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Determination of particle size distributions -- Electrical sensing zone method

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

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard 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*.

Annexes A to E of this International Standard are for information only.

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Determination of particle size distributions — Electrical sensing zone method

1 Scope

This International Standard gives guidance on the measurement of the size distributions of particles dispersed in an electrolyte solution using the electrical sensing zone method. It does not address the specific requirements of the particle size measurement of specific materials. The method described in this International Standard measures particle volumes and reports in the range about from 0,6 μm to 1 600 μm.

2 Terms and definitions

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

2.1
dead time
time during which the electronics are not able to detect particles due to the signal processing of a previous particle

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2.2
orifice
small-diameter hole through which suspension is drawn

2.3
sensing zone
volume of electrolyte solution within, and around, the orifice in which a particle is detected

2.4
sampling volume
volume of suspension that is analysed

3 Symbols

|	
D	orifice diameter, in μm
K_d	calibration constant of diameter
$\overline{K}_d$	calibration constant of mean diameter
$\sigma_{\overline{K}_d}$	standard deviation of mean calibration constant
m	mass of sample in beaker, in g
V_T	volume of electrolyte solution in which m is dispersed, in ml
V_m	analysis volume, in ml
ΔN_i	number of counts in a size interval i
ρ	mass of the particles per volume of the electrolyte it displaces, in g·ml^{-1}
$\overline{V}_i$	arithmetic mean volume for a particular size interval i, in ml

1

V_i volume of the particle obtained from the threshold, or channel boundary and instrument units; without an arbitrary calibration to particle diameter (i.e. $V_i = tIA$, where t = threshold level, I = current through aperture and A = attenuation factor), in ml

x diameter of a sphere with volume equivalent to that of the particle, in μm

x_{50}, x_{10}, x_{90} the values of x corresponding to the 50 %, 10 % and 90 % percentile points of the cumulative per cent undersize distributions, in μm

4 Principle

The response, i.e. the electrical pulse generated when a particle passes through the orifice, has been found both experimentally and theoretically to be proportional to the particle volume (see Bibliography). A dilute suspension of particles dispersed in an electrolyte solution is stirred to provide a homogeneous mixture and is drawn through a small orifice, or aperture, in an insulating wall. A current applied across two electrodes, placed on each side of the orifice, enables the particles to be sensed by the electrical impedance changes as they pass through the orifice. The particle-generated pulses 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.
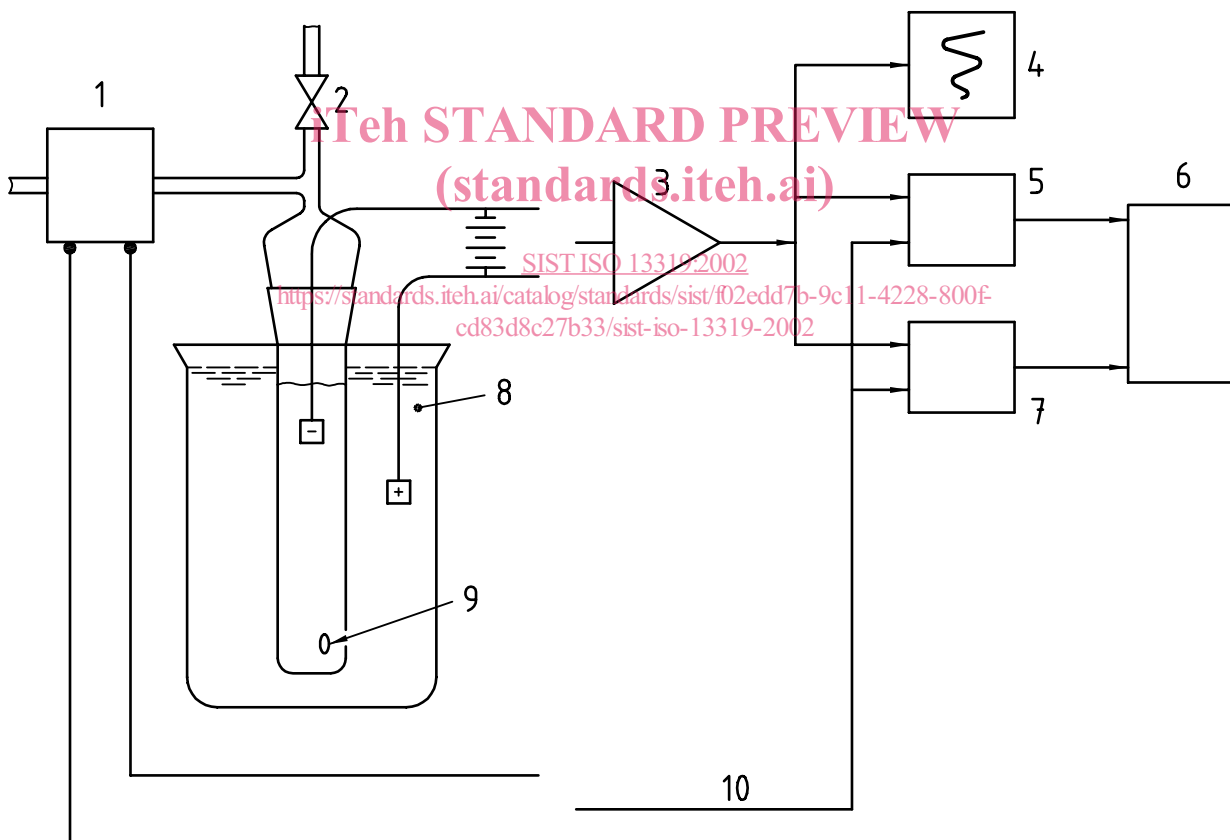
Key

