

Second edition
2007-07-01

Corrected version
2007-09-01

**Determination of particle size
distributions — Electrical sensing zone
method**

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

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 13319:2007

<https://standards.iteh.ai/catalog/standards/sist/28d4055f-42d0-473c-8d25-8712ccaaa6b9/iso-13319-2007>



Reference number
ISO 13319:2007(E)

© ISO 2007

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 13319:2007

<https://standards.iteh.ai/catalog/standards/sist/28d4055f-42d0-473c-8d25-8712ccaaa6b9/iso-13319-2007>



COPYRIGHT PROTECTED DOCUMENT

© ISO 2007

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword.....	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Symbols	2
5 Principle.....	3
6 General operation	4
6.1 Response.....	4
6.2 Size limits	4
6.3 Effect of coincident particle passage	4
6.4 Dead time.....	5
7 Repeatability of counts	6
8 Operational procedures	6
8.1 Instrument location.....	6
8.2 Linearity of the aperture/amplifier system	6
8.3 Linearity of the counting system	7
8.4 Choice of electrolyte solution	7
8.5 Preparation of electrolyte solution	7
8.6 Recommended sampling, sample splitting, sample preparation and dispersion	8
8.7 Choice of aperture(s) and analysis volume(s).....	9
8.8 Clearing an aperture blockage	10
8.9 Stability of dispersion	10
8.10 Calibration	11
9 Analysis	11
10 Calculation of results	12
11 Instrument qualification	12
11.1 General.....	12
11.2 Report	12
Annex A (informative) Calibration for the measurement of porous and conductive particles	13
Annex B (informative) Technique using two (or more) apertures	16
Annex C (informative) Chi-squared test of the correctness of instrument operation or sample preparation	18
Annex D (informative) Table of materials and electrolyte solutions.....	20
Annex E (informative) Mass integration method.....	30
Annex F (informative) Calibration and control of frequently used apertures	36
Bibliography	37

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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

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.

This corrected version of ISO 13319:2007 incorporates the following corrections:

- in 8.10.1, paragraph 2, line 2, “[13]” has been deleted, and “[11]. [12]” inserted;
- in E.2.4, the factor “ K_{da} ” has been added to the right hand side of Equation (E.1).

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 13319:2007

<https://standards.iteh.ai/catalog/standards/sis/38d4055f-42d0-473c-8d25-8712ccaaa6b9/iso-13319-2007>

