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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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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 $0,2 < Q_3(x) < 0,8$.

5 Characterization of polydisperse particles

5.1 Particle size distribution

The particle size distribution is determined by a method that provides traceable results and satisfies the following.

a) The relative expanded uncertainty (corresponding to a confidence level of 95 %) shall be fit for purpose.

b) The reference data for the range from $Q_{10,3}$ to $Q_{90,3}$ should be fit for purpose.

c) The particle size distribution should be measured using two or more types of measurement principles. For each measurement data, 95 % reliability or 5 % uncertainty should be indicated.

The requirements for the individual method are as follows.

d) Results shall be traceable to the International System (SI) of Units either by using CRMs with traceable certified values or by appropriate calibration of all relevant input parameters, in case of no direct calibration for length.

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

f) All results and characteristic values shall be given in terms of a volume-based particle size distribution $Q_3(x)$.

g) The number of particles measured shall be sufficiently high to achieve the required precision (see ISO 14488).

5.2 Aspect ratio

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

5.3 Apparent particle density

The apparent particle density shall be measured by any suitable method.

5.4 Refractive index

The refractive index of particle or bulk material shall be measured by any suitable method, e.g. by the liquid immersion method.

6 Estimation of size measurement uncertainties

The uncertainty of each quantile shall be estimated. The following uncertainty contributions shall be included in the combined uncertainty of the particle size distribution:

6.1 Uncertainty from sampling

6.1.1 Uncertainty of the average value *N* replicate measurement

At least four measurements shall be performed to obtain a sufficiently accurate value. This subclause describes the estimation of the uncertainty of *N* replicate measurements.

The average value of N measurements of the p percentile of the cumulative distribution of r type is calculated as;

$$\bar{x}_{p,r} = \frac{1}{N}\sum_{i=1}^{N} x_{p,r}(i) \tag{1}$$

where $x_{p,r}(i)$ is the particle size of p percentile of the cumulative distribution of r type quantity at the i-th measurement. The uncertainty of the average value of the particle size at certain cumulative distribution is defined by Formula (2).

$$u_{\text{rep},\,p,\,r} = \sqrt{\sum_{i=1}^{N}\left(x_{p,r}(i) - \bar{x}_{p,r}\right)^2 \Big/ \left\{N\,(N-1)\right\}}, \tag{2}$$

Formulae (1) and (2) are applied to various types of particle size distributions which are tried N more than three measurements.

An example for the estimation of an uncertainty of the average value N replicate measurement is shown in Annex A.

Proper subsampling, preferably by cross-riffling is required to obtain representative subsamples. The number of measurements to be performed depends on the desired uncertainty and the width of the distribution: the broader the distribution, the more measurements are needed to achieve a given uncertainty of characterization. More measurements result in a lower uncertainty of characterization. The final number of measurements should be chosen to achieve an uncertainty that is fit for purpose.

6.1.2 Uncertainty of a single measurement

6.1.2.1 Uncertainty of number-based size distribution

When the particle size distribution is determined by a single measurement using a number-based method, the following uncertainty should be calculated.

The uncertainty of number-based size distribution by a single measurement can be calculated with guidance from ISO 14488. In the case that the total number of particles counted is equal to n_t, the uncertainty of cumulative number-based size distribution is calculated by Formula (3):

$$u_{Q_{p,0}} = \sqrt{Q_0\left(x_{p,0}\right)\left\{1 - Q_0\left(x_{p,0}\right)\right\} \Big/ n_t}, \tag{3}$$

The uncertainty of particle size, $x_{p,0}$, is given by

$$u_{p,0} = u_{Q_{p,0}} \Big/ q_0\left(x_{p,0}\right) \tag{4}$$

where $q_0\left(x_{p,0}\right)$ is the number-based density distribution.

An example for the estimation of an uncertainty of a single measurement is shown in Annex B.

