

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

CERTIFICATE OF ANALYSIS

ERM[®]-FD101b

SILICA NANOPARTICLES IN AQUEOUS SOLUTION			
	Size distribution parameter: Weighting / Averaging	Certified value ²⁾ [nm]	Uncertainty ³⁾ [nm]
Hydrodynamic diameter from DLS (cumulants method) ¹⁾	Scattered light intensity-weighted / harmonic mean	89.5	2.3
Hydrodynamic diameter from DLS (distribution calculation algorithms) ¹⁾	Scattered light intensity-weighted / mean (arithmetic, harmonic, geometric) and modal	93	4
Hydrodynamic diameter from PTA ¹⁾	Number-weighted / modal	82	4
	Number-weighted / arithmetic mean	87	4
	Number-weighted / median	82	4
Stokes diameter from CLS ¹⁾ (turbidimetry)	Light extinction-weighted / modal	87	8
Area-equivalent diameter from EM ¹⁾	Number-weighted / modal	83.7	2.2
	Number-weighted / median	83.5	2.2
Mean particle diameter from SAXS (model fitting) ¹⁾	Scattered X-ray intensity-weighted / modal	82.5	1.8
	Volume-weighted / modal	81.7	1.8
	Number-weighted / modal	80.9	1.7
¹⁾ As obtained with the methods described overleaf, including, where applicable, the valid temperature range. ²⁾ Unweighted mean value of the means of accepted sets of data; each set being obtained in a different laboratory and/or with a different method of determination. The certified value and its uncertainty are traceable to the International System of Units (SI). ³⁾ The uncertainty of the certified value is the expanded uncertainty with a coverage factor $k = 2$ 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 one year after purchase.

Sales date:

Accepted as an ERM[®], Geel, October 2017

Signed:



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NOTE

European Reference Material ERM[®]-FD101b was produced and certified under the responsibility of the Directorate F – Health, Consumers and Reference Materials of the European Commission's Joint Research Centre according to the principles laid down in the technical guidelines of the European Reference Materials[®] co-operation agreement between BAM-JRC-LGC. Information on these guidelines is available on the internet (<http://www.erm-crm.org>).

DESCRIPTION OF THE MATERIAL

ERM-FD101b consists of silica nanoparticles suspended in an aqueous solution. The material is available in 10 mL pre-scored glass ampoules containing approximately 9 mL of suspension.

DETAILS OF THE ANALYTICAL METHODS USED FOR CERTIFICATION

Centrifugal liquid sedimentation (CLS): data obtained according to ISO 13318-1:2001 at gradient or suspension temperatures between 25 °C and 36 °C, using an effective particle density of 2.0 g/cm³.

Dynamic light scattering (DLS) - cumulants method: data obtained according to ISO 22412:2008 applying the cumulants method described in ISO 13321:1996 at a sample temperature of 25 °C.

Dynamic light scattering (DLS) – distribution calculation algorithms: data obtained applying distribution calculation algorithms such as non-negative least square (NNLS) and CONTIN for data analysis, using various averaging approaches and a sample temperature of 25 °C.

Electron microscopy (EM): data obtained with transmission and scanning electron microscopy, counting only particles with an equivalent diameter larger than 60 nm.

Particle tracking analysis (PTA): data obtained according to ISO 19430:2016 at a sample temperature of 25 °C.

Small-angle X-ray scattering (SAXS): data obtained according to ISO 17867:2015 at sample temperatures between 23 °C and 25 °C using 'model fitting' assuming homogeneous spheres and a Gaussian size distribution.

INDICATIVE VALUE			
	Size distribution parameter: Weighting / Averaging	Indicative value ²⁾ [nm]	Uncertainty ³⁾ [nm]
Mean particle diameter from SAXS (Guinier approximation) ¹⁾	((Volume) ²)-weighted / mean	87	6
<p>¹⁾ As obtained with small-angle X-ray scattering according to ISO 17867:2015 at sample temperatures between 23 °C and 25 °C using the Guinier approximation in different ranges of q varying between 0.025 nm⁻¹ and 0.070 nm⁻¹.</p> <p>²⁾ Unweighted mean value of the means of five accepted sets of data; each set being obtained in a different laboratory. The value is indicative and not certified because the measurements were made near the edge of the validity range of the Guinier approximation. The indicative value is valid in the temperature range of [20 °C, 30 °C].</p> <p>³⁾ The uncertainty of the indicative value is the expanded uncertainty, with a coverage factor $t = 2.78$, 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.</p>			

