

Certified Reference Material

BAM-N012

Particle size of iron oxide nanocubes determined by transmission electron microscopy (TEM) and small-angle X-ray scattering (SAXS)

Certified Values

Characteristics determined by TEM	Value	Uncertainty U^*
Median Area Equivalent Circular Diameter (ECD)	9.1 nm	0.8 nm
Median Area Equivalent Square Edge Length (ESL)**	8.1 nm	0.7 nm

* The uncertainty 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 98-3¹.

**Calculated as square root of a particle's area.

Informative Values*

Characteristics determined by SAXS	Value	Uncertainty U^{**}
Mean Edge Length (EL)	7.9 nm	0.7 nm
Particle Number Density (n)	$0.12 \cdot 10^{-6} \text{ mol} \cdot \text{L}^{-1}$	$0.05 \cdot 10^{-6} \text{ mol} \cdot \text{L}^{-1}$
Concentration (c)	$0.22 \text{ g} \cdot \text{L}^{-1}$	$0.08 \text{ g} \cdot \text{L}^{-1}$

* Informative values can be of interest to the user, but insufficient information is available to establish metrological traceability. Moreover, beginning agglomeration was detected in the scattering data that might have led to the significantly increasing EL observed in the long-term stability study. More details on this issue can be found in the certification report².

**The uncertainty 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 98-3¹.

End of Validity

Provided that the material is handled and stored appropriately, the validity of the certified values is:

- i) one day after opening of the bottle and
- ii) one year beginning with the dispatch to the consumer

Date of dispatch:

Material Description

BAM-N012 is provided in amber glass bottles containing 1 mL of iron oxide nanocubes dispersed in toluene, closed with polypropylene screw caps containing a solvent resistant butyl/PTFE septum.

Recommended Use

The intended purpose of BAM-N012 is the quality control and verification of electron microscopy (TEM and SEM) and SAXS measurements.

Handling

For TEM measurements apply one drop of at least 6 μL of the suspension onto a TEM grid, remove the drop with a filter paper and let it dry at ambient temperature in air for a few hours. For SAXS measurements use an ultrasonic bath for at least 5 minutes before filling the glass capillary with at least 50 μL of the suspension.

Transport and Storage

Toluene (UN 1294) is specified as dangerous goods, but it's supplied in excepted quantities (1 mL), so no special safety related measures must be applied for handling or storage other than standard laboratory precautions for organic solvents. The packaging is done in accordance with ADR 2021 and the ICAO Technical Instructions for road and air travel. The stability of the certified size parameters allows the dispatch of the material at ambient temperature. The short-time exposure to different temperature regimes (both high and low) are accounted for in the expanded uncertainty assessment of the TEM measurements. On receiving, the material must be stored at $(20 \pm 3)^\circ\text{C}$ in the dark.

Analytical Methods

The certified values were established with TEM measuring 3 bottles in duplicate with one traceably calibrated instrument. The values were verified with an ILC including two more transmission electron microscopes and one scanning electron microscope.