6.1.2.2 Uncertainty of volume-based size distribution (a log-normal size distribution)

When the uncertainty of the volume-based particle size distribution is calculated from the results of the number-based particle size distribution, the calculation is in general not simple, as described in ISO 14488. However, assuming the original particle size distribution follows a log-normal distribution, the volume-based uncertainty estimation method from a single measurement is analytically solved by Masuda et al [3],[4],[5]. An example for the estimation of uncertainty estimation for a volume-based cumulative size distribution transformed from the number-based size distribution is shown in Annex C.

6.2 Other uncertainty factors

Other uncertainty factors that do not influence precision, for example uncertainty related to calibration, or the between-unit homogeneity and the stability during storage and transport as mentioned in ISO 17034 shall be quantified. These uncertainties should be combined with the uncertainty estimated in 6.1.1 to obtain a combined standard uncertainty.

The total uncertainty can be represented by Formula (5). In addition, a list of uncertainties that might be relevant for imaging methods should be included.

$$u_{\mathrm{t}}^2 = \sum_{j=1}^{m} u_j^2 , \qquad (5)$$

where u_i indicates uncertainty due to the uncertainty contribution j (j = 1, ..., m), and m is the total number of uncertainty factor.

6.3 Expanded uncertainty of size distribution

The expanded uncertainty of p percentile of the cumulative distribution of r type quantity can be easily estimated by the following formulae:

$$U_{\mathrm{f},\, p,\, r} = k\sqrt{u_{\mathrm{rep},\, p,\, r}^2 + u_{\mathrm{t}}^2 + u_{\mathrm{hom}}^2 + u_{\mathrm{stab}}^2} \qquad (6)$$

for N replicate measurements

$$U_{\mathrm{f},\, p,\, 0} = k\sqrt{u_{p,\, 0}^2 + u_{\mathrm{t}}^2 + u_{\mathrm{hom}}^2 + u_{\mathrm{stab}}^2} \qquad (7)$$

for number-based size distribution obtained by a single measurement.

With u_{hom} and u_{stab} being the uncertainty contributions for the between-unit homogeneity and stability during storage and transport, respectively as assessed according to ISO 17034, and where k is the coverage factor. For a high number of degrees of freedom, a coverage factor of two is often chosen. If the number of degrees of freedom for the combined uncertainty is insufficiently large, the t factor from the student's t-distribution according to the effective number of degrees of freedom as calculated by the Welch Sattherthwaithe equation (see ISO/IEC Guide 98-3:2008, Formula G2.b) can be used instead.

This document recommends the combination of results from two different methods to derive the certified values. It is recommended to use the weighted average of the results as certified value, with the weights being inversely proportional to the measurement uncertainties of the results. Further guidance is given in ISO Guide 35.

Annex A
(informative)

Example calculation of the uncertainty estimation of particle size distribution determined by *N* replicate measurement

An example calculation method of uncertainty or reliability based on N times of the particle size measurement by using a scanning electron microscope is shown in this annex. The results of number-based size of various percentile cumulative distribution of spherical particles in each trial by use of microscopic method are shown in Table A.1. The measurement trial number N equals to 11 and the sample size of each measurement is 5 000. The uncertainty of the average value of median size due to 11 times the size measurements is calculated using Formula (2) with the data in Table A.1 and the result is shown as follows.

$$u_{\mathrm{rep},50,0} = 0{,}018\,6\,\mu\mathrm{m} \tag{A.1}$$

For other uncertainty factors, there are uncertainties of the standard scale for measurement, u_{mrs}, and of image warp at various screen positions, u_{meg}. u_{mrs} of the standard 2 µm length scale was 0,068 µm, and u_{meg} which was obtained from an image of the standard 2 µm length scale at various screen positions was 0,029 µm.

The expanded uncertainty of the average median size of number-based size distribution is obtained using Formula (6).

$$U_{\mathrm{f},50,0} = 2{,}0\sqrt{u_{\mathrm{rep},50,0}^2 + u_{\mathrm{mrs}}^2 + u_{\mathrm{meg}}^2} = 0{,}152\ \mu\mathrm{m} \tag{A.2}$$

The uncertainty and expanded uncertainty of the average value of size of p-percentile cumulative distribution can be also calculated in the same manner and the results are shown in Table A.2.

