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Nanotechnologies — Evaluation of the mean size of nano-objects in liquid dispersions by static multiple light scattering (SMLS)

Nanotechnologies — Évaluation de la taille moyenne des nano-objets dans les dispersions liquides par diffusion statique multiple de la lumière (SMLS)

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 229, *Nanotechnologies*.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Dispersions of nanoparticles in liquids are widely used in industry. Nanoparticles dispersed in liquids interact via a variety of weak and strong forces, which can lead to aggregation or agglomeration of objects (primary particles, agglomerates, aggregates, etc.). As a result, the dispersion state and the apparent mean particle size and size distribution can differ from those determined during product manufacturing, storage, and processing, particularly when using measurements requiring sample dilution or extensive preparation. Sample preparation can result in breaking or formation of aggregates or agglomerates and in some cases can also affect morphology of primary particles. Industrial stakeholders require analytical methods that are applicable to dispersions in their native state for reasons of product development, quality control and regulatory compliance.

While many methods exist for characterization of nanoparticle properties, in particular their size and size distribution, these methods typically require a specific and frequently complex sample preparation (e.g. dilution, stirring, shearing or pumping) and, therefore, do not yield characteristics specific to as-received dispersions. In addition, some experiments do not require measurement of a full particle size distribution with the mean particle size being the main measurand. Using the mean particle size measurement, it is possible to monitor other dispersion parameters of the system such as the state of agglomeration, aggregation or dissolution.

Static multiple light scattering (SMLS) based methods do not require sample preparation allowing, within limitations outlined in this document, direct measurement of the mean equivalent particle diameter in the native (as-received) state of dispersion. In addition, and beyond the scope of this document, SMLS is capable in some cases of monitoring in real time the temporal evolution of mean equivalent particle diameter due to agglomeration or aggregation processes.

This document describes a standardized method for evaluating the mean equivalent particle diameter in various sample types (including as-received samples) having a wide range of concentrations using the SMLS based method.

Nanotechnologies — Evaluation of the mean size of nano-objects in liquid dispersions by static multiple light scattering (SMLS)

1 Scope

This document provides guidance and requirements for the determination of the mean (spherical) equivalent diameter of nano-objects (i.e. particles, droplets or bubbles) dispersed in liquids using the static multiple light scattering (SMLS) technique. The technique is applicable to a wide range of materials and does not require dilution of concentrated samples.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO/TS 80004-1, *Nanotechnologies — Vocabulary — Part 1: Core terms*

ISO/TS 80004-2, *Nanotechnologies — Vocabulary — Part 2: Nano-objects*

ISO/TS 80004-4, *Nanotechnologies — Vocabulary — Part 4: Nanostructured materials*

ISO/TS 80004-6, *Nanotechnologies — Vocabulary — Part 6: Nano-object characterization*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/TS 80004-1, ISO/TS 80004-2, ISO/TS 80004-4, ISO/TS 80004-6 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
static multiple light scattering
SMLS
technique in which transmitted or backscattered light intensity is measured after multiple successive scattering events of incident light in a random scattering medium

3.2
transport mean free path
average distance that a photon travels before its direction vector in its initial direction of motion is reduced to 1/e of its initial magnitude by elastic scattering alone

[SOURCE: ISO 18115-1:2013, 4.299, modified — "an energetic particle" has been changed to "a photon"; "momentum" has been changed to "direction vector"; "initial value" has been changed to "initial magnitude"; notes to entry have been deleted.]

3.3
mean free path
mean distance between photon scattering events in a dispersion

[SOURCE: ISO 22493:2014, 3.2.4, modified — "electron" has been changed to "photon".]

3.4
volume fraction
quotient of the volume of a specified component and the total sample volume

3.5
refractive index
ratio of the speed of light (more exactly, the phase velocity) in a vacuum to the speed of that same light in a material

[SOURCE: ISO 18369-1:2017, 3.1.6.3, modified — “(more exactly, the phase velocity)” has been added; the alternative preferred term "index of refraction" and note 1 to entry have been deleted.]

3.6
equivalent particle diameter
diameter of the sphere with defined characteristics which behaves under defined conditions in exactly the same way as the particle being described

[SOURCE: ISO 21501-1:2009, 2.4]

3.7
absorption
reduction of intensity of a light beam not due to scattering

[SOURCE: ISO 13320:2020, 3.1.1]

4 Symbols and abbreviated terms

I_{BS}	backscattered light intensity
I_{T}	transmitted light intensity
l^*	transport mean free path
l	mean free path
g	asymmetry factor
Q_{e}	extinction efficiency factor
φ	volume fraction
D	mean equivalent particle diameter
λ	wavelength of the incident light (in vacuum)
R	sample half thickness
n	refractive index
T_0	light flux transmitted by the continuous phase
TEM	transmission electron microscopy
CCD	charge-coupled device

CMOS	complementary metal–oxide–semiconductor
ILC	interlaboratory comparison
RM	reference material
VAMAS	Versailles Project on Advanced Materials and Standards

5 Principles

5.1 Relevant theory

The SMLS technique is based on the principle of elastic light scattering from dispersed objects in a liquid. Incident light is scattered multiple times successively, which results in a loss of correlation of the incident light direction. The I_{BS} or I_T light depends on the incident light wavelength, particle concentration, particle size and shape, optical properties (n and absorption of both the continuous and dispersed phases), and the measurement geometry.

