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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

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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 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*.

A list of all parts in the ISO 14411 series can be found on the ISO website.

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

Introduction

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 metre. This material allows instrument manufacturers to demonstrate proper calibration of all input factors and hence demonstrate that their instrument results are traceable to the SI unit metre. 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 document 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 document describes the specifications for spherical polydisperse particulate reference materials with acceptable uncertainty in particle size distribution and describes protocols for their characterization. One potential use of these reference materials is the reliability test of the laser-diffraction instruments and other particle sizing methods.

This document expresses polydispersity and the related uncertainties in size. Small variations in size can imply large variations in cumulative distribution.

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

2 Normative references

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

ISO 17034, *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.

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

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

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

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

[SOURCE: ISO 26824:2013, 4.5, modified.]

3.1.2
apparent density
mass per unit volume of the material

Note 1 to entry: It is expressed in g/cm^3.

[SOURCE: ISO 5755:2012, 3.10]

3.1.3
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

[SOURCE: ISO 17034:2016, 3.3, modified — Notes to entry have been deleted.]

3.1.4
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 17034:2016, 3.2, modified — Notes to entry have been deleted.]

3.2 Symbols

Symbol	Description	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	-	-
$q_0(x)$	density distribution by number	m^{-1}	μm^{-1}
$q_3(x)$	density distribution by volume	m^{-1}	μm^{-1}
$Q_0(x)$	cumulative distribution by number	-	-
$Q_3(x)$	cumulative distribution by volume	-	-
r	type of quantity of distribution, $r = 0$: number, $r = 3$: volume	-	-
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
$u_{Q_{p,0}}$	uncertainty at p percentile of cumulative number-based size distribution	-	–
u_{hom}	uncertainty of the between-unit homogeneity	m	µm
$u_{p,r}$	uncertainty of particle size $x_{p,r}$	m	µm
u_{rep}	uncertainty of mean value due to N times of size measurement	m	µm
u_{stab}	uncertainty of stability of materials	m	µm
$x_{min,j}$	minimum particle size in size range j	m	µm

$x_{\max, j}$	maximum particle size in size range j	m	µm
$x_{p, r}$	particle size at p percentile of cumulative distribution of r type quantity, e.g. $x_{90,0}$ = particle size at 90 percentile of cumulative number-based size distribution, $x_{50,3}$ = median particle size of cumulative volume-based size distribution	m	µm
$\bar{x}_{p, r}$	average particle size of N times of size measurement $= \bar{x}_{p, r} = \frac{1}{N}\sum_{i=1}^{N} x_{p, r}$	m	µm

4 Requirements for preparing polydisperse particles

4.1 General description of a project for the production of a reference material

According to ISO 17034, "reference material production" is a process that covers all steps from project planning and design to the final distribution. In particular, it consists of the following steps.

a) Production planning: In this step, the desired characteristics of the material are established. It is decided whether a certified reference material (CRM) or a non-certified material should be produced, which material should be used, how much should be produced, desired measurand values and their uncertainties, required traceability and ways to achieve it as well as the planning of the homogeneity and stability assessment, material characterization and storage and distribution.

b) Processing and production control: This is the set of physical processes that converts the (often bulk) material into different candidate reference material units fit for distribution. Care shall be taken to ensure sufficient homogenization, avoid contamination and ensure proper packaging for long-term stability. Especially in the case of particulate reference materials, de-mixing during filling shall be avoided.

c) Assessment of homogeneity and stability: The between-unit variation of the values to be certified is assessed, usually by testing a representative number of units. Based on this assessment, an uncertainty of homogeneity (u_{hom}) is estimated. In addition, the minimum sample intake, i.e. the minimum amount of sample that is representative for the whole unit is established.

   The stability of the material under transport and storage conditions is assessed. Based on this assessment, uncertainty contributions of stability (u_{stab}) is estimated.

d) Characterization: It is the process to assess the assigned values of a CRM. Several characterization approaches are listed in ISO 17034 and ISO Guide 35. It is crucial that an approach is chosen that can ensure the envisaged traceability of the certified values.

e) Assignment of property values and their uncertainties: Based on the results of the characterization and homogeneity and stability assessment, the assigned values and their uncertainty are established.

f) Preparation of RM documents: The assigned property values and their uncertainties of CRMs are stated on the RM certificate, together with information on the intended use, instruction for use (which may include e.g. dispersion protocols).

g) Distribution and stability monitoring: If deemed necessary based on the material used. The stability of the material is assessed on regular intervals to detect changes that would make the certified values invalid.

Many of the above steps can be outsourced to third parties. However, production planning, selection of subcontractors, assignment of property values and their uncertainties, authorization of property values and their uncertainties and authorization of RM documents cannot be outsourced.

This document covers steps d) and e) of the above description, i.e. a part of the production planning (requirement of material characteristics) and characterization and property value assignment. All other steps shall be performed in accordance with ISO 17034. Guidance on the implementation of ISO 17034 is given in ISO Guide 35.

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

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

The requirements for the materials are as follows:

a) 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 0,90 or above in the size range between $x_{10,3}$ and $x_{90,3}$ of the material.

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

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

d) The apparent density of the material shall exceed the density of the dispersing liquid for the particles not to float in wet applications.

e) The number of fragmented particles contained in the material should be as small as possible and suitable for the intended use. The amount of fragmented or non-spherical particles, as well as the number of coarse outliers, shall be characterized by image analysis.

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

g) The particles should not break due to ultrasound pressure used for dispersion in liquid 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.

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

i) 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 % with reference to the particle size in dry conditions. The swelling behaviour shall be specified in the sample preparation procedure.

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

4.3 Size distribution for polydisperse particles

The size distribution for polydisperse particles shall be monomodal.

More than 90 percentile of cumulative volume-based size distribution should be within one decade.

The particle size distribution should be approximately represented by a log-normal distribution, at least in the region of $0,2 < Q_3(x) < 0,8$.