Table A.1 — Average size of number-based size distribution

Trial number	1	2	3	4	5	6	7	8	9	10	11	Average
$X_{10,0}$ [µm]	1,80	1,79	1,78	1,77	1,76	1,76	1,74	1,74	1,66	1,68	1,73	1,75
$X_{20,0}$ [µm]	2,11	2,09	2,11	2,04	2,06	2,04	2,04	2,04	1,96	1,98	2,04	2,05
$X_{30,0}$ [µm]	2,39	2,39	2,39	2,31	2,32	2,29	2,28	2,29	2,21	2,26	2,31	2,31
$X_{40,0}$ [µm]	2,66	2,67	2,66	2,61	2,60	2,58	2,58	2,58	2,49	2,52	2,59	2,59
$x_{50,0}$ [µm]	2,93	2,96	2,94	2,88	2,87	2,85	2,85	2,85	2,75	2,79	2,84	2,87
$X_{60,0}$ [µm]	3,28	3,30	3,28	3,20	3,19	3,17	3,21	3,20	3,12	3,13	3,14	3,20
$X_{70,0}$ [µm]	3,74	3,73	3,63	3,56	3,60	3,54	3,62	3,62	3,52	3,54	3,52	3,60
$X_{80,0}$ [µm]	4,29	4,30	4,15	4,07	4,10	4,07	4,21	4,14	4,06	4,07	4,04	4,14
$X_{90,0}$ [µm]	5,16	5,16	5,07	4,89	4,85	4,85	5,10	5,00	4,97	4,99	4,93	5,00

Table A.2 — Expanded uncertainty of number-based particle size distribution

Q_0 [%]	$\overline{x}_{p,0}$ [µm]	$u_{rep,\,0}$ [µm]	$U_{f,\,0}$ [µm]
10	1,75	0,012 8	0,150
20	2,05	0,014 6	0,151
30	2,31	0,017 3	0,152
40	2,59	0,017 3	0,152
50	2,87	0,018 6	0,152
60	3,20	0,018 6	0,152
70	3,60	0,023 2	0,155
80	4,14	0,027 6	0,158
90	5,00	0,034 3	0,163

Some particle size analysis methods, for example the laser diffraction method, give a volume-based result. In these cases, it is necessary to indicate the uncertainty of the volume-based particle size distribution. Particles in this annex were measured at an aspect ratio and it was found that 955 particles in 1 000 particles had 0,9 – 1,0 aspect ratio. As the sample could be estimated to be spherical particles from this result, the volume-based particle size distribution with the expanded uncertainty can be calculated from the number-based particle size distribution. The volume-based size of various percentile cumulative distribution is shown in Table A.3, and the uncertainty and expanded uncertainty of the average value of size of *p*-percentile cumulative distribution are shown in Table A.4, using the same procedure as for number-based size distribution.

Figure A.1 shows the number- and volume-based particle size distribution with expanded uncertainty.