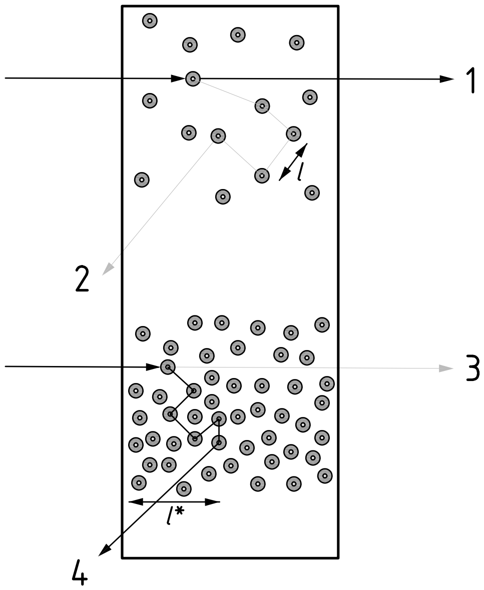
Light propagation in concentrated dispersions (Figure 1) can be characterised by two parameters: the mean free path (Formula (1)), l, and the transport mean free path, l^* [8],[9],[11]. The mean free path characterizes scattering phenomena at the microscopic level, while l^* describes multiple scattering at a macroscopic level as the penetration depth of radiation in a random medium (i.e. no significant correlation between scattering objects). Both parameters l and l^* are related by the Mie theory[14] under the hypothesis where $l > \lambda$ [11]:

$$l = \frac{2D}{3\varphi Q_e} \tag{1}$$

where D is the mean equivalent particle diameter, φ is the volume fraction of the material and Q_e is the extinction efficiency factor.

$$l^* = \frac{l}{(1-g)} \tag{2}$$

NOTE 1 The anisotropic scattering of light by an object can be characterized by the asymmetry factor g, which is the average cosine (cos θ) of the scattering angles weighted by the phase function or scattering diagram of the scatterer (e.g. $g = 0$ for isotropic Rayleigh scatterers and $0 < g < 1$ for Mie scatterers)[14]. Q_e takes into account scattering efficiency and light absorption phenomena.



Key

1 high I_T signal

2 low I_{BS} signal

3 low I_T signal

4 high I_{BS} signal

NOTE The I_{BS} dependence on the volume fraction is depicted.

Figure 1 — Schematic representation of the I_{BS}, I_T, l and l^*

Both Q_e and g are described by the Mie theory[8],[14] and depend on optical properties of the particles and the medium, particle size and wavelength of light.

The Mie theory is then used to determine either equivalent particle diameter or volume fraction, provided that the other is known, from I_T or I_{BS}. This is accomplished by comparing the experimental values of l or l^* with the values determined from the Mie theory.

For measuring (for instance) I_{BS} from the incident light, it is possible to derive an approximate[13]:

$$I_{BS} = \sqrt{\frac{\alpha^2}{l^*}} + \beta = \left[\alpha^2 \frac{3\varphi(1-g)Q_e}{2D}\right]^{1/2} + \beta \qquad (3)$$

Due to the influence of experimental geometry and the optical detector, an output calibration to convert the raw I_{BS} and the raw I_T (e.g. voltage signal) into an exploitable unit is used. The gain α and offset β in Formula (3) are determined with a set of samples of different volume fraction with known l^* (calculated theoretically with the Mie theory).

The light-flux transmitted through a sample can be expressed as[15] :

$$I_T(l,R) = T_0 e^{\left[-\frac{2R}{l}\right]} = T_0 e^{\left[-\frac{3R\varphi Q_e}{D}\right]} \qquad (4)$$

