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Preparation of particulate reference materials —

Part 2: Polydisperse spherical particles

Préparation des matériaux de référence à l'état particulaire —

Partie 2: Particules sphériques polydispersées

ICS: 19.120

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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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For an explanation on 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 the following URL: www.iso.org/iso/foreword.html.

The committee responsible for this document is ISO/TC 24/SC 4 Particle characterization

ISO 14411 consists of the following parts, under the general title *Preparation of particulate reference materials*:

— *Part 1: Polydisperse material based on picket fence of monodisperse spherical particles*

— *Part 2: Polydisperse spherical particles*

Part 1 is a Technical Specification.

Introduction

The measurement of particle size distribution can be accomplished by a number of techniques which measure some characteristics of the particle and usually equate this to a circular or spherical equivalent. Each of these techniques measures different properties of an irregular particle and thus has particular requirements for reference materials and method standardization. Often, the methods that are employed for particle size distribution are indirect in nature which rely on measuring some other property and converting this to a particle size distribution by means of equations based on ideal shapes (usually spherical) and sizes to that of the equivalent particle size distribution. Thus these techniques usually require or assume knowledge of some other constant in order to calculate the particle size distribution.

Even methods that do not require size calibration require reference materials for quality control and operation qualification. Such a reference material should be certified for its particle size distribution and the values should be traceable to the SI unit meter. This material allows instrument manufacturers to demonstrate proper calibration of all input factors and hence demonstration that their instrument results are traceable to the SI Unit meter. To achieve this, the reference material should be polydisperse and consist of spherical particles.

The heterogeneity of a particle size distribution poses statistical challenges for particle size analysis and therefore also for the production of reference materials for particle size analysis. This standard therefore describes the production of particulate reference materials consisting of spherical particles.

Preparation of particulate reference materials —

Part 2:
Polydisperse spherical particles

1 Scope

This international standard describes the specifications for spherical polydisperse particulate reference materials with acceptable uncertainty in particle size distribution and describes protocols for their characterisation. These reference materials will be fit for the purpose for their intended use. One potential use of these reference materials is the reliability test of the laser-diffraction and other related methods.

This international standard expresses polydispersity and the related uncertainties in size. Users should take notice that small variations in size may imply large variations in cumulative distribution.

This document describes the necessary requirements of particulate reference materials, which should be used to the reliability test of various types of particle size measurement apparatus. The requirements described in ISO 17034 should be applied for processing, homogeneity and stability assessment as well as for the preparation of certificates, which are not addressed in this international standard.

2 Normative references

The following referenced documents are indispensable for the application 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 13322-1:2004, *Particle size analysis — Image analysis methods — Part 1: Static image analysis methods*

ISO 14488:2007, *Particulate materials — Sampling and sample splitting for the determination of particulate properties*

ISO 14887:2000, *Sample preparation — Dispersing procedures for powders in liquids*

ISO 15901-3:2007, *Pore size distribution and porosity of solid materials by mercury porosimetry and gas adsorption — Part 3: Analysis of micropores by gas adsorption*

ISO 17034:2016, *General requirements for the competence of reference material producers*

3 Terms, definitions and symbols

3.1 Terms and definitions

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

3.1.1
aspect ratio
ratio of minimum Feret diameter to the maximum Feret diameter of a particle

[SOURCE: ISO 26824:2013, 4.5]

3.1.2
pycnometry
a method wherein particle density is obtained from the measured mass of sample with a given calibrated volume

[SOURCE: ISO 26824:2013, 2.4]

3.1.3
density
mass per unit volume of the material – expressed in g/cm^3

[SOURCE: ISO 5755:2012, 3.10]

3.1.4
reference material
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

[SOURCE: ISO 17034:2017, 3.3]

3.1.5
certified reference material
reference material characterized by a metrological valid procedure for one or more specified properties, accompanied by a certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability

[SOURCE: ISO 17034:2017, 3.2]

3.2 Symbols (and abbreviated terms)

For the purposes of this technical report, the following symbols apply.

Symbol	Quantity	Unit	derived unit
k	Coverage factor; numerical factor used as a multiplier of the combined standard uncertainty in order to obtain an expanded uncertainty	-	-
m	total number of uncertainty contributions	-	-
N	Total number of size measurements	-	-
n_t	number of particles counted	-	-
n_{min}	The minimum number of particles that should be counted for predetermined admissible maximum uncertainty with a defined level of confidence	-	-
$q_0(x)$	density distribution by number	m^{-1}	μm^{-1}
$q_3(x)$	density distribution by volume or mass	m^{-1}	μm^{-1}
$Q_0(x)$	cumulative distribution by number	-	-
$Q_3(x)$	cumulative distribution by volume or mass	-	-
r	type of quantity of distribution, r=0: number, r=3: volume or mass	-	-
s	standard deviation	m	µm
s_r	standard deviation of $Q_r(x)$	m	µm
s_g	geometric standard deviation	-	-
U_f	expanded uncertainty	m	µm
$U_{f,p,r}$	expanded uncertainty of p percentile of the cumulative distribution of r type quantity	m	µm
u	uncertainty	m	µm
u_{rep}	uncertainty of mean value due to N times of size measurement	m	µm
$u_{Q_{p,0}}$	uncertainty at p percentile of cumulative number-based size distribution	-	–

