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## Preparation of particulate reference materials —

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

*Préparation des matériaux de référence à l'état particulaire —  
Partie 1: Matériaux polydispersés composés d'un ensemble de  
particules sphériques monodispersées*

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# Contents

Page

|                                                                                                                                                                   |           |
|-------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------|
| <b>Foreword</b>                                                                                                                                                   | <b>iv</b> |
| <b>Introduction</b>                                                                                                                                               | <b>v</b>  |
| <b>1 Scope</b>                                                                                                                                                    | <b>1</b>  |
| <b>2 Normative references</b>                                                                                                                                     | <b>1</b>  |
| <b>3 Terms, definitions and symbols</b>                                                                                                                           | <b>1</b>  |
| 3.1 Terms and definitions                                                                                                                                         | 1         |
| 3.2 Symbols                                                                                                                                                       | 2         |
| <b>4 Material requirements for preparing the individual monodisperse fractions</b>                                                                                | <b>3</b>  |
| 4.1 General description                                                                                                                                           | 3         |
| 4.2 Requirements on the general properties of the material for individual pickets                                                                                 | 3         |
| <b>5 Characterization of the individual monodisperse fractions</b>                                                                                                | <b>4</b>  |
| 5.1 Particle size distribution                                                                                                                                    | 4         |
| 5.2 Aspect ratio                                                                                                                                                  | 5         |
| 5.3 Density                                                                                                                                                       | 5         |
| 5.4 Refractive index                                                                                                                                              | 5         |
| <b>6 Preparation of picket-fence distributions</b>                                                                                                                | <b>5</b>  |
| 6.1 General                                                                                                                                                       | 5         |
| 6.2 Preparation of individual pickets                                                                                                                             | 6         |
| 6.2.1 General                                                                                                                                                     | 6         |
| 6.2.2 Preparation of suspensions from dry powders                                                                                                                 | 6         |
| 6.2.3 Determination of the particle mass fraction of suspensions                                                                                                  | 6         |
| 6.3 Preparation of a picket fence distribution                                                                                                                    | 6         |
| 6.3.1 General                                                                                                                                                     | 6         |
| 6.3.2 Preparation from dry powders                                                                                                                                | 6         |
| 6.3.3 Preparation from suspensions                                                                                                                                | 7         |
| <b>7 Estimation of uncertainties</b>                                                                                                                              | <b>7</b>  |
| 7.1 General                                                                                                                                                       | 7         |
| 7.2 Uncertainty of a volume-based size distribution due to limited number of particles counted                                                                    | 7         |
| 7.3 Uncertainty of a number-based size distribution                                                                                                               | 8         |
| 7.4 Picket-fence distributions composed of more than two kinds of quasi-monodisperse particles                                                                    | 9         |
| 7.5 Uncertainty of a count base size distribution due to various number fraction                                                                                  | 10        |
| 7.6 Uncertainty of a mass base size distribution due to various mass fractions                                                                                    | 10        |
| 7.7 Uncertainty estimation based on the data before or after the mixing process                                                                                   | 10        |
| 7.8 Uncertainty due to microscopic scale measurement                                                                                                              | 12        |
| 7.9 Uncertainty due to surrounding particles in microscopic measurement                                                                                           | 12        |
| 7.10 Other uncertainty contributions                                                                                                                              | 12        |
| 7.11 Combined uncertainty                                                                                                                                         | 12        |
| <b>Annex A (informative) Picket-fence distributions composed of more than two kinds of quasi-monodisperse particles</b>                                           | <b>13</b> |
| <b>Annex B (informative) Example of reliability calculation for a mass-based cumulative size distribution transformed from the number-based size distribution</b> | <b>16</b> |
| <b>Annex C (informative) Example of uncertainty estimation due to mixture fraction and sample size</b>                                                            | <b>19</b> |
| <b>Annex D (informative) Uncertainty estimation of various cases</b>                                                                                              | <b>21</b> |
| <b>Bibliography</b>                                                                                                                                               | <b>28</b> |

## 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](http://www.iso.org/directives)).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on 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 the following URL: [www.iso.org/iso/foreword.html](http://www.iso.org/iso/foreword.html).

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

## Introduction

The measurement of the particle size distribution can be accomplished by a number of techniques which measure some 1-D characteristic of the particle and usually equate this to an equivalent size assuming ideal shapes (usually spherical). Thus, these techniques usually require or assume knowledge of some other constant in order to calculate the particle size distribution. Each of these techniques measures different properties which makes the equivalent particle size a method-defined measurand. Comparability of results therefore requires application of the same methods, which in turn requires standardization.