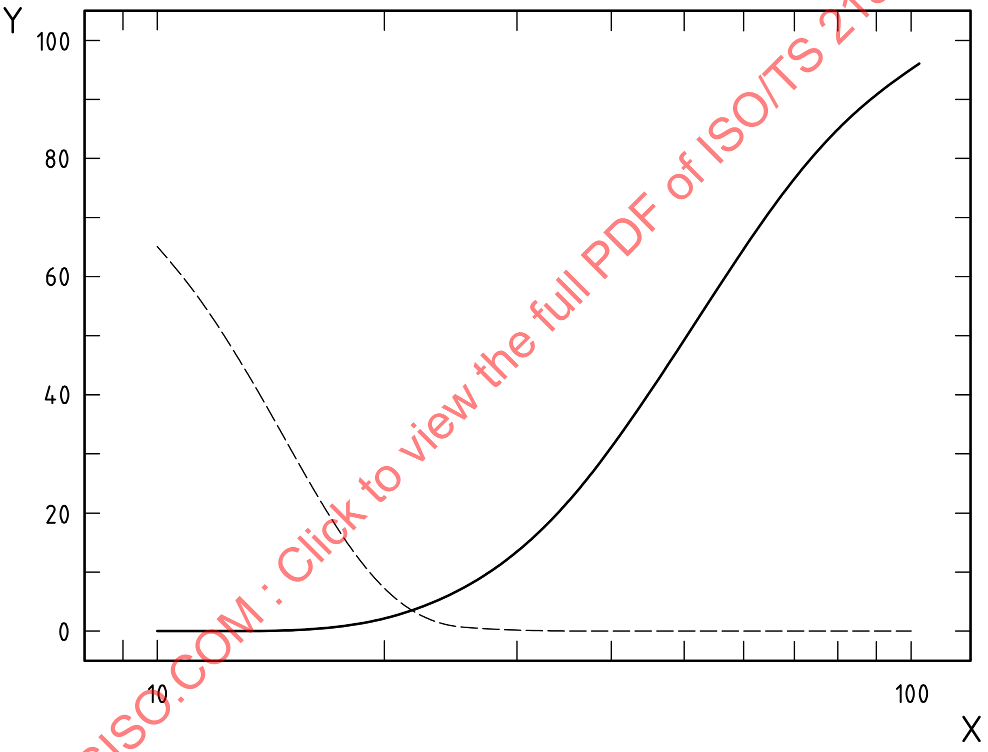
NOTE 2 Variations of I_{BS} and I_T as a function of l^* and l respectively are illustrated in Annex A. Variations of I_{BS} and I_T on mean equivalent particle diameter D for TiO_2 and melamine resin nanoparticles are illustrated in Annex B.

5.2 Key measurands

The measurand used in SMLS is a volume weighted mean equivalent (spherical) particle diameter. For a polydisperse suspension case, an effective l^* is defined that takes into account contributions to the signal from individual particles of various sizes (as described in 5.1). The diameter corresponding to the effective l^* is called the mean equivalent (spherical) particle diameter, also see Formula (2).

It can be shown that for particles smaller than the wavelength of light, the measured mean equivalent particle diameter linearly correlates with the mean volume diameter, D[10],[11]. In this case, backscattered light intensity scales approximately as D^3, meaning that "larger" (but still smaller than λ) particles contribute more to the signal.

The dependence of I_{BS} and I_T on the mean equivalent particle diameter is shown in Figure 2 by way of example. It is the calculated I_{BS} and I_T as a function of mean equivalent particle diameter in a 5 % volume fraction titanium dioxide aqueous dispersion.



Key

X D [nm]

Y intensity of light [%]

—— I_{BS}

– – – – I_T

Figure 2 — Calculated I_{BS} and I_T as a function of particle diameter for an aqueous dispersion (n = 1,33) of titanium dioxide (n = 2,50, φ = 5 %, λ = 880 nm)

The I_T and I_{BS} signals are instrument and sample dependent. Thus, as a rule of thumb, mean equivalent particle diameter estimation is obtained from I_T signal provided that it is not null and I_{BS} signal when I_T is null.

NOTE Although outside the scope of the document, for particles larger than λ, the measured mean equivalent particle diameter correlates with the mean surface diameter D[9],[10]. In this case, the I_{BS} signal scales as D^{-1}, meaning that "smaller" (but still larger than λ) particles contribute more to the backscattered intensity.

