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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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Foreword

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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.

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
α_i	Particle mass fraction for the suspension of picket *i*	kg/kg	mg/kg
$\alpha_i^{(0)}$	Particle number fraction for the suspension of picket *i*	—	—
Δx_i	Particle size interval in size range *i*	m	µm
δ	Uncertainty of the parameter given in the index NOTE: In other fields of measurement science and ISO/IEC Guide 98–3 (GUM), the symbol *u* is used instead.	—	—
δx_i	Uncertainty of the size x_i	m	µm
g	Parameter defined by Formula (A.1)	—	—
M	Parameter used in Formula (A.5)	—	—
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* 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_{picket}	Total number of pickets	—	—
p	Total number of uncertainty factors	—	—
$q_0(x)$	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	—	—
$Q_{0,i}^*$,$Q_{3,i}^*$	True cumulative distribution by number and mass with logarithmic abscissa	—	—
$Q_3(x)$	Cumulative distribution by volume or mass	—	—
ρ_i	Particle density of picket i	kg m^{-3}	g cm^{-3}
s	Standard deviation of the particle size distribution	m	µm
s_g	Geometric standard deviation	—	—
u	Parameter used in Formula (6) to give confidence level, u = 1,96 for 95 % probability reliability 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	µm
x_l	Diameter of the smallest picket	m	µm
x_u	Diameter of the largest picket	m	µm
$x_{50,0,i}$ $x_{50,3,i}$	Median diameter of particle i based on number and mass	m	µm
$x_{50,3}$	Median particle size of cumulative volume or mass distribution	m	µm
$x^*_{50,3}$	Most reliable median particle size of a cumulative volume or mass distribution with logarithmic abscissa	m	µm

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.

b) When dispersed in pure water, no colour bleeding is allowed. The optical homogeneity of the material is very important to be as uniform as possible. This applies for the particles within one monodisperse fraction, as well as for a comparison of the particles of two different monodisperse fractions.

c) The particle surface should be smooth without any contaminations or adhesions.

d) The apparent density of the material has to exceed the density of the dispersing liquid for the particles not to float in wet applications. Furthermore, the apparent density should not be too high for avoiding sedimentation effects. Therefore, a value within the range above 1 000 kg/m^3 and smaller than 2 500 kg/m^3 seems to be optimal for aqueous applications. Particles of higher densities can be used if a liquid with higher density or viscosity is used.

   The apparent density of the material is important to be as constant as possible for different particle sizes. Variations of ±0,5 % with respect to the mean value of the apparent density may be accepted.

e) The material should not contain any kind of fragmented particles or coarse outliers, e.g. agglomerates. Any other material coming in contact with the particles should not be dyed by adhering dust or abrasion.

f) The particles shall have a high chemical stability and be non-soluble in dispersant media.

g) 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.

h) 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. Nevertheless, 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.

i) The particles should not agglomerate under normal environmental conditions. Their electrostatic behaviour should allow for using them, e.g. on a vibratory chute without adhering to the chute itself.

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

k) The swelling of the material suspended in pure dispersant media should be as low as possible. In any case, it should 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

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

5 Characterization of the individual monodisperse fractions

5.1 Particle size distribution

Particle size should be determined by a method that provides traceable results. The requirements for the individual methods are given below.

— Results shall be traceable to the International System of Units (SI) either by using CRMs with traceable reference values or by being calibrationless.

— The methods shall be validated in a way that allows estimation of a measurement uncertainty.

— Uncertainty estimates for the various size fractions are available.

— All results and characteristic values have to be given in terms of a volume-based particle size distribution, $Q_3(x)$.

It should be ensured that the measured particle size distributions do not overlap. This is achieved if each distribution meets the following requirements:

a) The distribution width given in terms of the ratio x_{90}/x_{10} should be 1,12 or smaller.

b) The actual mass median particle diameter of each mono-disperse fraction should not deviate by more than 4 % from the nominal diameter calculated from the lognormal distribution.

c) The uncertainty of the actual mass median particle diameter, calculated from traceable results from a suitable optical method, $x_{50,3}$, should be in the range of 0,99 $x_{50,3}$* to 1,01 $x_{50,3}$* with 95 % reliability where $x_{50,3}$* is the most reliable mass median diameter. Larger uncertainties will result in larger uncertainty for the final distribution.

It is possible to compensate for not meeting one of the above criteria by setting stricter limits on others. For example, a higher ratio x_{90}/x_{10} is permissible if the actual mean particle diameters deviate less than 4 % from the nominal ones. Regardless of failing to meet an individual requirement, the basic requirement of non-overlapping distributions shall be met.

5.2 Aspect ratio

The aspect ratio should be measured by a suitable optical method measuring at least 10 000 particles by random sampling. Fewer particles would not allow demonstrating fulfilment of the criteria set for the aspect ratio.

5.3 Density

The particle density should be measured by pycnometry or hydrostatic balance.

5.4 Refractive index

The refractive index shall be measured by any suitable method, e.g. by the liquid immersion method.

6 Preparation of picket-fence distributions

6.1 General

A picket-fence distribution should include at least one complete decade in particle size.

A picket-fence distribution should contain an uneven number of mono-disperse fractions and not less than 7 different mono-disperse particle fractions within a range of one decade of particle size. All fractions should be equally spaced on a logarithmic scale.

The nominal diameter x_i of picket i is calculated from the lower diameter, x_{l}, the upper diameter, x_{u}, and the total number of pickets, n_{picket}, using Formula (1):

$$x_i = x_{\mathrm{l}} \cdot 10^{(i-1)\frac{\log(x_{\mathrm{u}})-\log(x_{\mathrm{l}})}{n_{\mathrm{picket}}-1}} \tag{1}$$

All pickets should consist of the same material to minimize differences in density or optical properties.

6.2 Preparation of individual pickets

6.2.1 General

In most cases, it will be impossible to weigh the particles as dry powders due to the intrinsic uncertainty of weighing. In addition, many particles are distributed as suspensions, so drying and weighing increases the risk of agglomeration.

NOTE The uncertainty of the balance alone for weighing 1 mg with an analytical balance (display 0,000 00 g) is about 15 %. Using a microbalance, this uncertainty is typically reduced to less than 0,5 %, but static influences (e.g. incomplete transfer from the weighing boat to the final vessel) increase this uncertainty.

If direct weighing is possible, 6.2.2 and 6.2.3 can be skipped. For particles available as suspensions, 6.2.2 can be skipped.

6.2.2 Preparation of suspensions from dry powders

Weigh an appropriate amount of the dry powder ($m_{p,i}$) into a known amount ($m_{l,i}$) of the chosen solvent and homogenize according to the instruction of the particle producer. The particle mass fraction of picket *i* is calculated as Formula (2):

$$\alpha_i = \frac{m_{p,i}}{m_{p,i} + m_{l,i}} \tag{2}$$

6.2.3 Determination of the particle mass fraction of suspensions

For particles available as suspensions, the particle mass fraction shall be determined. Follow the following steps.

a) Clean and dry a vessel and determine its mass, m_b.

b) Weigh an appropriate amount of the suspension (m_s) into the vessel. The amount taken should not contain less than 500 mg of solids.

c) Slowly evaporate the solvent to achieve constant mass (m_d). The final drying temperature should be high enough to evaporate all solvent, but should not cause shrinkage of particles. If in doubt, contact the provider of the suspension for appropriate drying conditions. Constant mass is achieved when subsequent weighings differ by less than 1 mg.

d) Determine the particle mass fraction of picket *i* in the suspension as given in Formula (3):

$$\alpha_i = \frac{m_d - m_b}{m_s - m_b} \tag{3}$$

6.3 Preparation of a picket fence distribution

6.3.1 General

Picket-fence distributions shall be prepared as “one-shot” materials directly from gravimetric weighing of the individual pickets to avoid errors from subsampling of a homogenized sample. The uncertainties of the weighing should be less than 0,1 % of the weighed mass.

