
Reference materials for particle size measurement — Specification of requirements

*Matériaux de référence pour la mesure de taille de particules —
Spécification des exigences*

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 24, *Particle characterization including sieving*, Subcommittee SC 4, *Particle characterization*.

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.

Reference materials for particle size measurement — Specification of requirements

1 Scope

This document is intended to support users of reference materials (RMs) for particle size analysis to identify suitable RMs (certified or not) for their needs. In line with the focus on users, questions on sample preparation that go beyond preparation of the sample as received by the user will not be covered by this document.

This document describes the fundamental requirements that RMs (certified or not) for the determination of particle size shall fulfil in order to be fit for a given purpose. The document is limited to a description of the fundamental principles – the discussion whether a certain numerical value is fit for purpose is beyond the scope of this document.

The scope of this document is limited to RMs (certified or not) in the form of particles. This document does not deal with any other form of RMs, like calibration grids.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions 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

kind of quantity

aspect common to mutually comparable quantities

Note 1 to entry: The division of 'quantity' according to 'kind of quantity' is to some extent arbitrary.

EXAMPLE The quantities diameter, circumference, and wavelength are generally considered to be quantities of the same kind, namely of the kind of quantity called length.

Note 2 to entry: Quantities of the same kind within a given system of quantities have the same quantity dimension. However, quantities of the same dimension are not necessarily of the same kind.

[SOURCE: ISO/IEC Guide 99:2007, 1.2, modified — Note 3 to entry and EXAMPLES 2 and 3 have been deleted.]

3.2

measurand

quantity intended to be measured

Note 1 to entry: The specification of a measurand requires knowledge of the *kind of quantity* (3.1), description of the state of the phenomenon, body, or substance carrying the quantity, including any relevant component, and the chemical entities involved.

[SOURCE: ISO/IEC Guide 99:2007, 2.3, modified — Notes 2 and 3 to entry and all the EXAMPLES have been deleted.]

3.3 operationally defined measurand method-defined measurand

measurand (3.2) that is defined by reference to a documented and widely accepted measurement procedure to which only results obtained by the same procedure can be compared

Note 1 to entry: A term for measurands that are independent of a procedure does not exist. The term “non-operationally defined measurand” is used in this document.

[SOURCE: ISO 17034:2016, 3.7, modified — the second term has been added and Note 1 to entry has been replaced.]

3.4 metrological traceability

property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of *calibrations* (3.12), each contributing to the measurement uncertainty

Note 1 to entry: For this definition, a ‘reference’ can be a definition of a measurement unit through its practical realization, or a measurement procedure including the measurement unit for a non-ordinal quantity, or a measurement standard.

[SOURCE: ISO/IEC Guide 99:2007, 2.41, modified — Notes 2 to 8 to entries have been deleted.]

3.5 monomodal material

material consisting of particles where the particle size density distribution has only one maximum

Note 1 to entry: A monomodal material is not monodisperse if the width of the distribution is larger than the limits described for *monodisperse materials* (3.6).

3.6 monodisperse material

material consisting of particles with narrow particle size distribution

Note 1 to entry: For this document, a material is considered monodisperse if the width of the distribution of the number-based diameter expressed as x_{90}/x_{10} is 1,12 or less (where x_{10} is 10 % percentile of the cumulative particle size distribution and x_{90} is 90 % percentile of the cumulative particle size distribution), which corresponds to a relative standard deviation of the distribution of 4,4 %. The limit 1,12 is taken from the requirements for monodisperse pickets from ISO/TS 14411-1. Such narrow size distributions are typically found in polymer latex materials.

3.7 spherical particle

particle with an aspect ratio of 0,95 or above in all three dimensions

Note 1 to entry: particles with small outgrowths or that are not smooth can nevertheless fulfil this definition of sphericity.

3.8 reference material RM

material, sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process

Note 1 to entry: RM is a generic term comprising both certified and non-certified RMs. There is no term explicitly referring to RMs without any assigned *certified value* (3.10). In this document, the term “reference material/RM” is used for the superordinate, i.e. certified and non-certified RMs, whereas “non-certified RM” is used to explicitly refer to materials without certified values.

Note 2 to entry: Properties can be quantitative or qualitative, e.g. identity of substances or species.

Note 3 to entry: Uses may include the *calibration* (3.12) of a measurement system, assessment of a measurement procedure, assigning values to other materials, and quality control.

[SOURCE: ISO Guide 30:2015, 2.1.1, modified — Note 1 to entry has been expanded and Note 4 to entry has been deleted.]

3.9

certified reference material

CRM

reference material (3.8) characterized by a metrologically valid procedure for one or more specified properties, accompanied by an RM certificate that provides the value of the specified property, its associated uncertainty, and a statement of *metrological traceability* (3.4)

Note 1 to entry: The concept of value includes a nominal property or a qualitative attribute such as identity or sequence. Uncertainties for such attributes may be expressed as probabilities or levels of confidence.

[SOURCE: ISO Guide 30:2015, 2.1.2, modified — Notes 2 to 4 to entry have been deleted.]

3.10

certified value

value, assigned to a property of a *reference material* (3.8) that is accompanied by an uncertainty statement and a statement of *metrological traceability* (3.4), identified as such in the RM certificate

[SOURCE: ISO Guide 30:2015, 2.2.3]

3.11

indicative value

information value

informative value

value of a quantity or property, of a *reference material* (3.8), which is provided for information only

Note 1 to entry: An indicative value cannot be used as a reference in a *metrological traceability* (3.4) chain

[SOURCE: ISO Guide 30:2015, 2.2.4]

3.12

calibration

operation that, under specified conditions, in a first step, establishes a relation between the quantity values with measurement uncertainties provided by measurement standards and corresponding indications with associated measurement uncertainties and, in a second step, uses this information to establish a relation for obtaining a measurement result from an indication.

Note 1 to entry: A calibration may be expressed by a statement, calibration function, calibration diagram, calibration curve, or calibration table. In some cases, it may consist of an additive or multiplicative correction of the indication with associated measurement uncertainty.

Note 2 to entry: Calibration should not be confused with adjustment of a measuring system, often mistakenly called "self-calibration", nor with verification of calibration.

Note 3 to entry: Often, the first step alone in the above definition is perceived as being calibration.