Table A.3 — Average diameter of volume-based size distribution

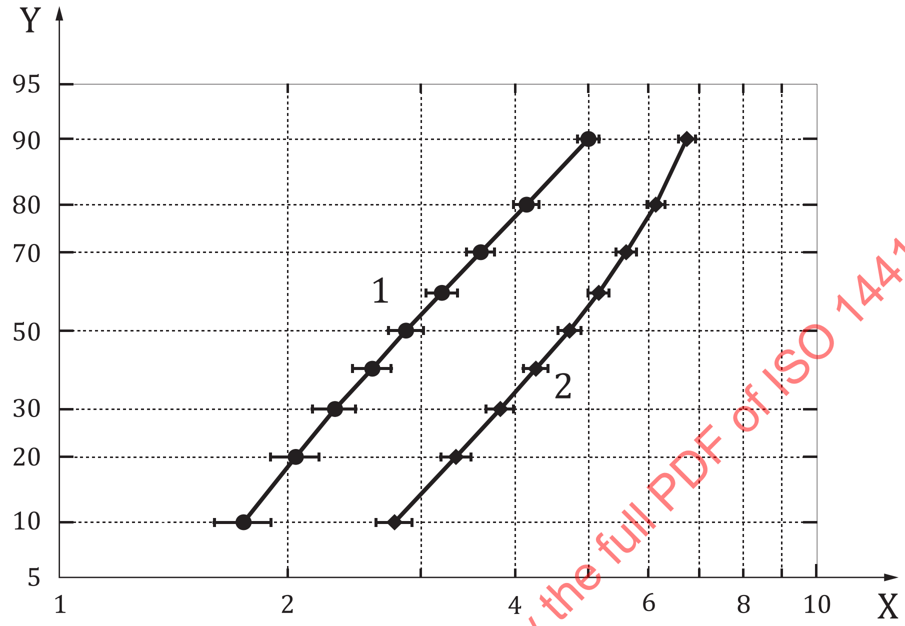
Trial number	1	2	3	4	5	6	7	8	9	10	11	Average
$X_{10,3}$ [µm]	2,84	2,87	2,80	2,76	2,73	2,72	2,78	2,77	2,71	2,74	2,74	2,77
$X_{20,3}$ [µm]	3,45	3,46	3,36	3,29	3,28	3,27	3,38	3,35	3,32	3,33	3,27	3,34
$X_{30,3}$ [µm]	4,00	3,98	3,80	3,73	3,73	3,72	3,89	3,88	3,81	3,79	3,75	3,82
$X_{40,3}$ [µm]	4,41	4,47	4,24	4,14	4,13	4,15	4,34	4,26	4,25	4,24	4,21	4,26
$x_{50,3}$ [µm]	4,88	4,90	4,76	4,57	4,51	4,57	4,80	4,72	4,76	4,73	4,69	4,72
$X_{60,3}$ [µm]	5,30	5,34	5,21	5,03	4,93	4,99	5,20	5,18	5,20	5,20	5,17	5,16
$X_{70,3}$ [µm]	5,81	5,85	5,66	5,46	5,39	5,41	5,62	5,64	5,64	5,60	5,65	5,61
$X_{80,3}$ [µm]	6,31	6,36	6,22	6,08	5,94	5,93	6,11	6,16	6,07	6,12	6,26	6,14
$X_{90,3}$ [µm]	6,93	6,99	6,85	6,65	6,43	6,57	6,70	6,78	6,70	6,81	6,82	6,75

Table A.4 — Expanded uncertainty of volume-based particle size distribution

Q_3 [%]	$\overline{x}_{p,3}$ [µm]	$u_{rep\,3}$ [µm]	$U_{f,\,3}$ [µm]
10	2,77	0,015 2	0,151
20	3,34	0,020 2	0,153
30	3,82	0,029 6	0,159
40	4,26	0,033 1	0,162
50	4,72	0,037 7	0,166
60	5,16	0,037 8	0,166

Table A.4 *(continued)*

Q_3 [%]	$\overline{x}_{p,3}$ [µm]	$u_{rep\ 3}$ [µm]	$U_{f,\ 3}$ [µm]
70	5,61	0,043 9	0,172
80	6,14	0,041 6	0,170
90	6,75	0,048 6	0,177



Key

X undersize, $Q(x)$ [%]

Y particle diameter, x [µm]

1 number-based size distribution

2 volume based size distribution

Figure A.1 — Particle size distribution with expanded uncertainty

Annex B
(informative)

Example calculation of the uncertainty estimation of number-based particle size distribution determined by a single measurement

Example calculations of uncertainty of number-based size distribution are shown in this annex. Particle size measurement was carried out singularly using the same powder sample as Annex A, and the total sample size was 55 000. Table B.1 shows the measurement data and the uncertainty calculated as follows.

