Standard (bottle part number CRM0020) has been produced to provide users of Malvern particle size analysers with a reliable, one-shot, polydisperse transfer standard that enables them to check the performance of the Hydro 2000µP sample dispersion unit on a regular basis.

This standard replaces Malvern's QAS2005 standard, which was first launched in 1999. QAS2005 was produced from a master batch of 250kg of glass beads (Batch 01), supplies of which were finally exhausted after 6 years. QAS3004 is produced from a new master batch of 1000kg of glass beads (Batch 02), representing the world's largest batch of particle size standards ever produced. The size of this master batch is designed to give users the assurance that QAS3004 will be available as a sizing reference for even longer than QAS2005.

Compliance with International Standards

As with all Malvern Quality Audit Standards, QAS3004 complies with the recommendations of ISO13320, USP <429> and EP 2.9.31 relating to the validation of laser diffraction systems using certified or standard reference materials. The glass bead particles present within QAS3004 are spherical, cover a decade in size and have precisely defined optical properties. In addition, the standard is used in conjunction with a clear measurement procedure, as outlined in this datasheet. As such, QAS3004 provides a reliable means of checking and documenting the consistent operation of a laser diffraction system, as part of FDA or other international laboratory accreditation schemes (e.g. ISO, NAMAS, and IAF).

Sample Variability

Polydisperse particle sizing standards are prone to segregation during transit, which can lead to sampling errors. To overcome this, Malvern's Quality Audit Standards are produced by one of Europe's leading suppliers of particle size standards, Whitehouse Scientific Ltd., who have used an extremely efficient riffle-splitting process to ensure that each one-shot sample is representative of the entire batch. Random sampling of QAS3004 bottles across the entire batch has shown that the relative standard deviation for the median (Dv50) particle size is of the order of 0.85%. This confirms that, as long as the entire contents of the bottle are used during a measurement in accordance with the instructions included on this datasheet, reproducible results can be obtained.

Shelf Life and Batch Numbering

Malvern's Quality Audit Standards are made of inert glass beads. Since they are also stored in sealed containers, they have an indefinite shelf life. Because of this, it has been possible to provide many years of continuous supply from a single master batch. For this reason, the only batch number for QAS3004 is 02.

Traceability

The pass/fail specifications set for Malvern's Quality Audit Standards have been developed via a fully documented programme of testing using a reference Mastersizer 2000 laser diffraction system. This reference system has been verified using NIST-traceable polystyrene latex standards. As such, although these standards are transfer standards, they are indirectly traceable to NIST.

Dispersant Selection

The Hydro 2000µP dispersion unit was designed primarily for the measurement of small sample quantities in organic dispersants. For this reason, the decision was made to characterise QAS3004 using Propan-2-ol as a dispersant, as this is miscible with most organic dispersants, as well as water. No specifications will be issued for the use of this standard in water.

Establishing Pass/Fail criteria and measurement procedures

An extensive and ongoing programme of dispersion unit testing is carried out by Malvern in order to characterize each Quality Audit Standard and establish the pass/fail criteria referenced on this datasheet. As testing continues, Malvern constantly assesses the average measurement values obtained over the entire population of units. As the population increases, slight adjustments to the pass/fail criteria may be required in order to ensure that these accurately reflect the expected performance of the units currently in field, as well as those leaving Malvern's production facility. Changes may also be made to the measurement procedure in order to ensure robust measurements can be made.

Given the above, it is important that the latest version of this datasheet is used, especially when carrying out an annual system OQ or PV. In case of doubt, the latest version number (MRK823-nn) can be verified by visiting Malvern's website. If there is any disagreement between the datasheet and the latest PV certificate and specification, the PV certificate and specification should be considered to take precedence over the datasheet, since these are subject to strict change control and are automatically updated and distributed to the Malvern support network.

Result Continuity

All the data used to set the original pass/fail criteria for the Batch 02 standards were generated using sample dispersion units which had first been tested using Batch 01 standards. This means that a unit which would have passed the earlier standard should also pass using Batch 02.

Target Value Specifications

In order to bring the specification for QAS3004 into line with ISO13320, USP<429> and EP 2.9.31, the limits for the Dv10 and Dv90 have been adjusted to ±5%, instead of ±6% as previously specified. In addition, the median values and limits for all Quality Audit Standards have been standardised to a precision of three decimal places. In order to achieve this, the source data used in setting the limits was reanalysed. This has caused a fractional change in the target values for QAS3004.

An analysis of the original measurement data used in setting the limits for QAS3004 suggests that the above changes should not cause the number of failed measurements to increase significantly.

Expected Results

The expected limits for the Hydro 2000µP dispersion units are set at ±3% for the Dv50 and ± 5% for the Dv10 and Dv90.

	Dv10 / µm	Dv50 / µm	Dv90 / µm
Lower Limit:	37.244	60.491	85.450
Target Value	39.204	62.362	89.947
Upper Limit:	41.164	64.233	94.444

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Start

Ensure the cell windows are clean and free from scratches

Thoroughly rinse the unit using Propan-2-ol such that the background recorded with the lid replaced and the system aligned is less than 100 units for detectors 1-19 and less than 20 units for detectors 20 and above.

Setup or create an SOP with the following settings:

Materials Tab:
Particle: Glass Beads (Typical). RI 1.52, Absorption 0.00
Dispersant: Propan-2-ol, RI 1.39

Model: Single mode spherical

Labels Tab:
Instructions Before Measurement: Check that the part no. of the standard being measured is QAS3004 (CRM0020). Enter the serial number of the dispersion unit into the samples details along with the bottle number for the standard. At the 'add sample' prompt, tap the base of the bottle on the bench, unscrew the cap and add the entire contents of the sample bottle into the sample well on the dispersion unit. Tap the bottle to ensure all the sample is added. Replace the lid of the dispersion unit before performing the first measurement. Carry out 3 measurements in total, with the first measurement being within 1 minute of adding the sample.