5.3 Method applicability and limitations

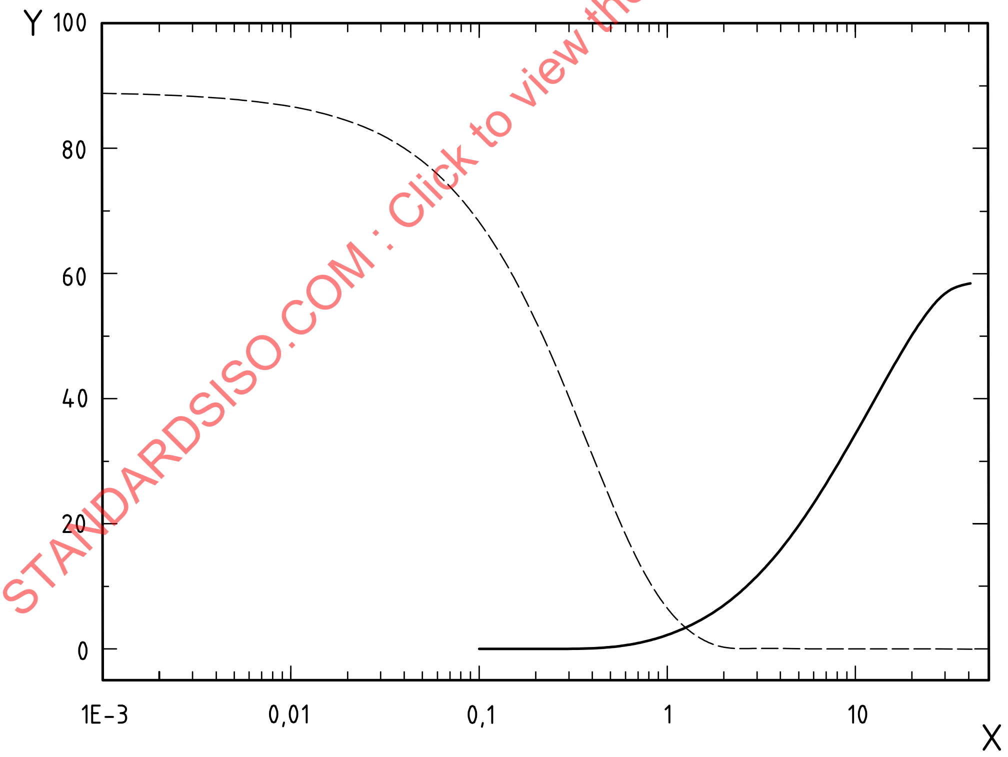
5.3.1 General

The SMLS technique can determine the mean equivalent particle diameter of nano-objects in concentrated dispersions as well as monitor stability of dispersions over time on the very same sample as it is a non-destructive method. It should be noted that this technique may be used for dispersions of solid particles in liquids (i.e. suspensions), liquid-in-liquid emulsions and bubble dispersions in liquids.

The SMLS technique does not allow for the analysis of particle size distributions. It yields a single parameter based on the I_{BS} or I_T measurement, which is converted into a mean equivalent particle diameter (when the particle concentration and n are known). This limits the applicability of the measurement technique to dynamic systems where at least one parameter (equivalent particle diameter or volume fraction) remains constant during the measurement. For mean equivalent particle diameter measurement, material n and volume fraction shall be known independently. The method has been successfully applied to measurements of metal oxides, metals, ceramics, emulsions and ultrafine bubble dispersions in water.

5.3.2 Sample concentration

The ability to analyse undiluted (as-received) samples that have not been modified by sample preparation is one of the main strengths of the SMLS method. Another feature of the method is the ability to measure mean equivalent particle diameter for a broad range of concentrations (from very low to very high concentration). 5.1 describes how I_T and I_{BS} relate to system parameters and the equivalent particle diameter and concentration. Using the Mie theory, I_{BS} and I_T can be calculated for a given material (such as 100 nm diameter silica beads in water). Figure 3 shows these data.



Key

X φ [%]

Y intensity of light [%]

—— I_{BS}

– – – – I_T

Figure 3 — Variation of I_T and I_{BS} with particle volume fraction for silica beads (n = 1,46) in water (n = 1,33) with D = 100 nm and λ = 880 nm

The calculated data in Figure 3 are plotted for dispersions with volume fractions varying from 0,001 % to 40 %. The method equally applies to very low concentration. Although there is no discrete boundary between single scattering and multiple scattering regimes, it should be noted that phenomenologically Formulae (3) and (4) are valid in both regimes. The effect of changing concentration is demonstrated experimentally in Annex E for TiO_2 dispersions in water.

The scattering by dispersed nano-objects depends heavily on several parameters, such as a material's n or particle size and the optical set-up of the instrument used for the evaluation. For this reason, the lower concentration limit is sample/instrument dependent. For the purpose of this document, the lower applicable concentration value is taken to be approximately 10^{-4} % volume fraction.

In the example given in Figure 3, the calculated data reach approximately 40 % volume fraction. Just like for the lower concentration limit, the upper limit depends on the sample and instrument properties.

5.3.3 Mean equivalent particle diameter

Particles with diameters ranging as widely as 10 nm to 100 µm can be evaluated by SMLS. These size limits are approximate and depend heavily on the samples. Indeed, just like for volume fraction, the actual lower and upper size limits are determined by a number of factors, such as the particles and continuous phase refractive indices, concentration, wavelength of light and instrument set-up.

5.3.4 Sample homogeneity and stability

SMLS is a non-destructive technique based on measurement of I_{BS} and I_T over time to characterise the physical evolution of a dispersion due to a destabilisation process (e.g. sedimentation, creaming, aggregation, agglomeration or coalescence)[10]. In all cases, the time scale of change in dispersion parameters should be slower than the time required for a single-time-point intensity measurement in the SMLS data sampling.

NOTE In practice a SMLS single point data acquisition interval at a given cell position is in the millisecond range.

For the mean equivalent particle diameter measurement of nano-objects by SMLS, the sample shall be homogeneous in terms of concentration (i.e. no sedimentation, creaming or floatation). Spatially dependent sampling of data along the measurement cell enables one to identify these local heterogeneities in the sample.

To identify concentration evolution due to sedimentation or creaming, the SMLS light source scans a sample cell vertically. Repeating the scans at fixed time intervals provides information about destabilisation kinetics (i.e. rate of change of concentration). In some systems this can be a critical indicator of the mean equivalent particle diameter measurement quality since the local volume fraction of measured objects in the dispersion should be uniform and stable. If local concentration change or non-uniformity is observed, then the global volume fraction should not be used for particle sizing measurement or homogenisation is required. In case of varying concentration with time or height, the mean equivalent particle diameter measurement can increase its uncertainty for I_{BS} and I_T according to Formulae (3) and (4).

5.4 Method characteristics

Table 1 summarises the characteristics of the technique for the measurement of key dispersion parameters.