[SOURCE: ISO/IEC Guide 99:2007, 2.39]

3.13

design qualification

DQ

process for verification that the proposed specification for the facility, equipment, or system meets the expectation for the intended use

[SOURCE: ISO 11139:2018, 3.220.1]

3.14
installation qualification
IQ

process of establishing by objective evidence that all key aspects of the process equipment and ancillary system installation comply with the approved specification

[SOURCE: ISO 11139:2018, 3.220.2]

3.15
performance qualification
PQ

process of establishing by objective evidence that the process, under anticipated conditions, consistently produces a product which meets all predetermined requirements

[SOURCE: ISO 11139:2018, 3.220.4]

3.16
operational qualification
OQ

process of obtaining and documenting evidence that installed equipment operates within predetermined limits when used in accordance with its operational procedures

[SOURCE: ISO 11139:2018, 3.220.3]

3.17
proficiency test

evaluation of participant performance against pre-established criteria by means of interlaboratory comparisons

[SOURCE: ISO/IEC 17043:2010, 3.7, modified — Notes to entry 1 and 2 have been removed.]

3.18
statistical quality control

part of quality control in which statistical methods are used (such as estimation and tests of parameters and sampling inspection)

EXAMPLE The use of quality control charts.

[SOURCE: ISO 12491:1997, 3.2, modified — the EXAMPLE has been added.]

4 Abbreviated terms

CRM	Certified reference material
DLS	Dynamic light scattering
DMA	Differential mobility analysis
DQ	Design qualification
ESZ	Electric sensing zone
IQ	Installation qualification
OQ	Operational qualification
PQ	Performance qualification
RM	Reference material

SAXS	Small angle X-ray scattering
SI	International system of units
SQC	Statistical quality control

5 Basic principles

5.1 Measurand definitions in particle size analysis

5.1.1 General

In general, two kinds of measurands can be distinguished.

- Non-operationally defined measurands are measurands where a physical unit can be directly related to a property of a particle and where no further information is required in order to interpret the value of this quantity. Examples for non-operationally defined measurands are a mass of a particle or a distance between two points.
- Operationally defined measurands are measurands that are the result of a specific set of operations. The quantity values of operationally defined measurands are only meaningful in connection with this set of operations. Deviation from the specified set of operations does not only result in a wrong result, but actually means that a different quantity is measured.

EXAMPLE 1 The impact toughness of a material as determined by for example, ISO 148-1. This is the energy required to break a sample of specified dimensions (1 cm × 1 cm × 5 cm) that has a notch of specified width depths with a hammer of specified dimensions. Deviation from the specifications of ISO 148-1 means that a different procedure was applied and that the values obtained are not comparable to the impact toughness of ISO 148-1. Note that the results of impact toughness measurements are expressed in joule, an SI unit. This shows that operationally defined measurands can be expressed in SI units.

Meaningful comparisons of numerical values can only be made for quantities of the same kind. This is immediately obvious for some non-operationally defined measurands: a comparison of a mass and a length is meaningless. As indicated in Note 2 to entry of 3.1, expression in the same unit is required but not sufficient in order to make results comparable. This is especially important for operationally defined measurands.

EXAMPLE 2 In the example of impact toughness above, the energy required to break a sample of different dimensions (e.g. 2 cm × 1 cm × 5 cm) is still expressed in J but it is impossible to say if a material with an impact toughness of 85 J measured on a 2 cm × 1 cm × 5 cm sample is tougher than a material with an impact toughness of 70 J as measured according to ISO 148-1.

This means that one should not expect that different operationally defined measurands yield the same value. Samples may exist that give the same result for two unrelated methods, but this may be due to coincidence. Conflicting values do not mean that one of the values is wrong, but simply reflect the different response for the sample measured.

As will be explained below, the same principle applies to results from different methods for particle size determinations: although the results can all be expressed (and traceable to) as metres, they are in fact different kinds of quantities and not comparable unless very specific conditions are met.

5.1.2 Operationally defined measurands in particle characterisation: Equivalent diameters

None of the methods used for particle sizing actually measures a particle diameter. Doing so requires applying a caliper to a particle or every individual particle of the sample. This is clearly impractical and all particle sizing methods actually measure particle properties different from particle diameters and relate these properties to the particle size. Examples of measured material properties for some particle characterisation methods are given in Table 1.

Table 1 — Selected measurement principles in particle characterisation, their measured properties and information on how this property is expressed

Method	Measured property	Result are expressed as distribution of
Sedimentation analysis	Speed of sedimentation	Diameters of spheres with the same sedimentation velocity (equivalent Stokes' diameter)
Dynamic light scattering, particle tracking analysis	Speed of diffusion	Diameters of spheres with the same diffusion coefficient (equivalent hydrodynamic diameter)
Differential mobility analysis	Electrical mobility of charged aerosol particles	Diameters of spheres with the same electrical mobility
Electrical sensing zone	Drop in resistance when a particle passes through an aperture	Diameters of spheres with the same volume
Image analysis	Length (diameter, circumference) or area of a projection or reflection of the particle	Diameters of circles with the same circumference or area, also direct measurement of maximum and minimum Feret diameter possible
Light scattering particle counters	Intensity of the light scattered by individual particles	Diameters of spheres of the same light scattering/extinction
Light extinction particle counters	Extinction of light caused by individual particles	
Ultrasonic attenuation spectroscopy	Frequency-dependent attenuation of ultrasound	Diameters of (usually spherical) particles which give the same attenuation spectrum
Single particle inductively coupled plasma mass spectrometry	Mass of the selected element(s) per particle	Diameters of spheres of the same mass of the selected element/compound
Small angle X-ray scattering	Angular distribution of elastically scattered X-rays	Diameters of (usually spherical) particles with the same angular distribution of X-rays
Laser diffraction	Angular distribution of scattered light	Diameters of spheres with the same angular distribution of light
Sieving analysis	Mass of material that passes a sieve	Mass fractions passing sieves of specified aperture size
NOTE Results can also differ in the way they are weighted (intensity, number, area etc.).		

These different properties are subsequently expressed as lengths, namely as diameters of spheres that show the same response, for example, having the same speed of sedimentation. These diameters are called “equivalent diameters”. Equivalent diameters are operationally defined measurands: they depend on the property measured (projected area, sedimentation velocity, etc.) and the definition to which shape the property should be equivalent (e.g. equivalent sphere, cube, tetrahedron).

As none of the methods used for particle sizing actually measure the particle diameter, all results of particle sizing methods are operationally defined. This also means that one should not expect that different methods yield the same value unless the particles measured fulfil very specific requirements (see 5.1.4). This non-comparability is clear when one looks at the properties actually measured, but is hidden by the expression of these properties in the dimension of length. It is not surprising that the speed of diffusion differs from the projected area but the fact that both are expressed as lengths of equivalent spheres falsely suggests otherwise.

Conceptually there is no difference between the determination of the equivalent diameter of a single particle and the determination of the distribution of equivalent diameters in an ensemble method: in each case, a property is measured and related to spherical particles that behave the same way for the chosen property. While relating the measured property to spherical particles is more complex for ensemble methods, it is conceptually not different from relating the property of a single particle to the same property of a sphere.