6.3.2 Preparation from dry powders

Weigh equal masses of the dry powders for each picket, $m_{x,i}$, into a vessel and homogenize.

NOTE This is the simplest approach for the preparation of a picket fence distribution, but the accuracy of weighing sets a limit to the use of this approach.

6.3.3 Preparation from suspensions

Prepare a picket fence distribution from *i* individual suspension as follows.

a) Determine the required mass, m_i, of each suspension so that $\alpha_i \cdot m_i$ is constant for all pickets.

b) Homogenize each suspension and weigh the amount calculated in a) into a vessel. Avoid weighing less than 0,5 g to minimize weighing errors.

c) The mass of the particles of picket *i* ($m_{x,i}$) is calculated as given in Formula (4):

$$m_{x,i} = \alpha_i \cdot m_i \tag{4}$$

The combined influence of the subsampling process can be assessed by preparing several suspensions from the same set of pickets and subject them to measurement.

7 Estimation of uncertainties

7.1 General

In order to represent particle size distribution of the reference material, the uncertainty of the picket-fence distribution shall be indicated. The following uncertainty contributions shall be included in the combined uncertainty of the particle size distribution.

7.2 Uncertainty of a volume-based size distribution due to limited number of particles counted

The detailed derivation of the formulae below is given in D.1.

For the general case, the maximum uncertainty of the cumulative distribution for the size range *i* is as given in Formula (5) to Formula (9):

$$\delta Q_{3,i} \le \left|\frac{\partial Q_{3,i}}{\partial Q_{0,1}}\delta Q_{0,1}\right| + \left|\frac{\partial Q_{3,i}}{\partial Q_{0,2}}\delta Q_{0,2}\right| + \cdots + \left|\frac{\partial Q_{3,i}}{\partial Q_{0,n}}\delta Q_{0,n}\right| = \delta Q_{3,i,N} \tag{5}$$

$$i>j \quad \frac{\partial Q_{3,i}}{\partial Q_{0,j}} = \frac{\left(x_j^{3} - x_{j+1}^{3}\right)\left(g_n - g_i\right)}{g_n^{2}} \tag{6}$$

$$i=j \quad \frac{\partial Q_{3,i}}{\partial Q_{0,j}} = \frac{x_j^{3} g_n - \left(x_j^{3} - x_{j+1}^{3}\right) g_i}{g_n^{2}} \tag{7}$$

$$i<j \quad \frac{\partial Q_{3,i}}{\partial Q_{0,j}} = \frac{0 - \left(x_j^{3} - x_{j+1}^{3}\right) g_i}{g_n^{2}} \tag{8}$$

$$i<j, j=n \quad \frac{\partial Q_{3,i}}{\partial Q_{0,j}} = \frac{0 - x_j^{3} g_i}{g_n^{2}} \tag{9}$$

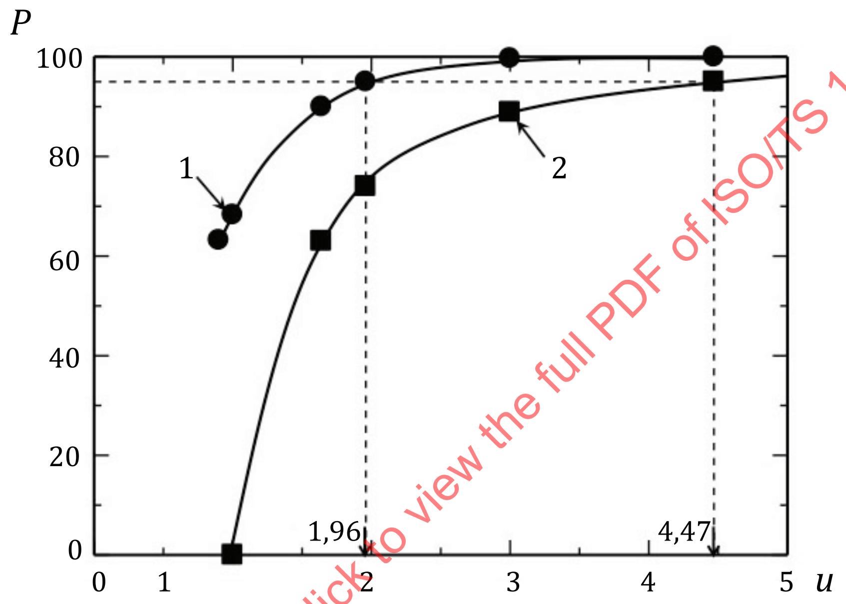
By use of Tschebyscheff theory, the uncertainty at 95 % confidence (= 5 % uncertainty) for the general distribution is determined by References [24] and [25]. The relation confidence reliability level and parameter u for general and normal distributions is shown in Figure 1. The parameter value of u is 4,47 for general distribution. The derivation of the 4,47 for the general distribution is indicated in Annex D.

$$\delta Q_{0,i,N} = 4,47 \sqrt{\frac{Q_{0,i}\left(1 - Q_{0,i}\right)}{N}} \tag{10}$$

The uncertainty δx_i for the cumulative mass distribution in size range i is calculated by Formula (11):

$$\delta x_{i,N} = \frac{\delta Q_{3,i,N}}{q_{3,i}} \tag{11}$$

By use of Formulae (D.9), (D.10), (D.18) and (D.19), the uncertainty of the particle diameter for cumulative mass distribution is fully determined.



Key

1 normal distribution

2 general distribution

P reliability level, expressed in per cent

u parameter, dimensionless

Figure 1 — Relation between reliability level and parameter u

7.3 Uncertainty of a number-based size distribution

The uncertainty δx_i for the cumulative number distribution in size range i is calculated by Formula (12):

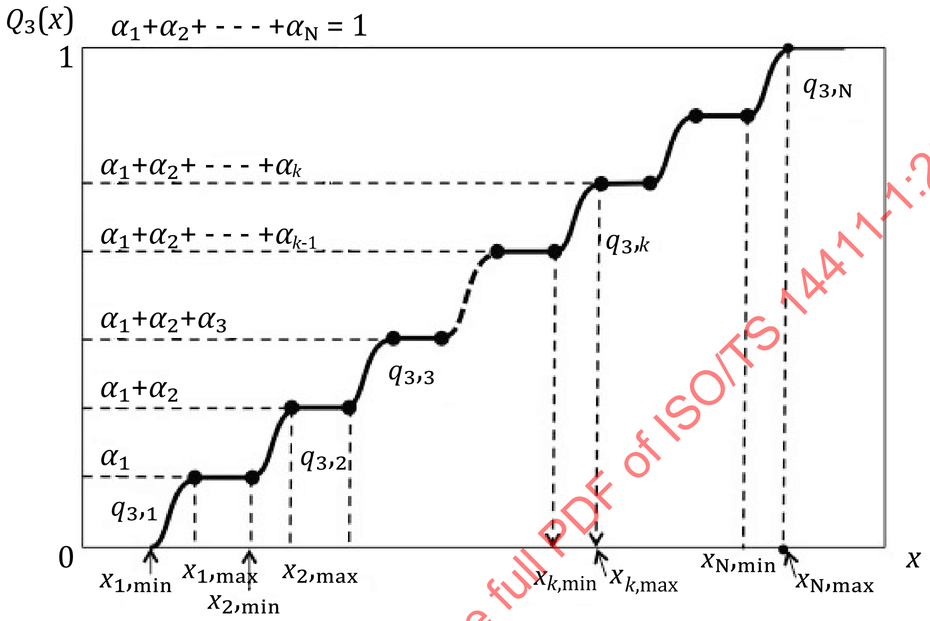
$$\delta x_{i,N} = \frac{\delta Q_{0,i,N}}{q_{0,i}} \tag{12}$$

where $\delta Q_{0,i,N}$ is calculated by Formula (D.18).

