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Determination of particle size distributions — Electrical sensing zone method

Détermination des répartitions granulométriques — Méthode de la zone de détection électrique

Reference number
ISO 13319:2007(E)

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Contents

Page

Foreword

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ISO 13319 was prepared by Technical Committee ISO/TC 24, *Sieves, sieving, and other sizing methods*, Subcommittee SC 4, *Sizing by methods other than sieving*.

This second edition cancels and replaces the first edition (ISO 13319:2000), which has been technically revised.

INTERNATIONAL STANDARD **ISO 13319:2007(E)**

Determination of particle size distributions — Electrical sensing zone method

1 Scope

This International Standard gives guidance on the measurement of the size distribution of particles dispersed in an electrolyte solution using the electrical sensing zone method. The method measures pulse heights and their relationship to particle volumes or diameters, and it reports in the range from approximately 0,4 µm to approximately 1 200 µm. It does not address the specific requirements of the measurement of specific materials. However, guidance on the measurements of conducting materials such as porous materials and metal powders is given.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 787-10, *General methods of test for pigments and extenders — Part 10: Determination of density — Pyknometer method*

ISO 9276-2:2001, *Representation of results of particle size analysis — Part 2: Calculation of average particle sizes/diameters and moments from particle size distributions*

3 Terms and definitions

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

3.1
dead time
time during which the electronics are not able to detect particles due to the signal processing of a previous pulse

3.2
aperture
small-diameter hole through which suspension is drawn

3.3
sensing zone
volume of electrolyte solution within, and around, the aperture in which a particle is detected

3.3
sampling volume
volume of suspension that is analysed

3.4
channel
size interval

3.5
envelope size

external size of a particle as seen in a microscope

3.6
envelope volume

volume of the envelope given by the three-dimensional boundary of the particle to the surrounding medium

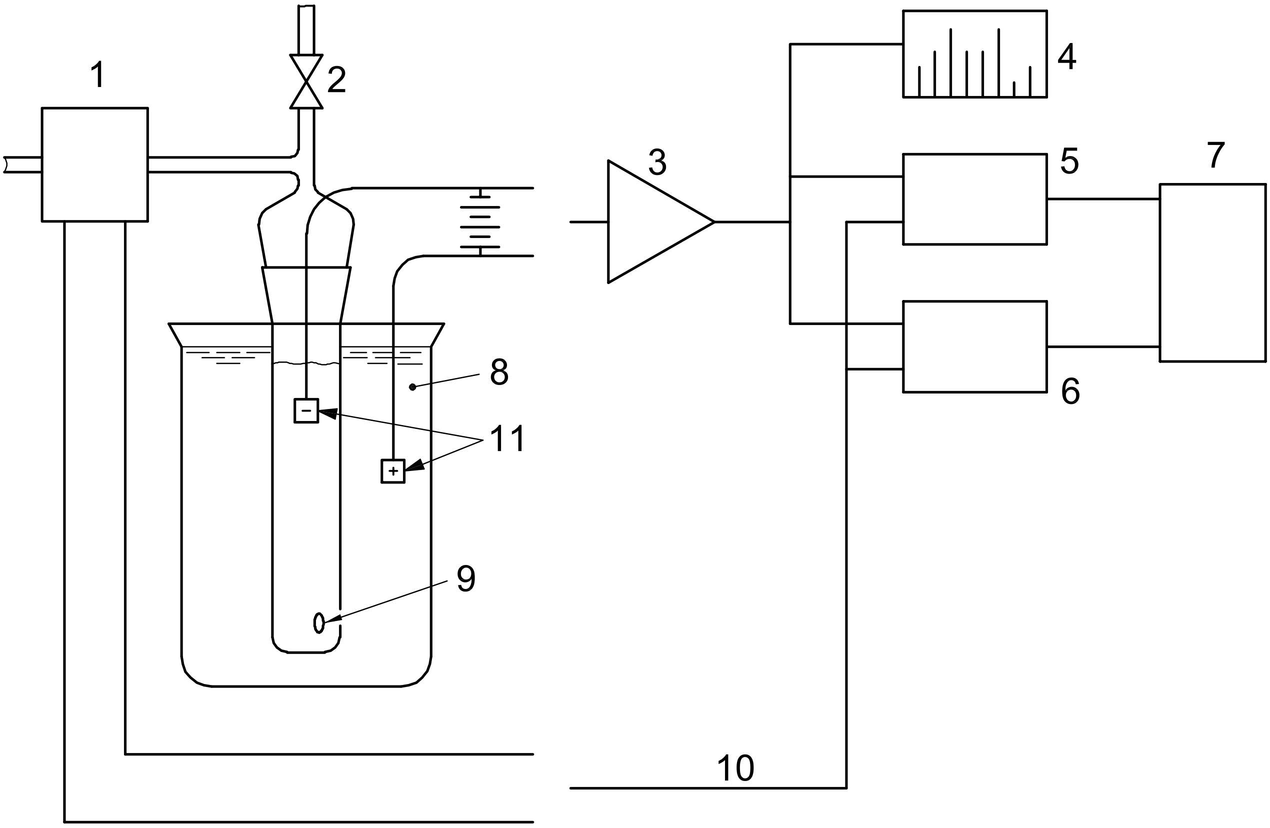
4 Symbols

For the purposes of this document, the following symbols apply.

A_p	amplitude of the most frequent pulse
A_x	amplitude of the electrical pulse generated by an arbitrary particle
d_p	modal diameter of a certified particle size reference material
$\overline{d}$	mean particle diameter of a size interval or channel
d_L	particle diameter at the lower boundary of a size interval or channel
d_U	particle diameter at the upper boundary of a size interval or channel
D	aperture diameter
K_d	calibration constant of diameter
$\overline{K}_d$	mean calibration constant of diameter
$K_{d\,\mathrm{a}}$	arbitrary calibration constant of diameter
m	mass of sample
ΔN_i	number of counts in a size interval i
V_T	volume of electrolyte solution in which a mass, m, is dispersed
V_m	analysis volume
$\overline{V}_i$	arithmetic mean volume for a particular size interval i
V_i	volume of the particle obtained from a threshold or channel boundary
x	diameter of a sphere with volume equivalent to that of the particle
x_{50}, x_{10}, x_{90}	values of x corresponding to the 50 %, 10 % and 90 % points of the cumulative percent undersize distributions
ρ	mass of the particles per volume of the electrolyte displaced
$\sigma_{\overline{K}_d}$	standard deviation of mean calibration constant

5 Principle

A dilute suspension of particles dispersed in an electrolyte solution is stirred to provide a homogeneous mixture and is drawn through an aperture in an insulating wall. A current applied across two electrodes, placed on each side of the aperture, enables the particles to be sensed by the electrical impedance changes as they pass through the aperture. The particle pulses thus generated are amplified and counted, and the pulse height is analysed. After employing a calibration factor, a distribution of the number of particles against the volume-equivalent diameter is obtained. This distribution is usually converted to percentage by mass versus particle size, where the size parameter is expressed as the diameter of a sphere of volume and density equal to that of the particle. See Figure 1.



Key

1	volumetric metering device
2	valve
3	pulse amplifier
4	oscilloscope pulse display
5	counting circuit
6	pulse-height analyser
7	output
8	stirred suspension of particles in electrolyte solution
9	aperture
10	counter start/stop triggered by the volumetric device
11	electrodes

Figure 1 — Diagram illustrating the principle of the electrical sensing zone method

6 General operation

6.1 Response

The response (i.e. the electrical pulse generated when a particle passes through the aperture) has been found both experimentally and theoretically to be proportional to the particle volume if the particles are spherical [1]-[3].This has also been shown to be true for particles of other shapes; however, the constant of proportionality (i.e. the instrument's calibration constant) may be different [4]. In general, particles should have a low conductivity with respect to the electrolyte solution, but particles with high conductivity can be measured e.g. metals [5], carbon [6], silicon and many types of cells and organisms, such as blood cells [7], [8]. For porous particles, the response may vary with the porosity [9], [10]. Recommendations for the measurement of conducting particles and porous particles are given in Annex A.

As the response is proportional to the volume of particles, the pulse amplitude provides a relative scale of particle volumes. By calibration, this scale may be converted to spherical diameter. The calibration constant based on diameter may be calculated by Equation (1):

$$K_d = \frac{d_\mathrm{p}}{\sqrt[3]{A_\mathrm{p}}} \tag{1}$$

The size, x, of any particle can be calculated by Equation (2):

$$x = K_d \cdot \sqrt[3]{A_x} \tag{2}$$

6.2 Size limits

The lower size limit of the electrical sensing zone method is generally considered to be restricted only by thermal and electronic noise. It is normally stated to be about 0,6 µm but, under favourable conditions, 0,4 µm is possible. There is no theoretical upper size limit, and for particles having a density similar to that of the electrolyte solution, the largest aperture available (normally 2 000 µm) may be used. The practical upper size limit is about 1 200 µm, limited by particle density. In order to increase the possibility of keeping the particles in homogeneous suspension, the viscosity and the density of the electrolyte solution may be increased, for example by addition of glycerol or sucrose. The homogeneity may be checked by repeated analyses at a range of stirrer speeds. The results of this should be compared to establish the lowest speed at which recovery of the largest particles is maintained.

The size range for a single aperture is related to the aperture diameter, D. The response has been found to depend linearly in volume on D, within about 5 % under optimum conditions, over a range from 0,015 D to 0,8 D (i.e. 1,5 µm to 80 µm for a 100 µm aperture) although the aperture may become prone to blockage at particle sizes below the maximum size where the particles are non-spherical. In practice, the limitation of thermal and electronic noise and the physical limitation of non-spherical particles passing through the aperture usually restricts the operating range to 2 % to 60 % of the aperture size. This size range can be extended by using two or more apertures (see Annex B). In practice, this procedure can be avoided by the careful selection of the diameter of one aperture, to achieve an acceptable range.

