QUALITY AUDIT STANDARD QAS3001-B MEASUREMENT PROTOCOLS

0.4g one-shot bottles of polydisperse glass-bead transfer standard, Part No.CRM0016.

Suitable for: Hydro MV, Hydro SM

Hydro 2000S, Hydro 2000S+, Hydro 2000SM, Autosampler 2000 with Hydro 2000S using 2 bottles per test

STP2520



MRK0794-10 06-2015



Introduction

Malvern's QAS3001B Quality Audit Standard (bottle part number CRM0016) has been produced to provide users of Malvern particle size analysers with a one-shot, polydisperse transfer standard that enables them to check the performance of their sample dispersion units on a regular basis.

Compliance with International Standards

QAS3001B complies with the recommendations of ISO13320, USP <429> and EP 2.9.31 relating to the validation of laser diffraction systems. The glass bead particles present within QAS3001B 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, QAS3001B 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 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 QAS3001B bottles has shown that the relative standard deviation for the median (Dv50) particle size is of the order of 0.2%. 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 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 OAS3001B 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 reference laser diffraction systems which 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 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 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 (MRK794-nn) can be verified 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

QAS3001B has been designed for use with the following dispersion units:

Mastersizer 3000 Hydro MV, Hydro SM

Mastersizer 2000 Hydro 2000SM, Hydro 2000S, Hydro 2000S+,

Hydro 2000S with Autosampler 2000

Spraytec Wet Dispersion Unit (STP2520)

Note: Specifications for the **Mastersizer S** dispersion units are provided in an earlier version of this document (MRK0794-09). Please contact your local Malvern representative if you wish to obtain a copy of this document.

Mastersizer 2000

The specifications for the Mastersizer 2000 dispersion units are set at \pm 3% for the Dv50 and \pm 5% for the Dv10 and Dv90.

	Dv10 / μm	Dv50 / μm	Dv90 / μm
Lower Limit	34.735	58.779	83.944
Target Value	36.563	60.597	88.362
Upper Limit	38.391	62.415	92.780

Mastersizer 3000

The specifications for the Mastersizer 3000 dispersion units are set at \pm 2.5% for the Dv50, \pm 3% for the Dv10 and \pm 4% Dv90.

	Dv10 / μm	Dv50 / μm	Dv90 / μm
Lower Limit	35.597	60.054	85.928
Target Value	36.698	61.594	89.508
Upper Limit	37.799	63.134	93.088

Spraytec

The Spraytec software does not provide an equivalent of the 'Monomodal' or 'Single Mode' analysis models offered with the Mastersizer systems. For this reason, the target values are different, especially for the Dv90:

	Dv10 / μm	Dv50 / μm	Dv90 / μm
Lower Limit	35.255	58.179	90.753
Target Value	37.111	59.978	95.529
Upper Limit	38.967	61.777	100.305

Aaterial Safety Data Sheet [MSDS]