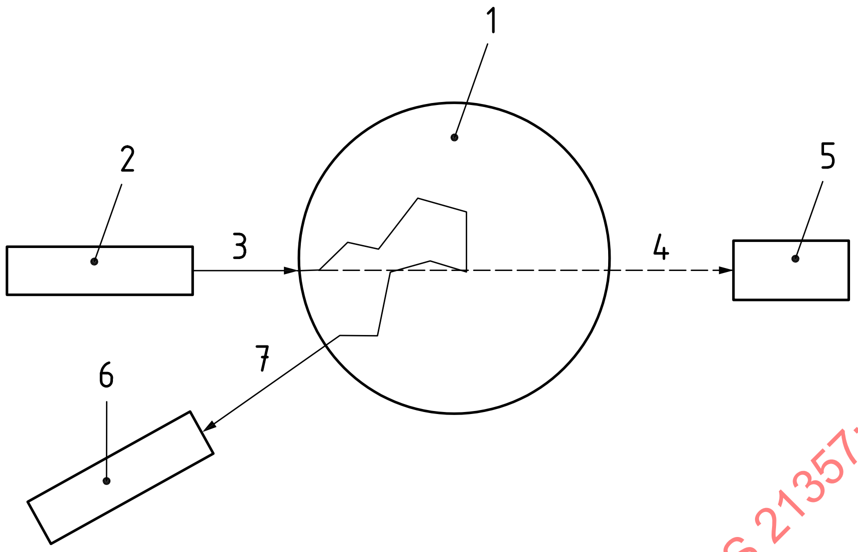
Table 1 — SMLS measurement characteristics

Method feature	**Applicability**	**Notes**
Sample preparation	Generally not required[a]	Sample preparation procedure may not be required (see 7.2).
Concentration range	Approximately from 0,000 1 % to 40 % volume fraction	Dilution (ISO 14488) of the sample is not necessary; and most samples are measured in as-received form. Sample concentration limits are discussed in 5.3.2.
Equivalent particle diameter range	Approximately from 10 nm to 100 µm	The lower and upper size limits are discussed in 5.3.3.
Stability of suspension	Sample should be stable during the time required for the measurement	The size measurement as a function of aging time can be performed on the same sample. The allowed range of change of the intensity is discussed in 5.3.4.
Homogeneity	Homogeneous over the whole sample height	The mean particle size and concentration can vary based on the position in the sample. This is due to non-uniform sample mixing and processes such as sedimentation, creaming or other instabilities. Spatially dependent sampling of data along the measurement cell enables one to identify these local heterogeneities in the sample. The allowed range of homogeneity of suspension is discussed in 5.3.4.

[a] Sample preparation can mean any action done onto the sample including dilution, dispersion, sonication, etc. For samples that have settled or creamed homogenisation prior to the mean equivalent particle diameter measurements is required to have uniform concentration.

6 Apparatus

The common geometry for this kind of measurement is shown in Figure 4. Light from the source is directed into a liquid dispersion. Two sensors (e.g. photodiodes, CCD or CMOS) allow for a simultaneous measurement of the backscattered and transmitted light. The incident light wavelength should be chosen in order to minimize light absorption by the dispersed particles: near-infrared light is advantageous to utilize in the measurement of highly absorbent products. The temperature of the sample should not vary by more than 0,5 °C during the measurement to ensure the repeatability of the result. Temperature of the dispersion affects a number of parameters such as refractive indices, viscosity of continuous phase and the dispersion state.



Key

1	sample	5	I_T sensor
2	light source	6	I_{BS} sensor
3	incident light	7	backscattered light
4	transmitted light		

NOTE Both the backscattered and transmitted paths are shown.

Figure 4 — Schematic of an SMLS experimental set-up

The I_{BS} sensors should be placed outside of the coherent backscattering cone (interference phenomenon that persists after multiple scattering events, see Reference [16] for definition). Speckle noise resulting from interfering scattering waves which leads to a light intensity spatial fluctuation is negligible as speckle size is smaller than detector size.

The post treatment algorithms shall be capable of calculating the mean equivalent particle diameter, D, or the volume fraction, φ, by using the Mie theory with the optical properties of both the dispersed and continuous phases as inputs.

The incident light source, and the backscattered and transmitted light detectors can be mounted onto a moving plate or on a multiple detector array that allows for a repetitive spatial scanning of the sample over time (thereby monitoring the dispersion state and aging) to detect changes in local equivalent particle diameter due to material physical destabilisation (ISO/TR 13097).

7 Measurement procedure

7.1 Instrument preparation

The instrument preparation procedures may vary between manufacturers, so following the manufacturer's instructions is critical.

The instrument shall be turned on and allowed to stabilise at a given temperature. Light source stability is critical for reliable measurements. The instrument should be equilibrated prior to a measurement following the manufacturer's recommendation; typically, a 30 min warm-up is recommended.

7.2 Sample handling

For mean equivalent particle diameter determination, no dilution is required. The user should ensure that the particle concentration is uniform throughout (i.e. no creaming or sedimentation) at the beginning and during the measurement of the mean equivalent particle diameter. The sample may be shaken prior to loading into the equipment if the sample is not homogeneous, but care shall be taken not to introduce bubbles, as they can influence the measurement results due to the fact that bubbles act as light scattering centres of unknown number, size and n. All techniques to remove air bubbles are appropriate, as long as they do not alter the dispersion state. In some cases, sonication is required to disperse nanoparticles.