6.3 Effect of coincident particle passage

Ideal data would result if particles traversed the aperture singly, when each particle would produce a single pulse. When two or more particles arrive in the sensing zone together, the resulting pulse will be complex. Either a single large pulse will be obtained, resulting in a loss of count and effectively registering a single larger particle, or the count will be correct but the reported size of each will be increased, or some particles will not be counted. These effects will distort the size distribution obtained but can be minimized by using low concentrations. Table 1 shows counts per millilitre for the coincidence probability to be 5 %.

Table 1 — Counts for 5 % coincidence probability for typical aperture diameters

Aperture diameter D µm	Analysis volume [a] V_m ml	Count for 5 % coincidence [b] N
1 000	2	80
560	2	455
400	2	1 250
280	2	3 645
200	2	10 000
140	2	29 150
100	0,5	20 000
70	0,5	58 500
50	0,05	16 000
30	0,05	74 000
20	0,05	250 000

[a] For other sampling volumes, use *pro rata* values.

[b] Calculated using the equation $N = \dfrac{4 \times 10^{10} V_m}{D^3}$

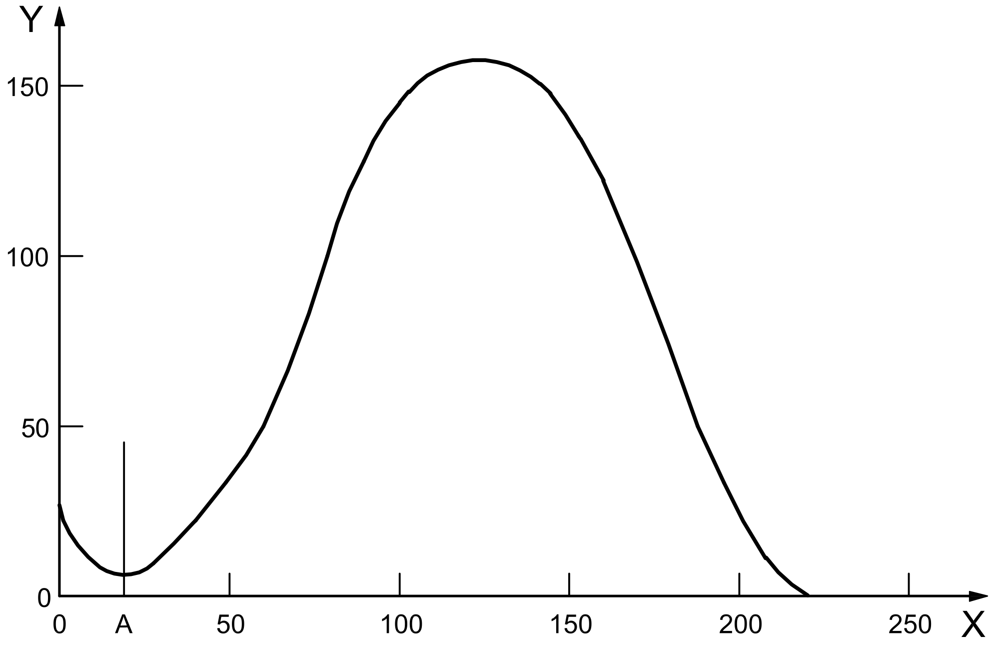
Counts per millilitre should always be less than these quoted values. Since particle size distributions should not be a function of concentration, the effect of coincidence can be tested by obtaining a distribution at one concentration and comparing it with that obtained when the concentration is halved. In such a test, repeat such dilutions until the reduction in count in a channel with the largest number decreases in proportion to the dilution. This should always be done when analysing very narrow size distributions, as this is where the effect of coincidence is most noticeable.

6.4 Dead time

In instruments using digital pulse processing routines, to analyse the signal it is scanned at high frequency. Information on pulse parameters, such as maximum pulse height, maximum pulse width, mid-pulse height, mid-pulse width and pulse area, is stored for subsequent analysis. In this case, analog-to-digital conversion of the pulse with storage of the size value for the pulse is not performed in real time and dead time losses are avoided.

In instruments in which pulse-height analysis routines are used in real time to process the data, it is possible that the analyser may not count particles for a given time after receiving a pulse, since it takes a finite time to process each pulse. Dead time is not a function of pulse height. Therefore, the loss will be proportional to the counts in each channel and will not affect the size distribution.

To minimize the effect of dead time, the analyser should be used with the lower threshold set to exclude thermal and electronic noise, as indicated at A in Figure 2. Additionally, the concentration of particles should be maintained below 5 % coincidence levels.



Key

X	channels
Y	counts

NOTE Counts at channels below A are noise counts. True particle counts are at the higher channels.

Figure 2 — Typical results

7 Repeatability of counts

In a correctly performed analysis, the number of counts in a size interval is a random variable which follows a Poisson distribution. In this, the variance is equal to the expected (mean) value. This indicates that the standard deviation of a number of counts, n, with mean, N, approximates to $\sqrt{N}$. Both the variance and the standard deviation can be used in statistical tests on the correctness of instrument operation or sample preparation. The statistical chi-squared test can be used to test whether obtained data follow a Poisson distribution or not. In this, the apparent and the theoretical variance for a given number of measurements and a given probability are related. An example is given in Annex C. This statistical test can be performed on single size intervals, groups of size intervals, or on the total particle count.

8 Operational procedures

8.1 Instrument location

The instrument should be sited in a clean environment that is free from electrical interference and vibration. If organic solvents are to be used, the area should be well ventilated.

8.2 Linearity of the aperture/amplifier system

The linearity of the aperture/amplifier system can be checked using four materials consisting of near mono-sized particles with a certified modal diameter. In a suitable electrolyte solution, the instrument is calibrated with particles at about 0,3 D (see 8.10.2). Three further sizes of particles are then added to the suspension, one of size of about 0,1 D, one of size of about 0,2, D and one of size about 0,5 D. The suspension is re-analysed and the size corresponding to these extra peaks must correspond to the quoted size of the particles to within 5 %.

8.3 Linearity of the counting system

The linearity of the counting system can be tested by obtaining three counts at an arbitrary concentration. The concentration is then reduced and three further counts obtained. Coincidence-corrected counts shall be used. The ratio of the mean of the counts should be the same as the dilution. If the agreement is not within 5 %, the test should be repeated comparing the two lowest dilutions. Subsequent analyses should be carried out at the dilution giving the best results.

8.4 Choice of electrolyte solution

8.4.1 General

An electrolyte solution should be selected in which the sample is stable. The electrolyte solution should not dissolve, flocculate, react or, once a good dispersion is achieved, not change the state of dispersion of the sample in the measurement time, typically up to five minutes. Particles insoluble in water can be analyzed in a variety of aqueous electrolyte solutions. Particles soluble in water can often be analyzed in methanol or in Iso-propanol. See Annex D for recommended electrolyte solutions. When using small apertures (20 µm, 30 µm and 50 µm) or large apertures (400 µm, 560 µm, 1 000 µm and 2 000 µm), special care shall be taken due to their particular characteristics.

8.4.2 Special considerations for small apertures ($D \leqslant 50$ µm)

Where possible, the electrolyte solution should consist of a 4 % sodium chloride solution or one of equivalent conductivity. It should be membrane filtered twice at 0,2 µm.

8.4.3 Special considerations for large apertures ($D \geqslant 400$ µm)

To prevent turbulence that can cause noise signals due to fast flow through the aperture, the viscosity of the electrolyte solution may be increased by the addition of glucose or glycerol; 10 % glycerol is recommended for 560 µm and 400 µm apertures, and 30 % glycerol for the 2 000 µm and 1 000 µm apertures.

8.5 Preparation of electrolyte solution

An electrolyte solution should be well filtered with a membrane filter for which the pore size is less than the diameter of the smallest particle measured, as it is essential that its background count should be as low as practicable. It should be noted that quoted values for filters are not absolute. Usually a mean pore size is given. The width of the distribution of pores around this mean varies depending on filter type and manufacturer. This will affect the choice of filter size used. All glassware and apparatus used should be pre-rinsed with filtered electrolyte solution or other suitable liquids. Background counts should not exceed the values given in Table 2 or yield a total equivalent volume in excess of 0,1 % of the total volume of particles subsequently measured in the analysis volume.

Table 2 — Counts for background for typical aperture diameters

Aperture diameter D µm	Analysis volume [a] V_m ml	Background counts [b]
1 000	2	2
560	2	10
400	2	25
280	2	75
200	2	200
140	2	600
100	0,5	400
70	0,5	1 200
50	0,05	300
30	0,05	1 500
20	0,05	5 000

[a] For other analysis volumes, use *pro rata* values.

[b] Suggested maximum counts.

8.6 Recommended sampling, sample splitting, sample preparation and dispersion

8.6.1 General

See ISO 14488 for guidance on the sampling and sample-splitting procedure. Select a dispersant and a dispersion method from the recommendations in ISO 14887 or Annex D. The expertise of the laboratory performing the analysis with respect to the sample under test may also be utilized.

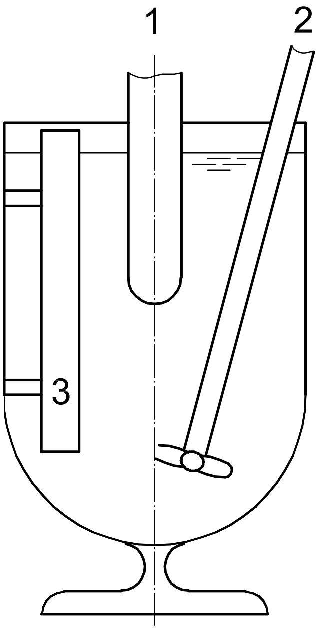
8.6.2 Method 1: Using a paste

The sample should be subdivided to about 0,2 cm^3. If the sample is in the form of a powder, it should be worked and kneaded gently with a flexible spatula with a few drops of suitable dispersant to break down agglomerates. Transfer a mass of about 20 mg to 50 mg of the paste to a round-bottomed beaker and thin it with dispersant, followed by a few drops of electrolyte solution. Almost fill the beaker with electrolyte solution and place it in an ultrasonic bath with suitable power and frequency for 1 min, stirring occasionally. A stop watch is recommended for a reproducible dispersion technique. One suitable design of beaker of 400 ml capacity with a baffle is shown in Figure 3. If the sample is not required to be fully dispersed, it may be added to the electrolyte solution and dispersant while stirring.