EXAMPLE In laser diffraction, the diffraction pattern of a sample is measured. Applying a chosen theory that models the diffraction pattern of spherical particles, the particle size distribution of an ensemble of spherical particles is calculated that show the same diffraction pattern.

5.1.3 Required detail of procedure description for operationally defined measurands

As discussed above, the results of operationally defined measurands are only meaningful within the clearly specified measurement procedure. In general, a measurement procedure for particle characterisation consists of the following steps, each of which can influence the measurement result:

- sample preparation/dispersion: dispersing medium (e.g. air, liquid), kind and amount of energy used (air pressure, ultrasound, stirring), addition of dispersion facilitating agents, geometry of the sample cell, etc.;
- measurement of a property of the dispersed particles: property measured (e.g. sedimentation velocity, diffraction pattern, diffusion coefficient), instrument parameters (e.g. geometry, laser wavelength);
- evaluation, i.e. relating the measured property to the particle size (distribution) of equivalent particles assumption of shape of the particles (spheres, cylinders, spheroids, etc.): the model, data evaluation algorithm;

In an extreme case, the description may be so specific that results are valid only for a specific instrument using a specific evaluation algorithm.

In other cases, the measured property is independent of many of these instrument-related parameters and the same result can in principle be obtained by a variety of instrument configurations.

The level of detail required for a clear definition of the kind of quantity depends on the type of material and can range from extreme detailed to rather simple.

It is the responsibility of producers of all RMs, in particular of CRMs, to clearly define the detail of the measurement procedure to which the assigned values refer.

5.1.4 Conditions for equivalent diameters to coincide with the actual particle diameter

While results of particle sizing methods are operationally defined and only meaningful in the context of the measurement method, there are some samples for which the equivalent diameter approaches the geometric diameter of a sphere within the measurement uncertainty. The equivalent diameter can coincide with the actual particle diameter under the following conditions.

- a) The material consists of spherical particles. Spherical particles are the only particles that can be characterised by a single length, the diameter.
- b) The material is a monodisperse material. The response of different sizing techniques is weighted differently depending on the property measured and how it is measured (e.g. for dynamic light scattering (DLS): scattering intensity scales with the sixth power of the diameter). As size polydispersity decreases, this weighting becomes less significant. For ideally monodisperse particles, there is no influence due to weighting.
- c) There are no other factors that influence the particle diameter as measured. A plethora of factors can influence the apparent or real particle diameter even for spherical, monodisperse materials. For example, particles can shrink in air or in vacuum, which means the actual diameter in air or in an electron microscope can differ from the actual diameter in suspension. Molecules of the dispersing liquid can adhere to the particle in dispersion, thus indicating a larger diameter in suspension than in air. Molecules of dissolved salts can adhere to the particle when turned into an aerosol, thus increasing the particle diameter of aerosolized particles compared to the same particles in suspension.

The relative influence of most of these effects decreases as the particle diameter increases: an adsorbing liquid layer of 1 nm to 2 nm is relevant for a particle diameter of 10 nm but irrelevant for particles with diameters of 10 μm .

When these conditions are met the equivalent diameter often coincides with the actual diameter and different equivalent diameters based on different measurement principles will often have the same

value. In these cases, it is therefore possible to use values determined by one method as reference values for another, unrelated method.

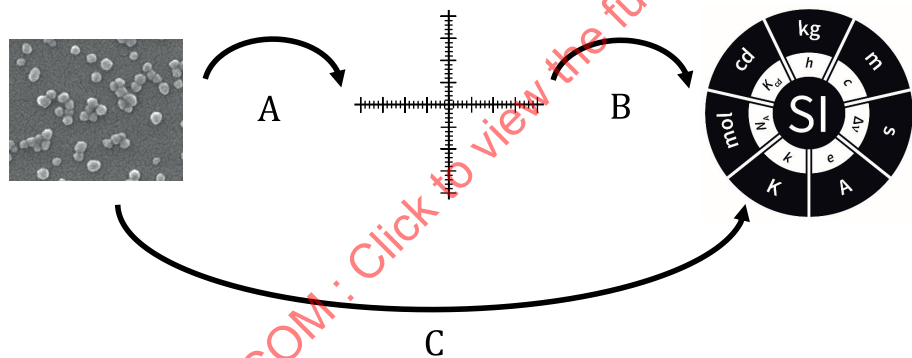
5.2 Metrological traceability of size measurement results

5.2.1 General

As 3.3 highlights, metrological traceability is the property of a measurement result, i.e. the numerical value that is assigned to a measurand of a certain kind of quantity. Traceability describes by which calibrations (or comparisons) a measurement result is related to the stated reference.

EXAMPLE The goal is the determination of the length of a structure in a micrograph. The calibration of the image magnification relates the number of pixels to the stated distance of lines on the calibration grid. The stated distance of the lines of the calibration grid is related by measurement to the SI length unit (metre). This two-step calibration makes the measured length traceable to the metre.

In the example above, the traceability of the measurement result is achieved by two sequential calibration steps. The term “traceability chain” is used to describe such linear, sequential schemes that establish traceability. However, many measurements have several, unrelated input quantities. The value used for each of these input quantities shall be traceable to a stated reference to ensure that the final result is traceable to the given reference. Such multiple references result in a “traceability network” rather than a linear chain. It is irrelevant for the reference to which a measurement result is traceable whether this traceability was achieved in one or multiple steps. Three examples of traceability networks in particle sizing are shown in Figures 1, 2 and 3.