Sampling for the purpose of the SMLS measurement should follow the guidelines described in ISO 14488. The sample should be placed in a sample cell made from optically transparent glass. For concentrated media, because sample viscosity can be high, cylindrical measurement cells are ideally suited for good sampling. Before measurements, the sample cell should be cleaned and sealed. The following procedures should be followed to ensure good sample handling.

a) Load sample in measurement cell.

b) Ensure that the cell is sealed.

c) Homogenise the sample (if needed by the experiment) appropriately. In some cases the particle concentration of as-received dispersions may not be homogeneous. Re-dispersion is then advised.

d) Clean the exterior of the cell with a non-abrasive tissue and an appropriate solvent (if necessary) until no visible marks are present. Verify that the cell surface dries without visible residue.

e) Introduce the cell into the equipment.

f) Allow sample to reach thermal equilibrium in the instrument. If a home-built system is not able to do this then another thermal stabilisation should be designed.

7.3 System settings

7.3.1 General

Any instrument qualification (or re-qualifications) shall follow the manufacturer's instructions.

For SMLS measurement of mean equivalent particle diameter in a nanodispersion, several parameters of that nanodispersion shall be known. 7.3.2 to 7.3.4 elaborate on the main parameters such as n and volume fraction of the dispersed material and its dispersion homogeneity.

7.3.2 Procedure to verify sample homogeneity

If the sample has a tendency to cream or sediment, there are variations of I_T or I_{BS} as a function of vertical position in the sample. It should be noted that the very top and bottom of the sample cell are affected most by such processes. Measurement zone in the middle third of the sample cell is recommended for equivalent particle diameter measurement. If the range of I_T or I_{BS} spatial variation is greater than 1 %, then the sample should be re-homogenised.

The 1 % threshold is only indicative and for some experiments another threshold may be considered.

7.3.3 Volume fraction

The volume fraction of dispersed objects in a sample can be evaluated by an independent method or known a priori from the preparation of the sample.

7.3.4 Refractive index

n of the continuous phase (liquid) and of the objects in the liquid phase are required for the evaluation of the mean equivalent particle diameter in a sample. This generally means that the user shall know the identity of the object material or measure n using an independent method.

8 Performance qualification

Performance qualification is based on equivalent particle diameter measurements for an RM.

NOTE CRM is used for trueness evaluation and RMs are often used for repeatability and stability evaluation.

For industrial applications, it is recommended to use an RM that is similar to the sample to be measured (i.e. in terms of *n*, equivalent particle diameter and concentration).

Instrument performance should be periodically verified using an appropriate RM and applying predetermined fit for purpose qualification criteria (Annex C) that entail best laboratory practices. For this comparison, a measurement result using an RM is deemed compliant (i.e. no significant difference exists between the measurement result and the reference value), if the absolute difference between the measurement result (mean value) and reference value is less than or equal to the combined expanded uncertainties of the measurement result and reference value at a predetermined confidence level (typically 95 %). See for example Reference [12] for practical guidelines. Alternatively, a pre-set absolute differential threshold (e.g. 10 %) may be applied to compare the verification measurement result with the reference value, if this approach is fit for purpose and meets industry standards of practise.

An RM is used to validate the SMLS method through a VAMAS interlaboratory study. The results are reported in Annex F.

9 Data record

This clause deals with the data that need to be recorded by the operator and/or the data recording equipment. The sample properties should be included in the record keeping. Experimental conditions should be recorded. See Table 2.

Table 2 — Minimum data record for SMLS measurements

Data description	**Value**
Measurement date	
Measurement location	
Instrument make	
Instrument serial number	
Software version	
Date of last qualification	
Transmitted light intensity sensor qualification	
Backscattered light intensity sensor qualification	
Sample dispersed phase *n*	
Sample continuous phase *n*	
Sample volume fraction	
Temperature of the sample	
Mean equivalent particle diameter	Measurement 1
	Measurement 2
	……..
	Measurement *n*

10 Measurement uncertainty

Mean equivalent particle diameter evaluation using SMLS relies on the accurate measurement of I_{BS} or I_T. Random errors can arise from I_T or I_{BS} variation resulting, for example, from spatial and temporal variation of sample properties as well as from measurement temperature, light source intensity, and detector response fluctuations.

A typical uncertainty due to the intensity measurement of 1 % to 5 % is expected. The precision of the method can be affected by uncertainties related to the I_{BS} or I_T variation, fluctuations of temperature, light source, detector response, etc. The trueness of the method can be affected by the use of incorrect values of refractive indices, volume fraction, particle density.

Following the discussion in 5.3.1 and I_{BS} and I_T variation in Figure 3, it should be noted that for very low turbidity samples, using I_{BS} can introduce large uncertainties; thus I_T is used. For high turbidity samples, I_T measurement adds higher uncertainty; and I_{BS} should be used in measurement.