The data of the 11th line (gray colour commune) corresponds to the region close to median size. The uncertainty of cumulative size distribution is obtained using Formula (3) (note that the 11th line gives the data in fractions whereas the following formulae use percentiles).

$$Q_0 = 0,460, \qquad 1 - Q_0 = 0,540 \tag{B.1}$$

$$u_{Q_{46},0} = \sqrt{\frac{0,460 \times 0,540}{55\,000}} = 0,002\ 125 \tag{B.2}$$

The uncertainty of particle size x_{46} is obtained from Formula (4).

$$u_{46,0} = \frac{u_{Q_{46},0}}{q_0\left(x_{46,0}\right)} = \frac{0,002\ 125}{0,377\ 2} = 0,005\ 63\ \ \mu\text{m} \tag{B.3}$$

For other uncertainty factors, there are uncertainties of the standard scale for measurement, u_{mrs}, and of image warp at various screen positions, u_{meg}. u_{mrs} of the standard 2 µm length scale was 0,068 µm, and u_{meg} which was obtained from an image of the standard 2 µm length scale at various screen positions was 0,029 µm. The expanded uncertainty for the number-based size distribution is obtained using Formula (7).

$$U_{f,\ 46,\ 0} = 2.0\sqrt{u_{46,0}^2 + u_{mrs}^2 + u_{meg}^2} = 0,148\ 3\ \ \mu\text{m} \tag{B.4}$$

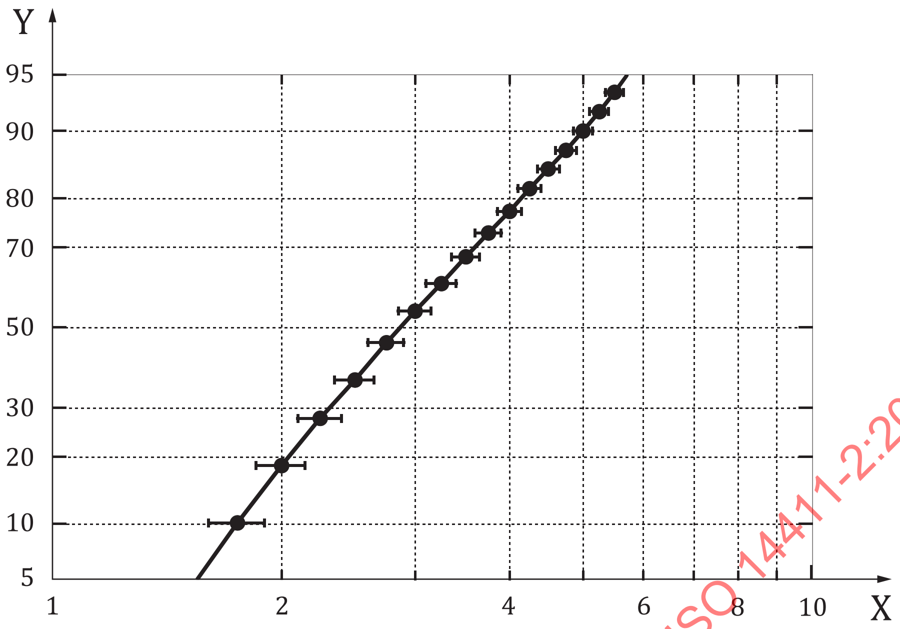
Table B.1 also shows the calculated results of expanded uncertainty for all number-based particle size range.

Table B.1 — Expanded uncertainty of number-based size distribution on a single measurement

j	$x_{min,\ j}$ [µm]	$x_{max,\ j}$ [µm]	n_j [-]	q_0 [-/µm]	Q_0 [-]	u_{Q0} [-]	u_0 [µm]	$U_{f,\ 0}$ [µm]
1	0,00	0,25	0	0,000 00	0	0,000 00	-	-
2	0,25	0,50	0	0,000 00	0,000 00	0,000 00	-	-
3	0,50	0,75	0	0,000 00	0,000 00	0,000 00	-	-
4	0,75	1,00	1	0,000 07	0,000 02	0,000 02	0,250 0	0,521 4
5	1,00	1,25	544	0,039 56	0,009 91	0,000 42	0,010 7	0,149 4
6	1,25	1,50	1 716	0,124 80	0,041 11	0,000 85	0,006 8	0,148 5
7	1,50	1,75	3 311	0,240 80	0,101 31	0,001 29	0,005 3	0,148 2
8	1,75	2,00	4 567	0,332 15	0,184 35	0,001 65	0,005 0	0,148 2