7.4 Picket-fence distributions composed of more than two kinds of quasi-monodisperse particles

A picket-fence distribution composed of three-kinds of quasi-monodisperse particles is indicated in Annex A.

The cumulative distribution composed of m kinds of quasi-monodisperse particles is shown in Figure 2.



Key

x particle diameter, expressed in micrometers

$Q_3(x)$ mass base cumulative distribution, dimensionless

$q_{3,k}$ mass base distribution density, expressed in reciprocal of micrometers

α_k mass fraction of particles included in picket k, dimensionless

Figure 2 — Particle size distribution composed of more than two kinds of quasi-monodisperse particles

The density distribution by mass is given as Formula (13):

$$q_3(x) = \sum_{j=1}^{m} \alpha_j \, q_{3,j}(x) \tag{13}$$

where α_j is the mass fraction of the sample j.

For the cumulative distribution by mass, the size x is in the size range of sample k, Formula (14) is satisfied.

$$Q_3(x) = \alpha_1 + \alpha_2 + \cdots + \alpha_{k-1} + \alpha_k \int_{x_{k,\min}}^{x} q_{3,k}(x)\,dx \tag{14}$$

7.5 Uncertainty of a count base size distribution due to various number fraction

The number fraction of *i* kind of particles is calculated as given in Formula (15):

$$\alpha_i{}^{(0)} = \frac{N_i}{(N_1 + \cdots + N_m)} \tag{15}$$

The symbol N_i indicates particle number of picket *i*.

The uncertainty due to *m* kinds of number fraction is calculated by Formula (16):

$$\delta Q_{0,i,\alpha} = \sqrt{\left(\frac{\partial Q_0}{\partial \alpha_1{}^{(0)}} \delta \alpha_1{}^{(0)}\right)^2 + \cdots + \left(\frac{\partial Q_0}{\partial \alpha_m{}^{(0)}} \delta \alpha_m{}^{(0)}\right)^2} \tag{16}$$

7.6 Uncertainty of a mass base size distribution due to various mass fractions

The mass fraction of *i* kinds of particles is calculated as Formula (17):

$$\alpha_i = \frac{m_i}{(m_1 + \cdots + m_m)} \tag{17}$$

The symbol m_i indicates particle mass of picket *i*.

The uncertainty due to *m* kinds of mass fraction is calculated by Formula (18):

$$\delta Q_{3,i,\alpha} = \sqrt{\left(\frac{\partial Q_3}{\partial \alpha_1} \delta \alpha_1\right)^2 + \cdots + \left(\frac{\partial Q_3}{\partial \alpha_m} \delta \alpha_m\right)^2} \tag{18}$$

The calculation method of the terms in Formula (16) and Formula (17) is shown in D.2 and D.3.

Examples of uncertainty estimation to prepare sample preparation are shown in Annex C.

7.7 Uncertainty estimation based on the data before or after the mixing process

7.7.1 General

The uncertainty of *m* kinds of quasi-monodisperse particles can be estimated by use of the data before or after the mixing process. Both methods are acceptable in calculating the uncertainty. For the uncertainty estimation based on the data after mixing process, it is not necessary to include the uncertainty due to the mixture fraction, but the total sample size should be large enough. For the uncertainty estimation based on the data before mixing process, it is necessary to include the uncertainty contributions due to the mixture and the sample size of each size distribution. The calculation method of the uncertainty based on the data before mixing process is shown in D.2 and D.3.

7.7.2 Uncertainty of count and mass base cumulative distribution based on the data after the mixing process

Formula (19) and Formula (20) are used to calculate the uncertainty. In this case, the particle size distribution based on the mixed particles is examined.

$$\delta Q_0 = \sqrt{(\delta Q_{0,i,N})^2 + (\delta Q_{\text{ad}})^2} \tag{19}$$

$$\delta Q_3 = \sqrt{\left(\delta Q_{3,i,N}\right)^2 + \left(\delta Q_{ad}\right)^2} \tag{20}$$

The values of $\delta Q_{0,i,N}$ due to total sample size N is calculated by Formula (D.18) and δQ_{ad} is an additional uncertainty contribution. The value of $\delta Q_{3,i,N}$ is calculated by Formula (21):

$$\delta Q_{3,i,N} = \left|\frac{\partial Q_{3,i}}{\partial Q_{0,1}}\delta Q_{0,1}\right| + \left|\frac{\partial Q_{3,i}}{\partial Q_{0,2}}\delta Q_{0,2}\right| + \cdots + \left|\frac{\partial Q_{3,i}}{\partial Q_{0,n}}\delta Q_{0,n}\right| \tag{21}$$

The uncertainty of particle size is calculated by Formula (22) and Formula (23):

$$\delta x_0 = \frac{\delta Q_0}{q_{0,i}} \tag{22}$$

$$\delta x_3 = \frac{\delta Q_3}{q_{3,i}} \tag{23}$$

7.7.3 Uncertainty of count and mass base cumulative distribution based on the data before the mixing process

In this case, the data of particle size distribution before the mixing process is examined. The uncertainty due to mixture fraction should be considered.

Formula (24) and Formula (25) are used to calculate the uncertainty:

$$\delta Q_0 = \sqrt{\left(\delta Q_{0,N}\right)^2 + \left(\delta Q_{0,i,\alpha}\right)^2 + \left(\delta Q_{ad}\right)^2} \tag{24}$$

$$\delta Q_3 = \sqrt{\left(\delta Q_{3,N}\right)^2 + \left(\delta Q_{3,i,\alpha}\right)^2 + \left(\delta Q_{ad}\right)^2} \tag{25}$$

The term $\delta Q_{0,N}$ indicates the uncertainty of count base cumulative distribution and the value is calculated by the size distribution and number fraction of each picket. The term $\delta Q_{0,i,\alpha}$ indicates uncertainty due to number fraction of each picket.

$$\delta Q_{0,i,\alpha} = \sqrt{\left(\frac{\partial Q_0}{\partial \alpha_1{}^{(0)}}\delta \alpha_1{}^{(0)}\right)^2 + \cdots + \left(\frac{\partial Q_0}{\partial \alpha_m{}^{(0)}}\delta \alpha_m{}^{(0)}\right)^2} \tag{26}$$

$$\delta \alpha_i{}^{(0)} = \sqrt{\left(\frac{\partial \alpha_i{}^{(0)}}{\partial N_1}\delta N_1\right)^2 + \cdots + \left(\frac{\partial \alpha_i{}^{(0)}}{\partial N_m}\delta N_m\right)^2} \tag{27}$$

The uncertainty of the number of particles of picket i depends on the uncertainty of the particle diameter of picket i, the uncertainty of mass of picket i and the uncertainty of the density of the particles of picket i.

$$\delta N_i = N_i \cdot \sqrt{\frac{9}{x_i^2}\delta x_i^2 + \frac{1}{m_i^2}\delta m_i^2 + \frac{1}{\rho_i^2}\delta \rho_i^2} \tag{28}$$

The detailed derivation in Formula (28) is shown in D.4.

