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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

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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 mateials* (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 outgrows 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 Sesign qualification

ESZ Electric sensing zone

IQ Installation qualification

OQ Operational qualification

PQ Performance qualification

RM Reference material

SAXS Small angle X-ray scatteringPQ

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.

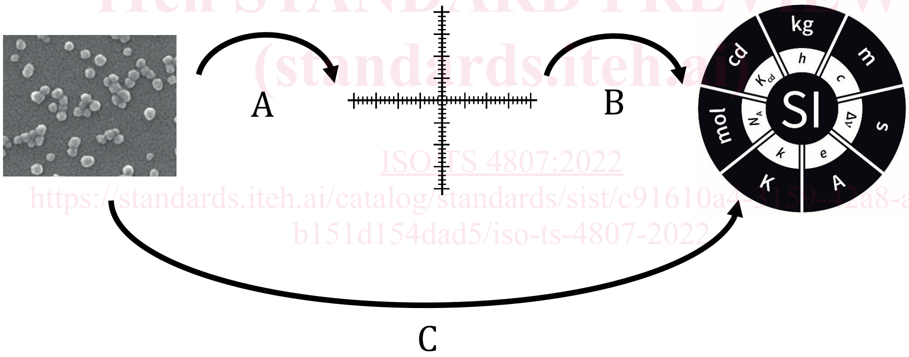
5.2 Metrological traceability of size measurement results

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

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Figure 1 — Traceability chain/network of image analysis