European Commission

ERM European Reference Materials

JOINT RESEARCH CENTRE
Directorate F – Health, Consumers and Reference Materials

CERTIFICATE OF ANALYSIS

ERM®-ERM-FD069

CORUNDUM

Volume-weighted equivalent diameter Laser diffraction, Fraunhofer approximation			Volume-weighted equivalent diameter Laser diffraction, Mie theory			Number-weighted area-equivalent diameter Optical microscopy		
Diameter[1)]	Certified value [4)] [µm]	U [5)] [µm]	Diameter[2)]	Certified value[4)] [µm]	U [5)] [µm]	Diameter[3)]	Certified value[4)] [µm]	U [5)] [µm]
$x_{5,3}$	13.9	0.6	$x_{5,3}$	15.0	0.6	$x_{5,0}$	12.4	2.9
$x_{10,3}$	17.4	0.4	$x_{10,3}$	18.1	0.5	$x_{10,0}$	15.8	2.9
$x_{25,3}$	24.9	0.30	$x_{25,3}$	25.1	0.8	$x_{25,0}$	19.9	2.5
$x_{50,3}$	36.8	0.4	$x_{50,3}$	36.7	1.5	$x_{50,0}$	23.9	2.6
$x_{75,3}$	52.3	0.6	$x_{75,3}$	52.8	2.2	$x_{75,0}$	30	4
$x_{90,3}$	68.6	0.9	$x_{90,3}$	70.5	2.7	$x_{90,0}$	40	6
$x_{95,3}$	79.8	1.7	$x_{95,3}$	82	3	$x_{95,0}$	46	8

1) As defined by ISO 13320 applying the Fraunhofer approximation and applicable to both dry and wet dispersion.

2) As defined by ISO 13320 applying the Mie theory using a refractive index of 1.77 -0i and applicable to both dry and wet dispersion.

3) As defined by the application of optical microscopy for image analysis according to ISO 13322.

4) Certified values are values that fulfil the highest standards of accuracy. The given values represent the unweighted mean value of the means of accepted sets of data, each set being obtained in a different laboratory and/or with a different instrument. The certified value and its uncertainty are traceable to the International System of Units (SI)."

5) The uncertainty is the expanded uncertainty of the certified value with a coverage factor k = 2 (laser diffraction, Fraunhofer) and k = 2.57 (laser diffraction, Mie and optical microscopy), respectively, corresponding to a level of confidence of about 95 % estimated in accordance with ISO/IEC Guide 98-3, Guide to the Expression of Uncertainty in Measurement (GUM:1995), ISO, 2008.

This certificate is valid for five years after purchase.

Sales date:

The minimum amount of sample to be used is 100 mg (laser diffraction), or 7 mg and analysis of at least 5000 particles (optical microscopy).

Geel, January 2018

Signed:

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Additional Material Information

Equivalent diameter laser diffraction, wet dispersion, application of Mie theory		Equivalent diameter dynamic image analysis	
	Diameter[1)]		Diameter[2)]
$x_{5,0}$	10.1	$x_{5,3}$	11.8-12.9
$x_{10,0}$	11.0	$x_{10,3}$	15.2-15.7
$x_{25,0}$	13.1	$x_{25,3}$	19.1-22.7
$x_{50,0}$	17.0	$x_{50,3}$	26.6-33.8
$x_{75,0}$	23.1	$x_{75,3}$	39.1-47.9
$x_{90,0}$	31.5	$x_{90,3}$	49.8-61.6
$x_{95,0}$	38.2	$x_{95,3}$	56.4-70.8

Volume weighted mean diameter

	Laser diffraction, Mie theory	Laser diffraction, Fraunhofer approximation
	Diameter [3)] [μm]	Diameter [3)] [μm]
$\bar{x}_{1,3}$	41.3	40.5

These values refer to values that were obtained in the course of the study. They are usually derived from single measurements only and are stated without an uncertainty and give merely information about other material properties that may be of interest for the user.

1) As defined by ISO 13320 applying the Mie theory . The value is the unweighted mean of three sets of results obtained by different laboratories using instruments from the same manufacturer. The values are traceable to the international system of Units (SI).

2) As defined by ISO 13322:2, dynamic image analysis. The values represent the range of two sets of results obtained by two different laboratories. The values are traceable to the international system of Units (SI).