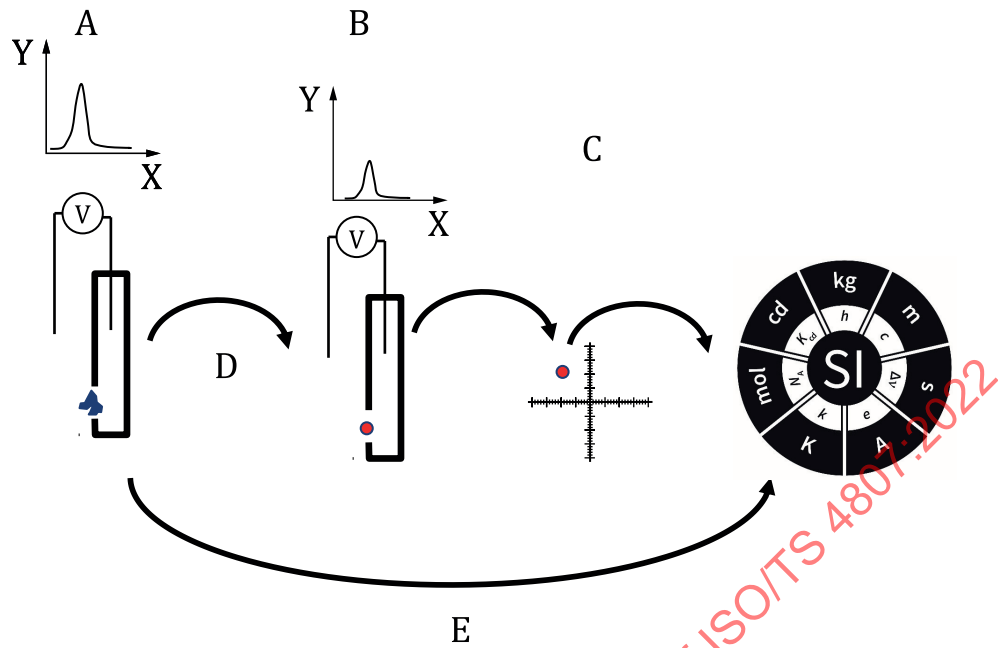


Key

- A calibration of the magnification, pixels/gridline
- B calibration of the calibration grid, gridlines/metre
- C diameter, traceable to the SI via calibration of magnification and grid

NOTE SI Logo from BIPM.org under Creative Commons Licence CC BY-ND 4.0.

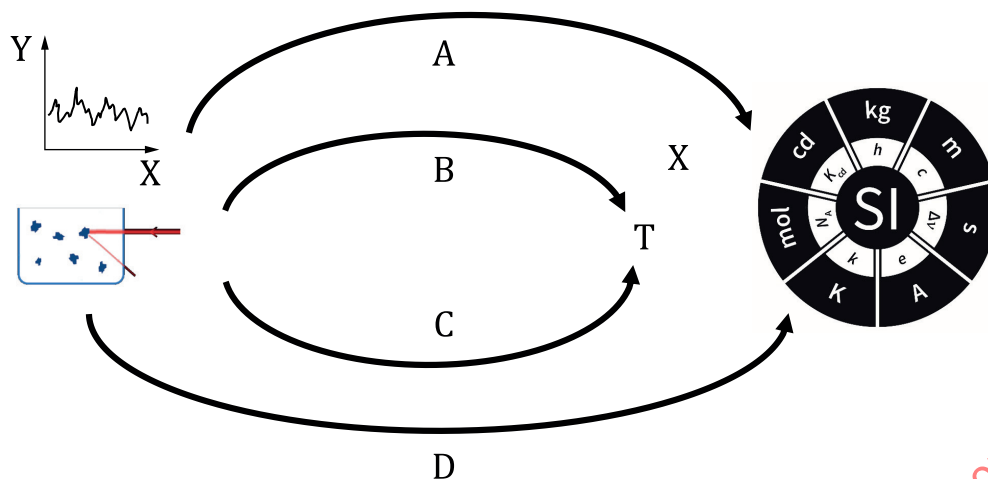
Figure 1 — Traceability chain/network of image analysis

**Key**

- X time
- Y voltage
- A measured pulse height of the sample
- B calibration of the pulse height with a CRM of diameter of d_{CRM}
- C measurement of d_{CRM} by image analysis via calibration of the magnification and calibration of the grid
- D relation of the measured pulse height of the sample to the pulse height of the CRM
- E equivalent diameter of the sample, traceable to the SI via calibration of the pulse height of the ESZ and the calibration of the magnification and grid for the determination of d_{CRM}

NOTE SI Logo from BIPM.org under Creative Commons Licence CC BY-ND 4.0.

Figure 2 — Traceability chain/network of electrical sensing zone

**Key**

X time

Y intensity

A calibration of the clock

B calibration of the temperature sensor

C viscosity determined via calibrated temperature sensor

D equivalent diameter, traceable to the SI via calibration of the clock and temperature sensor

T temperature

NOTE 1 No calibration in length is required to obtain results traceable to the metre in the case of dynamic light scattering.

NOTE 2 SI Logo from BIPM.org under Creative Commons Licence CC BY-ND 4.0.

Figure 3 — Traceability chain/network of dynamic light scattering

An important feature of the calibrations involved in establishing metrological traceability is that each calibration (or comparison) contributes to the measurement uncertainty, even if this contribution may be small or insignificant. In contrast, factors that do not contribute to the measurement uncertainty are also not part of the traceability chain/network.

EXAMPLE 1 In the example above, the calibration of a number of pixels/distance between gridlines can only be performed within a certain uncertainty. The distance between gridlines has an uncertainty that is derived from the accuracy with which this distance was determined. Both of these uncertainties are part of the combined measurement uncertainty of the image analysis, hence both are part of the traceability network.

EXAMPLE 2 A laboratory performs a DLS experiment and uses a CRM to demonstrate proper functioning of the instrument. It compares the certified values with the measured values, but does not use the certified values to adjust any parameters of the evaluation algorithm. Therefore, the CRM used is not part of the traceability chain and the uncertainty of the certified value does not contribute to the overall measurement uncertainty. The difference between verification and calibration is further explained in [5.2.2](#).

It is possible to convert input factors from part of the traceability network to the definition of the property and vice versa by defining an input factor, which normally should be measured, as a certain number. This number then becomes part of the definition of the measurands and, as it is defined without uncertainty, also does not contribute to the measurement uncertainty.

EXAMPLE 3 The measured particle size in sedimentation analysis depends, among other, on the density of the particle. If the value for this density is derived from measurements, these measurements need to be traceable and the uncertainty of those results contributes to the overall measurement uncertainty. On the other hand, one can also assume a certain value for the density, for example, 2,2 g/cm³ for amorphous silicon dioxide. In this case, the definition of the measurand is “as obtained by sedimentation analysis, using a density value of 2,2 g/cm³ for silicon dioxide”.

The definition of an operationally defined measurand itself is unrelated to the traceability of the measurement result. The fact that a measurand is operationally defined does not mean that the quantity values assigned to this measurand cannot be metrologically traceable. Traceability of the quantity values is ensured by proper calibration of the relevant input factors. If this is achieved, then the results are comparable with the results of another laboratory for the same operationally defined property.

EXAMPLE 4 Proper calibration of magnification and image processing ensures SI-traceability of the area-equivalent circular diameter from an image analysis. As such, this value can be compared to the results from other laboratories, as long as these are also the area-equivalent circular diameters of image analysis. Despite its traceability to the metre, the result is not comparable to a result from laser diffraction (even if this result is also traceable to the SI), as the two refer to different kinds of quantity.