NOTE The use of high energy ultrasonic baths and probes, blenders and mixers can cause both agglomeration and fracture of particles.

8.6.3 Method 2: Alternative method applicable to low-density particles of less than 50 µm

Subdivide the sample into portions of about 1 g. Mix a portion with the dispersant and add it to the electrolyte solution. Then place the beaker (see Figure 3) containing the suspension in an ultrasonic bath for about 45 s. After stirring this stock suspension well, withdraw 5 ml using a pipette and add to approximately 400 ml of electrolyte solution. Place in the ultrasonic bath for a further 15 s. When using this method, it is important that at least two samples are withdrawn from the stock suspension and analysed to ensure repeatability of the aliquot sampling and the analysis.



Key

1 aperture tube
2 stirrer
3 baffle

Figure 3 — Example of a beaker with baffle and stirrer

8.6.4 Suspensions and emulsions

Suspensions and emulsions should be diluted by addition of smaller volumes of diluent to the emulsion, not by addition of the emulsion to a larger volume of diluent. Dilution should be performed stepwise with mixing performed at each step. To avoid “dilution shock”, oil-in-water emulsions may be initially diluted with distilled or de-ionized water.

8.6.5 Verification of the dispersion

A small sample of the dispersion may be placed on a microscope slide and used to verify the degree of dispersion and to estimate the size range of the particles using an optical microscope.

8.7 Choice of aperture(s) and analysis volume(s)

From the microscope examination (8.6.5), estimate the diameter of the largest particles present. Choose an aperture for the size analysis such that the diameter of the largest particles to be analysed is less than approximately 60 % of the diameter of the aperture, selected to reduce the possibility of blocking the aperture. For particles that are spherical or nearly spherical, an aperture such that the diameter of the largest particles is less than 80 % of the diameter of the aperture may be chosen. If there is a considerable proportion of sample below the lower size limit of that aperture (1,5 % of its diameter), a second and possibly a third smaller aperture will be needed (see Annex B). An alternative method to determine the amount of particles not accounted for with a specific aperture is to perform a mass balance (see Annex E).

Select a suitable analysis volume with reference to Table 1 or select a suitable time of accumulation. It may be necessary to analyse a number of analysis volumes or to accumulate for a long time to obtain an acceptable precision, e.g. 50 000 particles will yield a precision (relative standard deviation) of 0,4 %. Counting fewer particles will reduce the precision, but this may be necessary when using the larger apertures (see Clause 7 and Annex C).

8.8 Clearing an aperture blockage

Apertures below 100 µm in diameter may become blocked with extraneous particles, particularly if care is not exercised in the clean handling, careful filtration and thorough rinsing of beakers and associated equipment. A blockage or a partial blockage can be seen by means of the viewing optics provided with the analyser. A blockage may also be indicated by measuring the flow time through the aperture or by measuring the electrical resistance of the aperture. A blockage will cause a longer flow time and a higher resistance. A blockage can also be revealed by an examination of the particle pulse train, which is recorded with some instruments. A blockage will cause a clearly visible shift in the pulse train. In some instruments, there are means to automatically detect and remove blockages. Blockages can also be removed by one of the following techniques.

a) Back flushing: Reversing the flow through the aperture may be sufficient to clear a blockage.

b) Boiling: It is possible to use the heating effect of the current to boil the blockage out. This is done by using a high aperture current.

c) Brushing: It is often possible to brush the particles off the aperture by using a small high-quality soft-hair brush with the hairs cut short. Care should be taken not to damage the aperture.

d) Air pressure.

e) Ultrasonic cleaning: With the aperture tube filled with electrolyte solution, the end is dipped into a low-power ultrasonic bath for about 1 s. Repeat this operation as necessary. This method is very effective but extreme care should be taken as it is possible to damage the aperture.

CAUTION — This method should not be used for apertures of 50 µm or less.

8.9 Stability of dispersion

With the most suitable aperture fitted, and the suspension prepared, dry the outside of the beaker and place it on the sample stand of the instrument. Adjust the stirrer for maximum effect without creating a vortex which will entrain bubbles. Then check the stability of the dispersion during the analysis time. Make a full size distribution analysis as soon as possible after dispersion; then stir the dispersion for 5 min to 10 min and then reanalyse. Cumulative counts are recorded at size levels close to 30 % and 5 % of the aperture diameter (denoted as $x_{\max}$ and $x_{\min}$ respectively). Changes in the counts greater than those expected from statistics will indicate that the dispersion is not stable (see Clause 7 and Annex C). Additional verification of stability can also be performed in instruments that record raw pulse data. Inhomogeneity across the pulse train during the time of analysis may indicate a change in the stability of dispersion. Table 3 details some possible causes.

Table 3 — Examples of suspected phenomena in dispersion

Change in count at		Suggests
$x_{\max}$	$x_{\min}$	
No change	no change	stable dispersion
Increase	increase	crystallization, precipitation
Decrease	decrease	dissolution
Decrease	increase	size reduction, deflocculation
Increase	decrease	flocculation, agglomeration
Decrease	no change	settling of large particles

8.10 Calibration

8.10.1 General

Electrical sensing zone instruments are calibrated using polymer latex microspheres of known size and narrow size distribution.

Another method, which is an absolute method, is the mass integration method. Here the weighed mass is compared to the mass found as determined by the instrument [13]. This calibration method is directly traceable and there is no assumption made about the shape, porosity or electric conductivity of the particles. The mass integration method is described in Annex E.

Special care shall be taken for porous particles. Such particles may have an interconnected pore system which, at least partly, is being filled with electrolyte solution during the sample preparation procedure. This electrolyte solution will, to a certain extent, not contribute to the impedance change in the sensing zone when the particle passes through it. Therefore, a porous particle generates a pulse with lower amplitude than a solid particle of equivalent envelope volume. The difference is not negligible; for some porous materials the size can be as little as half that of the envelope size. For the calibration for the measurement of porous particles, see Annex A.

8.10.2 Calibration procedure — Microsphere calibration

Microspheres with narrow size distribution with a single mode, characterized by a variety of other methods, are available. They should be characterized traceably to the metre, to a Community Bureau of Reference reference material (BCR), a National Institute of Standards and Technology reference material (NIST) or similar reference material. The calibration method used depends on the assayed size parameter of the microspheres and the analyser used (contact the instrument manufacturers for details). One method is to obtain a histogram (frequency) plot of the number of particles against channels of equal width (on a linear scale). The size at the centre of the channel with the greatest number of particles corresponds closely to the modal size of the calibration material if the distribution is symmetrical. If the distribution is not symmetrical, the fractional channel position is calculated from the counts in channels on either side of the central channel. The calibration factor is the ratio of the modal size of the calibration material to the size reported on the instrument.

Calibration should be checked on a regular basis to ensure that the change in the calibration constant is less than 1,0 %, or every time an aperture tube or an electrolyte solution is changed. See Annex F for a method for calibration of frequently used apertures.

9 Analysis

Most powders have a particle size range that is sufficiently narrow for a satisfactory analysis to be carried out using one aperture. Where the size range of a powder is too wide for a single aperture, two or more apertures should be used. If over 1,5 % by volume of the particles fall in the smallest size interval, it is advisable to use the multiple aperture method (see Annex B). For certain sample materials, a mass balance may also be performed (see Annex E).

When the particles are dispersed satisfactorily, following the foregoing procedures, the analysis can begin. Select the analysis volume, or the number of repeat measurements of the analysis volume, or the time for accumulation, in such a way that a particle size distribution with sufficient precision is obtained (see Clause 7 and Annex C). Counting fewer particles will reduce the precision, but this may be necessary when using the larger aperture tubes. It is advisable that at least three, and preferably five, replicates be measured. To ensure that the sample subdivision has been carried out well, the whole procedure should be repeated with at least one other, but preferably more, sample(s) from the stock suspension or from the dry powder subdivision. Report all the measured data on a suitable data sheet.

10 Calculation of results

Modern instruments measure the volume and the number of particles within various size channels directly, so no data conversion is needed. Some analysers count the number of particles above, or between, variable equivalent-volume particle diameters, and therefore conversion of data to volume percentage may be required. In the event of requiring number data to be converted to, and presented as, volume data, it is usual for the method of Simpson's rule [13] to be used. Since the volume of each particle is measured, the numbers of particles within a size interval (size channel) can be multiplied by the arithmetic mean volume of the channel in order to present the total particle volume within the channel. In this way, the total volume of all particles within all size channels can be calculated, and the percentage by volume size distribution calculated. For the calculation to be reasonably accurate, the size interval should be narrow, i.e. a large number of channels should be counted. For a more accurate method and the calculation of moments of the distribution, see ISO 9276-2. Modern analysers perform the calculation automatically. The volume-percentage distribution so calculated is identical to the mass (or weight) distribution if all the particles have the same specific gravity (immersed density).

11 Instrument qualification

11.1 General

The instrument is qualified through the verification of the linearity of the aperture/amplifier system (see 8.2), verification of the linearity of the counting system (see 8.3) and the verification of the calibration constant (see Annex F).

11.2 Report

It is essential that the whole qualification procedure and all data be reported in full detail on a suitable data sheet.