The each term of $\delta Q_{0,i,\alpha}$ is determined by use of Formula (27) and Formula (28). The term $\delta Q_{3,i,\alpha}$ calculated by Formula (18) is uncertainty due to mass fraction. For the uncertainty of mass base cumulative distribution, the data of mass base size distribution and mass fraction of each picket are used.

The detailed calculation method is shown in D.3.

In this case, the uncertainty of particle size for count and mass base is calculated by Formula (29) and Formula (30):

$$\delta x_0 = \frac{\delta Q_0}{q_{0,i}} \tag{30}$$

$$\delta x_3 = \frac{\delta Q_3}{q_{3,i}} \tag{31}$$

7.8 Uncertainty due to microscopic scale measurement

In order to obtain particle size by microscopic method, a calibrated scale with values traceable to the International System of Units shall be used. The uncertainty due to the microscopic scale measurement changes with magnification factor and position in a screen. The corresponding deviation is indicated by δ_1.

7.9 Uncertainty due to surrounding particles in microscopic measurement

It is important to know the uncertainty due to surrounding particles in a screen in the microscopic measurement. This factor depends on magnification factor and particle diameter. The corresponding deviation is indicated by δ_2.

7.10 Other uncertainty contributions

When other uncertainty contributions (e.g. microscope resolution, threshold setting in digital image analysis, etc.) are known, they shall be included to calculate total uncertainty, δ_3.

7.11 Combined uncertainty

The combined uncertainty for a cumulative count and mass distribution is given as Formula (32):

$$\delta Q_{0,t} = \sqrt{\sum_{i=1}^{p} \left(\delta Q_{0,i}\right)^2} \tag{32}$$

$$\delta Q_{3,t} = \sqrt{\sum_{i=1}^{p} \left(\delta Q_{3,i}\right)^2} \tag{33}$$

where $\delta Q_{0,i}$ and$\delta Q_{3,i}$ are the individual uncertainty contribution i, $1 \le i \le p$, and p is the total number of uncertainties. The combined uncertainty of particle size for count and mass base is calculated by Formula (34) and Formula (35):

$$\delta x_{0,t} = \frac{\delta Q_{0,t}}{q_{0,i}} \tag{34}$$

$$\delta x_{3,t} = \frac{\delta Q_{3,t}}{q_{0,i}} \tag{35}$$

The example calculation of the uncertainty based on limited number of sample size is shown in Annex B.

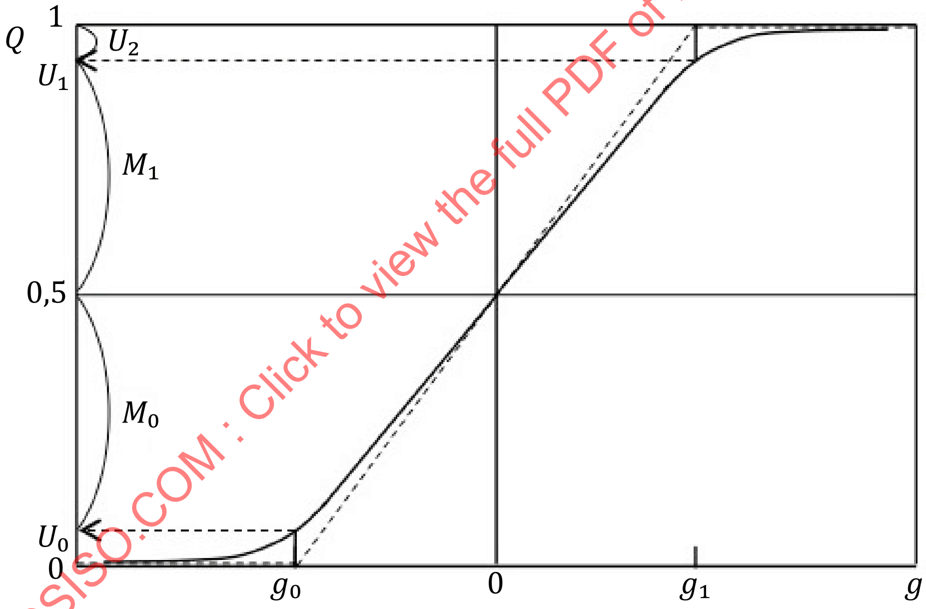
Annex A
(informative)

Picket-fence distributions composed of more than two kinds of quasi-monodisperse particles

For the sake of illustrative clarity, a simplified example using only three rather than the recommended seven pickets is shown.

In order to calculate the uncertainty of picket-fence distributions composed of three kinds of nearly mono-disperse particles, a modified size distribution with maximum and minimum particle size based on log-normal distribution is considered. Figure A.1 shows the relationship between cumulative distribution and a parameter g defined by Formula (A.1):

$$g = \frac{\ln x - \ln x_{50}}{\ln s_g} \tag{A.1}$$



Key

g parameter defined Formula (A.1), dimensionless

Q undersize distribution, dimensionless

g_0, g_1 parameters corresponding to the minimum and the maximum particle diameters, dimensionless

M_0, M_1 fractions of particles between minimum particle and median particle, and median particle and maximum particle, respectively, dimensionless

U_0, U_1 undersize distributions of the log-normal distribution corresponding to the minimum and the maximum particle diameters, dimensionless

U_2 fraction of particles having parameters from g_1 to infinity, dimensionless

Figure A.1 — Corrected distribution with known maximum and minimum particle sizes

The parameters g_0 and g_1 in Figure A.1 correspond to the minimum and the maximum particle diameters, respectively. The solid line shows the perfect log-normal distribution and the dotted line indicates the cumulative distribution truncated by the minimum and the maximum particle sizes. The undersize in this case is represented as Formulae (A.2), (A.3), and (A.4):

$$g < g_0 \quad Q(g) = 0 \tag{A.2}$$

$$g_0 \le g \le g_1 \quad Q(g) = \frac{1}{M}\frac{1}{\sqrt{2\pi}}\int_{g_0}^{g} \exp\left(-\frac{\varsigma^2}{2}\right)d\varsigma \tag{A.3}$$

$$g > g_1 \quad Q(g) = 1 \tag{A.4}$$

where

$$M = M_0 + M_1 = 1 - U_0 - U_2 \tag{A.5}$$

As the particle size distribution is truncated at the minimum size and the maximum size, M, is smaller than unity. In order to calculate the uncertainty of picket-fence distribution composed of three kinds of nearly mono-disperse particles, the true particle size distribution is represented by Formula (A.6) to Formula (A.11). When the three kinds of nearly mono-disperse particles that follows Formula (A.2) to Formula (A.5) are uniformly mixed with N_1, N_2 and N_3 particles, the mass fraction of the particle i is α_i. The parameter g and minimum and maximum particle diameters are as follows:

$$g_1 = \frac{\ln x - \ln x_{50,3,1}}{\ln s_{g,1}} \tag{A.6}$$

$$g_2 = \frac{\ln x - \ln x_{50,3,2}}{\ln s_{g,2}} \tag{A.7}$$

$$g_3 = \frac{\ln x - \ln x_{50,3,3}}{\ln s_{g,3}} \tag{A.8}$$

$$x_{1,max} = x_{50,3,1}\exp\left(g_{1,max}\ln s_{g,1}\right),\ x_{1,min} = x_{50,3,1}\exp\left(g_{1,min}\ln s_{g,1}\right) \tag{A.9}$$

$$x_{2,max} = x_{50,3,2}\exp\left(g_{2,max}\ln s_{g,2}\right),\ x_{2,min} = x_{50,3,2}\exp\left(g_{2,min}\ln s_{g,2}\right) \tag{A.10}$$

$$x_{3,max} = x_{50,3,2}\exp\left(g_{3,max}\ln s_{g,3}\right),\ x_{3,min} = x_{50,3,3}\exp\left(g_{3,min}\ln s_{g,3}\right) \tag{A.11}$$

where g_i is the parameter g of particle i. In Formula (A.6) to Formula (A.11), the mass median diameter of particle i is represented by $x_{50,3,i}$. The maximum particle diameter of particle i is smaller than the minimum particle diameter of particle i + 1.