5.2.2 Establishing versus verifying traceability in particle characterisation

There is an important difference between establishing and verifying traceability: as stated above, establishing traceability requires calibration of input parameters and these calibrations all contribute to the measurement uncertainty. Although results of properly calibrated instruments should deliver traceable results, it is good practice to check the correctness of the calibration to verify the traceability of results by measuring a CRM. Agreement of the measurement result with the certified values confirms:

- a) correctness of the calibration; an incorrect calibration (e.g. wrong line width for a grid) will lead to deviating results;
- b) proper calibration of all other relevant input parameters; insufficient calibration of (even unknown) relevant input parameters will lead to deviating results.

However, as no adjustment of instrument parameters is performed after this verification, this CRM used to verify traceability is important, but not a part of the traceability chain.

EXAMPLE An electrical sensing zone (ESZ) instrument is calibrated with CRM A by adjusting the nominal aperture size based on the certified diameter of this CRM. Subsequently, CRM B is used to verify that the calibration was appropriate and that all other input factors are properly calibrated. CRM A is part of the traceability chain, as its values are used to adjust the measurement result, but CRM B is not.

Many particle characterisation instruments are calibrated by the manufacturer, but the users cannot directly calibrate their instruments or verify correct calibration of the relevant input parameters. An example is the distance and angle of the detector from the sample in laser diffraction, where calibration by the user requires dismantling of the instruments. In this case users need to assume proper calibration of the input parameters by the manufacturer (an assumption that can be supported by certification of the manufacturer to relevant ISO standards, reputation, experience of the manufacturer, results of instruments from the manufacturer in proficiency tests etc.). CRMs (often particle size CRMs) are subsequently used to verify the assumption of proper calibration by the manufacturer. This means that measurements of these CRMs are important to verify traceability, but do not actually establish traceability.

5.3 Types of RMs

5.3.1 Certified and non-certified RMs/certified and non-certified values

Within the framework of ISO 17034, the term “reference material (RM)” is the superordinate of “certified reference material (CRM)”. There is currently no generally accepted term for non-certified materials. Each RM, certified or not, shall be sufficiently homogeneous and stable for one or more clearly stated measurand to serve as a reference in a measurement. Homogeneity and stability for one measurand does not usually imply homogeneity and stability for other measurands. This means a material may be an RM for one measurand, but not an RM for a different measurand.

Although the term CRM suggests that being certified or not is a status of the complete material, it is more accurate to refer to certified values and non-certified values. Certified values shall come with stated uncertainties and stated traceability, whereas neither uncertainty nor traceability is required

for non-certified values. ISO Guide 30 uses the term “indicative value,” as well as the terms “information value” and “informative value,” to mean “non-certified value”.

CRMs can be used for calibration and assigning values to other materials for that specific certified property value. Non-certified RMs can be used for statistical quality control, such as demonstrating a measurement system is under statistical control, performs as expected and provides reliable results where the trueness of the measurement result is not critical.

Certified values refer to a clearly defined measurand and a material is only a CRM for this specific measurand. For all other measurands, the material is not a CRM and can possibly not even be a non-certified RM.

EXAMPLE The characterisation data obtained from several laboratories for the certified reference material BCR-066, have shown that the Stokes diameter as measured by sedimentation analysis is sufficiently homogeneous to serve as RM for sedimentation analysis. In addition, certified values were assigned for the Stokes diameter. The material is therefore a CRM for the Stokes diameter as determined by sedimentation analysis. No information on homogeneity when using image analysis is available and no value is assigned. The material cannot be used as a CRM or non-certified RM for image analysis or other particle size analysis technique that does not measure Stokes diameter.

The certified value of any CRM shall consist of a clear definition of the certified quantity, a value and its uncertainty and the unit of this value. In addition, it shall be stated whether the uncertainty is a standard or expanded uncertainty. For the latter, the coverage factor or a level of confidence shall be given.

For any non-certified RM, the property for which it has been found sufficiently homogeneous and stable, together with the degree of homogeneity for the measurands for which it should serve as a reference, shall be stated on a document (“product information sheet”). Non-certified RMs may have, but do not necessarily have, assigned values. Their values may have, but do not necessarily have, associated uncertainty estimates.

Note that CRMs can have both certified and non-certified value assignments. Such materials can be used as a CRM for the certified value(s) but can only be used as a non-certified RM for the non-certified value(s).

5.3.2 Primary/secondary/tertiary CRMs

CRMs can also be classified as primary, secondary or tertiary.^[11] In this classification, primary CRMs have values that are directly assigned with traceability to the stated reference. Values for secondary CRMs can then be assigned by calibration with these primary CRMs, and values of tertiary CRMs by calibration with secondary CRMs.

EXAMPLE 1 The distance between the gridlines of a calibration grid are directly measured, making this calibration grid a primary CRM. Values to the distance between gridlines of a second calibration grid can be assigned by calibration with the first grid, making the second grid a secondary CRM. If this CRM is used to assign values to spherical latex particles, the latex particles are the tertiary CRM.

Within a given chain of CRMs, the certified values of the secondary CRMs will always have a higher uncertainty than the certified value of the primary CRM, as the uncertainty of the values of the primary CRM and the uncertainty of the calibration of the secondary with the primary CRM are propagated to obtain the uncertainty of the certified value of the secondary CRM. However, the values of a secondary CRM can have smaller uncertainties than an unrelated primary CRM.

EXAMPLE 2 Calibration grid A was characterised rather imprecisely and the certified average distance between its gridlines has a standard uncertainty of 1,3 %. In contrast, calibration grid B was characterised with a highly accurate method and its certified average distance between gridlines has a standard uncertainty of 0,5 %. This grid B is used to calibrate grid C. Also, here a highly accurate method is used with results of a similar standard measurement uncertainty. The combined standard uncertainty of the certified value of this grid C (0,7 %) is smaller than the standard uncertainty of the primary CRM A.

For the use of a CRM, its position in the calibration hierarchy is irrelevant. What is relevant is the magnitude of the uncertainty of its certified value.

5.3.3 Spherical/non-spherical and monodisperse/polydisperse RMs

The question of sphericity and monodispersity is especially relevant to all RMs for particle characterisation. As discussed above, many particle size analysis methods give the results as physical properties of spheres that give the same measurement signal as the sample in question. This measurement result will only correspond to the actual material property if the material consists of perfectly spherical and solid particles. That means that the equivalent spherical diameters of monodisperse, spherical particles are often the same across different methods.

In practice, there are generally five different types of RMs with respect to degree of polydispersity and shape, as follows.