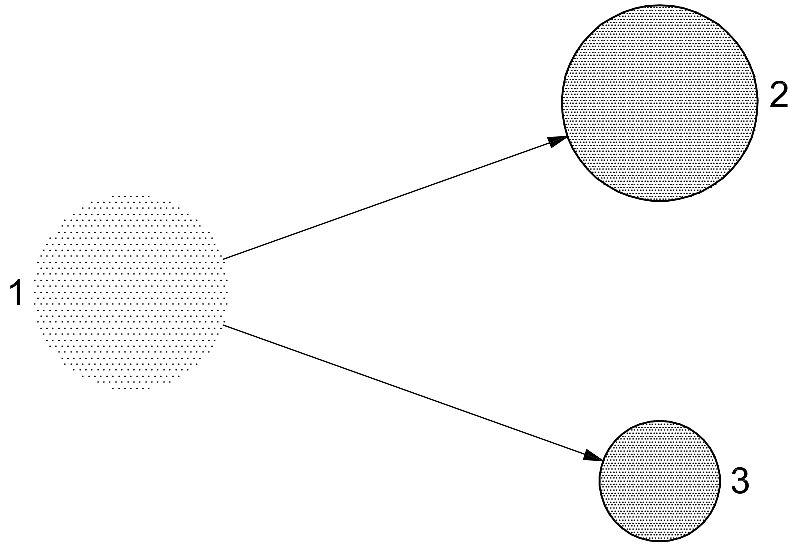
Annex A
(informative)

Calibration for the measurement of porous and conductive particles

A.1 General

Particles of conductive materials (e.g. metal particles) can be accurately measured by the electrical sensing zone method, provided that the voltage applied across the aperture does not prohibit the formation of a surface layer, the so-called Helmholz layer. This voltage may typically be 10 V to 15 V [4].

Porous particles have a pore structure which may be filled with electrolyte solution during the sample preparation procedure. This electrolyte solution will, at least to a certain extent, not contribute to the impedance change in the sensing zone of the aperture when the particle passes through it. Therefore, a porous particle generates a pulse with lower amplitude than a solid particle of equivalent envelope volume. If the calibration of the instrument is done with solid particles and no correction is carried out for the effect of the porosity, the measured size will be too small (see Figure A.1). A technique to solve this problem is to fill the pores either with an organic substance that is solid at room temperature or with a solvent that is immiscible with water [14], [15]. However, this technique cannot be used with porous particles made of natural polymers because they change size in organic solvents. Porous particles can be accurately measured after calibration of the measuring instrument using the mean size of a narrowly sized fraction of the material under investigation as described in A.3.



Key

1 porous particle
2 reported size with correction for the porosity (envelope size)
3 reported size without correction for the porosity

Figure A.1 — Diagram illustrating the response of a porous particle

A.2 Particles of conductive materials

To obtain acceptable results for conductive particles, such as metal particles, a distribution is obtained under normal conditions. The analysis is then repeated using half the current and twice the gain. The distributions should be the same. If they are not, the procedure should be repeated using an even lower current. Some metal particles are highly conductive (e.g. copper, silver and platinum). These particles do not easily form surface layers but they can be correctly sized if a very low voltage is applied and the barrier increased by adding a 0,5 % solution of Cetrimide.

A.3 Porous particles

A.3.1 General

In order to compensate for the effects of the porosity, the scaling of pulse height is done with the material under test. A narrow size-range fraction of that material is prepared by sieving or a similar separation method. The mean diameter is then determined by computer-aided microscopy, so-called image analysis, or by measurements on photographs. Finally the material is analysed by the electrical sensing zone (ESZ) instrument and the known mean diameter is used for calibration of the instrument [9], [10].

A.3.2 Sample preparation

In the case of sieving, the sieves should have openings as close as possible to the modal diameter of the sample (e.g. 5 µm to 10 µm on each side). Wet sieving with electro-formed sieves is preferred. The sieving fluid may be chosen according to the expertise of the laboratory to fit the actual material as well as possible. The size of porous materials may vary with the ion strength of the suspension medium. Therefore, the fraction shall be transferred to the electrolyte solution in which the size is to be expressed. The sample is then allowed to stand overnight or treated in an ultrasonic bath in order to substitute the liquid inside the particle for electrolyte solution.

A.3.3 Microscopy and ESZ measurements

An aliquot of the suspension of the sieved fraction is transferred to a microscope slide and covered with a cover glass. Then photographs are taken, or the particle size is measured directly with an image analyser. It is important that the sample is not allowed to dry. At least 400 particles should be measured [9]. The number of particles to be measured can also be calculated using the procedure described in ISO 13322-1. The modal diameter of the number distribution should be the preferred size parameter for calibration, but the median diameter either in number or in volume distribution may also be used. Finally, the particle size is measured with the ESZ instrument which has been calibrated with microspheres.

A.4 Calculations for microscopy

A.4.1 General

The mean diameters of the microspheres and the sieved fraction are used to calculate a response factor which can be used for future calibrations.

A.4.2 Symbols

d_m	certified mean diameter of the microspheres used for primary calibration
d_micr	mean diameter of the sieved fraction as determined using microscopy
d_ESZ	mean diameter of the sieved fraction as determined using the ESZ instrument

d_{ref} reference diameter of the microspheres

f_{resp} response factor

A.4.3 Calculations

The mean diameters are used to calculate a response factor

$$f_{\mathrm{resp}} = \frac{d_{\mathrm{micr}}}{d_{\mathrm{ESZ}}} \tag{A.1}$$

The microspheres may then be given a reference diameter

$$d_{\mathrm{ref}} = f_{\mathrm{resp}} \cdot d_{\mathrm{m}} \tag{A.2}$$

This reference diameter may then be used for future calibrations.

Annex B
(informative)

Technique using two (or more) apertures

B.1 General

When it is necessary to use more than one aperture to obtain a complete analysis, it is necessary to divide the suspension into two or more fractions of a suitable particle size that can be measured by each aperture. This is usually accomplished by wet sieving.

B.2 Separation

After analysis with the larger aperture, the beaker and the remaining suspension are weighed and the suspension passed through an electroformed or monofilament sieve with apertures equal to approximately half the diameter of the smaller aperture. The filtrate is collected in a clean weighed beaker. The first beaker is washed out on to the sieve with fresh electrolyte solution and the sieve itself is similarly washed. All the washings are collected in the second beaker. The dilution factor is calculated from the masses of the original and final suspensions.

B.3 Calibration

Suitable combinations of apertures similar to those in the Table B.1 are selected. To ensure that the overlapping regions will match, the diameter of the smaller aperture shall be more than 20 % of the diameter of the larger aperture.

An electrolyte concentration is chosen such that the solution has a resistance low enough for use with the small aperture. For example, 10 g/l sodium chloride is suitable for most aperture sizes. The electrolyte solution is prepared as recommended (see 8.5).

Both apertures are calibrated using the microsphere method (see 8.10.2) with the same calibration material. This will ensure that the size ranges in the overlap region will coincide.

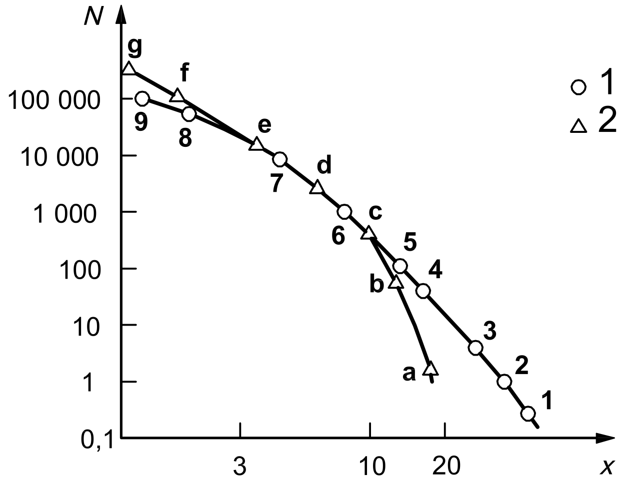
Table B.1 — Suitable aperture diameter combinations

Particle diameter range (approx.) µm	**Aperture diameter combination** µm
0,6 to 84	30/140
1,0 to 120	50/200
1,4 to 168	70/280
2,8 to 240	140/400
0,6 to 240	30/140/400

B.4 Analysis

Each fraction is analysed with the respective aperture and the analyses are combined, allowing for the different dilutions. Full operational details or on-board computer programs are available from the instrument manufacturers, but the basic method is illustrated as follows.

Obtain the particle size distribution results from the larger aperture, as coincidence-corrected cumulative numbers of oversize particles, N, versus equivalent particle diameter, x, per unit volume of original suspension, and present, for example on log-log axes, the size distribution denoted by the points **1** to **9** in Figure B.1. After correcting for coincidence and any dilution, present the results from the smaller aperture in a similar way, for example as the points **a** to **g** in Figure B.1.



Key

1 140 µm analysis

2 30 µm analysis

Figure B.1 — Illustration of the procedure for the overlap of distributions from two apertures

If the dilution factor is not known, the exact data for the smaller aperture should be multiplied by the factor which produces overlapping agreement with the central portion of the larger aperture data, as in Figure B.1.

The full size distribution will then be represented in this example by the points 1, 2, 3, 4, 5, 6, 7, e, f, g, or the points 1, 2, 3, 4, 5, 6, d, e, f, g, from which a cumulative mass or volume percentage distribution may be calculated by normal methods. In the overlap region, the counts should agree to within 10 %.

Annex C
(informative)

Chi-squared test of the correctness of instrument operation or sample preparation

C.1 General

Ideally, the number of particles in a repeated analysis should follow a Poisson distribution. In this, the variance is equal to the expected (mean) value. A significantly larger variance indicates that the particles are not homogeneously distributed in the sample dispersion; a significantly smaller variance indicates that the instrument may be counting something else as well, for example a regular electrical disturbance. The chi-squared test is suitable for testing whether the numbers of particles from repeated measurements follow a Poisson distribution or not. It can be applied to the number of particles in a single size interval, in a group of size intervals, or to the total particle count.