3) As defined by ISO 13320 applying the Mie theory using a complex refractive index of 1.77 -0i and applicable to both dry and wet dispersion. The value is the unweighted mean of four sets of results obtained by different laboratories using instruments from the same manufacturer. The values are traceable to the international system of Units (SI).

4) As defined by ISO 13320 applying the Fraunhofer approximation and applicable to both dry and wet dispersion. The value is the unweighted mean of eight sets of results obtained by different laboratories using instruments from different manufacturers. The values are traceable to the international system of Units (SI).

DESCRIPTION OF THE MATERIAL

The material consists of approximately 40 g corundum powder with a top particle size of 150 μm. It is packed in glass bottles with glass inserts to facilitate mixing.

ANALYTICAL METHODS USED FOR CERTIFICATION

Laser diffraction according to ISO 13320:2009 applying the Fraunhofer approximation and Mie theory

Optical microscopy with image analysis according to ISO 13322:2014

PARTICIPANTS

Agfa-Gevaert, Research and Development Materials, Mortsel, BE

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Escubed Ltd., Leeds, UK
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European Commission, Joint Research Centre, Directorate F – Health, Consumers and Reference Materials, Geel, BE
(measurements under the scope of ISO/IEC 17025 accreditation BELAC No. 268-TEST; accreditation for the production of reference materials BELAC No. 268-RM)

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Industrial Technology Research Institute (ITRI), Shinchu, TW

MVA Scientific Consultants, Duluth, US
(measurements under the scope of ISO/IEC 17025 accreditation A2LA 2096.01)

National Institute of Metrology (NIM), Beijing, CN

National Institute of Standards and Technology (NIST), Gaithersburg, US

National Measurement Institute Australia (NMIA), Lindfield, AU

Particle Analytical ApS, Hørsholm, DK

Solvias AG, Kaiseraugst, CH

Sympatec GmbH, Clausthal-Zellerfeld, DE

Umicore, Analytical Competence Center, Olen, BE
(measurements under the scope of ISO/IEC 17025 accreditation BELAC No. 401-TEST)

SAFETY INFORMATION

The usual laboratory safety precautions apply.

INSTRUCTIONS FOR USE AND INTENDED USE

Preparation of the material

Before opening a bottle, the bottle must be gently inverted several times to ensure the homogeneity of the powder. Take several sub-samples at different depths, typically top, middle and bottom of the bottle, using spatula or special sampler device (using rotating/spinning riffler).

For laser diffraction in compressed air, sub-samples should be mixed and shacked in a separate container and then the sample can be introduced in the dry dispersing system of the laser diffraction instrument.

Suspensions are prepared preferably with deionised water with addition of a surfactant like 0.1 mol/L $Na_4P_2O_7$ or isopropanol.

For optical measurements, measure at least 5000 particles.

Use of the certified values

The main purpose of these materials is to assess method performance, i.e. for checking accuracy of analytical results/calibration.

Comparing an analytical result with the certified value

A result is unbiased if the combined standard uncertainty of measurement and certified value covers the difference between the certified value and the measurement result (see also ERM Application Note 1, https://crm.jrc.ec.europa.eu).

When assessing the method performance, the measured values of the CRMs are compared with the certified values. The procedure is summarised here:

- Calculate the absolute difference between mean measured value and the certified value (Δ_{meas}).
- Combine the measurement standard uncertainty (u_{meas}) with the standard uncertainty of the certified value (u_{CRM}): $u_{\Delta} = \sqrt{u_{meas}^2 + u_{CRM}^2}$
- Calculate the expanded uncertainty (U_{Δ}) from the combined uncertainty (u_{Δ}) using an appropriate coverage factor, corresponding to a level of confidence of approximately 95 %
- If $\Delta_{meas} \leq U_{\Delta}$ then no significant difference exists between the measurement result and the certified value, at a confidence level of approximately 95 %.

Use in quality control charts

The materials can be used for quality control charts. Using CRMs for quality control charts has the added value that a trueness assessment is built into the chart.

STORAGE

The material should be stored at (18 ± 5) °C.

However, the European Commission cannot be held responsible for changes that happen during storage of the material at the customer's premises, especially of opened samples.

LEGAL NOTICE

Neither the European Commission, its contractors nor any person acting on their behalf:

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NOTE

A detailed certification report is available at https://crm.jrc.ec.europa.eu/.

A paper copy is obtainable from the Joint Research Centre, Directorate F – Health, Consumers and Reference Materials on request.

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