- a) Spherical, monodisperse materials: These are usually polymer latex materials which, due to the production process, tend to be highly monodisperse and spherical.
- b) Spherical, polydisperse but monomodal materials: Such materials have a broader, but continuous size distribution. Typical examples are materials consisting of glass beads. Due to the production process, their sphericity is less ideal than those of polymer particles.
- c) Monodisperse particles having a regular shape: They can be applied to check accuracy and precision of shape measurement for imaging instruments. They typically consist of regularly shaped particles, for which length and breadth or maximum and minimum Feret diameter have been certified by means of an optical or electron microscopy technique combined with image analysis.
- d) Non-spherical, polydisperse materials: These materials approach many samples that are measured in a laboratory. Their irregular shape and broad size distribution means that results obtained from one method are usually not comparable with results from other methods.
- e) Picket-fence mixtures of monodisperse spherical particles^[2]: These are mixtures of spherical, monodisperse particles which each have individually been characterized for their particle diameter. The idea is to combine the advantage of the highly accurate assessment of particle diameters for spherical, monodisperse materials with a broader distribution. At the time of the writing of this document, such mixtures had to be prepared by the users themselves from individual materials, although bimodal materials exist.

For monodisperse materials, the effect of the basis for the weighting is insignificant, meaning that the number-based average is equal to the volume-based average is equal to the scattered light intensity-based average. This only rarely occurs for routine samples, since real materials always contain some degree of polydispersity but can be the case for RMs.

This means that for a monodisperse material of spherical particles it may be possible under the conditions described in 5.1.4 to apply the certified value for one operationally defined measurand to another quantity.

EXAMPLE 1 The average area-equivalent circular diameter of highly spherical and monodisperse latex particles has been assigned by image analysis. As the particles are spherical, the average area-equivalent circular diameter approaches the geometric diameter of a sphere within the measurement uncertainty. As the material consists of spherical particles, the intensity-weighted hydrodynamic diameter is expected to also approach the geometric diameter of a sphere within the measurement uncertainty (assuming that the particle did not swell or shrink in either of the measurements). Therefore, the diameter as obtained by image analysis is expected to also apply to the diameter as obtained by DLS. Note that shrinking of particles in the electron microscope or layers of water that moves with the particles in DLS makes this assumption invalid.

The use of monodisperse, spherical particles has practical advantages and disadvantages. The main advantage is that a considerable number of spherical or near spherical CRMs are available. Many of them consist of latex particles which are inherently of a high sphericity. In contrast, relatively few non-spherical, polydisperse CRMs for operationally defined measurands exist.

The main disadvantage of monodisperse spherical RMs is that they rarely represent practical samples measured routinely. Real test samples are often non-spherical and have different physicochemical

properties (e.g. density, optical). Measurement of monodisperse, spherical particles therefore possibly does not allow a proper assessment of the performance of a method, laboratory or operator.

EXAMPLE 2 A bias in measuring spherical, monodisperse particles clearly indicates an instrument, laboratory or operator problem. On the other hand, successful measurement of spherical, monodisperse particles does not always show suitability of an instrument or laboratory proficiency for polydisperse, irregularly shaped particles. Hence, accurate measurement of spherical, monodisperse particles is necessary, but not sufficient to ensure quality for irregular, polydisperse materials.

5.4 Porous/dense RMs

The question of porosity is relevant as some sizing methods only probe the external surface of a particle (e.g. image analysis), whereas others probe particle mass (e.g. sedimentation analysis). Pores in a particle change the apparent density of the particle defined as particle mass per total enclosed volume and therefore will result in differences between sizing methods based on mass and surface. The same is true for particles with an inner core and an outer shell of a different composition like coated particles or lipid-based particles for drug delivery. Porosity and internal structure therefore can be a reason why results of different methods disagree even for monodisperse, spherical particles.

6 Practical handling

Whatever material is used, it is important to follow the RM producer's instructions for use. These usually include instructions for homogenisation, sampling and minimum sample size, sample preparation and storage.

7 Requirements for specific uses

7.1 General

For all the uses given below, the chosen material shall at least meet the requirements specified in the definition of RM, i.e. it shall be sufficiently homogeneous and stable to serve as reference in the chosen measurement. For some applications certified values are required, i.e. the chosen material shall be a CRM with certified values for the method in question.

ISO/IEC 17025 points out that the CRMs used should be produced by a competent producer. Producers meeting the requirements of ISO 17034 are considered competent and will ensure that the requirements of homogeneity and stability are met and that valid procedures have been applied to assign any certified values. Accreditation to ISO 17034 is one way to demonstrate adherence to these requirements.

7.2 Instrument verification/design qualification

The goal of this step is to demonstrate that an instrument design in principle can deliver correct results. This activity is performed by the instrument producers and is therefore not part of this document.

7.3 Installation qualification

7.3.1 General

The goal of the installation qualification is to demonstrate that the instrument is in compliance with the manufacturer's specifications, following delivery to a customer.

7.3.2 Type of material

The material used for installation qualification shall be an RM.

The type of material required or if any material is used at all depends on the scope of the installation qualification. Specifications in terms of accuracy and precision can also be checked subsequently within the scope of either the operational or performance, or both, qualifications.

If only sufficient precision is checked, the only requirement is that the material used is sufficiently homogeneous and stable for the time of the installation qualification. This means that in principle any sample of which several identical subsamples can be taken are sufficient.

If also accuracy is demonstrated, an assigned value is necessary. This can be an in-house certified value (see 7.3.3). Therefore, installation qualification is often performed with RMs provided by the producer of the equipment.

7.3.3 Kind of quantity of the assigned value

The assigned value of the RM shall be of the same kind of quantity as the one measured by the instrument

This requirement stems from the fact that only values for the same kind of quantity can be compared. This requirement can be fulfilled in three ways:

- a) The material is spherical or non-spherical, monodisperse or polydisperse and the assigned value is a certified value obtained by the method or instrument as the one to be tested. In this case, the assigned value is by definition of the same kind of quantity. Often, a manufacturer will provide its own RM with specifications for an instrument in question and IQ is successful if the measurement result is within these specifications.
- b) The material is spherical or non-spherical, monodisperse or polydisperse and the assigned value is a certified value where the certified values have been obtained from a network of competent laboratories applying the method in question. This operationally defined certified value also ensures that the instrument delivers the same kind of quantity than the certified value.
- c) The material is a monodisperse, spherical material meeting the criteria of 5.1.4 with certified values assigned by other methods than the instrument in question. Due to the sphericity and monodisperse nature of the material, results of other methods should be applicable to the method in question.

In practice, spherical and non-spherical RMs are suitable for this purpose. However, it is advisable to select polydisperse, irregularly shaped material as these allow a better test of potential misalignments during transport.

7.3.4 Degree of homogeneity

The homogeneity of the material shall be significantly better than the between-run variation.

The variation observed on different subsamples of the material is a combination of the intrinsic method variation and the variation between subsamples. A meaningful assessment of the precision of an instrument is ideally based on replicate measurements of a single specimen. If that is not possible, the standard among subsamples of the RM shall be less than the intrinsic method variance, ideally $< 1/3$.