$$x_{i,max} \le x_{i+1,min} \tag{A.12}$$

Then, the cumulative distribution is represented by Formula (A.13) to Formula (A.19):

$$x < x_{1,min} \quad Q_3(g) = 0 \tag{A.13}$$

$$x_{1,min} \le x \le x_{1,max} \quad Q_3(g) = \frac{\alpha_1}{M}\frac{1}{\sqrt{2\pi}}\int_{g_{1,min}}^{g} \exp\left(-\frac{\varsigma^2}{2}\right)d\varsigma \tag{A.14}$$

$$x_{1,max} < x < x_{2,min} \quad Q_3(g) = \alpha_1 \tag{A.15}$$

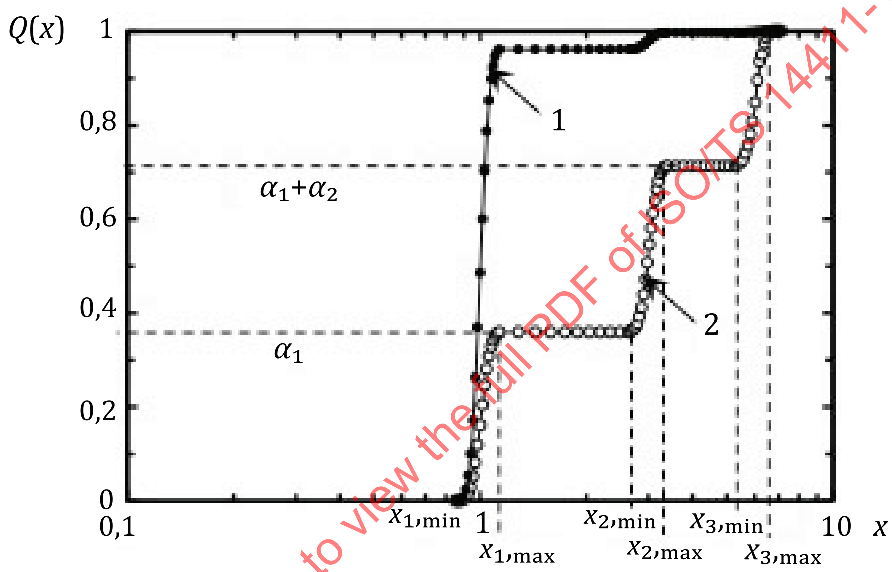
$$x_{2,\min} \le x \le x_{2,\max} \qquad Q_3(g) = \alpha_1 + \frac{\alpha_2}{M}\frac{1}{\sqrt{2\pi}}\int_{g_{2,\min}}^{g} \exp\left(-\frac{\varsigma^2}{2}\right)d\varsigma \tag{A.16}$$

$$x_{2,\max} < x < x_{3,\min} \qquad Q_3(g) = \alpha_1 + \alpha_2 \tag{A.17}$$

$$x_{3,\min} \le x \le x_{3,\max} \qquad Q_3(g) = \alpha_1 + \alpha_2 + \frac{\alpha_3}{M}\frac{1}{\sqrt{2\pi}}\int_{g_{3,\min}}^{g} \exp\left(-\frac{\varsigma^2}{2}\right)d\varsigma \tag{A.18}$$

$$x_{3,\max} < x \qquad Q_3(g) = 1 \tag{A.19}$$

A typical distribution composed of three kinds of nearly mono-disperse particles is shown in Figure A.2.



Key

1 count base distribution

2 mass base distribution

x particle diameter, expressed in micrometers

$Q(x)$ cumulative distribution, dimensionless

α_k mass fraction of particles included in picket k, dimensionless

Figure A.2 — Particle size distribution composed of three kinds of quasi- monodisperse particles

Annex B
(informative)

Example of reliability calculation for a mass-based cumulative size distribution transformed from the number-based size distribution

In this example case, the particles are composed of three kinds of quasi-monodisperse particles and the particle properties before mixing are as follows.

The mass fractions of particles 1,2, and 3 are 0,35, 0,35 and 0,30, respectively.

$$x_{50,0,1} = 1 \ \mu\text{m} \ s_{g,1} = 1{,}05 \tag{B.1}$$

$$x_{50,0,2} = 3 \ \mu\text{m} \ s_{g,2} = 1{,}05 \tag{B.2}$$

$$x_{50,0,3} = 6 \ \mu\text{m} \ s_{g,3} = 1{,}05 \tag{B.3}$$

Total sample size: $N = 90\,000$.

In this example case, the mass fraction of α_1, α_2, α_3 is selected as 0,35, 0,35 and 0,3. Formulae (B.4), (B.5) and (B.6) are used to calculate the number of particle 1, 2 and 3:

$$\alpha_1 = \frac{x_{50,0,1}{}^3 N_1}{x_{50,0,1}{}^3 N_1 + x_{50,0,2}{}^3 N_2 + x_{50,0,3}{}^3 N_3} \tag{B.4}$$

$$\alpha_2 = \frac{x_{50,0,2}{}^3 N_2}{x_{50,0,1}{}^3 N_1 + x_{50,0,2}{}^3 N_2 + x_{50,0,3}{}^3 N_3} \tag{B.5}$$

$$\alpha_3 = \frac{x_{50,0,3}{}^3 N_3}{x_{50,0,1}{}^3 N_1 + x_{50,0,2}{}^3 N_2 + x_{50,0,3}{}^3 N_3} \tag{B.6}$$

where N_1, N_2, N_3 are the total numbers of particles of picket 1, 2 and 3. By use of the above equations, the values of N_1, N_2, N_3 are determined. By use of Formulae (A.2), (A.3) and (A.4), the sample data are obtained.