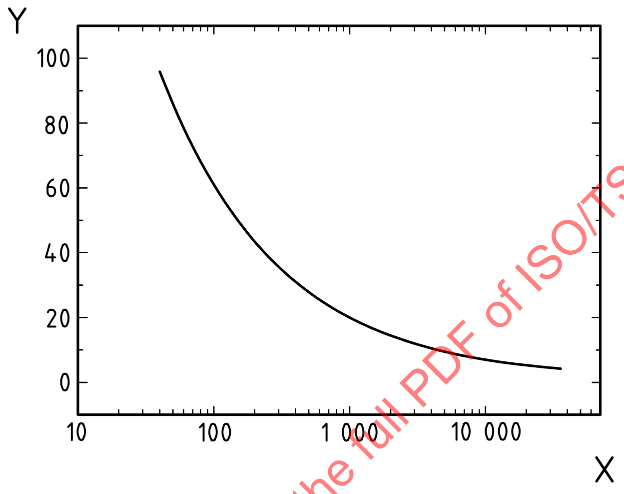
As described in 7.3.3, the mean equivalent particle diameter measurement with SMLS is sensitive to the instabilities in the dispersion.

Annex A

(informative)

I_{BS} and I_T versus l^* and l

Clause 5 describes in detail the relationships between I_{BS} and l^* and I_T and l which is illustrated in Figures A.1 and A.2 respectively. These figures illustrate Formulae (3) and (4).

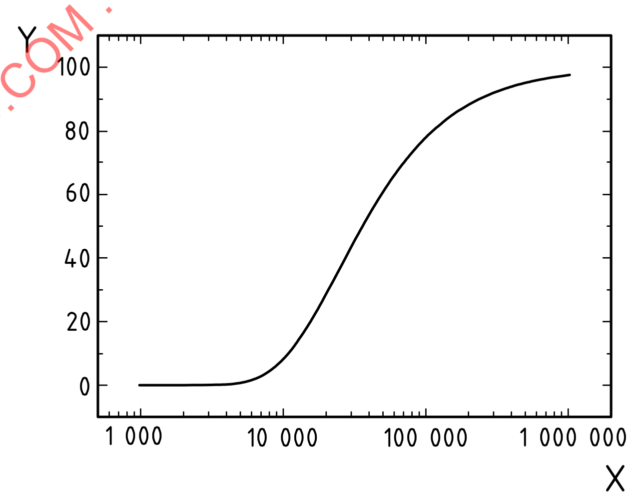


Key

X l^* [µm]

Y I_{BS} [%]

Figure A.1 — I_{BS} as a function of l^*



Key

X l [µm]

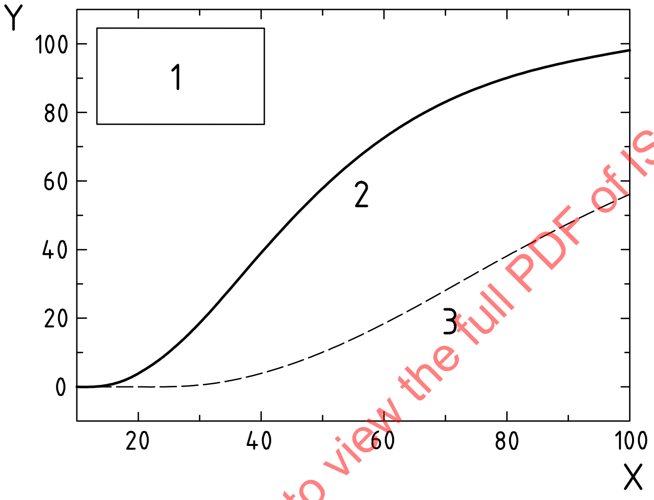
Y I_T [%]

Figure A.2 — I_T as a function of l

Annex B
(informative)

I_{BS} and I_T as a function of D for titanium dioxide and melamine resin particles

As the mean equivalent particle diameter *D* increases from 10 nm to 100 nm, I_{BS} increases but I_T decreases. It should be noted that TiO_2 due to its higher *n* shows higher I_{BS} values than melamine resin. Figures B.1 and B.2 show the calculated (not experimental) values for I_{BS} as a function of mean equivalent particle diameter *D*.



Key

X *D* [nm]

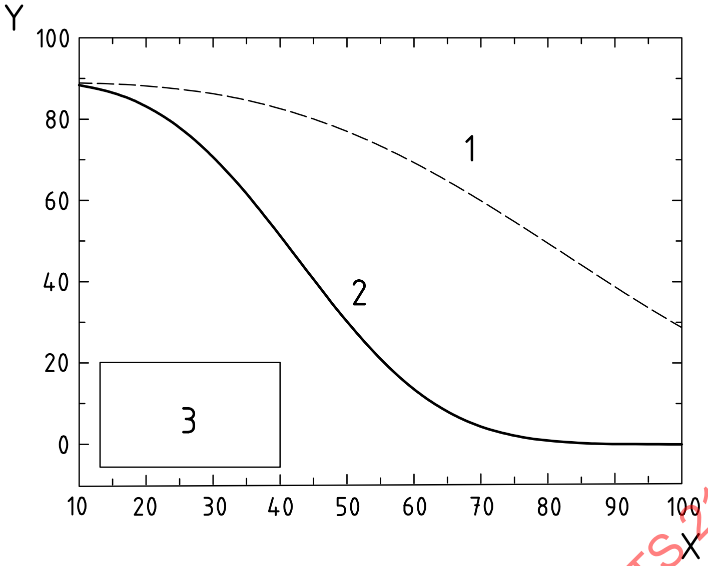
Y I_{BS} [%]