NOTE The $1/3$ is derived from the error propagation: the variation among subsamples is one uncertainty contribution. If this uncertainty is $< 1/3$ of the intrinsic method variation, it does not significantly contribute to the combined uncertainty anymore and such a material allows a reliable assessment of instrument performance. Measurements of a less homogeneous material largely determine the variation between subsamples.

7.4 Calibration

7.4.1 General

Calibration is an operation to establish a relation between the instrument response signal and the measurand. As calibration has a direct influence on the measurement result, the RM used for calibration is part of the traceability network of the measurement results.

Particle sizing methods that require calibration with particles include for example the electrical sensing zone method and dynamic image analysis, whereas, for example, laser diffraction, static image analysis and dynamic light scattering do not require calibration with particles but traceability is ensured by calibration of the individual input factors (see [Figure 3](#)).

This discussion in this subclause is only applicable to particle CRMs used for calibration of instrument signals. It does not apply to calibration of other input factors like wavelengths of light sources, temperature, etc.

The following requirements for RMs used for calibration shall be met.

7.4.2 Type of material

Only certified RMs (CRMs) shall be used for calibration.

As the RM used for calibration is part of the traceability network of the measurement results, the assigned value shall be certified and traceable and shall have an uncertainty statement. In practice, the certified value and its uncertainty shall be documented in writing. This means, any RM used for calibration shall fulfil all requirements of a CRM.

7.4.3 Traceability of the certified values

The certified values of CRMs shall be traceable to the intended reference.

The CRM used for calibration links the instrument signal to the measurand. The certified values therefore need to be traceable to the intended reference of the measurement result.

7.4.4 Kind of quantity of the certified property

The certified property of CRMs used for calibration shall be the same kind of quantity as the one measured by the instrument calibrated

Relating the instrument signal to a material property via calibration is only meaningful if the certified value of the CRM used is of the same kind of quantity (i.e. measurand) as the intended measurement result. This is particularly important for operationally defined quantities.

EXAMPLE An electrical sensing zone (ESZ) instrument is calibrated with a CRM with certified particle size values obtained by sedimentation analysis. However, unless specific requirements are met, particle size as determined by ESZ is unrelated to particle size as determined by sedimentation analysis because of the difference in kind of quantity. Hence, calibrating with the material certified for sedimentation analysis is in practice calibrating with a random number and cannot establish traceability.

There are in principle two ways to ensure that the certified value of the CRM used for calibration and the measurement result are of the same kind of quantity:

- a) the certified value is operationally defined and was obtained by the method to be calibrated;
- b) the certified value was obtained method other than the measurement method to be calibrated but the material consists of monodisperse spherical particles and the conditions listed in [5.1.4](#) are met.

Option a) is theoretically possible, but rarely used, as it requires a starting point in the traceability chain. It can be possible for an instrument or method to be calibrated with a spherical, monodisperse

CRM. This calibrated instrument or method can then be used to assign a value to non- spherical/ polydisperse CRMs.

In practice, particle CRMs used for calibration usually consist of spherical particles whose diameters have been determined by methods other than the one of current interest. If the conditions listed in [5.1.4](#) are met, the equivalent diameter of the method to be calibrated coincides with the geometrical diameter. In addition, calibration often requires monodisperse particles to simplify the calculations required for calibration.

Therefore, CRMs used for calibration typically consist of monodisperse, spherical particles in a simple matrix.

7.4.5 Uncertainty of the certified value

The uncertainty of the certified values shall be sufficiently small.

The uncertainty of the certified values of the CRM used for calibration contributes to the measurement uncertainty of the measurement result. If only one CRM is used, the measurement uncertainty can never be smaller than the uncertainty of the certified value.

No prescription of the admissible uncertainty can be given. What is “sufficient” depends on the purpose of the measurement result (some measurements will require a higher accuracy than others). The uncertainty of a certified value of a CRM used for calibration also cannot be seen in isolation: If the other contributions to the measurement uncertainty are large, a larger uncertainty for calibration and hence, for the certified value of the CRM used for calibration is acceptable.

7.5 Operational qualification/demonstration of proficiency

7.5.1 General

The goal of OQ is to show that an instrument is able to produce correct results. Demonstration of proficiency aims to show that a laboratory or operator is proficient in the application of a method. Both aspects are related – a proficient operator shall perform the OQ test and demonstration of proficiency is only meaningful with an instrument that has passed OQ. This means that OQ assumes a proficient operator and demonstration of proficiency assumes an instrument having passed OQ. As both tasks are governed by the same principles, they are treated together in this document.

In order to demonstrate proficiency, the value obtained is compared with a reference value. OQ and demonstration of method proficiency requires comparison with a “true” value. This means, contrary to routine quality control discussed in [7.6](#) and which aims to show that the same value as usual is obtained, the goal here is to show that the correct value is obtained. This results in the following requirements for materials used for OQ/demonstration of performance.

7.5.2 Type of material

Only CRMs shall be used for OQ/demonstration of proficiency.

Any comparison requires a value with which to compare. To make meaningful comparisons, both values to be compared shall have measurement uncertainties. In practice, the assigned value and its uncertainty shall be documented in writing. In addition, the certified value needs to be traceable to a defined reference.

This means, any material used for OQ/demonstration of proficiency shall have a document listing a traceable value and its metrological traceability, which is one requirement of a CRM.

7.5.3 Kind of quantity of the certified property

7.5.3.1 General

The certified property of CRMs shall be the same kind of quantity as the one measured by the instrument

Only values of the same kind of quantity can be compared in a meaningful way. This is especially important for operationally defined quantities.

EXAMPLE A comparison of an equivalent hydrodynamic diameter obtained by DLS and a Stokes diameter obtained by centrifugal liquid sedimentation can allow some conclusions about a material but does not allow an assessment of the accuracy of either of the two values.

There are two ways to ensure that the certified value of the CRM used for OQ/demonstration of proficiency and the measurement result are of the same kind of quantity:

- a) the certified value is operationally defined and was obtained by the measurement method to be assessed;
- b) the certified value was obtained by a method other than the measurement method to be assessed but the requirements of [5.1.4](#) are met.

The certified values of materials used for approach a) and b) are of the same metrological quality. Materials of each of the approaches have their specific advantages and disadvantages and users should decide which material fits their requirements best.

7.5.3.2 Certified values of operationally defined properties for non-spherical, polydisperse CRM

If the certified values were obtained by the method to be assessed, they are obviously of the same kind of quantity. However, methods are sometimes implemented in not exactly the same way, for example differences in data evaluation. Therefore, to ensure that a value of an operationally defined property is also applicable to the method to be assessed, users should verify that one of the following two conditions apply.