Additional Material Information		
	Size distribution parameter: Weighting / Averaging	Value
Stokes diameter from CLS ¹⁾ (refractometry)	Mass-weighted / modal	84 nm
Hydrodynamic diameter from AF4	Scattered light intensity-weighted / modal ²⁾	86 nm
	Scattered light intensity-weighted / modal ³⁾	83 nm
Properties other than size		Value ⁷⁾
Zeta potential ⁴⁾		-49 mV
pH ⁵⁾		8.7
Electrolytic conductivity ⁶⁾		0.03 mS/cm
¹⁾ obtained with centrifugal liquid sedimentation (CLS) according to ISO 13318-1:2001, at 25 °C ± 1 °C, using an effective particle density of 2.0 g/cm ³ . ²⁾ obtained with asymmetrical flow field-flow fractionation (AF4) using MALS detector at 92°, at 35 °C ± 1 °C, via calibration of the particle elution time using a linear scale. ³⁾ obtained as in ²⁾ but via calibration of the particle elution time using a double logarithmic scale. ⁴⁾ obtained with electrophoretic light scattering (ELS) at 25 °C ± 1 °C. ⁵⁾ determined with a potentiometric method at 22 °C ± 1 °C. ⁶⁾ determined using a folded capillary cell (Malvern Zetasizer Nano ZS instrument) at 25 °C ± 1 °C. ⁷⁾ unweighted mean of six independent results provided by one laboratory.		

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SAFETY INFORMATION

This material should be handled with care. Nanoparticles can have an impact on environment and human health. Any spillage of the suspension should be handled according to the usual laboratory safety precautions. For further details refer to the safety data sheet.

INSTRUCTIONS FOR USE AND INTENDED USE

The intended use of ERM-FD101b is to check the performance of instruments and/or methods that characterise the size distribution of particles around 100 nm, that are either suspended in a liquid medium or deposited onto a suitable substrate. The certified values are regarded as reliable estimates of the true values and ERM-FD101b can therefore be used for calibration purposes.

Before opening, the ampoule should be gently inverted several times to ensure the homogeneity of the suspension and to re-suspend any settled particles. Remove any suspension that remains in the upper part (conical tip) of the ampoule by gently flicking the conical part with the forefinger while tilting the ampoule. The ampoule is pre-scored and can be opened by applying moderate pressure with one's thumb to snap off the conical part. The contents of an ampoule should be used the same day as opened and should be gently homogenised before every measurement, without introducing air bubbles.

DLS method: Aliquots of ERM-FD101b shall be measured as-received, i.e. without dilution. The measurement temperature shall be within the range $[25 \pm 5]$ °C. The viscosity and refractive index of the dispersing medium (water) at 25 °C are 0.8872 mPa·s and 1.330, respectively. The value of the viscosity must be adjusted when tests are not performed at 25 °C.

CLS (turbidity) method: Aliquots of ERM-FD101b shall be measured as-received, i.e. without dilution. The effective density of the silica particles is 2.0 g/cm^3 . The temperature of the sample (for cuvette methods) or of the density gradient (for disc methods) shall be within the range $[25^\circ\text{C}, 36^\circ\text{C}]$.

Electron microscopy method: The material should be transferred to a suitable grid/substrate; after drying, at least 250 discrete (non-overlapping) particles of the large particle population should be counted and measured. If necessary, ERM-FD101b can be diluted with purified water before transferring the particles to the grid/substrate.

Particle tracking analysis: ERM-FD101b should be diluted with purified water (filtered through a membrane with nominal pore size of $0.1 \mu\text{m}$) to a particle concentration suitable for the user's instrument. Neither the as-received, nor the diluted silica suspension shall be treated by filtration, centrifugation or sonication. The measurement temperature shall be within the range $[25 \pm 5]^\circ\text{C}$.

Small-angle X-ray scattering: ERM-FD101b should be either analysed as-received, i.e. without dilution, or diluted in 2-fold in purified water. The measurement temperature shall be within the range $[25 \pm 5]^\circ\text{C}$.

The minimum amount of sample to be used is for:

- CLS (turbidimetry): $100 \mu\text{L}$;
- CLS (refractometry): $340 \mu\text{L}$;
- DLS: $100 \mu\text{L}$;
- EM: $2.5 \mu\text{L}$ of an as-received aliquot and analysis of at least 250 discrete particles;
- PTA: $1 \mu\text{L}$ of an as-received aliquot and analysis of at least 500 tracks per measurement;
- SAXS: $20 \mu\text{L}$.

STORAGE

The material shall be stored at $18^\circ\text{C} \pm 5^\circ\text{C}$. Samples must not be allowed to freeze, as this will irreversibly compromise the integrity of the material.

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

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NOTE

A detailed technical report is available on <https://crm.jrc.ec.europa.eu>. A paper copy can be obtained from the Joint Research Centre on request.

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