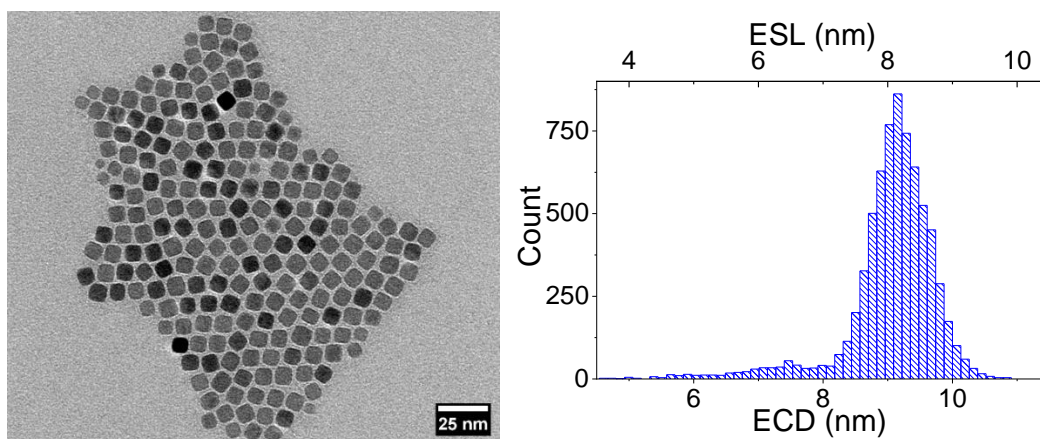


Figure 1: Representative TEM micrograph (left) and particle size distribution of the area equivalent circular diameter (ECD, bottom x-axis) and area equivalent square edge length (ESL, top x-axis) (right).

The informative values were established by SAXS measurements. They are taken from the homogeneity study that included 10 bottles measured in triplicates with one SAXS instrument.

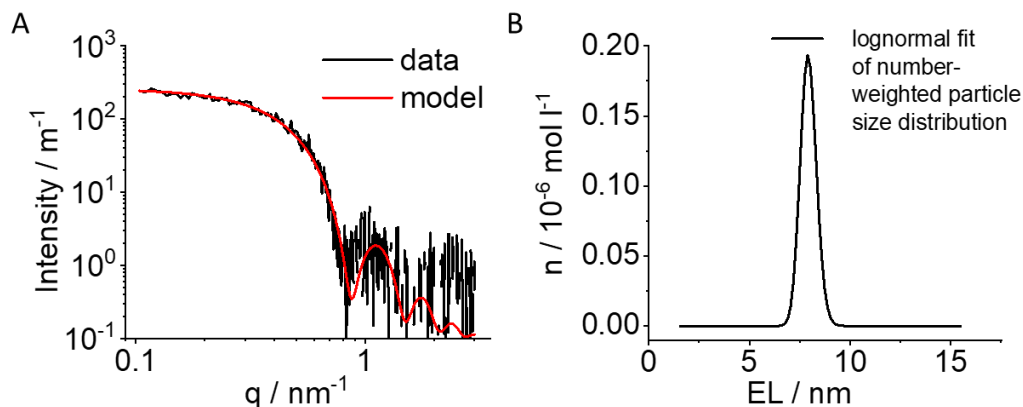


Figure 2: Analysis of the SAXS data by application of the model of spherical particles. Given are (A) the measured scattering data in terms of the scattering intensity as a function of the scattering vector q (black curve) and the best fit of the model

(red curve) and (B) the corresponding distribution of the particles' edge length EL. The size distribution is number-weighted, and the distribution's shape is a lognormal function.

Metrological Traceability

The transmission electron microscope used for the measurement of the certified values was calibrated using the following standards:

Calibration standard (Certificate number):

MAG-I-CAL (Serial Number 2145) – directly traceable to the Si (1 1 1) lattice spacing as a constant of nature. (https://www.microtonano.com/download/MAG-I-CAL%20instruction%20book_v6.2.pdf)

Instrument calibration standards:

ISO 29301:2017 Microbeam analysis – Analytical electron microscopy – Methods for calibrating image magnification by using reference materials with periodic structures.

Literature

¹ISO/IEC Guide 98-3, *Guide to the Expression of Uncertainty in Measurement (GUM:1995)*, ISO, 2008. ISO Guide 35:2017 *Reference materials – Guidance for characterization and assessment of homogeneity and stability*.

² Certification Report, *Certified Reference Material BAM-N012 Cubical iron oxide nanoparticles in toluene*, 2022

³ ISO GUIDE 35:2017 *Reference materials – Guidance for characterization and assessment of homogeneity and stability*, ISO, 2017

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Bundesanstalt für Materialforschung und -prüfung (BAM)

Dr. S. Richter
Committee for Certification

Dr. H. Bresch
Head of TF22 (Nanoplattform)

This Reference Material is offered by:

Bundesanstalt für Materialforschung und -prüfung (BAM)

Richard-Willstätter-Str. 11, D-12489 Berlin, Germany

P: +49 30 8104 2061

E-Mail: sales.crm@bam.de

F: +49 30 8104 72061

Internet: www.webshop.bam.de