1. IDENTIFICATION OF THE SUBSTANCE AND THE	Ingestion:	Wash out mouth with water.	Respiratory Protection:	Respiratory Protection: Respiratory protective device	14. TRANSPORTATION INFORMATION	N INFORMATION
COMPANY /UNDERTIAKING Product Name: Malvern Quality Audit standards	Inhalation:	Remove to fresh air.	Hand protection:	with particle filter. Protective gloves.	UN No:	
65997-17-3			Eye Protection:	Safety glasses. Ensure eye	Shipping Name:	"NOT SUBJEC
er:	5. FIRE FIGHTING MEASURES	SURES	•	bath is to hand.	IMDG/IMO	
	Extinguishing Media:	Non-Flammable substance,	Skin Protection:	Protective clothing.	UN NO:	
)	not applicable. Suitable		,	IATA / ICAO	
tion of prod		extinguishing media for the	9. PHYSICAL AND CHEMICAL PROPERTIES	AICAL PROPERTIES	UN No:	
Company: Whitehouse Scientific Ltd.		surrounding fire should be	State:	Solid.		
		used.	Colour:	White.	15. REGULATORY INFORMATION	ORMATION
Chester, CH3 7PB, England	Protection of	Wear self-contained	Odour:	Odo urless.	Hazard Symbols:	
Tel: +44 (0) 1244 332626	fire-fighters:	breathing apparatus. Wear	Melting Point/Range °C	Melting Point/Range°C: Approximately 730°C	No significant hazard.	
	ì	protective clothing to prevent	Relative Density:	2.6 g/cm3 (20°C)	Note: The regulatory information give	information give
email: info@whitehousescientific.com		contact with skin and eyes.			indicates the principal regulations spe	al regulations spe
			10. STABILITY AND REACTIVITY	CTIVITY	applicable to the product described in	duct described in
2. COMPOSITION / INFORMATION ON INGREDIENTS	6. ACCIDENTAL RELEASE MEASURES	SE MEASURES	Stability:	Stable under normal	sheet. The user's attention is drawn to	ention is drawn to
Hazardous Ingredients: SODA LIME GLASS 100.000%	Personal Precautions:	Refer to section 8 below for	,	conditions.	existence of additional provisions whi	al provisions whi
EINECS: 2660460		personal protection details.			these regulations. Refer to all applicat	fer to all applicat
	Clean-Up Procedure:	Transfer to a suitable	11. TOXICOLOGICAL INFORMATION	FORMATION	international and local regulations or	al regulations or
		container. Material can create	Chronic Toxicity:	Overexposure to dust may cause		
3. HAZARDS IDENTIFICATION		slippery conditions underfoot.		irritation of eves and throat.	16. OTHER INFORMATION	NOIL
Main Hazards: No significant hazard.		Avoid creating dust.	Routes of Exposure:	No data available.	Other Information:	
		n	_		Complies with Directives (2001/58/EC),	ives (2001/58/EC),
4. FIRST AID MEASURES (SYMPTOMS)	7. HANDLING AND STORAGE	DRAGE	12. ECOLOGICAL INFORMATION	RMATION	(91/155/EEC), (67/548/EEC) as amended	/EEC) as amended
Skin contact: There may be mild irritation at	Handling Requirement	Handling Requirements: Ensure that there is sufficient	Mobility:	No data available.	(hazard information and packaging fo	and packaging fo
		ventilation of the area. Avoid	Persistence and degrad	Persistence and degradability: No data available.	(CHIP3) Regulation, EH40	.H40.
Eve contact: There may be irritation and		direct contact with the	Bioaccumulative Potential:	tial: No data available.		
		substance. Avoid the			Legal Disclaimer:	
Ingestion: No Symptoms.		formation or spread of dust	13. DISPOSAL CONSIDERATIONS	RATIONS	The information contained in this safe	tained in this safe
		in the air.	Disposal Operations:	Disposal Operations: Contact waste disposal services.	correct to the best of our knowledge,	our knowledge,
	Storage Conditions:	Store in cool, well ventilated	Disposal of Packaging:	Disposal of Packaging: Contact waste disposal services.	belief at the date of its publication. The	its publication. The
)	area.			given is designed only as guidance for	y as guidance for
4. FIRST AID MEASURES (ACTIONS)		Keep bottles tightly closed.	NB: The user's attentio	NB: The user's attention is drawn to the possible	use, processing, storage, transportatio	ige, transportatio
Skin contact: Wash immediately with plenty			existence of regional or national regulations	r national regulations	release and it is not to be considered	o be considered a
	8. EXPOSURE CONTRO	8. EXPOSURE CONTROLS / PERSONAL PROTECTION	regarding disposal.		quality specification. The information	The information
Eye contact: Bathe the eye with running	Hazardous ingredients: SODA LIME GLASS	:: SODA LIME GLASS			the specific material designated and n	designated and n
water for at least 15 minutes.	TWA (8hr exposure limit): 5mg/m3 (OES)	rit): 5mg/m3 (OES)			for such material used in combination	d in combination
Also rinse under the eyelids.	Engineering Methods:	Engineering Methods: Ensure that there is exhaust			materials or in any process unless spec	rocess unless spec
If irritation persists, consult a		ventilation of the area.				



Malvern Instruments limited

Grovewood Road, Malvern Worcestershire, WR14 1XZ, UK Tel +44 1684 892456 Fax +44 1684 892789 www.malvern.com

Hydro MV or Hydro SM on the Mastersizer 3000

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 wate

With the sample tank filled, turn the pump/stirrer on to full speed and then turn it off for about 10 sec to allow air to dissipate. Then, set the pump speed to 3000rpm.

Setup or create an SOP with the following settings:

Sample: Particle Type: Spherical

Name: Glass Beads (typical) Refractive index: 1.52

Different blue-light properties: unchecked

Dispersant: Refractive index: 1.33

Sample:

ons Before Measurement:

Add 2 drops of 5% Igepal CA-630 or Nonidet P-40 surfactant to the tank and allow them to

Absorption index: 0.00

Check that the part No. of the standard being measured is QAS3001B (CRM0016). Enter the serial No. of the dispersion unit into the sample details along with the bottle number for the standard. 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. Allow 30 to 50 seconds for the sample to disperse before performing the first measurement starting within 1 minute of adding the sample

Measurement:

Background measurement duration (seconds): 15 Sample measurement duration (seconds): 15

Don't perform blue light measurement: checked

Number of measurements: 3 Delay between measurement (Pre-measurement delay (s): 0

Obscuration lower limit (%): 10 Obscuration higher limit (%): 30

Auto start measurement, when obscuration is in range: unchecked

Enable filtering: unchecked

Sample Dispersion:

Stirrer Speed: 3000 rpm (Hydro MV SOP only) Tank Fill Behaviour: Manual. Degas after fill enabled (Hydro MV SOP only) Ultrasound Mode: None (Hydro MV SOP only)

Cleaning: Clean type: Normal (Hydro MV SOP only)

Clean cycles: 3 (Hydro MV SOP only)
Ultrasonication: Enable 'No ultrasound during clean' (Hydro MV SOP only)

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

Limit the result size range: unchecked Result Type: Volume Distribution (recon

Extend the Result: All options unchecked

Use default sizes

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

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Hydro SM using Propan-2-ol on the Mastersizer 3000

(only for use as regular internal performance checks not engineers annual OQ or PV)

Carry out sufficient rinses with Propan-2-ol in order to yield a good background.

With the dispersion unit filled, adjust the pump speed up to 3000rpm and turn the pump off for about 3s to allow air to dissipate

Then slowly adjust the pump speed back up to 3000rpm. Top up the Propan-2-ol in the dis unit to replace the volume of air displaced.

If the background is unstable, replace the lid on the unit to reduce thermal instability and wait until the background stabilises. This should occur in under 5

Setup or create an SOP with the following settings: Sample

Particle Type: Spherical

Material: Name: Glass Beads (typical)

Refractive index: 1.52 Absorption index: 0.00

Different blue-light properties: unchecked Dispersant: Refractive index: 1.39

nstructions Before Measurement:

Check that the part No. of the standard being measured is QA53001B (CRM0016). Enter the serial No. of the dispersion unit into the sample details along with the bottle number for the standard. Ensure that the entire content of the sample bottle s emptied into the tank at the 'add sample' stage. Half fill the empty bottle with Propan-2-ol, replace nam in the enjoy bottle with Hopainz-or, repract the cap, shake well and empty the contents into the tank. Allow 30 to 50 seconds for the sample to disperse before performing the first measurement starting within 1 minute of adding the sample.

nstructions After Measurement

If thermal instability was observed during the measurement, as indicated by data instability or the reporting of negative data on the first three detector channels, the measurement should be repeated using a longer thermal equilibration time prior to the background measurement.

Measurement:

Background measurement duration (seconds): 15 ample measurement duration (seconds): 15 Don't perform blue light measurement: checked

Number of measurements: 3 Delay between measurement (s): 0 Pre-measurement delay (s): 0

Obscuration:

Obscuration lower limit (%): 10

Obscuration higher limit (%): 30

Auto start measurement, when obscuration is in

Enable filtering: unchecked

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:

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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.

Hydro 2000SM, Hydro 2000S and Hydro 2000S+ using deionised water on the Mastersizer 2000

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

If ultrasonics are available, turn off the pump/stirrer and turn on the ultrasonics for 30sec to allow air bubbles to dissipate.

With the dispersion unit filled, adjust the which is a speak to 3000 pm 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 3000 pm. Top up the water in the dispersion unit o replace the volume of air displaced

Add 4 drops of 5% Igepal CA-630 or Nonidet P-40 surfactant to the tank and allow them to disperse before measuring the background. If this oncentration of surfactant causes bubble rmation, clean the unit and repeat the procedure

Setup or create an SOP with the following settings:

Materials Tab: Particle: Glass Beads (Typical). RI 1.52,

Water, RI 1.33

Single mode spherical

Labels Tab:

Instructions Refore Measurement: Check that the part no. of the standard being measured is QAS3001B (CRM0016). Enter the serial number of the dispersion unit into the samples details along with the bottle number for the standard. Ensure that the entire contents of the sample bottle are emptied into the tank at the "add sample" prompt Half fill the empty bottle with deignised water replace the cap, shake well and empty the contents into the tank. Allow 30 to 50 seconds for the sample to disperse before performing the first measurement in the series, with the first measurement starting within 1 minute of adding the sample. Carry out 3 measurements in total.

Instructions After Measurement: If hubbles are se instructions Arter measurement: in Dubies are seen on the surface of the water or are detected as data instabilities on the first three detector channels in the scattering display, the measurement can be repeated using only 2 drops of surfactant.

Measurement Tab:

Rackground measurement time: 15 seconds surement time: 15 second

Sampler Settings Tab:

Pump/stir: 3000 rpm Tank Fill: Manual

Cycles Tab:

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

Run the SOP and follow the on-screen instruction

STOP

Hydro 2000SM, Hydro 2000S and Hydro 2000S+ using Propan-2-ol on the Mastersizer 2000

Carry out sufficient rinses with Propan-2-ol in order to yield a good background.

With the dispersion unit filled, adjust the pump speed up to 3000rpm and turn the pump off for about 3 sec to allow air to dissipate. Then slowly adjust the pump speed back up to 3000rpm. Top up the Propan-2-ol in the dispersion unit to replace the volume of air

If the background is unstable, replace the lid on the unit to reduce thermal instability and wait until the background stabilises. This should occur in under 5 minutes.