Symbol	Quantity	Unit	derived unit
$u_{p,0}$	uncertainty of particle size $x_{p,0}$	m	µµ
$x_{\min,j}$,	minimum particle diameter in size range j	m	µm
$x_{\max,j}$	maximum particle diameter in size range j	m	µm
$x_{p,r}$	particle diameter at p percentile of cumulative distribution of r type quantity, e.g. $x_{90,0}$ = particle diameter at 90 percentile of cumulative number distribution, $x_{50,3}$ = median particle size of cumulative volume or mass distribution	m	µm
$\overline{x}_{p,r}$	average particle diameter of N times = $\overline{x}_{p,r} = \frac{1}{N}\sum_{i=1}^{N} x_{p,r}$	m	µm

4 Requirements for preparing polydisperse

4.1 General description

The material shall be stable and appropriate for its intended use.

4.2 Requirements on the general properties of the material for polydisperse particles

The requirements for the materials are as follows:

1. The aspect ratio or the ellipse ratio of the particles shall be characterized by image analysis. The mean aspect ratio or the mean ellipse ratio shall be 0,80 or above in the size range between $x_{10,3}$ and $x_{90,3}$ of the material.

2. When dispersed in liquid, bleeding of colour or absorbing material should not occur. The material should be chemically and physically homogeneous, and be non-soluble in the dispersant medium.

3. The particle surface should be smooth with a minimum of contaminations or adhesions.

4. The apparent density of the material has to exceed the density of the dispersing liquid for the particles not to float in wet applications.

5. The material should contain the minimum number of fragmented particles suitable for the intended use. The amount of fragmented or non-spherical particles as well as the amount of coarse outliers shall be characterized by image analysis.

6. The material should be easily dispersible in the chosen liquid. No particle agglomerates or flocculation should be detectable after dispersion. It is allowed to support the particle dispersion using dispersing agents or ultrasound.

7. The particles should not be disrupted by ultrasound pressure in dispersant media. The mechanical strength should be as high as possible since the material should be able to withstand a typical dry dispersion procedure without getting crushed. It is not possible to define a concrete value since there are several different dry dispersion procedures not allowing for a reliable theoretical calculation of stress parameters.

8. The material should provide a shelf life of at least 2 years after production without appreciably changing its physical properties. All-important storage conditions have to be known, e.g. necessary UV-/light-protection.

9. The swelling of the material suspended in pure dispersant media should be as low as possible. Swelling shall not exceed a value of 0,8 % referred to the particle diameter in dry condition. The swelling behaviour shall be specified in the sample preparation procedure

10. The size of the particle-liquid interface in dispersion should be negligible compared to the particle diameter.

4.3 Requirements on size distribution for polydisperse particles

The size distribution shall satisfy the following requirements:

1. Distribution shall be monomodal.
2. More than 90 percentile of cumulative mass distribution should be within one decade.
3. The particle size distribution should be approximately represented by a log-normal, at least in the region of 0,2 < $Q_3(x)$ < 0,8.

5 Characterization of polydisperse particles

5.1 Particle size distribution

Particle size distribution is determined by a method that provides traceable results, and satisfies the following requirements:

1. The relative expanded uncertainty (corresponding to a confidence level of 95 %) of the most reliable mass median diameter $x_{50,3}$* shall be not larger than 5 %.
2. The reference data for the range from $Q_{10,\,3}$ to $Q_{90,\,3}$ should be provided with adequate quality.
3. The particle size distribution should be measured by two or more types of different measurement principles. For each measurement data, 95% reliability or 5% uncertainty should be indicated. Using an image analysis method with an electron microscope or optical microscope should be suitable. Other methods such as the electrical sensing zone method with focus flow system can be used as appropriate.

The requirements for the individual methods are:

1. Results shall be traceable to the International System of Units (SI) either by using CRMs with traceable reference values or by appropriate calibration of all relevant input parameters, in case of no direct calibration for length.
2. The methods shall be validated in a way that allows estimation of a measurement uncertainty.
3. For each dataset, the expanded uncertainty on a 95 % confidence level should be given.
4. All results and characteristic values shall be given in terms of a volume-based particle size distribution $Q_3(x)$.
5. The number of particles measured shall be sufficiently high to achieve the required precision.

5.2 Aspect ratio

The aspect ratio shall be measured by a suitable image analysis method measuring at least 1 000 particles by random sampling. Fewer particles would not allow demonstrating fulfilment of the criteria set for the aspect ratio.

5.3 Particle density

The particle density shall be measured by any suitable methods.