This unsatisfactory situation of fundamental lack of comparability could be improved by a better understanding of the effects influencing the various methods. Since the sample material represents the link between the different methods, it is of central importance that it should meet as many physical assumptions of the considered methods as possible. A feasible approach is mixing known amounts of spherical, monodisperse particle fractions to create a polydisperse mixture (“picket fence distribution”).

The individual particles should be spherical, as many sizing methods assume the particles to be spherical. Using particles that are in fact spherical fulfils this assumption, so the results of the various methods should be the same as far as the particle shape is concerned. A further advantage of spherical particles is that their size can be described by a single parameter only, the particle diameter.

The individual fractions of the mixture need to be monodisperse, as only then it is possible to trace the particle diameter back to the standard meter with an acceptable uncertainty and to get mixtures of theoretically known particle size distributions in the end.

These materials should be used as follows.

The monodisperse particle fractions can be used to demonstrate equivalence of results with these ideal particles. If a method gives deviating results, the method is not yet fully understood and further investigation of the deviation is needed. The polydisperse mixtures can be used to challenge measurement methods to see what the output is. Final outcome should be a comprehensive understanding of the methods including particle dispersion, particle transport, physical principle and evaluation leading to better comparability of results. The approach described in this document is based on Reference [22] and Reference [23].

A second approach is developing a theoretical framework for more accurate measurement of particle size distributions. Also, this approach is fundamentally limited to spherical particles of equal density, to be applicable to different methods.

This document describes preparation protocols of picket fence distributions of spherical, quasi-monodisperse particulate reference materials.

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# Preparation of particulate reference materials —

## Part 1:

## Polydisperse material based on picket fence of monodisperse spherical particles

### 1 Scope

This document describes the preparation of polydisperse spherical particles based on a picket fence of quasi-monodisperse reference materials, the characterization of its monodisperse components with acceptable uncertainty and the estimation of the uncertainty of the mixture of these particles. This type of material is normally suitable for all particle characterization methods within the appropriate limits of the techniques. An example of using these reference materials in a reliability calculation for a mass-based cumulative size distribution is provided.

This document limits itself to the technical specificities of preparation beyond the general requirements for certified and non-certified reference materials as described in ISO Guide 30, ISO Guide 31, ISO Guide 35 and ISO 17034.

### 2 Normative references

There are no normative references in this document.

### 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:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

##### 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

##### **pycnometry**

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****apparent particle density**

particle mass in the dry status divided by the volume it would occupy including all pores, closed or open, and surface fissures

[SOURCE: ISO 13317-4:2014, 3.1]

**3.1.4****hydrostatic balance**

method to measure particle density based on particle dynamic sedimentation velocity with known fluid density and viscosity condition

**3.1.5****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 Guide 30:2015]

**3.1.6****certified reference material**

*reference material* (3.1.5) characterized by a metrologically 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 Guide 30:2015]

**3.1.7****nominal value**

designated diameter in terms of a target value in a given specification

Note 1 to entry: The nominal value is the target diameter for an individual picket as calculated from the upper and lower size of the *picket fence distribution* (3.1.8), the number of pickets and the requirement of equal spacing of pickets on a lognormal scale. Actual values may differ from the nominal ones due to the availability of suitable material

**3.1.8****picket fence distribution**

mixture of several monodisperse particle fractions (pickets)

**3.2 Symbols**

| Symbol           | Quantity                                                                                                                                                           | Unit  | Derived unit  |
|------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------|---------------|
| $\alpha_i$       | Particle mass fraction for the suspension of picket <i>i</i>                                                                                                       | kg/kg | mg/kg         |
| $\alpha_i^{(0)}$ | Particle number fraction for the suspension of picket <i>i</i>                                                                                                     | —     | —             |
| $\Delta x_i$     | Particle size interval in size range <i>i</i>                                                                                                                      | m     | $\mu\text{m}$ |
| $\delta$         | Uncertainty of the parameter given in the index<br>NOTE: In other fields of measurement science and ISO/IEC Guide 98-3 (GUM), the symbol <i>u</i> is used instead. | —     | —             |
| $\delta x_i$     | Uncertainty of the size $x_i$                                                                                                                                      | m     | $\mu\text{m}$ |
| <i>g</i>         | Parameter defined by <a href="#">Formula (A.1)</a>                                                                                                                 | —     | —             |
| <i>M</i>         | Parameter used in <a href="#">Formula (A.5)</a>                                                                                                                    | —     | —             |
| $m_b$            | Mass of the vessel for the dry mass determination                                                                                                                  | kg    | mg            |
| $m_d$            | Dry mass (vessel and particles) in the dry mass determination                                                                                                      | kg    | mg            |
| $m_i$            | Mass of suspension <i>i</i> used for the preparation of the picket-fence distribution                                                                              | kg    | mg            |

