

MALVERN VALIDATION INITIATIVE

Measurement protocols for Quality Audit Standard

QAS3006

For use on the **Morphologi G3**
Sample Dispersion Unit (SDU)

MRK1061-02
10-2013



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INTRODUCTION

Malvern's QAS3006 Audit Standard has been produced to provide users of Malvern particle size analysers with a reliable one-shot polydisperse transfer standard to enable them to check the performance of their sample dispersion units on a regular basis.

Sample dispersion units are set up at the Malvern facility under ideal conditions and are then distributed across the world and used in widely divergent operating environments.

With the passage of time, all analytical instruments need to be checked or re-calibrated and a transfer standard such as QAS3006 is the best method of checking whether an instrument is continuing to perform correctly.

In the case of the Morphologi sample dispersion unit (SDU), subtle changes in injection pressure, injection time, sample settling time, cleanliness and wear of the SDU components over time can all have an effect on the unit's performance characteristics. Using a Quality Audit Standard to establish a baseline performance for the

SDU and to carry out routine performance checks provides a reliable means of checking and documenting the continued consistency of operation of the unit. This can form part of a user's procedures to meet the requirements 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 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. Comprehensive sampling of pots taken from the whole production run of the QAS3006 reference material has allowed us to average the data obtained at fixed size percentiles and establish pass/fail criteria for the purposes of Performance Verification (PV) testing of the SDU. This process has also confirmed 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 and are stored in sealed containers. For this reason they have an indefinite shelf life. It has also been possible to provide many years of continuous supply from a single, large master batch. As a result, the only batch number for QAS3006 is 02.

TRACEABILITY

These samples are traceable to the UK's National Physical Laboratory (NPL) by a transfer method since they have been characterised on a reference Morphologi instrument which in turn has been verified using a reference slide certified and traceable to NPL.

The pass/fail specifications set for Malvern's Quality Audit Standards have been developed via a fully documented programme of testing using reference Morphologi instrument which in turn has been verified using a reference slide certified and traceable to NPL. As such, although these standards are transfer standards, they are indirectly traceable to internationally recognised length standards.

ESTABLISHING PASS/FAIL CRITERIA AND MEASUREMENT PROCEDURES

An on-going 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 sample dispersion 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 all units. 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 (MRK1061-n) can be obtained by visiting Malvern's website. If there is any disagreement between the datasheet and the latest OQ procedure, the OQ certificate and specification

should be considered to take precedence over the datasheet.

EXPECTED RESULTS

QAS 3006 is designed for use on the Morphologi G3 Sample Dispersion Unit (SDU). Expected limits can be found in the PV certificate and specification documents. In addition, the QAS3006_PV_xM_Vx.y report which is provided within the Morphologi software contains custom calculations that will automatically check each parameter is within specification, and will provide an overall PASS/FAIL. The pass fail limits applied within the PV certificate and within the Morphologi report are summarised below.

	Mean / μm	Dn10 / μm	Dn50 / μm	Dn90 / μm	Dv10 / μm	Dv50 / μm	Dv90 / μm	No Particles
Lower Limit	38.53	19.91	35.37	57.52	32.93	49.26	74.49	25000
Upper Limit	45.84	28.07	42.70	69.48	39.69	65.84	86.10	-

continued over

QAS 3006 SAMPLE PREPARATION

Dismantle the SDU chamber assembly:

Remove the air pipe if fitted. Unclip the SDU chamber from the instrument and unscrew the cap.

Clean the SDU chamber:

Clean chamber with anti-static cleaning fluid and clean room wipe. Allow to dry naturally. It is important that the chamber is completely dry before use. If it remains moist too long then blow through with compressed air. Also ensure the red O ring at the bottom of the chamber is free of particles, clean and dry.

Clean the sample dispersion spool:

Clean the metal spool parts with anti-static cleaning fluid and a clean room wipe. Allow it to dry naturally before assembling. See the instrument user manual for more information regarding the cleaning process.

Clean Slide:

Check that the large glass slide (110x180mm) is free from any form of contamination (particularly grease) and is scratch free. If it is greasy, clean with a mild detergent or alcohol-based glass cleaner. Check that the slide is clean and free of dust and dirt. A 'clean-air' aerosol can be used to blow off any dry dust particles as a final step in the cleaning procedure. Fit the glass plate into the XY stage.

Load sample dispersion spool:

Tap the QAS 3006 sample vial to ensure all material is at the bottom of the vial before opening. Open the vial and tip the contents against the side of the central funnel of the sample dispersion spool, ensuring no material is lost during transfer. Try to ensure that the entire contents are transferred into the sample holder inside the spool. Tap the vial several times while inverted to ensure all material is transferred to the funnel and none is trapped on the shoulder of the vial. Tap the cap of the vial to transfer any remaining sample to the spool.

Assemble the spool following the instructions in the instrument user manual. Place it into the top of the SDU chamber, being careful not to tilt or invert the spool.

Load the SDU chamber:

Fit the screw on cap to the SDU chamber, ensuring that it is completely tight, as this completes the sealing of the sample spool. Slide the chamber into the SDU arm and attach the air pipe to the top of the chamber cap.

QAS 3006 MEASUREMENT

Select the QAS measurement SOP:

Select Measure -> SOP... and select QAS3006_2M_PV_Vx.y.vsp if the 2Mpixel CCD is used or QAS3006_5M_PV_Vx.y if the 5Mpixel CCD is used, Vx.y indicates the SOP version. For software version up to and including v8.10 use SOP versions \leq 1.1, and for v8.11 and later use SOP version \geq V2.0.

You can confirm the camera resolution by reading the CCD model number on the label at the back of the camera:

FWX20c = 2 Mpixel CCD

FQX50c = 5 Mpixel CCD

Note: Do NOT rotate the camera. Take care not to knock the camera when fitting the cover.

Perform the measurement:

Click on the start arrow to start the measurement. You will be given an opportunity to modify the measurement name. Append the Bottle No of the bottle used for the measurement.

View & Print result:

Click on the QAS 3006 xM PV Report tab (2Mpixel or 5Mpixel CCD). This report contains custom calculations that will automatically check each parameter is within specification, reports a PASS/FAIL for each parameter and reports an overall PASS/FAIL.

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 (8 hr 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.

Legal Disclaimer: The information contained in this safety Data Sheet is correct to the best of our knowledge, information and belief at the date of its publication.

The information given is designed only as guidance for safe handling, use, processing, storage, transportation, disposal and release and it is not to be considered a warranty or quality specification.

The information relates only to the specific material designated and may not be valid for such material used in combination with any other materials or in any process unless specified in the text.