C.2 Symbols

χ^2	chi-squared statistical distribution
n	number of counts
N_i	number of particles in a count
$\overline{N}$	mean of particles in counts N_i $(i = 1, 2, 3 \ldots n)$
p	significance level of statistical test
df	degrees of freedom

C.3 Theory

C.3.1 General

For n repeated measurements, N_1, N_2, ..., N_n, the hypothesis of a Poisson distribution can be tested by comparison of the observed and the theoretical variances. The test statistic is

$$\chi^2 = \frac{\sum_{i=1}^{n}\left(N_i - \overline{N}\right)^2}{\overline{N}} \tag{C.1}$$

where

$$\overline{N} = \frac{\sum_{i=1}^{n} N_i}{n} \tag{C.2}$$

This test statistic has an approximate χ^2-distribution with $n-1$ degrees of freedom. The hypothesis of a Poisson distribution is rejected on the p % level if the test statistic is greater than the (100 – p)-percentile of the χ^2-distribution (see statistical tables).

Worked examples

Table C.1 — Chi-squared test of the number of particles of n experiments

$p = 0{,}05$, df = 5

n	N_i	$\overline{N}$	$N_i - \overline{N}$	$\frac{\left(N_i - \overline{N}\right)^2}{\overline{N}}$
1	936	982	–46	2,15
2	1 016	982	34	1,18
3	971	982	–11	0,12
4	1 004	982	22	0,49
5	999	982	17	0,29
6	968	982	–14	0,20
	$\overline{N}$ = 982			Sum = 4,43 = χ^2

For p = 0,05 and df = 5, the critical value of the chi-squared distribution is 11,07 (see statistical tables). The observed value is 4,43 (see Table C.1), which is smaller than the critical value. Therefore it is likely that the results follow a Poisson distribution.

Table C.2 — Chi-squared test of the number of particles of n experiments

$p = 0{,}05$, df = 6

n	N_i	$\overline{N}$	$N_i - \overline{N}$	$\frac{\left(N_i - \overline{N}\right)^2}{\overline{N}}$
1	936	964	–28	0,80
2	1 016	964	52	2,75
3	971	964	7	0,05
4	1 004	964	40	1,63
5	999	964	35	1,25
6	968	964	4	0,02
7	856	964	–108	11,88
	$\overline{N}$ = 964			Sum = 18,38 = χ^2
NOTE A partial blockage of the aperture occurred during the measurement No. 7.				

For p = 0,05 and df = 6, the critical value of the chi-squared distribution is 12,59 (see statistical tables). The observed value is 18,36 (see Table C.2), which is greater than the critical value. Therefore it is likely that the results do not follow a Poisson distribution.

Annex D
(informative)

Table of materials and electrolyte solutions

D.1 Key to electrolyte solutions

Unless otherwise stated, the electrolytes are dissolved in water.

Salts and solvents for non-aqueous use should be water-free.

a	10 g/l sodium chloride solution (often interchangeable with b)
b	Isoton II (often interchangeable with a)
c	4 g/l sodium hydroxide solution
d	0,1 mol/l hydrochloric acid solution (+ 1 g/l Cetrimide)
e	20 g/l to 50 g/l trisodium orthophosphate dodecahydrate solution or 20 g/l to 50 g/l sodium pyrophosphate decahydrate solution
f	200 g/l sodium chloride solution
g	10 g/l sodium nitrate solution
h	40 g/l zinc chloride solution
i	20 g/l to 50 g/l sodium sulfate solution
j	10 g/l potassium silicate solution
k	80 g/l ammonium thiocyanate in dimethylformamide
l	50 g/l lithium chloride in methanol (often interchangeable with m)
m	50 g/l ammonium thiocyanate or magnesium perchlorate in propan-2-ol (often interchangeable with l)
n	50 g/l ammonium thiocyanate or lithium chloride in acetone
p	100 g/l to 400 g/l lithium iodide in butan-2-ol
q	40 g/l to 50 g/l ammonium thiocyanate in butan-2-ol
r	40 g/l to 50 g/l lithium iodide in acetone
s	78,5 g/l sodium silicate solution
t	10 g/l sodium carbonate solution
u	60 g/l ammonium thiocyanate in dimethylformamide
v	40 g/l ammonium thiocyanate (in ethanol) + 5 % (volume fraction) formamide

w	2,23 g/l sodium pyrophosphate decahydrate solution
x	100 g/l sodium chloride in 85 % (volume fraction) propan-2-ol in acetonitrile
y	8 g/l sodium hydroxide solution
z	7 g/l hydrochloric acid solution
aa	80 g/l ammonium thiocyanate in 33,3 % (volume fraction) methanol in propan-1,2-diol
bb	60 g/l lithium iodide in bis(2-hydroxyethyl) ether
cc	50 g/l ammonium thiocyanate in 50 % (volume fraction) methanol in cyclohexane
dd	3 g/l sodium chloride solution
ee	40 g/l ammonium thiocyanate in 70 % (volume fraction) propan-2-ol in dichloroethane
ff	100 g/l concentrated hydrochloric acid in propan-2-ol
gg	40 g/l ammonium thiocyanate in butanone
hh	40 g/l ammonium thiocyanate in dimethyl formamide
ii	50 g/l ammonium thiocyanate in 33,33 % (volume fraction) of dimethylformamide, tetrahydrofuran and trichloroethylene
jj	45 g/l lithium chloride in 90 % (volume fraction) acetone in methanol
kk	10 g/l potassium chloride in 90 % (volume fraction) formic acid in water
ll	7,5 g/l ammonium thiocyanate in 90 % (volume fraction) butanone in trichloroethylene
mm	40 g/l ammonium thiocyanate in 50 % (volume fraction) propan-2-ol + 40 % (volume fraction) chloroethane + 10 % (volume fraction) sample
nn	38 g/l lithium chloride in 50 % (volume fraction) propan-2-ol in benzene
oo	100 g/l ammonium thiocyanate in 50 % (volume fraction) propan-2-ol in dichloromethane
pp	10 g/l hydrochloric acid in cyclohexanol
qq	Up to 250 g/l lithium iodide in 50 % (volume fraction) propan-2-ol in trichloromethane
rr	50 g/l ammonium thiocyanate in 33,33 % (volume fraction) of propan-2-ol, trichloroethane and tetrahydrofuran
ss	50 g/l ammonium thiocyanate in 33,33 % (volume fraction) of dimethylformamide, trichloroethane and tetrahydrofuran
tt	40 g/l tetrabutyl ammonium perchlorate in 50 % (volume fraction) propan-2-ol in dichloroethane
uu	60 g/l ammonium thiocyanate in bis(2-methoxyethyl) ether
vv	40 g/l ammonium thiocyanate in 83 % (volume fraction) butanone in light petroleum with 40 g/l potassium nitrate solution (ww)
ww	40 g/l potassium nitrate solution

xx 40 g/l tetrabutyl ammonium perchlorate in dimethyl formamide

yy 65 g/l sodium acetate in 66,7 % (volume fraction) ethanol in water

yz 30 g/l lithium chloride in 50 % (volume fraction) propan-2-ol in methanol

zz Karuhn's medium

D.2 Materials and recommended electrolyte solutions

See Table D.1.

NOTE 1 Where more than one electrolyte solution is given, they are indicated in preferred order.

NOTE 2 G signifies that the addition of glycerol can often help to suspend large particles;

S signifies that the addition of sucrose can often help to suspend large particles;

satd. signifies that the electrolyte solution should be presaturated with sample.

Table D.1 — Materials and recommended electrolyre solutions

Material	Electrolyte solutions (see D.1)	Comments
Acetylsalicylic acid	a satd., b satd.	Aerosol OT dispersant
Acrylic emulsion or powder	a, b, e	See chemical name
Aldactone A	a, b	(Spironolactone)
Alumina	e	Any aluminas
	a, b	Coarse powders only
Aluminium	a	Alkaline electrolyte solutions react
Aluminium oxide	—	See alumina
Aluminium silicate (Andalusite)	e	
Ammonium perchlorate	q satd., m	Common ion effect depresses solubility
Ammonium phosphate	m	Up to 80 g/l ammonium thiocyanate may be used to increase the common ion effect
Amphotericin (B)	a satd., b satd.	
Anionic bitumen emulsion	c	
Antimony	a	Daxad has been used as dispersant
Asphalt emulsion	—	See anionic or cationic bitumen emulsion
Attapulgite	e	
Avicel (microcrystalline cellulose)	a	
Azodicarbamide	m	Slightly soluble in water
Ball clay	—	See clay
Barium ferrite	r	See ferrites
Barium sulfate	i	Common ion effect suppresses solubility
Bark	a	

Table D.1 (*continued*)

Material	**Electrolyte solutions** (see D.1)	**Comments**
Barytes	e	
Bauxite	a	
Beef extract (dried)	m	
Bentonite	e	
Benzioc acid	b	
Benzyl procaine penicillin	a satd.	
Beryllia	e, a, b	
Bitumen emulsion	—	See anionic or cationic bitumen emulsion
Bone	e	
Boron carbide	a, b	Electrolyte solution has been presaturated with boric acid
Brick dust	l	
Bronze	s, t	The recommended electrolyte concentration should not be exceeded because of chemical attack
Cadmium sulfide	e	
Calamine	e	
Calcined magnesia	—	See magnesia
Calcite	—	See calcium carbonate
Calcium carbonate	l, m, a satd.	
Calcium chromate	l satd.	
Calcium dihydrogen phosphate	l	
Calcium oxide	l	
Calcium stearate	l, a, b, e	Difficult to wet; alcohols, or non-ionic dispersant with ultrasonics and spatulation, has been used
Calcium trihydrogen phosphate	l	
Carbon (and carbon black)	e, a	Activated carbon will release gases during passage through the aperture to give spurious results. Disperse the powder in a little warm glycerol with a spatula, add a little electrolyte solution and boil for a few minutes. Cool, add the remainder of electrolyte solution and count. Dispersion under vacuum was used
Carbonyl iron	a(+G), b(+G)	
Carbonyl nickel	a(+G), b(+G)	
Carborundum	—	See alumina and emery
Casein	m	
Cationic bitumen emulsion	d	
Cellulose	n, m	n is generally more suitable, but it cannot be used with apertures above 280 µm unless the flow rate is reduced, owing to whistling high-frequency noise interference on rapid flow through the aperture and/or vapour bubble formation in the aperture
Cement	l, m	

Table D.1 (*continued*)