Setup or create an SOP with the following

Materials Tab:

Particle: Glass Beads (Typical). RI 1.52, Absorption 0.00

Dispersant: Propan-2-ol, RI 1.39 Single mode spherical

Labels Tab:

Instructions Before Measurement: Check that the part no. of the standard being measured is OAS3001B (CRM0016). Enter the serial number QAS3001B (CRM0016). Enter the serial number of the dispersion unit into the samples details along with the bottle number for the standard. Ensure that the entire contents of the sample bottle is emptied into the tank at the "add sample" prompt. Half fill the empty bottle with Propan-2-ol, replace the cap, shake well and empty the contents into the tank. Allow 30 to 50 seconds for the sample to disperse before For seconds for the sample to disperse before performing the first measurement in the series, with the first measurement being within 1 minute of adding the sample.

Instructions After Measurement: If thermal instability was observed during the neasurement, as indicated by data instability or the reporting of negative data on the first three detector channels, the measurement should be repeated using a longer thermal equilibration time prior to the background

Measurement Tab:

Background measurement time: 15 seconds

Sampler Settings Tab:

Pump/stir: 3000 rpm Tank Fill: Manual

Cycles Tab:

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

Run the SOP and follow the on-screen instructions.

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Hydro 2000S with the Autosampler 2000 using deionised water

Add the contents of two bottles of QAS3001B (CRM0016) 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 tha 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

Next, adjust the pump speed up to 3000rpm and turn the pump off for about 3 sec to allow air to dissipate. Then slowly adjust the pump speed back up to 3000rpm

Setup or create an SOP with the following settings:

Materials Tab:

Particle: Glass Beads (Typical). RI 1.52,

Absorption 0.00 Dispersant: Water RI 1 33 Model: Single mode spherical

Labels Tab:

Enter the serial number of the Hydro 2000S and Autosampler into the samples details along with the Bottle Numbers for the standards.

Measurement Tab:

Background measurement time: 15 seconds Measurement time: 15 seconds

Autosampler Settings Tab:

Sample Quantity: 3ml Sample Transfer: By volume Sub-sample volume: 15ml In-pot mixing time: 15 sec Dispersal Delay: 15 sec Dispersant equilibration delay: 30 sec

Sampler Settings Tab:

Pump/stir: 3000 rpm Ultrasound: None

Cycles Tab:

Select a single aliquot and 3 measurement

Full wash with one clean cycle for the autosampler and Hydro 2000S. Select 'Create Average result'.

Associate the sample on the autosampler trav Run the measurement in accordance with the

instructions in the autosampler manual

STOP

Spraytec with Wet Measurement Cell

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 deionised water.

With the dispersion unit filled, adjust the pump/stirrer speed to 3000rpm 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 3000rpm. Top up the water in the dispersion unit to replace the volume of air displaced.

Add 4 drops of 5% Igepal CA-630 or Nonidet P-40 surfactant to the tank and allow them to disperse before measuring the background. If this concentration of surfact causes bubble formation, clean the unit and repeat the procedure using 2 drops of

Setup or create an SOP with the following settings:

SOP Welcome Tab:

Use Template: Default Wet Measurement

SOP Type Tab:

Hardware Configuration: Wet measurement cell.

Measurement Type: 1 second Sampling Period:

Time: 1 minute 0 seconds Lens Either 300mm or 750mm, depending on the system

Documentation:

Instructions Before Measurement: Check that the part no. of the standard being measured is OAS3001B (CRM0016). Enter the serial number of the dispersion unit into the samples details along with the bottle number for the standard. Ensure that the entire contents of the sample bottle are emptied into the tank at the inspection stage entire contents or intersample obtained when replace the cap, shake well and empty the contents into the tank. At time inspection stage, the contents into the tank. Allow 30 to 50 seconds for the sample to disperse before performing the first measurement in the series, with the first measurement starting within 1 minute of adding the sample.

Instructions After Measurement: If bubbles are seen on the surface of the water or are detected as data instabilities on the first three detector channels in the scattering display, the measurement can be repeated using only 2 drops of surfactant.

Measurement Tab:

Background measurement time: 10 seconds Inspection: Enabled

Analysis Tab:

Particle: RI 1.52, Absorption 0.00 spersant: RI 1.33 Multiple scattering analysis: Enabled

Derived Parameters: Select Dx(10), Dx(50), Dx(90) and Obs (%)

Run the SOP and follow the on-screen instructions

Mode: Average Scatter Data (recommended)

When the measurement ends, a Particle Size History (PSH) data file will be created. the data in this file, using the following averaging op

Compare the results contained in this average with the specifications overleaf.

Concentration-weighted average: Enabled Selection Mode: All

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