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 — Pycnometer method*

ISO 13319:2007

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

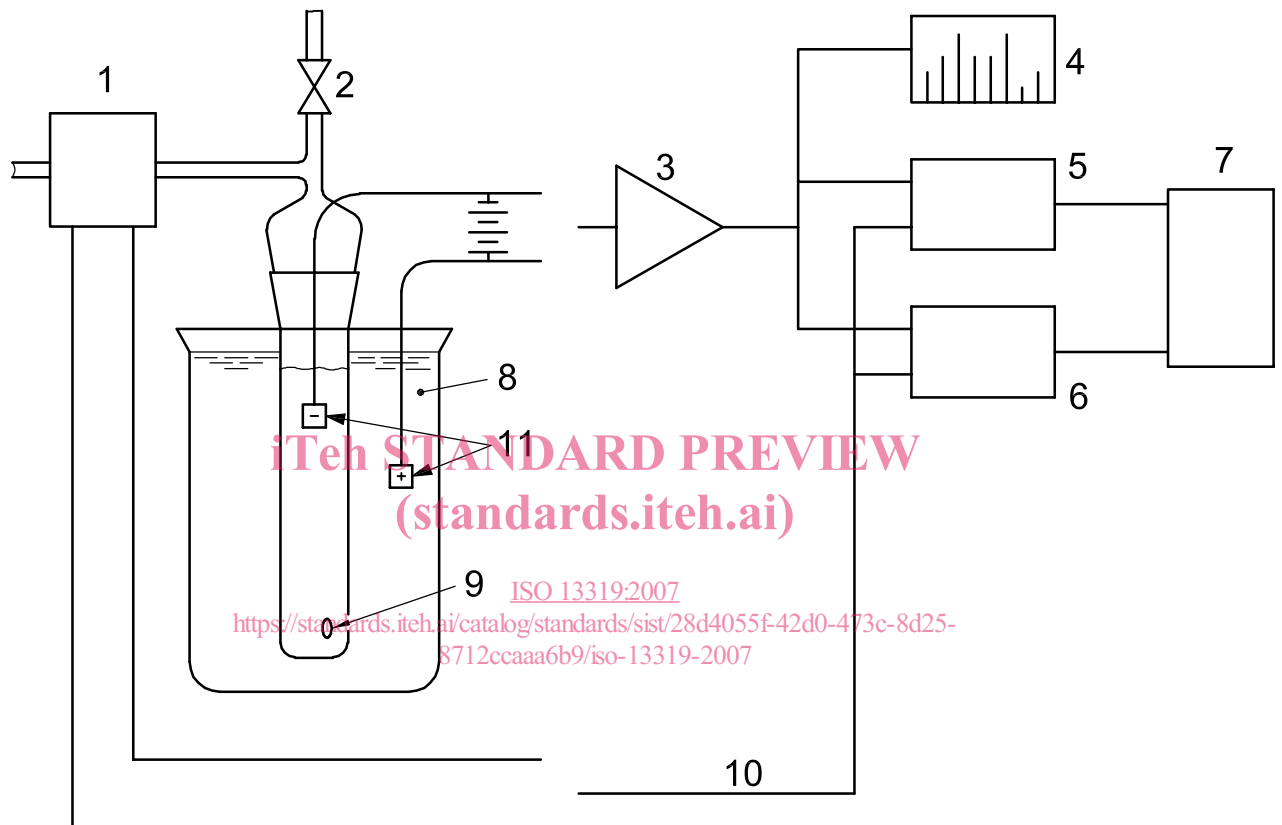
4 Symbols

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
\bar{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
\bar{K}_d	mean calibration constant of diameter
K_{da}	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
\bar{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
$\sigma_{\bar{K}_d}$	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 | 7 | output |
| 2 | valve | 8 | stirred suspension of particles in electrolyte solution |
| 3 | pulse amplifier | 9 | aperture |
| 4 | oscilloscope pulse display | 10 | counter start/stop triggered by the volumetric device |
| 5 | counting circuit | 11 | electrodes |
| 6 | pulse-height analyser | | |

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):

$$K_d = \frac{d_p}{\sqrt[3]{A_p}} \quad (1)$$

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

$$x = K_d \cdot \sqrt[3]{A_x} \quad (2)$$

iTeh STANDARD PREVIEW
(standards.iteh.ai)

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 $N = \frac{4 \times 10^{10} V_m}{D^3}$

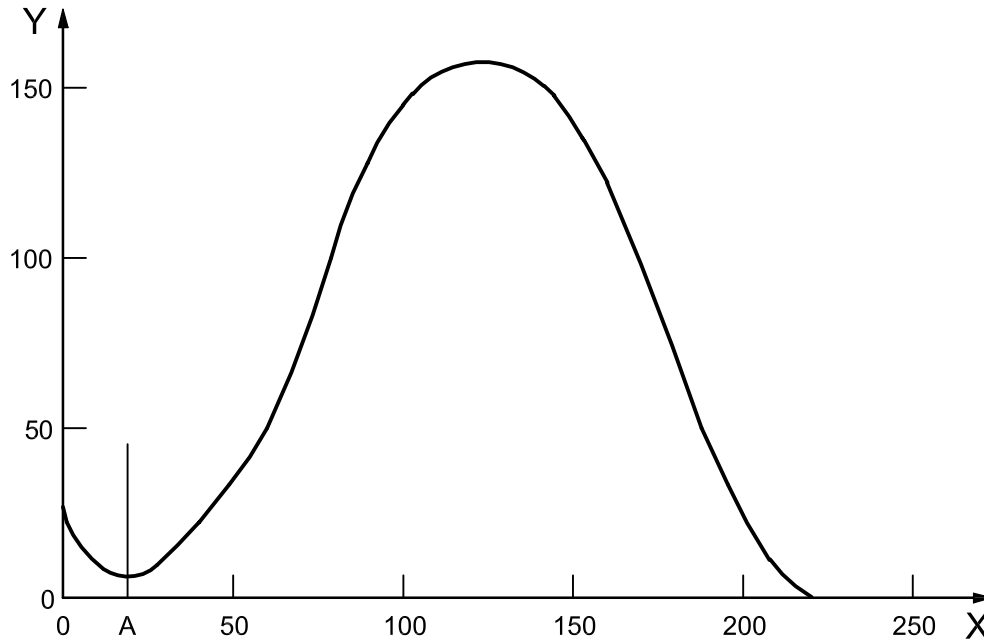
Counts per millilitre should always be less than these quoted values. Since particle size distributions should not be a function of concentration, the effect of coincidence can be tested by obtaining a distribution at one concentration and comparing it with that obtained when the concentration is halved. 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.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

Figure 2 — Typical results

[ISO 13319:2007](https://standards.iteh.ai/catalog/standards/sist/28d4055f-42d0-473c-8d25-8712ccaaa6b9/iso-13319-2007)

7 Repeatability of counts

<https://standards.iteh.ai/catalog/standards/sist/28d4055f-42d0-473c-8d25-8712ccaaa6b9/iso-13319-2007>

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 ($D \leq 50 \mu\text{m}$)

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 ($D \geq 400 \mu\text{m}$)

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 <i>D</i> μm	Analysis volume ^a <i>V_m</i> 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 <i>pro rata</i> values. ^b Suggested maximum counts.		


 (standards.iteh.ai)

8.6 Recommended sampling, sample splitting, sample preparation and dispersion

[ISO 13319:2007](https://standards.iteh.ai/catalog/standards/sist/28d4055f-42d0-473c-8d25-8712ccaaa6b9/iso-13319-2007)

8.6.1 General

<https://standards.iteh.ai/catalog/standards/sist/28d4055f-42d0-473c-8d25-8712ccaaa6b9/iso-13319-2007>

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.

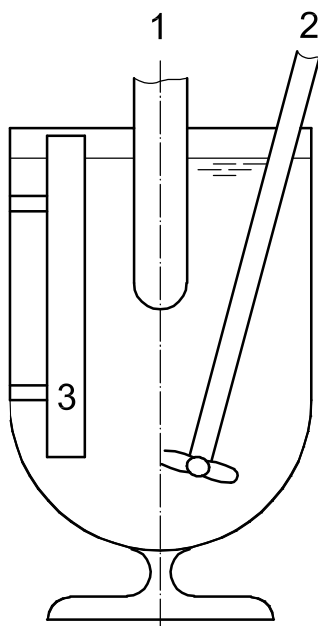
8.6.2 Method 1: Using a paste

The sample should be subdivided to about 0,2 cm³. 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

iTeh STANDARD PREVIEW

Figure 3 — Example of a beaker with baffle and stirrer

8.6.4 Suspensions and emulsions

ISO 13319:2007

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).