Measurement Tab:
Background measurement time: 15 seconds
Measurement time: 15 seconds

Sampler Settings Tab:
Pump/stir: 3000 rpm
Ultrasound: None

Cycles Tab: Select a single aliquot and 3 measurement cycles.
Select 'Create Average result'

Run the SOP and follow the on-screen instructions, adding the sample as instructed. Once the sample has been dispersed, continue with the measurement.

Stop

Cleaning Routine

1) Set the measurement mode to 'Add Sample' in the manual measurement mode dialogue and note the obscuration level.

2) Stop the pump and open the drain. Inject 20mls of dispersant directly into the dispersion unit via the sample well on the top of the unit.

3) Close the drain and inject 25mls of dispersant into the dispersion unit via the dispersion inlet tube on the front of the unit.

4) Turn on the pump and check the obscuration figure on the screen. It should have halved compared to the previous reading.

5) Repeat steps 2-4 until the obscuration has dropped to zero.

Material Safety Data Sheet [MSDS]

1. IDENTIFICATION OF THE SUBSTANCE AND THE COMPANY /UNDERTAKING
Product Name: Malvern Quality Audit Standards
Cas-Number: 65997-17-3
EINECS-Number: 2660460
Product Code: 0390
Synonyms: GLASS BEADS
Use/description of product: Soda Lime Glass
Company: Whitehouse Scientific Ltd. Whitchurch Road, Waverton, Chester, CH3 7PB, England
Tel: +44 (0) 1244 332626
Fax: +44 (0) 1244 335098
email: info@whitehousescientific.com

2. COMPOSITION / INFORMATION ON INGREDIENTS
Hazardous Ingredients: SODA LIME GLASS 100.000%
EINECS: 2660460
CAS: 65997-17-3

3. HAZARDS IDENTIFICATION
Main Hazards: No significant hazard.

4. FIRST AID MEASURES (SYMPTOMS)
Skin contact: There may be mild irritation at the site of contact.
Eye contact: There may be irritation and redness.
Ingestion: No Symptoms.
Inhalation: Exposure may cause coughing or wheezing.

4. FIRST AID MEASURES (ACTIONS)
Skin contact: Wash immediately with plenty of soap and water.
Eye contact: Bathe the eye with running water for at least 15 minutes. Also rinse under the eyelids. If irritation persists, consult a specialist.
Ingestion: Wash out mouth with water.
Inhalation: Remove to fresh air.

5. FIRE FIGHTING MEASURES
Extinguishing Media: Non-Flammable substance, not applicable. Suitable extinguishing media for the surrounding fire should be used.
Protection of fire-fighters: Wear self-contained breathing apparatus. Wear protective clothing to prevent contact with skin and eyes.

6. ACCIDENTAL RELEASE MEASURES
Personal Precautions: Refer to section 8 below for personal protection details.
Clean-Up Procedure: Transfer to a suitable container. Material can create slippery conditions underfoot. Avoid creating dust.

7. HANDLING AND STORAGE
Handling Requirements: Ensure that there is sufficient ventilation of the area. Avoid direct contact with the substance. Avoid the formation or spread of dust in the air.
Storage Conditions: Store in cool, well ventilated area. Keep bottles tightly closed.

8. EXPOSURE CONTROLS / PERSONAL PROTECTION
Hazardous ingredients: SODA LIME GLASS
TWA (8hr exposure limit): 5mg/m3 (OES)
Engineering Methods: Ensure that there is exhaust ventilation of the area.
Respiratory Protection: Respiratory protective device with particle filter.
Hand protection: Protective gloves.
Eye Protection: Safety glasses. Ensure eye bath is to hand.
Skin Protection: Protective clothing.

9. PHYSICAL AND CHEMICAL PROPERTIES
State: Solid.
Colour: White.
Odour: Odourless.
Melting Point/Range°C: Approximately 730°C
Relative Density: 2.6 g/cm3 (20°C)

10. STABILITY AND REACTIVITY
Stability: Stable under normal conditions.

11. TOXICOLOGICAL INFORMATION
Chronic Toxicity: Overexposure to dust may cause irritation of eyes and throat.
Routes of Exposure: No data available.

12. ECOLOGICAL INFORMATION
Mobility: No data available.
Persistence and degradability: No data available.
Bioaccumulative Potential: No data available.

13. DISPOSAL CONSIDERATIONS
Disposal Operations: Contact waste disposal services.
Disposal of Packaging:Contact waste disposal services.
NB: The user's attention is drawn to the possible existence of regional or national regulations regarding disposal.

14. TRANSPORTATION INFORMATION
ADR / RID
UN No: -
Shipping Name: "NOT SUBJECT TO ADR"
IMDG / IMO
UN No: -
IATA / ICAO
UN No: -

15. REGULATORY INFORMATION
Hazard Symbols: No significant hazard.
Note: The regulatory information given above only indicates the principal regulations specifically applicable to the product described in the safety data sheet. The user's attention is drawn to the possible existence of additional provisions which complete these regulations. Refer to all applicable national, international and local regulations or provisions.

16. OTHER INFORMATION
Other Information: Complies with Directives (2001/58/EC), (1999/45/EC), (91/155/EEC), (67/548/EEC) as amended and Chemicals (hazard information and packaging for supply) 2002 (CHIP3) Regulation, EH40.

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