Material	**Electrolyte solutions** (see D.1)	**Comments**
Ceramic powders	e	
Cerium oxide	e	
Chalk	—	See calcium carbonate
China clay (kaolin)	—	See clay
Cloramphenicol	a satd.	
Chocolate	m satd.	Melt in beaker over water bath at 40 °C to 50 °C, spatulate with 50 g/l alcohol-soluble fraction of Span 80 in cyclohexanol. Cool and add electrolyte solution. Presaturation with defatted solids (from petroleum ether) is essential for milk-chocolate analysis, and sucrose may be used for dark chocolate
Chromium powder	e	
Clay	e	Usually very fine. It is suitable for 30 µm or 50 µm apertures. It is common to make up a dispersion and leave for one or two days before analysis. When the mass integration method is used, the density should be measured for the material suspended in the same electrolyte solution for a similar time; this because of water present in the lattice structure
Coal	e, a, b, m, w	
Cocoa	l	
Coffee	l, m	For powder from coffee beans only
Coke	a, b, e	
Copper	a, b	No reaction during time for analysis
DDT	a satd., e satd.	
Diatomite	—	See clay
Dolomitic lime	l	
Dust (flue, coal, etc.)	e, a, b	e is a better choise if metal particles are suspected in the sample, e.g. gas mains dust
Electrolyte solutions (particles in)	—	To measure particles in electrolyte solutions, e.g. plating solutions, measure directly, or if the particle size range is reduced because the aperture resistance is too low, dilute with filtered distilled water
Emery	a(+G), b(+G)	Usually in very narrow size ranges, e.g. 75 µm to 85 µm
Emulsions (including lubrication and coolant emulsions for rolling mills)	a, b	Solids or oil in water can be measured. Two-stage dilution of, for example, 1:100 for each stage is preferable to reduce coagulation. The stability of emulsion over the analytical period should be maintained NOTE Measurement of water in oil is not possible at present.
Encapsulated particles	l, m	Depending on the encapsulant material
Explosives: HMX, PETN, RDX	a satd.	See also ammonium perchlorate and guanidine perchlorate
Feldspar	a, b	

Table D.1 (*continued*)

Material	**Electrolyte solutions** (see D.1)	**Comments**
Ferrites	e	Addition of 500 g/l glycerol reduces re-agglomeration rate Heat-treated ferrites are magnetic and cannot be dispersed to primary particles; the agglomerate size will be fairly stable (1 µm to 20 µm) and measurable and it is preferable to measure before heat-treatment
Fibres (paper pulp)	a	
Fibres (wool top)	m, x	
Fibres (in glycerine)	b	
Filters	—	See membrane filters
Flint	a, e	
Flour	m, l	Usual range 10 µm to 125 µm; disperse by spatulation in anhydrous electrolyte solution, then use ultrasonics
Fly ash	w	
Fullers earth	e	
Garnet	e	
Glass powder	a, b	Almost any electrolyte solution may be used
Gold	a, b, e, g(+G)	
Graphite	e	Satisfactory for coarse graphites; very slow flocculation at 1 µm level
	y	For fine graphite
Graphite in oil	—	See oil
Griseofulvin	a satd.	Add 0,1 g/l Goulac for dispersion
Gypsum	m	
Guanidine perchlorate	q	
Herbicides	a, b	Usually insoluble
Indomethacin	z satd.	
Injection fluids (particles in parenteral fluids)	—	Usually need no added electrolyte solution. If no electrolyte is present, add filtered sodium chloride solution. Iron dextran apparently has never been analysed successfully, possibly because of complex formation giving unstable counts
Ink, ball point	aa	
Ink, in toluene	cc	All particles are very fine; many particles are not measurable. Highly coloured suspensions make observation of aperture difficult
Ink, printing	zz	
Ink, silk screening	m, bb	
Ion-exchange resin	a, f	Saturate sample electrolyte solution f to exhaust resin before analysing
Indium	e(+S)	Extremely dense (22 g/cm^3): with 500 g/l added sugar, analysis can be performed up to 60 µm to 80 µm
Iron	dd(+G), e	

Table D.1 (*continued*)

Material	**Electrolyte solutions** (see D.1)	**Comments**
Iron oxide	a(+G), b(+G)	
Kaolinite	—	See clay
Kerosene (paraffin) (particles)	ee	Add up to 20 % (volume fraction) kerosene
Ketchup (catsup)	a, b	It has been reported that the electrical sensing zone method may not respond to envelope volume for tomato cells
Latex (rubber)	a, b, e	
Latex (synthetic)	a, b, e	
Lead	a(+S), f(+S)	Methanol has been used as a dispersant
Lead(II) oxide and lead(III) oxide	a(+G)	Up to 250 g/l glycerol has been used
Lead, red	e	
Lignite dust	e	
Lime	a satd., l, m	
Lycopodium powder	a, b	
Magnesia	m, l	Reactive in water when finely divided
Magnesia, calcined	e(+G)	
Magnesium	e(+S)	Very slow reaction; will not affect accuracy of results in usual analytical time
Magnesium hydroxide	p, e satd.	
Membrane filters		See particles captured by membrane filters
Mica	e, a, b	
Molybdenium disulfide	y, e	
	m	More suitable for MoS_2 in oil
Mud	a, b, e	
Neomycin	l satd.	
Nickel	a	See also Raney nickel
Nylon, particles in	kk	Wet first with dispersant, then add electrolyte solution
Ocean sediment	—	See sediment
Oil, cutting	a, b	
Oil, hydraulic and lubricating	ll	Will accept up to 50 % (volume fraction) of added oil (DTD 585)
(Oil specifications are for lubrication effectiveness, not composition, DTD 585 from one manufacturer may not dissolve in the electrolyte solution suitable for DTD 585 from another manufacturer.)	mm nn	Electrolyte solution will accept 33 % (volume fraction) to 50 % (volume fraction) Skydrol
	oo	Will accept 50 % (volume fraction) volume of oil (MIL 5606 B, MIL 7808 E and DTD 585)
	pp, qq, rr, ss	
	tt	For MIL 5606
	uu	For lube oil
	vv, zz	

Table D.1 (*continued*)

Material	Electrolyte solutions (see D.1)	Comments
Orange extract	a, b	
Paint (oil based)	gg, zz	
Paper pulp	a, b	
Particles captured by membrane filters	ff	Dissolve the membrane in 30 % (volume fraction) dimethyl formamide in acetone before adding an equal volume of electrolyte solution. The electrolyte solution and solvent mixture should be filtered and stored separately
	gg, hh, ii, jj	Ideal for particles in oil: filter the known volume of oil through membrane, then wash through with filtered petrol, carbon tetrachloride, trichloro-ethylene, etc. and dry the filter before dissolving. Extra dimethyl formamide increases membrane solubility
Peanut butter	m	
Penicillin	a satd.	
Phenacetin	a satd.	
Phenothiazine	a satd.	
Phosphors	a, b, e	Size range approximately 1 µm to 40 µm
Photographic emulsions	a, i	40 g/l potassium nitrate solution may also be used
Pigments	e, a, b	
Plaster of Paris	l, k	Electrolyte solution should be anhydrous
Plastics	a, b, e	
Plating solutions	—	Analysed with no added electrolyte or diluted with filtered distilled water
Pollens	a, b	
Polyethylene	a, b	
Polypropylene	a, b	
Polystyrene	a, b	
Poly(styrene-divinylbenzene)	a, b, e	Useful for calibration of apertures with most organic electrolytes
Polytetrafluoroethylene	a	Almost any electrolyte solution is suitable. For finer powders, use alcohols or ketones to ensure thorough wetting
Polyvinyl acetate	a	
Polyvinyl chloride	a, b	
Polyvinyl propylodone	a	
Polyvinyl toluene	a, b	
Porcelain	e	
Potassium chloride	l	
Potassium sulfate	l	
Potato starch	a	

Table D.1 (*continued*)

Material	**Electrolyte solutions** (see D.1)	**Comments**
Powdered milk	m	For fraction insoluble in alcohol
	a	For fraction insoluble in water. Disperse in a few drops of 200 g/l sodium hydroxide solution
Quartz	e	
Raney nickel in xylene	l, m	Söow reaction with m
River sediment	—	See sediment
River water	a, b	
	e	If no calcium salts are present
Rouge	a	
Rust in gasoline	e	Filter out and resuspend in electrolyte solution
Rutile	e	
Sand	a, b, e	
Sediment	a, b, e	
Shale	e	
Silica	e	
Silica gel	l	
Silicates	e	
Silicon carbide	a, b, e	
Silicon nitride	m	
Silver halide	ww	
	a	For silver bromide
Silver oxide	a, g	
Slag (basic)	a, e	
Sodium (metal)	—	Dispersions are usually in oil or grease, and electrolyte solutions made from ammonium thiocyanate and alcohols or ketones are suitable (see oils). Coupling agents, e.g. 1,1,2-trichloroethane, may be needed
Sodium chloride	msatd.	
Sodium hydrogen carbonate	m	
Sodium hydroxide	xx satd.	Still not fully stable; use multi-channel models
Soya flour	m, b	
Spironolactone	a, b	
Starch	a, e, l, m	Aqueous electrolytes suitable for hydrated form only
Stearates	a, l	Difficult to wet. Spatulate with alcohol and use ultrasonics
Steel	a(+G), f(+G)	
Sugar	m satd.	
Sulfadimidine	a satd.	
Sulfur	a, e	Wet by spatulation with alcohol before diluting with electrolyte solution

Table D.1 (*continued*)

Material	**Electrolyte solutions** (see D.1)	**Comments**
Superphosphate (of lime)	m	
Talc	e	
Tantalum	a (+G), e(+G), f(+G)	
Tin	a(+G), f(+G)	
Tin oxide	e	Disperse in 50 g/l Calgon 1) solution
Titanium dioxide	e	
Tomato juice	a	
Toner, xerographic	a	
Tungsten	a, e(+G/S), f(+G)	
Tungsten carbide	e(+G), a(+G)	Can cold-sinter within hours of milling to cause permanent agglomeration
Uranium	e(+G), f(+G)	
Uranium dioxide	e, c	Can cold-sinter (see tungsten carbide)
Viscose	c	Or dilute with filtered distilled water
Water contaminants in	a	Dilute as required into electrolyte solution
Whiting	l, m, a satd.	See calcium carbonate
Wool fibre	m	
Yeast	a	
Yttrium iron garnet	e(+G)	Magnetic and needs frequent redispersing by ultrasonics
Zeolite	—	See clay
Zinc	yy	10 g/l Aerosol O.T. dispersant
Zinc cadmium sulfide	e	Range usually 1 µm to 15 µm
Zinc oxide	l, a, e satd.	Slightly soluble in water
Zinc stearate	l	See calcium stearate
	a, b, e	Wet
	h, j	
Zinc sulfide	l	Not very suitable as the sulfide slowly oxidises to sulfate
Zirconium oxide	a	Wet with 40 g/l sodium pyrophosphate solution

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1) Example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement of this product by ISO.