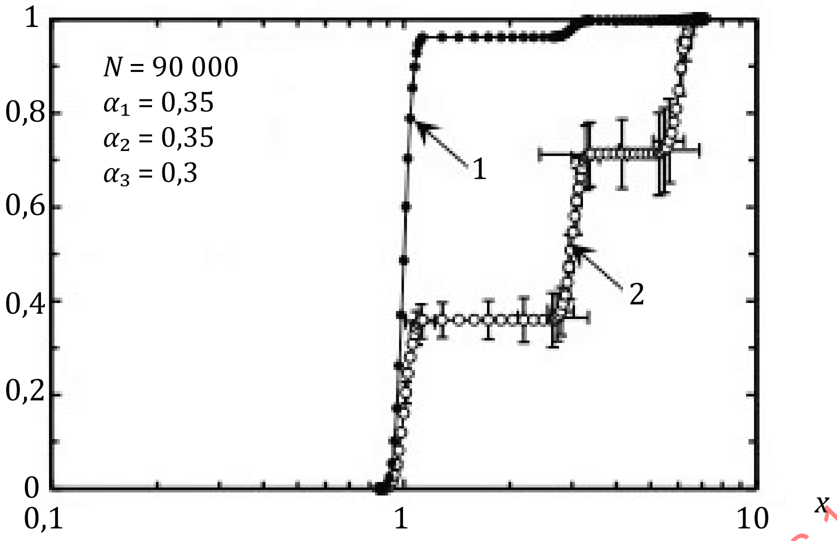
The sample data of the quasi-polydisperse particles based on a picket-fence, quasi-monodisperse particles is shown in Table B.1. The count base data are transformed into mass base distribution data, q_3, and the cumulative size distribution, $Q_3(x)$. In Table B.1, the symbol aE-b means ax10^{-b}.

The first part of the table indicates the range number with particle diameter from x_1 to x_2 µm. The symbol n_i indicates number of particles included in the size range i. Based on the count data, the cumulative distributions of Q_0 and Q_3 are determined. The uncertainty of particle diameter, δx_3 is calculated by use of Formula (7). The uncertainty of mass base cumulative distribution, δQ_3, is calculated by use of Formula (4).

Table B.1 — Particle size distribution

i	x_1 (μm)	x_2 (μm)	n_i	Q_0	Q_3	$\delta x_i^{(3)}$ (μm)	$\delta Q_i^{(3)}$
1	8.38E-01	8.53E-01	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00
2	8.53E-01	8.67E-01	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00
3	8.67E-01	8.82E-01	1.70E+01	1.89E-04	4.68E-05	1.75E-02	5.52E-05
4	8.82E-01	8.97E-01	6.75E+02	7.69E-03	2.00E-03	4.06E-03	5.35E-04
5	8.97E-01	9.12E-01	1.48E+03	2.41E-02	6.50E-03	4.19E-03	1.27E-03
6	9.12E-01	9.27E-01	2.61E+03	5.31E-02	1.48E-02	4.35E-03	2.44E-03
7	9.27E-01	9.41E-01	4.26E+03	1.01E-01	2.91E-02	4.42E-03	4.26E-03
8	9.41E-01	9.56E-01	6.30E+03	1.71E-01	5.12E-02	4.60E-03	6.87E-03
9	9.56E-01	9.71E-01	8.21E+03	2.62E-01	8.14E-02	5.03E-03	1.02E-02
10	9.71E-01	9.86E-01	9.68E+03	3.69E-01	1.19E-01	5.66E-03	1.42E-02
11	9.86E-01	1.00E+00	1.05E+04	4.86E-01	1.61E-01	6.52E-03	1.86E-02
12	1.00E+00	1.02E+00	1.03E+04	6.00E-01	2.04E-01	7.79E-03	2.29E-02
13	1.02E+00	1.03E+00	9.19E+03	7.03E-01	2.45E-01	9.79E-03	2.67E-02
14	1.03E+00	1.05E+00	7.70E+03	7.88E-01	2.80E-01	1.26E-02	3.00E-02
15	1.05E+00	1.06E+00	5.82E+03	8.53E-01	3.08E-01	1.72E-02	3.24E-02
16	1.06E+00	1.08E+00	4.08E+03	8.98E-01	3.29E-01	2.48E-02	3.41E-02
17	1.08E+00	1.09E+00	2.65E+03	9.28E-01	3.42E-01	3.78E-02	3.52E-02
18	1.09E+00	1.10E+00	1.62E+03	9.46E-01	3.51E-01	6.07E-02	3.59E-02
19	1.10E+00	1.12E+00	9.09E+02	9.56E-01	3.56E-01	1.05E-01	3.63E-02
20	1.12E+00	1.13E+00	5.06E+02	9.61E-01	3.59E-01	1.82E-01	3.66E-02
21	1.13E+00	1.29E+00	0.00E+00	9.61E-01	3.59E-01	0.00E+00	3.71E-02
22	1.29E+00	1.44E+00	0.00E+00	9.61E-01	3.59E-01	0.00E+00	3.81E-02
23	1.44E+00	1.59E+00	0.00E+00	9.61E-01	3.59E-01	0.00E+00	3.94E-02
24	1.59E+00	1.74E+00	0.00E+00	9.61E-01	3.59E-01	0.00E+00	4.09E-02
25	1.74E+00	1.89E+00	0.00E+00	9.61E-01	3.59E-01	0.00E+00	4.28E-02
26	1.89E+00	2.04E+00	0.00E+00	9.61E-01	3.59E-01	0.00E+00	4.50E-02
27	2.04E+00	2.19E+00	0.00E+00	9.61E-01	3.59E-01	0.00E+00	4.76E-02
28	2.19E+00	2.34E+00	0.00E+00	9.61E-01	3.59E-01	0.00E+00	5.06E-02
29	2.34E+00	2.49E+00	0.00E+00	9.61E-01	3.59E-01	0.00E+00	5.39E-02
30	2.49E+00	2.65E+00	0.00E+00	9.61E-01	3.59E-01	0.00E+00	5.78E-02
31	2.65E+00	2.68E+00	2.80E+01	9.62E-01	3.61E-01	8.56E-01	4.92E-02
32	2.68E+00	2.72E+00	3.80E+01	9.62E-01	3.65E-01	6.12E-01	4.98E-02
33	2.72E+00	2.76E+00	5.90E+01	9.63E-01	3.70E-01	3.84E-01	5.05E-02
34	2.76E+00	2.80E+00	9.40E+01	9.64E-01	3.78E-01	2.35E-01	5.13E-02
35	2.80E+00	2.83E+00	1.61E+02	9.65E-01	3.93E-01	1.34E-01	5.24E-02
36	2.83E+00	2.87E+00	1.98E+02	9.68E-01	4.12E-01	1.33E-01	6.63E-02
37	2.87E+00	2.91E+00	2.78E+02	9.71E-01	4.39E-01	9.27E-02	6.77E-02
38	2.91E+00	2.95E+00	3.13E+02	9.74E-01	4.71E-01	8.06E-02	6.89E-02
39	2.95E+00	2.99E+00	3.31E+02	9.78E-01	5.07E-01	7.44E-02	6.98E-02
40	2.99E+00	3.02E+00	3.53E+02	9.82E-01	5.46E-01	6.77E-02	7.04E-02
41	3.02E+00	3.06E+00	2.92E+02	9.85E-01	5.80E-01	7.91E-02	7.07E-02
42	3.06E+00	3.10E+00	2.66E+02	9.88E-01	6.12E-01	8.37E-02	7.06E-02
43	3.10E+00	3.14E+00	2.15E+02	9.90E-01	6.39E-01	9.94E-02	7.04E-02
44	3.14E+00	3.18E+00	1.84E+02	9.92E-01	6.63E-01	1.11E-01	6.99E-02
45	3.18E+00	3.21E+00	1.32E+02	9.94E-01	6.80E-01	1.49E-01	6.94E-02
46	3.21E+00	3.25E+00	1.13E+02	9.95E-01	6.96E-01	1.66E-01	6.87E-02
47	3.25E+00	3.29E+00	6.10E+01	9.96E-01	7.05E-01	2.95E-01	6.83E-02
48	3.29E+00	3.33E+00	2.60E+01	9.96E-01	7.09E-01	6.68E-01	6.83E-02
49	3.33E+00	3.37E+00	1.80E+01	9.96E-01	7.12E-01	9.33E-01	6.83E-02
50	3.37E+00	3.40E+00	9.00E+00	9.96E-01	7.13E-01	1.81E+00	6.84E-02
51	3.40E+00	3.59E+00	0.00E+00	9.96E-01	7.13E-01	0.00E+00	6.92E-02
52	3.59E+00	3.78E+00	0.00E+00	9.96E-01	7.13E-01	0.00E+00	7.06E-02
53	3.78E+00	3.97E+00	0.00E+00	9.96E-01	7.13E-01	0.00E+00	7.21E-02
54	3.97E+00	4.16E+00	0.00E+00	9.96E-01	7.13E-01	0.00E+00	7.38E-02
55	4.16E+00	4.35E+00	0.00E+00	9.96E-01	7.13E-01	0.00E+00	7.56E-02
56	4.35E+00	4.54E+00	0.00E+00	9.96E-01	7.13E-01	0.00E+00	7.76E-02
57	4.54E+00	4.72E+00	0.00E+00	9.96E-01	7.13E-01	0.00E+00	7.98E-02
58	4.72E+00	4.91E+00	0.00E+00	9.96E-01	7.13E-01	0.00E+00	8.22E-02
59	4.91E+00	5.10E+00	0.00E+00	9.96E-01	7.13E-01	0.00E+00	8.48E-02
60	5.10E+00	5.29E+00	0.00E+00	9.96E-01	7.13E-01	0.00E+00	8.76E-02
61	5.29E+00	5.38E+00	3.00E+00	9.97E-01	7.15E-01	4.43E+00	8.95E-02
62	5.38E+00	5.48E+00	9.00E+00	9.97E-01	7.21E-01	1.41E+00	9.00E-02
63	5.48E+00	5.57E+00	7.00E+00	9.97E-01	7.26E-01	1.73E+00	9.06E-02
64	5.57E+00	5.66E+00	2.00E+01	9.97E-01	7.40E-01	5.70E-01	8.94E-02
65	5.66E+00	5.75E+00	2.20E+01	9.97E-01	7.57E-01	4.83E-01	8.75E-02
66	5.75E+00	5.85E+00	2.90E+01	9.97E-01	7.80E-01	3.35E-01	8.40E-02
67	5.85E+00	5.94E+00	3.30E+01	9.98E-01	8.08E-01	2.64E-01	7.89E-02
68	5.94E+00	6.03E+00	4.50E+01	9.98E-01	8.48E-01	1.64E-01	7.02E-02
69	6.03E+00	6.13E+00	5.20E+01	9.99E-01	8.96E-01	1.11E-01	5.74E-02
70	6.13E+00	6.22E+00	4.10E+01	9.99E-01	9.35E-01	1.04E-01	4.45E-02
71	6.22E+00	6.31E+00	1.50E+01	1.00E+00	9.51E-01	2.36E-01	3.86E-02
72	6.31E+00	6.40E+00	2.20E+01	1.00E+00	9.74E-01	1.10E-01	2.76E-02
73	6.40E+00	6.50E+00	1.10E+01	1.00E+00	9.86E-01	1.53E-01	2.00E-02
74	6.50E+00	6.59E+00	7.00E+00	1.00E+00	9.94E-01	1.49E-01	1.29E-02
75	6.59E+00	6.68E+00	3.00E+00	1.00E+00	9.98E-01	2.10E-01	8.15E-03
76	6.68E+00	6.77E+00	2.00E+00	1.00E+00	1.00E+00	0.00E+00	0.00E+00
77	6.77E+00	6.87E+00	0.00E+00	1.00E+00	1.00E+00	0.00E+00	0.00E+00
78	6.87E+00	6.96E+00	0.00E+00	1.00E+00	1.00E+00	0.00E+00	0.00E+00
79	6.96E+00	7.05E+00	0.00E+00	1.00E+00	1.00E+00	0.00E+00	0.00E+00
80	7.05E+00	7.15E+00	0.00E+00	1.00E+00	1.00E+00	0.00E+00	0.00E+00