Table B.1 *(continued)*

j	$x_{\min, j}$ [µm]	$x_{\max, j}$ [µm]	n_j [-]	q_0 [-/µm]	Q_0 [-]	u_{Q0} [-]	u_0 [µm]	$U_{f, 0}$ [µm]
9	2,00	2,25	5 123	0,372 58	0,277 49	0,001 91	0,005 1	0,148 2
10	2,25	2,50	4 870	0,354 18	0,366 04	0,002 05	0,005 8	0,148 3
11	2,50	2,75	5 186	0,377 16	0,460 33	0,002 13	0,005 6	0,148 3
12	2,75	3,00	4 526	0,329 16	0,542 62	0,002 12	0,006 5	0,148 4
13	3,00	3,25	3 857	0,280 51	0,612 75	0,002 08	0,007 4	0,148 6
14	3,25	3,50	3 602	0,261 96	0,678 24	0,001 99	0,007 6	0,148 6
15	3,50	3,75	2 935	0,213 45	0,731 60	0,001 89	0,008 9	0,148 9
16	3,75	4,00	2 445	0,177 82	0,776 05	0,001 78	0,010 0	0,149 2
17	4,00	4,25	2 299	0,167 20	0,817 85	0,001 65	0,009 8	0,149 2
18	4,25	4,50	1 750	0,127 27	0,849 67	0,001 52	0,012 0	0,149 8
19	4,50	4,75	1 469	0,106 84	0,876 38	0,001 40	0,013 1	0,150 2
20	4,75	5,00	1 292	0,093 96	0,899 87	0,001 28	0,013 6	0,150 3
21	5,00	5,25	1 131	0,082 25	0,920 44	0,001 15	0,014 0	0,150 5
22	5,25	5,50	941	0,068 44	0,937 55	0,001 03	0,015 1	0,150 9
23	5,50	5,75	795	0,057 82	0,952 00	0,000 91	0,015 8	0,151 2
24	5,75	6,00	590	0,042 91	0,962 73	0,000 81	0,018 8	0,152 6
25	6,00	6,25	563	0,040 95	0,972 96	0,000 69	0,016 9	0,151 7
26	6,25	6,50	431	0,031 35	0,980 80	0,000 59	0,018 7	0,152 5
27	6,50	6,75	316	0,022 98	0,986 55	0,000 49	0,021 4	0,153 9
28	6,75	7,00	292	0,021 24	0,991 85	0,000 38	0,018 0	0,152 2
29	7,00	7,25	175	0,012 73	0,995 04	0,000 30	0,023 5	0,155 2
30	7,25	7,50	123	0,008 95	0,997 27	0,000 22	0,024 9	0,156 0
31	7,50	7,75	67	0,004 87	0,998 49	0,000 17	0,034 0	0,162 7
32	7,75	8,00	31	0,002 25	0,999 05	0,000 13	0,058 1	0,188 1
33	8,00	8,25	27	0,001 96	0,999 55	0,000 09	0,046 3	0,174 4
34	8,25	8,50	12	0,000 87	0,999 76	0,000 07	0,075 1	0,210 8
35	8,50	8,75	6	0,000 44	0,999 87	0,000 05	0,110 2	0,265 5
36	8,75	9,00	4	0,000 29	0,999 95	0,000 03	0,108 3	0,262 2
37	9,00	9,25	0	0,000 00	0,999 95	0,000 03	-	-
38	9,25	9,50	0	0,000 00	0,999 95	0,000 03	-	-
39	9,50	9,75	0	0,000 00	0,999 95	0,000 03	-	-
40	9,75	10,00	3	0,000 22	1,000 00	0,000 00	-	-
41	10,00	10,25	0	0,000 00	1,000 00	0,000 00	-	-
42	10,25	10,50	0	0,000 00	1,000 00	0,000 00	-	-



Key

X undersize, $Q(x)$ [%]

Y particle diameter, x [µm]

Figure B.1 — Number-based particle size distribution with expanded uncertainty on a single measurement