- a) Is the evaluation algorithm used for value assignment of the CRM the same as the one used in the method to be assessed?

EXAMPLE Certified values obtained by DLS using cumulants analysis are not suitable to assess the performance of DLS using evaluation by inverse Laplace transform (e.g. "CONTIN") algorithm.

- b) Have different instruments been used to assign the value or is the instrument to be assessed the same as was used for the value assignment?

Differences between instruments (general setups, instrument parameters used, specific implementation of the evaluation algorithm etc.) can mean that values obtained by one instrument cannot be reproduced by other instruments. If several instruments were used during value assignment and all give the same value, there is evidence that for the CRM in question this concern is not relevant. The more different instruments have been used, the higher the probability that there is no significant influence of instrument-specific effects.

CRMs that are produced by an instrument manufacturer and characterised by this instrument naturally meet requirement a).

CRMs of this kind are characterised using a network of competent laboratories applying the method in question. Characterisation in a single laboratory is not a valid strategy, as it is impossible to demonstrate the absence of laboratory bias^[9].

One advantage of this approach is that it allows assigning values to materials consisting of irregularly shaped particles with a broad particle size distribution. In addition, it can be applied to any material composition. This means that materials characterised by this approach can be much closer to the

materials typically measured in practice, therefore allowing a better assessment of the accuracy of test results of the samples measured in the laboratory.

Another advantage is that the irregularity and polydispersity of the CRMs (in practice, materials characterised by that approach consist of polydisperse, irregular particles) can allow a more thorough assessment of instrument/laboratory performance than materials consisting of monodisperse, spherical particles.

The major disadvantage of polydisperse, non-spherical materials is that their selection requires more care and information from the users. They need to check whether the definition of the measurand of the certified value includes their method. This can require not only checking which evaluation algorithms were used, but also confirming that a representative variety of instruments have been used to determine the certified value. This information should be readily available from any CRM producer, but obtaining it is an additional step.

A second disadvantage is limited availability because of multiple combinations of methods and materials, relatively few CRMs certified for operationally defined particle properties are available. Users therefore may be unable to find a suitable CRM for their intended application.

A third disadvantage can be that in some cases, uncertainties of certified values are larger than those of spherical CRMs. If uncertainties are excessively large, this can render the CRM unsuitable.

7.5.3.3 Certified values of (near) spherical, monodisperse CRMs

The basic assumption when using spherical, monodisperse CRMs is that if a material is spherical, the equivalent diameter is equal to the geometric diameter. If a material is monodisperse, different ways of weighting (number-weighted, volume-weighted, signal intensity-weighted) will not significantly influence the mean or median value provided that other physical properties (e.g. refractive index) are the same for all particles. For that reason, spherical, monodisperse CRMs, usually characterised by image analysis methods, can often be used for other methods as well. Before using such a material, users should verify the following:

- a) Is the assumption "true diameter equals equivalent diameter" justified? Image analysis usually only measures the particle itself, whereas other methods can measure the particle with the immediate hydrodynamic layer. This layer can become important for very small particles. In addition, especially if particles have been characterised by electron microscopy, the particle can have shrunk when exposed to the electron beam and the high vacuum in the instrument chamber, and as a result, the measured area-equivalent circular diameter can possibly not be the same as when measured in suspension.
- b) Is the distribution sufficiently narrow to make the average indeed independent from the weighting? Otherwise, the often number-based mean can differ from the volume-based or intensity-based mean of other methods.

CRMs of this kind can in principle be characterised by a single laboratory using image analysis as reference method.

One advantage of CRMs of this kind is that users do not have to investigate with which method the CRM was characterised. They also have the possibility to use the CRM for different methods or instruments.

Another advantage is that such CRMs are readily available at many different particle sizes, fitting a wide range of applications.

A major disadvantage is that the sphericity and narrow distribution is possibly not a relevant test on how the method performs for polydisperse, non-spherical particles. Good performance on such materials is required, but not sufficient to show that accurate results can be obtained for irregular-shaped, polydisperse materials.

A fourth disadvantage is the relatively limited chemistry of materials available: Most materials of this kind consist of polymer particles, which can differ significantly (chemistry, density) from the materials measured routinely.

7.5.4 Uncertainty of certified values

The uncertainty of the certified values shall be sufficiently small.

Comparison of the measurement result with the certified value is only possible within the respective uncertainties. If the uncertainty of the certified value is too large, the comparison will not give any useful information.

No prescription of the admissible uncertainty can be given. What is “sufficient” depends on the purpose of the measurement (some measurements will require a higher accuracy than others) and also the other uncertainty contribution. As a rule of thumb, the standard uncertainty of the certified value should ideally be smaller than 1/3 of the standard deviation between results of an interlaboratory comparison test.

NOTE The 1/3 is derived from the error propagation: the measurement uncertainty can be estimated as the standard deviation of an interlaboratory comparison. If the uncertainty of the certified value is $< 1/3$ of this value, then the contribution of the uncertainty of the certified value no longer contributes significantly to the performance assessment.

7.6 Statistical quality control/performance qualification

7.6.1 General

SQC has the purpose to show that a method as implemented by a laboratory performs consistently in time. The emphasis here is to obtain the same result each time and not to ensure that any of the results is actually correct. In practice, this application means establishing control charts as described in the ISO 7870 series. This results in following requirements for RMs used for SQC.

7.6.2 Type of material

The material used for SQC shall be an RM.

The key requirements for materials used for SQC are homogeneity and stability, hence certified and non-certified RMs are suitable.

RMs do not necessarily have assigned values, but even the requirement of sufficient homogeneity and stability for RMs is possibly not straightforward for particle size RMs: a material can be homogeneous for one method and inhomogeneous for others. For example, a polydisperse material homogeneous for DLS is possibly not homogeneous for electron microscopy if the fine fraction which is not seen by DLS is inhomogeneous. It is therefore advisable to establish the homogeneity and stability for the method in question.

If this is not possible because no RM for the method chosen is available, one can often find a material for which the homogeneity and stability has been established with other methods but which, based on the knowledge of the method, should still provide sufficient evidence of sufficient homogeneity and stability.

EXAMPLE A material that is homogeneous for percentiles of the number-based distribution as determined by image analysis has a high probability also to be homogeneous for electrical sensing zone measurements, as both methods determine number-based distributions.

7.6.3 Kind of quantity

The assigned value of the RM shall be of the same kind of quantity as the one measured by the instrument.

The purpose of the SQC is to assess or demonstrate consistency rather than trueness. However, even assessment of consistency requires comparison of the individual results with a target value. As described below, this does not mean that the RM producer shall assign a target value.