By use of Formulae (D.9), (D.10), (D.18) and (D.19), the uncertainty of particle diameter for cumulative mass distribution is fully determined. Figure B.1 shows the results of calculation.



Key

1 count base distribution

2 mass base distribution

x particle diameter, expressed in micrometers

$Q(x)$ cumulative distribution, dimensionless

N total sample number, dimensionless

α_k mass fraction of particles included in picket k, dimensionless

Figure B.1 — Size distribution included with uncertainty for picket-fence, quasi-monodisperse particles

It is found that the uncertainty increases around the region of particle diameter from $x_{1,\max}$ to $x_{2,\min}$.

The following results are obtained regarding cumulative mass distribution.

$$x_{50,0} = 1{,}003 \ \mu\text{m}, \ x_{50,3} = 2{,}980 \ \mu\text{m} \quad \text{(B.7)}$$

$$\delta x_{50,3} = 0{,}077 \ \mu\text{m} \quad \text{(B.8)}$$

$$\frac{\delta x_{50,3}}{x_{50,3}} = \frac{0{,}077}{2{,}980} = 0{,}026 \quad \text{(B.9)}$$

The sample number, N, in this case is relatively large, then the uncertainty becomes small and satisfies the criteria of reference particles. This is due to the fact that the geometric standard deviations of the three kinds of particles are extremely small.

Annex C
(informative)

Example of uncertainty estimation due to mixture fraction and sample size

C.1 Example 1: Preparation from dry powders

As an example, a picket is to be prepared from 235 µm particles made of polystyrene crosslinked with divinylbenzene (PSDVB). Specifications from the manufacturer are as follows:

— mean number-based diameter: 235 µm ± 3,7 µm;

— standard deviation of the size distribution: 7,3 µm;

— particle density: 1,05 g/cm^3.

An analytical and a microbalance are available. The weighing uncertainties are given as follows:

— analytical balance (<60 g; display 0,01 mg): U = 0,000 13 g + 6,54 × 10^{-6} × mass (g);

— microbalance (<5,2 g; display 1 µg): U = 0,001 7 mg + 9,70 * 10^{-6} × mass (mg).

Weighing 200 mg on the analytical balance (28 000 particles) has an uncertainty of 0,13 mg (0,07 %). Using the microbalance, an uncertainty of 0,036 mg is obtained. Such amounts can be weighed on aluminium foil, where the foil is the dropped with the particles into the dispersant.

Weighing 10 mg on the microbalance (1 500 particles) has an uncertainty of 0,001 8 mg or less than 1 particle. Using the analytical balance would introduce a weighing uncertainty of 0,13 mg or 18 particles.

C.2 Example 2: Preparation from suspensions

As an example, a picket is to be prepared from a suspension of 10 µm particles made of polystyrene. The specifications of the manufacturer are as follows:

— mean number-based diameter: 10,00 µm ± 0,08 µm;

— standard deviation of the size distribution: 0,09 µm;

— nominal particle mass fraction: 10 %;

— particle density: 1,05 g/cm^3.

An analytical and a microbalance are available. The weighing uncertainties are given as follows:

— Analytical balance (<60 g; display 0,01 mg): U = 0,000 13 g + 6,54 × 10^{-6} × mass (g);

— Microbalance (<5,2 g; display 1 µg): U = 0,001 7 mg + 9,70 × 10^{-6} × mass (mg).