Annex E
(informative)

Mass integration method

E.1 General

The mass integration method is generally believed to be close to an absolute method. Here the volume concentration of the suspension as determined by the instrument is compared to the true volume concentration determined from the mass per unit volume and the immersed density of the particles. This method is directly traceable and there is no assumption made about the shape, porosity or electrical conductivity of the particles [11], [12]. The method can be used both for calibration and for a mass balance. In a mass balance, it is possible to determine whether the smallest particles are measured or if they are out of the range of the aperture. Some instruments have a dead time in the pulse processing circuitry which can be significant and can correspond to an effective loss in the mass of the particles analysed. A loss will influence the result of a mass balance or a mass calibration procedure, therefore it is important to keep it low. The loss will depend on the total number of particles counted. Therefore, a low particle concentration shall be used (e.g. with a coincidence risk lower than 5 %). For highest possible accuracy, coincidence-corrected counts should be used.

E.2 Calibration procedure

E.2.1 Volume V_m of analysed suspension

In some instruments, the accuracy of V_m is guaranteed to better than 0,5 % for all settings, but in others only one metered volume may be guaranteed by the manufacturer. For these instruments, it is not sufficient to accept the nominal value of the analysis volumes. Using a suspension of particles, at a statistically valid count level (see Clause 7), five times measure the total coincidence-corrected particle count with the guaranteed volume. Switch to another analysis volume and obtain the total coincidence-corrected particle counts at least five times. The ratio of the total number of counts will be the ratio of the guaranteed volume to the selected volume. All counts should be recorded. The counts shall follow a chi-squared distribution, see Annex C.

E.2.2 Immersed density of particles

Using a balance weighing to an accuracy of 0,1 mg, a relative-density bottle, pipettes and the usual particle-dispersion method, the immersed density, ρ, of the material in the electrolyte solution to be used for the analysis is determined (see ISO 787-10).

E.2.3 Sample preparation

A narrow size-range fraction of the material under test is prepared by sieving or a similar separation method. At least 99 % of the mass of the particles should lie within a size range of no more than 10:1, so that all can be measured using one aperture.

A suspension is prepared by dispersing a mass, m, of the powder in a volume, V_T, of the electrolyte solution and the dispersing agent.

E.2.4 Determination of the calibration factor

The calibration factor, K_d for the instrument is then determined by measuring very carefully the size distribution using an accurately known analysis volume, V_m, ensuring that the aperture is absolutely clear. A number of analysis volumes may be counted or the analysis time may be long so that many particles are measured (e.g. 50 000 particles yields a relative standard deviation of 0,4 %). Fewer particles may be measured but this will lower the precision of the measurement. The particle concentration used should be less than 5 % coincidence limit. The coincidence-corrected counts shall be recorded and used in the calculations.

K_d is calculated from the formula:

$$K_d = \left(\frac{V_\mathrm{m} m}{V_\mathrm{T} \rho \sum\limits_{i=1}^{n} \Delta N_i \overline{V}_i} \right)^{1/3} \tag{E.1}$$

where

$$\overline{V}_i = \frac{\frac{\pi}{6}\left(d_\mathrm{L}^3 + d_\mathrm{U}^3\right)}{2} \tag{E.2}$$

E.2.5 Secondary calibration

It is not necessary to perform the full mass-integration calibration procedure for every analysis or for every aperture. It is recommended that, having calibrated for the aperture and material under test, a secondary standard, such as a microsphere suspension, be analysed and used as an intermediate standard to calibrate other apertures within their linear range (see 8.10.2) and for regular checks of the stability of calibration. Analyses reported using this secondary calibration should be identified.

E.2.6 Example of calibration by mass integration

See Table E.1.

Table E.1 — Example of calibration by mass integration

Diameter µm	$\overline{V}_i$ µm^3	N_i	ΔN_i	$\Delta N_i \overline{V}_i$ µm^3	$\sum \Delta N_i \overline{V}_i$ µm^3
15-17	$2{,}169\,79 \times 10^3$	487	487	$1{,}056\,69 \times 10^6$	$1{,}056\,69 \times 10^6$
17-19	$3{,}081\,90 \times 10^3$	898	411	$1{,}266\,66 \times 10^6$	$2{,}323\,35 \times 10^6$
19-21	$4{,}220\,21 \times 10^3$	1 301	403	$1{,}700\,74 \times 10^6$	$4{,}024\,09 \times 10^6$
21-23	$5{,}609\,84 \times 10^3$	1 691	390	$2{,}187\,84 \times 10^6$	$6{,}211\,93 \times 10^6$
23-25	$7{,}275\,93 \times 10^3$	2 190	499	$3{,}630\,69 \times 10^6$	$9{,}842\,62 \times 10^6$
25-27	$9{,}243\,61 \times 10^3$	2 921	731	$6{,}757\,08 \times 10^6$	$1{,}659\,97 \times 10^7$
27-29	$1{,}153\,80 \times 10^4$	4 175	1 254	$1{,}446\,87 \times 10^7$	$3{,}106\,84 \times 10^7$
29-31	$1{,}418\,43 \times 10^4$	6 678	2 503	$3{,}550\,33 \times 10^7$	$6{,}657\,17 \times 10^7$
31-33	$1{,}720\,76 \times 10^4$	11 602	4 924	$8{,}473\,00 \times 10^7$	$1{,}513\,02 \times 10^8$
33-35	$2{,}063\,29 \times 10^4$	20 384	8 782	$1{,}811\,98 \times 10^8$	$3{,}325\,00 \times 10^8$
35-37	$2{,}448\,56 \times 10^4$	31 583	11 199	$2{,}742\,14 \times 10^8$	$6{,}067\,14 \times 10^8$
37-39	$2{,}879\,06 \times 10^4$	42 342	10 759	$3{,}097\,58 \times 10^8$	$9{,}164\,72 \times 10^8$

Table E.1 (*continued*)

Diameter µm	$\bar{V}_i$ µm³	N_i	ΔN_i	$\Delta N_i \bar{V}_i$ µm³	$\sum \Delta N_i \bar{V}_i$ µm³
39-41	$3{,}357\ 32 \times 10^{4}$	50 083	7 741	$2{,}598\ 90 \times 10^{8}$	$1{,}176\ 36 \times 10^{9}$
41-43	$3{,}885\ 84 \times 10^{4}$	54 548	4 465	$1{,}735\ 03 \times 10^{8}$	$1{,}349\ 86 \times 10^{9}$
43-45	$4{,}467\ 14 \times 10^{4}$	56 803	2 255	$1{,}007\ 34 \times 10^{8}$	$1{,}450\ 59 \times 10^{9}$
45-47	$5{,}103\ 73 \times 10^{4}$	57 840	1 037	$5{,}292\ 57 \times 10^{7}$	$1{,}503\ 52 \times 10^{9}$
47-49	$5{,}798\ 12 \times 10^{4}$	58 369	529	$3{,}067\ 21 \times 10^{7}$	$1{,}534\ 19 \times 10^{9}$
49-51	$6{,}552\ 84 \times 10^{4}$	58 659	290	$1{,}900\ 32 \times 10^{7}$	$1{,}553\ 19 \times 10^{9}$
51-53	$7{,}370\ 39 \times 10^{4}$	58 804	145	$1{,}068\ 71 \times 10^{7}$	$1{,}563\ 88 \times 10^{9}$
53-55	$8{,}253\ 28 \times 10^{4}$	58 894	90	$7{,}427\ 95 \times 10^{6}$	$1{,}571\ 37 \times 10^{9}$
55-57	$9{,}204\ 03 \times 10^{4}$	58 936	42	$3{,}865\ 69 \times 10^{6}$	$1{,}575\ 24 \times 10^{9}$
57-59	$1{,}022\ 52 \times 10^{5}$	58 963	27	$2{,}760\ 80 \times 10^{6}$	$1{,}578\ 00 \times 10^{9}$
59-61	$1{,}131\ 92 \times 10^{5}$	58 976	13	$1{,}471\ 50 \times 10^{6}$	$1{,}579\ 47 \times 10^{9}$
61-63	$1{,}248\ 86 \times 10^{5}$	58 987	11	$1{,}373\ 75 \times 10^{6}$	$1{,}580\ 84 \times 10^{9}$
63-65	$1{,}373\ 59 \times 10^{5}$	58 993	6	$8{,}241\ 54 \times 10^{5}$	$1{,}581\ 66 \times 10^{9}$
65-67	$1{,}506\ 36 \times 10^{5}$	58 993	0	$0{,}000\ 00 \times 10^{5}$	$1{,}581\ 66 \times 10^{9}$

$K_{d\,\mathrm{a}} =$ 294,1

$V_\mathrm{m} =$ 100,000 ml

$m =$ 25,2 mg $= 25{,}2 \times 10^{-3}$ g

$V_\mathrm{T} =$ 333,695 0 ml

$\rho =$ 2,23 g/ml

$$\sum \Delta N_i \bar{V}_i = 1{,}581\,66 \times 10^{9}\,(\mu\mathrm{m})^{3} = 1{,}581\,66 \times 10^{9} \times 10^{-12}\,\mathrm{ml}$$

$$K_d = 294{,}1\sqrt[3]{\frac{100{,}00 \times 25{,}2 \times 10^{-3}}{333{,}6950 \times 2{,}23 \times 1{,}581\,66 \times 10^{9} \times 10^{-12}}}$$

$K_d =$ 379,1

The mean particle volume of a size interval $\Delta N_i \bar{V}_i$ and the total particle volume $\sum_{i=1}^{n} \Delta N_i \bar{V}_i$ may be calculated by the computer software provided with the particle size analyser. For precise information about the specific software, the instrument manufacturer or the instrument operation manual should be consulted.