| Symbol                       | Quantity                                                                                                                                                                                             | Unit               | Derived unit       |
|------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------|--------------------|
| $m_{l,i}$                    | Mass of solvent of picket $i$                                                                                                                                                                        | kg                 | mg                 |
| $m_{p,i}$                    | Mass of particles of picket $i$ in suspension                                                                                                                                                        | kg                 | mg                 |
| $m_s$                        | Mass of suspension used for the dry mass determination                                                                                                                                               | kg                 | mg                 |
| $m_{x,i}$                    | Mass of the particles of picket $i$ in the final picket fence distribution                                                                                                                           | kg                 | mg                 |
| $N, N_i$                     | Total number of particles and particle number of picket $i$                                                                                                                                          | —                  | —                  |
| $n_i$                        | Number of particles in size range $i$                                                                                                                                                                | —                  | —                  |
| $n_{\text{picket}}$          | Total number of pickets                                                                                                                                                                              | —                  | —                  |
| $p$                          | Total number of uncertainty factors                                                                                                                                                                  | —                  | —                  |
| $q_0(x)$                     | Density distribution by number                                                                                                                                                                       | $\text{m}^{-1}$    | $\mu\text{m}^{-1}$ |
| $q_3(x)$                     | Density distribution by volume or mass                                                                                                                                                               | $\text{m}^{-1}$    | $\mu\text{m}^{-1}$ |
| $Q_0(x)$                     | Cumulative distribution by number                                                                                                                                                                    | —                  | —                  |
| $Q_{0,i}^*, Q_{3,i}^*$       | True cumulative distribution by number and mass with logarithmic abscissa                                                                                                                            | —                  | —                  |
| $Q_3(x)$                     | Cumulative distribution by volume or mass                                                                                                                                                            | —                  | —                  |
| $\rho_i$                     | Particle density of picket $i$                                                                                                                                                                       | $\text{kg m}^{-3}$ | $\text{g cm}^{-3}$ |
| $s$                          | Standard deviation of the particle size distribution                                                                                                                                                 | m                  | $\mu\text{m}$      |
| $s_g$                        | Geometric standard deviation                                                                                                                                                                         | —                  | —                  |
| $u$                          | Parameter used in <a href="#">Formula (6)</a> to give confidence level, $u = 1,96$ for 95 % probability reliability<br>NOTE This corresponds to the coverage factor $k$ in ISO/IEC Guide 98-3 (GUM). | —                  | —                  |
| $x, x_i$                     | Particle diameter and particle diameter in size range $i$                                                                                                                                            | m                  | $\mu\text{m}$      |
| $x_l$                        | Diameter of the smallest picket                                                                                                                                                                      | m                  | $\mu\text{m}$      |
| $x_u$                        | Diameter of the largest picket                                                                                                                                                                       | m                  | $\mu\text{m}$      |
| $x_{50,0,i}$<br>$x_{50,3,i}$ | Median diameter of particle $i$ based on number and mass                                                                                                                                             | m                  | $\mu\text{m}$      |
| $x_{50,3}$                   | Median particle size of cumulative volume or mass distribution                                                                                                                                       | m                  | $\mu\text{m}$      |
| $x_{50,3}^*$                 | Most reliable median particle size of a cumulative volume or mass distribution with logarithmic abscissa                                                                                             | m                  | $\mu\text{m}$      |

## 4 Material requirements for preparing the individual monodisperse fractions

### 4.1 General description

The material of the individual pickets shall be suitable for particle size measurement using image analysis methods within dry or aqueous environment.

### 4.2 Requirements on the general properties of the material for individual pickets

The material of the individual pickets shall meet the following requirements.

- a) The particles shall be spherical without significant macroscopic concavities, outgrowths or pores.

The aspect ratio of all particles shall exceed a value of 0,95. A typical mean aspect ratio should be 0,97. Alternatively, the ellipse ratio shall exceed 0,95, a typical value should be 0,97 or above.