The vessel has a mass of 30 g, thus making the use of the microbalance impossible.

a) Uncertainty of the particle mass fraction

This uncertainty involved three weighing steps: Mass of the vessel, mass of the suspension and finally the dry mass. The standard uncertainties are as follows:

— vessel (30 g): 0,000 33 g;

— suspension + vessel (36 g): 0,000 37 g;

— dry particles + vessel (30,6 g): 0,000 33 g.

Using these data, the dry mass content ± standard uncertainty is: 0,100 0 ± 0,000 11 g/g (0,11 %). It might be argued that the definition of constant mass ("subsequent weighings should not differ by more than 1 mg) should be part of the uncertainty. In this case, the uncertainty would increase to 0,2 %.

NOTE If the manufacturer specifies systematic and random uncertainties separately, the uncertainty can be decreased by using the same balance for weighing.

b) Uncertainty of an individual picket

500 mg of the suspension are used to create the picket fence.

The standard uncertainty of weighing 0,5 g is 0,000 13 g (0,03 %), which is negligible compared to the uncertainty of the determination of the dry mass content.

The mass of particles added and their standard uncertainty is 50,00 mg ± 0,058 mg (0,12 %) or (910 ± 1)* 10^5 particles.

NOTE The uncertainty of weighing is small enough to allow also weighing of 100 mg suspension. In this case, the uncertainty of weighing (0,13 %) is no longer negligible and a total uncertainty of the added particle mass of $\sqrt{0{,}11^2 + 0{,}13^2} = 0{,}17\,\%$ is obtained. The mas of particles added is therefore 10,00 mg ± 0,02 mg which corresponds to (181,9 ± 0,3) * 10^5 particles.

Annex D
(informative)

Uncertainty estimation of various cases

D.1 Uncertainty of a volume-based size distribution due to limited number of particles counted

In order to estimate the uncertainty for the general size distribution, it is necessary to obtain the relationship between cumulative distribution based on volume and that based on count. The data of general particle size distribution obtained by use of the microscope is shown in Table D.1. For the particle diameter from x_{i-1} to x_i, n_i particles are detected among the total sample size of N. The frequency distributions based on number and volume are indicated as $q_{0,i}\Delta x_i$ and $q_{3,i}\Delta x_i$, respectively.

Table D.1 — Date of general particle size distribution

Size range µm	Interval µm	Number	Count ratio	Mass ratio
$x_0 - x_1$	Δx_1	n_1	$q_{0,1}\Delta x_1$	$q_{3,1}\Delta x_1$
$x_1 - x_2$	Δx_2	n_2	$q_{0,2}\Delta x_2$	$q_{3,2}\Delta x_2$
$x_2 - x_3$	Δx_3	n_3	$q_{0,3}\Delta x_3$	$q_{3,3}\Delta x_3$
$x_{i-1} - x_i$	Δx_i	n_i	$q_{0,i}\Delta x_i$	$q_{3,i}\Delta x_i$
$x_{n-1} - x_n$	Δx_n	n_n	$q_{0,n}\Delta x_n$	$q_{3,n}\Delta x_n$

The cumulative distribution based on number in this size range i is as given in Formula (D.1):

$$Q_{0,i} = \frac{n_1 + n_2 + \cdots + n_i}{n_1 + n_2 + \cdots + n_n} = \frac{x_1{}^0 n_1 + x_2{}^0 n_2 + \cdots + x_i{}^0 n_i}{x_1{}^0 n_1 + x_2{}^0 n_2 + \cdots + x_n{}^0 n_n} \quad \text{(D.1)}$$

The uncertainty for the size range i is obtained based on ISO 14488.

$$\delta Q_{0,i,N} = u\sqrt{\frac{Q_{0,i}\,(1 - Q_{0,i})}{N}} \quad \text{(D.2)}$$

For the general size distribution, the uncertainty is given by Formula (D.3) based on Tschebyscheff theory under the uncertainty value equal to $1/u^2$ case.

$$Q_{0,i}{}^* - u\sqrt{\frac{Q_{0,i}{}^*\,(1 - Q_{0,i}{}^*)}{N}} \le Q_{0,i} \le Q_{0,i}{}^* + u\sqrt{\frac{Q_{0,i}{}^*\,(1 - Q_{0,i}{}^*)}{N}} \quad \text{(D.3)}$$

where $Q_{0,i}{}^*$ indicates the true cumulative distribution on count base.

For a 5 % uncertainty (95 % confidence level) case, the value of u is determined as given in Formula (D.4):

$$\frac{1}{u^2}=0{,}05 \quad u=4{,}47 \tag{D.4}$$

On the other hand, the cumulative distribution based on volume in the size range i is as given in Formula (D.5):

$$Q_{3,i}=\frac{x_1{}^3 n_1+x_2{}^3 n_2+\cdots+x_i{}^3 n_i}{x_1{}^3 n_1+x_2{}^3 n_2+\cdots+x_n{}^3 n_n} \tag{D.5}$$

The value of n_i is shown as Formula (D.6):

$$\begin{aligned} &n_1=N Q_{0,1},\ n_2=N\left(Q_{0,2}-Q_{0,1}\right),\ n_3=N\left(Q_{0,3}-Q_{0,2}\right) \\ &n_i=N\left(Q_{0,i}-Q_{0,i-1}\right),\ n_n=N\left(Q_{0,n}-Q_{0,n-1}\right) \end{aligned} \tag{D.6}$$

Substituting Formula (D.1) into Formula (3), the Formula (D.7) is obtained.

$$Q_{3,i}=\frac{x_1{}^3 Q_{0,1}+x_2{}^3\left(Q_{0,2}-Q_{0,1}\right)+\cdots+x_i{}^3\left(Q_{0,i}-Q_{0,i-1}\right)}{x_1{}^3 Q_{0,1}+x_2{}^3\left(Q_{0,2}-Q_{0,1}\right)+\cdots+x_n{}^3\left(Q_{0,n}-Q_{0,n-1}\right)}=\frac{g_i}{g_n} \tag{D.7}$$

where

$$g_i\equiv x_1{}^3 Q_{0,1}+x_2{}^3\left(Q_{0,2}-Q_{0,1}\right)+\cdots+x_i{}^3\left(Q_{0,i}-Q_{0,i-1}\right) \tag{D.8}$$

For the general case, maximum uncertainty of the cumulative distribution for the size range i is as given in Formula (D.9) to Formula (D.13):

$$\delta Q_{3,i}\le\left|\frac{\partial Q_{3,i}}{\partial Q_{0,1}}\delta Q_{0,1}\right|+\left|\frac{\partial Q_{3,i}}{\partial Q_{0,2}}\delta Q_{0,2}\right|+\cdots+\left|\frac{\partial Q_{3,i}}{\partial Q_{0,n}}\delta Q_{0,n}\right|=\delta Q_{3,\mathrm{i,N}} \tag{D.9}$$

$$i>j \quad \frac{\partial Q_{3,i}}{\partial Q_{0,j}}=\frac{\left(x_j{}^3-x_{j+1}{}^3\right)\left(g_n-g_i\right)}{g_n{}^2} \tag{D.10}$$

$$i=j \quad \frac{\partial Q_{3,i}}{\partial Q_{0,j}}=\frac{x_j{}^3 g_n-\left(x_j{}^3-x_{j+1}{}^3\right)g_i}{g_n{}^2} \tag{D.11}$$

$$i<j \quad \frac{\partial Q_{3,i}}{\partial Q_{0,j}}=\frac{0-\left(x_j{}^3-x_{j+1}{}^3\right)g_i}{g_n{}^2} \tag{D.12}$$