1	Volumetric metering device	6	Output
2	Valve	7	Pulse-height analyser
3	Pulse amplifier	8	Stirred suspension of particles in electrolyte solution
4	Oscilloscope pulse display	9	Aperture
5	Counting circuit	10	Counter start/stop

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

5 General operation

5.1 Response

If the particles are spherical, the electrical response is proportional to the volume of the particles. 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. In general, particles should have a low conductivity with respect to the electrolyte solution, but conducting particles can be measured.

5.2 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 a lower limit is possible. There is no theoretical upper limit, and for particles having a density similar to that of the electrolyte solution, the largest orifice available (normally 2 000 μm) may be used.

When the particle density is high, the upper size limit is reached when the particles can no longer be kept in homogeneous suspension. In this case, the viscosity and/or the density of the electrolyte solution has to be increased, for example by the addition of glycerol or sucrose.

The size range for a single orifice sensor is proportional to the orifice diameter, D. The response has been found to depend linearly on D over a range from 0,015 D to 0,80 D (i.e. 1,5 μm to 80 μm for a 100 μm orifice), although the orifice may become prone to blockage at levels greater than 0,60 D. This range can be extended by using two or more sensors (see annex B) but in practice this procedure can be avoided by the careful selection of the diameter of one sensor, to achieve an acceptable range.

The response of the instrument is dependent on the effective electrical resistance of the particle, which is usually high. The measurement of conducting particles (e.g. metals, carbon, silicon and many types of cells and organisms, such as blood cells) requires more time to implement. The particles can become electrically translucent (i.e. give a smaller electrical pulse than their volume indicates) if a voltage, typically of 10 V to 15 V or more, is applied between the electrodes. To obtain acceptable results, a distribution is obtained under normal conditions. The analysis is then repeated using half the current and twice the gain (1/attenuation). The distributions should be the same. If they are not, the procedure should be repeated using an even lower current.

5.3 Effect of coincident particle passage

Ideal data would result if particles traversed the orifice 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 particle distribution obtained but can be minimized by using low concentrations. Table 1 shows counts per millilitre for the coincidence to be 5 % (i.e. approximately only one particle in twenty is affected). 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.