E.3 Mass balance

E.3.1 General

The mass balance compares the volume of the particles measured by the instrument with that calculated from the true particle concentration as determined by weighing the powder sample dispersed in the weighed volume of electrolyte solution for that measurement. The ratio of these two values gives the fraction of powder that is accounted for in the measurement. This ratio gives guidance as to whether the smallest particles are accounted for. See also E.1.

E.3.2 Symbols

M_m mass of particles measured by the instrument

M_b mass of particles accounted for in a measurement

E.3.3 Calibration

Calibration should preferably be done by the mass calibration procedure. The procedures described in E.2.4 to E.2.6 shall be followed. The volume V_m of analysed suspension shall be determined as described in E.2.1 and the immersed density of the particles as described in E.2.2. The instrument may also be calibrated by the microsphere method.

E.3.4 Sample preparation

A suspension is prepared by dispersing a mass, m, of the powder in a volume, V_T, of the electrolyte solution and the dispersing agent. Weigh the sample and the electrolyte solution with highest possible accuracy.

E.3.5 Procedure

When the particles are dispersed satisfactorily, the analysis can begin. Measure very carefully, on a linear scale, the size distribution ensuring that the aperture is absolutely clear. For the calculation to be reasonably accurate, the size interval should be narrow (i.e. a large number of channels should be counted). The coincidence-corrected counts shall be recorded and used in the calculations.

E.3.6 Calculations

The mass measured by the instrument may be calculated by the formula

$$M_\mathrm{m} = \frac{\sum \Delta N_i \overline{V}_i \left(\rho \cdot V_\mathrm{T}\right)}{V_\mathrm{m}} \qquad \text{(E.3)}$$

The particles accounted for, in percent, may be calculated by the formula

$$M_\mathrm{b} = \frac{M_\mathrm{m}}{m} \times 100\ \% \qquad \text{(E.4)}$$

E.3.7 Results

The ratio of the volume of the particles measured by the instrument and the true particle concentration, as determined by weighing the powder sample dispersed in the weighed volume of electrolyte, should deviate from unity by no more than 3 %. A value deviating by more than 3 % indicates that not all particles can be measured using one aperture. Then the two (or more) aperture technique (see Annex B) should be used to achieve highest possible accuracy.

E.3.8 Report

It is essential that the whole mass integration procedure and all data be reported in full detail on a suitable data sheet.

E.3.9 Example of mass balance

See Table E.2.

Table E.2 — Example of mass balance

Diameter µm	$\overline{V}_i$ µm^3	N_i	ΔN_i	$\Delta N_i \overline{V}_i$ µm^3	$\sum \Delta N_i \overline{V}_i$ µm^3
19,33 to 21,91	$4,644\ 45 \times 10^{3}$	487	487	$2,261\ 85 \times 10^{6}$	$2,261\ 85 \times 10^{6}$
21,91 to 24,49	$6,598\ 91 \times 10^{3}$	898	411	$2,712\ 15 \times 10^{6}$	$4,974\ 00 \times 10^{6}$
24,49 to 27,07	$9,038\ 52 \times 10^{3}$	1 301	403	$3,642\ 52 \times 10^{6}$	$8,616\ 52 \times 10^{6}$
27,07 to 29,65	$1,201\ 72 \times 10^{4}$	1 691	390	$4,686\ 72 \times 10^{6}$	$1,330\ 32 \times 10^{7}$
29,65 to 32,23	$1,558\ 90 \times 10^{4}$	2 190	499	$7,778\ 92 \times 10^{6}$	$2,108\ 22 \times 10^{7}$
32,23 to 34,80	$1,979\ 83 \times 10^{4}$	2 921	731	$1,447\ 25 \times 10^{7}$	$3,555\ 47 \times 10^{7}$
34,80 to 37,38	$2,470\ 70 \times 10^{4}$	4 175	1254	$3,098\ 26 \times 10^{7}$	$6,653\ 73 \times 10^{7}$
37,38 to 39,96	$3,037\ 87 \times 10^{4}$	6 678	2503	$7,603\ 78 \times 10^{7}$	$1,425\ 75 \times 10^{8}$
39,96 to 42,54	$3,685\ 89 \times 10^{4}$	11 602	4924	$1,814\ 93 \times 10^{8}$	$3,240\ 68 \times 10^{8}$
42,54 to 45,12	$4,420\ 18 \times 10^{4}$	20 384	8782	$3,881\ 80 \times 10^{8}$	$7,122\ 48 \times 10^{8}$
45,12 to 47,69	$5,244\ 34 \times 10^{4}$	31 583	11 199	$5,873\ 14 \times 10^{8}$	$1,299\ 56 \times 10^{9}$
47,69 to 50,27	$6,165\ 35 \times 10^{4}$	42 342	10 759	$6,633\ 30 \times 10^{8}$	$1,962\ 89 \times 10^{9}$
50,27 to 52,85	$7,190\ 39 \times 10^{4}$	50 083	7 741	$5,566\ 08 \times 10^{8}$	$2,519\ 50 \times 10^{9}$
52,85 to 55,43	$8,323\ 24 \times 10^{4}$	54 548	4 465	$3,716\ 33 \times 10^{8}$	$2,891\ 13 \times 10^{9}$
55,43 to 58,01	$9,569\ 31 \times 10^{4}$	56 803	2 255	$2,157\ 88 \times 10^{8}$	$3,106\ 92 \times 10^{9}$
58,01 to 60,58	$1,093\ 11 \times 10^{5}$	57 840	1 037	$1,133\ 56 \times 10^{8}$	$3,220\ 28 \times 10^{9}$
60,58 to 63,16	$1,241\ 67 \times 10^{5}$	58 369	529	$6,568\ 42 \times 10^{7}$	$3,285\ 96 \times 10^{9}$
63,16 to 65,74	$1,403\ 42 \times 10^{5}$	58 659	290	$4,069\ 93 \times 10^{7}$	$3,326\ 61 \times 10^{9}$
65,74 to 68,32	$1,578\ 66 \times 10^{5}$	58 804	145	$2,289\ 06 \times 10^{7}$	$3,349\ 55 \times 10^{9}$
68,32 to 70,90	$1,767\ 91 \times 10^{5}$	58 894	90	$1,591\ 12 \times 10^{7}$	$3,365\ 46 \times 10^{9}$
70,90 to 73,47	$1,971\ 30 \times 10^{5}$	58 936	42	$8,279\ 45 \times 10^{6}$	$3,373\ 74 \times 10^{9}$
73,47 to 76,05	$2,189\ 75 \times 10^{5}$	58 963	27	$5,912\ 32 \times 10^{6}$	$3,379\ 65 \times 10^{9}$
76,05 to 78,63	$2,424\ 23 \times 10^{5}$	58 976	13	$3,151\ 50 \times 10^{6}$	$3,382\ 81 \times 10^{9}$
78,63 to 81,21	$2,674\ 88 \times 10^{5}$	58 987	11	$2,942\ 37 \times 10^{6}$	$3,386\ 08 \times 10^{9}$
81,21 to 83,79	$2,942\ 25 \times 10^{5}$	58 993	6	$1,765\ 35 \times 10^{6}$	$3,387\ 84 \times 10^{9}$
83,79 to 86,37	$3,226\ 87 \times 10^{5}$	58 993	0	0,000 00	$3,387\ 84 \times 10^{9}$

$V_\mathrm{m} =$ 100,00 ml

$m =$ 25,2 mg $= 25{,}2 \times 10^{-3}$ g

$V_\mathrm{T} =$ 333,695 0 ml

$\rho =$ 2,23 g/ml

$$\sum \Delta N_i \overline{V}_i = 3{,}387\,84 \times 10^{9} \left(\mu\mathrm{m}^{3}\right) = 3{,}387\,84 \times 10^{9} \times 10^{-12}\,\mathrm{ml}$$

Using Equations (E.3) and (E.4) gives

$$M_\mathrm{m} = \frac{3{,}387\,84 \times 10^{9} \times 10^{-12} \times 2{,}23 \times 333{,}695\,0}{100{,}00}\,\mathrm{g} = 0{,}025\,21\ \mathrm{g}$$

$$M_\mathrm{b} = \frac{0{,}025\,21}{25{,}2 \times 10^{-3}} \times 100 = 100{,}0$$

The recovery of particles is 100 % with the aperture in use.

Annex F
(informative)

Calibration and control of frequently used apertures

This procedure is for the apertures that are used continually or very frequently. Twenty calibrations should be made consecutively using microspheres (see 8.10.2) over a short period of time (e.g. within a day or preferably during a week). Calculate the mean calibration constant $\overline{K}_d$ and its standard deviation σ_{K_d} from this data. Plot the data in a Shewhart chart with warning limits at $\overline{K}_d \pm 2\sigma_K$, and action limits at $\overline{K}_d \pm 3\sigma_K$, The mean calibration constant $\overline{K}_d$ is used in the subsequent analyses. The use of the mean ensures that random errors in the determination of the constant are reduced. Repeat the calibration immediately if the result falls between $\pm 2\sigma_K$ and $\pm 3\sigma_K$ (the warning area). Take action in the form of cleaning or repair if two consecutive results are outside $\pm 2\sigma_K$, or if one result is outside $\pm 3\sigma_K$, from the mean.

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