1 particles in water, φ = 10 %, λ = 880 nm

2 TiO_2

3 melamine resin

Figure B.1 — I_{BS} as a function of mean equivalent particle diameter for TiO_2 (n = 2,50) and melamine resin particles (n = 1,68) in water (n = 1,33)



Key

X D [nm]

Y I_T [%]

1 melamine resin

2 TiO_2

3 particles in water, $\varphi = 0,1$ %, $\lambda = 880$ nm

Figure B.2 — I_T as a function of mean equivalent particle diameter for TiO_2 ($n = 2,50$) and melamine resin particles ($n = 1,68$) in water ($n = 1,33$)

Annex C
(informative)

Instrument qualification

Qualification of an instrument is performed by verifying the operation of the transmitted and backscattered light sensors. This is a pass or fail test for the equipment. Prior to the experiment, each instrument should be checked with appropriate qualification materials supplied by the manufacturer or independently certified.

Each instrument has a reference value for both transmitted and backscattered light measurements. These values should be measured and saved when the instrument is first installed, repaired or modified and maintained for future reference. The required intensity achieved at any subsequent instrument qualification should be within 1 % of the assigned reference value. This test ensures that the I_{BS} and I_T detectors are operating properly.

The instrument qualification is performed at the temperature prescribed by the manufacturer. Thermal equilibrium shall be reached for the qualification material. The recommended minimum time to attain thermal equilibrium is 30 min. Sample cell surfaces shall be clean and free from scratches and defects.

Annex D
(informative)

Comparative analysis of Latex suspensions at various concentrations

Latex polystyrene spheres (60 nm in diameter) are dispersed in distilled water with varied volume fraction of the material to test SMLS. The volume fraction varies from 10^{-4} % to 10 %, and corresponding *D* (nm) is reported in Table D.1.

Volume fraction (%)	*D* (nm) (by SMLS)
10	52,0 ± 2,0
1	52,8 ± 1,2
0,1	57,6 ± 2,2
0,01	57,3 ± 1,9

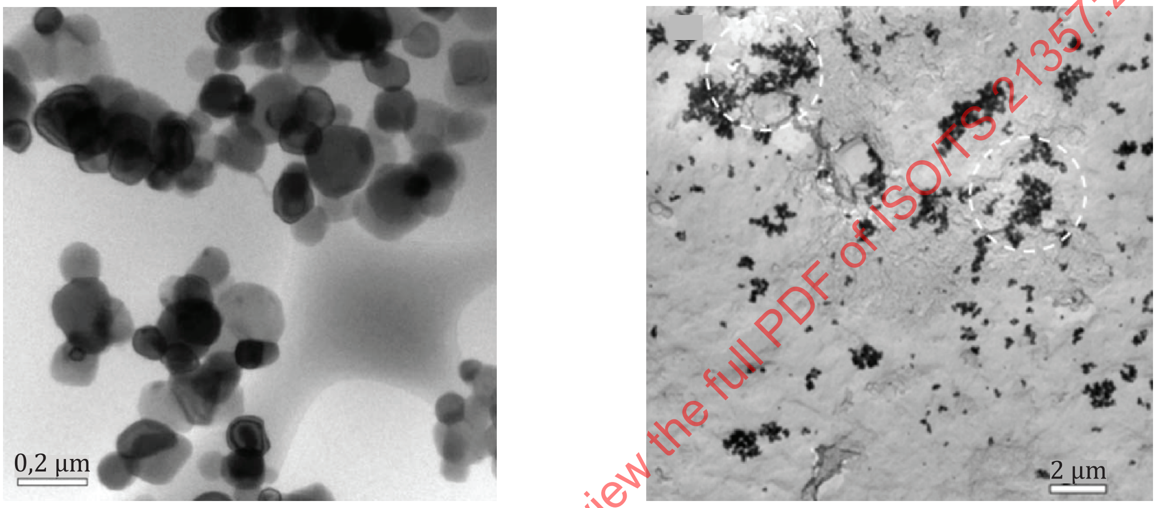
Table D.1 — Values of mean equivalent particle diameter obtained by SMLS

The uncertainty in the values of *D* in Table D.1 correspond to the standard deviation of three independent measurements.

Annex E
(informative)

Analysis of titanium dioxide suspensions at different concentrations

Dry titanium dioxide powder is dispersed in distilled water to obtain samples of concentrations varying from 10^{-4} % to 10 % volume fraction. The samples are also imaged in Cryo-TEM as in Annex D.



a) At 0,01 % volume fraction

b) At 10 % volume fraction

Figure E.1 — Cryo-TEM images of TiO_2 particles

Cryo-TEM images report that particle size increases at high volume fraction (Figure E.1) and is in good agreement with SMLS measured values as illustrated in Figure E.2.