5.4 Dead time

In some modern instruments, pulse-height analysis routines are used to process the data. Since it takes a finite time to process each pulse, it is possible that the analyser may not count particles for a given time after receiving a pulse. This means that, for a relatively high count rate, a significant proportion of the counts may be lost. Since dead time is not a function of the pulse height, the loss will be proportional to the counts in each channel and will not affect the size distribution. However, if concentration is to be reported or the mass integration method of calibration (see 6.11.3) is to be used, the effect can be kept to a minimum by using dilute suspensions (e.g. at < 5 % coincidence) and setting up the instrument so that the pulses in the lowest channels are not counted. This is

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done by first obtaining a count distribution and observing the number of counts per channel. A typical result is shown in Figure 2. By restricting the counts in the lowest channel to that shown by A, the dead time will be minimized.

In normal operation, this dead time will not cause any distortion of the size distribution since all particles will have the same chance of not being counted, provided that a large number of particles, at least 100 000, are counted. However dead time will affect the accuracy of the mass integration method of calibration (see 6.11.3), when there will be an apparent loss of mass.
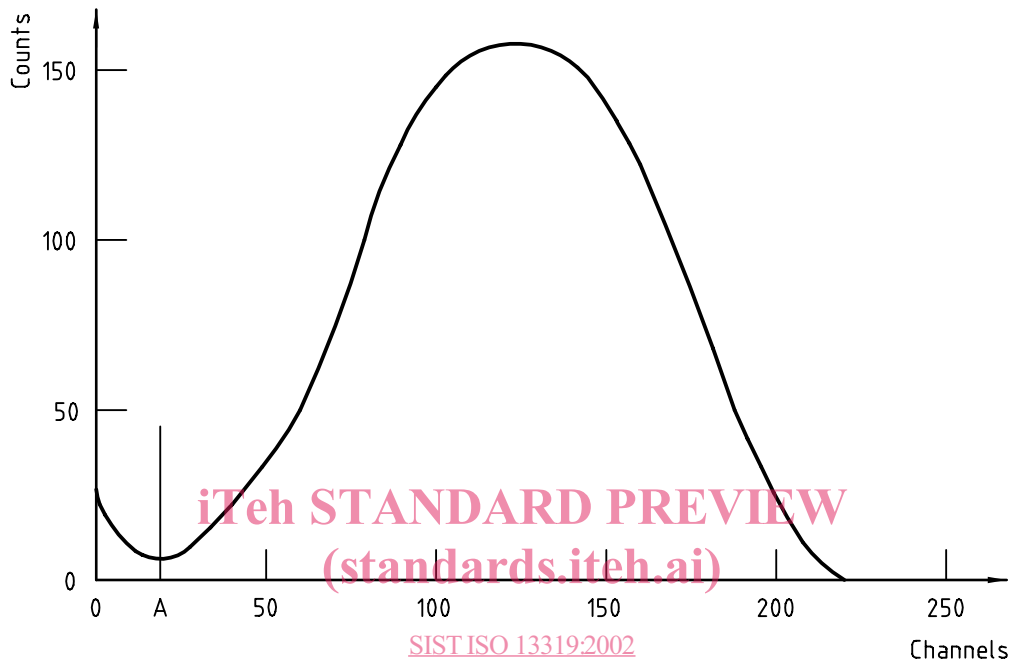
Counts at channels below A are noise counts. True particle counts are at the higher channels

Figure 2 — Typical results

5.5   Repeatability of counts

It has been shown that, in a correctly performed analysis, the number of counts in each channel is a random variable which follows Poisson's law. This means that the standard deviation of a number of counts N approximates to $\sqrt{N}$. Thus, in a series of replicate runs the number of counts in a channel, $N_{i,1}$, $N_{i,2}$, $N_{i,3}$, etc., which yield a mean count N_i with 95 % confidence, the replicate counts $N_{i,n}$ should be in the range $N_i \pm 1{,}96\sqrt{N_i}$; i.e. if the count N_i is 100 000, the uncertainty is $\pm$ 619. If 20 replicate analyses yield more than one outside this range, the sample preparation procedure should be re-examined (see 6.7). This statistical test can be performed on single channels, groups of channels, or on the total particle count.

6   Operational procedures

6.1   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.

6.2   Linearity of the sensor/amplifier system

The linearity of the sensor/amplifier system can be checked using three monodisperse particles of certified diameter. In a suitable electrolyte solution, the instrument is calibrated with particles at about $0,3D$ (see 6.11.2). Two further sizes of microspheres are then added to the suspension (one of size $\approx 0,2D$ and one $\approx 0,5D$). The suspension is re-analysed and the size corresponding to these extra peaks has to correspond to the quoted size of the particles to within 5 %.

6.3   Linearity of 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. 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; (coincidence may prevent the true count being obtained if the concentration is too high). Subsequent analyses should be carried out at the dilution giving the best results.

6.4   Volume V_{m} of analysed suspension

If particle concentrations are to be determined or the mass integration method of calibration (see 6.11.3) is to be used, it is necessary to check the volume V_{m} of the analysed suspension, which is usually only guaranteed at one metered value by the manufacturer. This value of the analysed volume should be known. Using a suspension of particles, at a statistically valid count level (see 6.3), measure the total particle count with that volume three times. Switch to another analysis volume and obtain the total particle counts at least three 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.

6.5   Choice of electrolyte solution

An electrolyte solution should be selected in which the sample is stable. The electrolyte solution should not dissolve, flocculate, react or otherwise interfere with the state of dispersion of the sample in the measurement time, typically up to 5 min.

Particles insoluble in water can be analysed in aqueous electrolytes, such as 50 g/l hydrated trisodium orthophosphate solution, or 10 g/l sodium chloride solution. Particles soluble in water can often be analysed in 50 g/l lithium chloride solution in methanol, or 50 g/l ammonium thiocyanate solution in iso-propanol. See annex A for other recommended electrolyte solutions for many common materials.

6.6   Preparation of electrolyte solution

The 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 is practicable. All glassware and other apparatus used should be pre-rinsed with filtered electrolyte solution. Background counts should not exceed the values given in Table 1 or yield a total equivalent volume in excess of 0,1 % of the total volume of particles subsequently measured in the same sampling volume.

Table 1 — Counts for background and 5 % coincidence for typical orifice diameters

Orifice diameter D μm	Analysis volume [a] V_m ml	Background counts [b]	Count for 5 % coincidence [c] N
1 000	2	2	80
560	2	10	455
400	2	25	1 250
280	2	75	3 645
200	2	200	10 000
140	2	600	29 150
100	0,5	400	20 000
70	0,5	1 200	58 500
50	0,05	300	16 000
30	0,05	1 500	74 000
20	0,05	5 000	250 000

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

[b] Suggested maximum counts.

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

6.7 Recommended sample preparation and dispersion

6.7.1 General

A dispersant should be selected from the recommendations in ISO 14887 or annex A.

6.7.2 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. A mass of about 20 mg to 50 mg of the paste is transferred into a round-bottomed beaker and thinned with dispersant, followed by a few drops of electrolyte solution. The beaker is nearly filled with electrolyte solution and placed in a low-power ultrasonic bath for 1 min, stirring occasionally. A suitable design of beaker of 400 ml capacity with a baffle is shown in Figure 3. The ultrasonic bath should be in the range 50 W to 100 W, 60 kHz to 80 kHz, and a stop watch is recommended for a reproducible dispersion technique.

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

If the sample is not required to be fully dispersed, it may be added to the electrolyte solution and dispersant while stirring.