BRITISH STANDARD

BS ISO 13317-1:2001

Determination of particle size distribution by gravitational liquid sedimentation methods —

Part 1: General principles and guidelines

ICS 19.120

National foreword

This British Standard reproduces verbatim ISO 13317-1:2001 and implements it as the UK national standard. Together with BS ISO 13317-2:2001 and BS ISO 13317-3:2001 it supersedes BS 3406-2:1984, which is withdrawn.

The UK participation in its preparation was entrusted by Technical Committee LBI/37, Sieves, screens and particle sizing, to Subcommittee LBI/37/4, Particle sizing by methods other than sieving, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this subcommittee can be obtained on request to its secretary.

Cross-references

The British Standards which implement international publications referred to in this document may be found in the BSI Standards Catalogue under the section entitled “International Standards Correspondence Index”, or by using the “Find” facility of the BSI Standards Electronic Catalogue.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

This British Standard, having been prepared under the direction of the Sector Committee for Materials and Chemicals, was published under the authority of the Standards Committee and comes into effect on 15 July 2001

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INTERNATIONAL STANDARD

ISO 13317-1

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Determination of particle size distribution by gravitational liquid sedimentation methods —

Part 1:
General principles and guidelines

Détermination de la distribution granulométrique par les méthodes de sédimentation par gravité dans un liquide —

Partie 1: Principes généraux et lignes directrices

Reference number
ISO 13317-1:2001(E)

Contents

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 part of ISO 13317 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 13317-1 was prepared by Technical Committee ISO/TC 24, *Sieves, sieving and other sizing methods*, Subcommittee SC 4, *Sizing by methods other than sieving*.

ISO 13317 consists of the following parts, under the general title *Determination of particle size distribution by gravitational liquid sedimentation methods*:

- *Part 1: General principles and guidelines*
- *Part 2: Fixed pipette method*
- *Part 3: X-ray gravitational technique*

Annexes A to D of this part of ISO 13317 are for information only.

Introduction

Gravitational sedimentation particle size analysis methods are among those in current use for determining size distribution of many powders. Typically, the gravitational methods apply to samples in the 0,5 µm to 100 µm size range and where the sedimentation condition for a Reynolds number $< 0{,}25$ is satisfied.

No single method of size analysis can be specified to cover the many different types of material encountered, but it is possible to recommend procedures that may be applied in the majority of cases. The purpose of this part of ISO 13317 is to obtain uniformity in procedure for any gravitational method selected to facilitate comparisons of size analysis made in different laboratories.

Gravitational sedimentation methods may be undertaken:

— as part of a research project involving an investigation of the particle size distribution of a material;

— as part of a control procedure for the production of a material where the particle size distribution is important;

— as the basis of a contract for the supply of material specified to be within stated specification limits.

Determination of particle size distribution by gravitational liquid sedimentation methods —

Part 1:
General principles and guidelines

1 Scope

This part of ISO 13317 covers methods for determining the particle size distributions of particulate materials, typically in the size range 0,5 µm to 100 µm, by gravitational sedimentation in a liquid.

NOTE This part of ISO 13317 may involve hazardous materials, operations and equipment. This part of ISO 13317 does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this part of ISO 13317 to establish appropriate safety and health practices and to determine the applicability of the regulatory limitations prior to its use.

The methods of determining the particle size distribution described in this part of ISO 13317 are applicable to slurries or to particulate materials which can be dispersed in liquids. A positive density difference between the discrete and continuous phases is necessary, although gravitational photosedimentation can be used for emulsions where the droplets are less dense than the liquid in which they are dispersed. Particles should not undergo any physical or chemical change in the suspending liquid. The usual precautions need to be taken with hazardous material, and explosion proof analysers are required when examining volatile liquids with a low flash point.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 13317. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 13317 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 758, *Liquid chemical products for industrial use — Determination of density at 20 °C.*

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

ISO 2591-1, *Test sieving — Part 1: Methods using test sieves of woven wire cloth and perforated metal plate.*

ISO 8213, *Chemical products for industrial use — Sampling techniques — Solid chemical products in the form of particles varying from powders to coarse lumps.*

ISO 9276-1, *Representation of results of particle size analysis — Part 1: Graphical representation.*

ISO 13317-2, *Determination of particle size distribution by gravitational liquid sedimentation methods — Part 2: Fixed pipette method.*

ISO 13317-3, *Determination of particle size distribution by gravitational liquid sedimentation methods — Part 3: X-ray gravitational technique.*

ISO 14887, *Sample preparation — Dispersing procedures for powders in liquids.*

3 Terms, definitions and symbols

3.1 Terms and definitions

For the purposes of this part of ISO 13317, the following terms and definitions apply.

3.1.1
terminal settling velocity
velocity of a particle through a still liquid at which the force due to gravity on the particle is balanced by the drag exerted by the liquid

3.1.2
Stokes diameter
equivalent spherical diameter of the particle that has the same density and terminal settling velocity as the real particle in the same liquid under creeping flow conditions

3.1.3
open pores
cavities that are connected to the external surface of the particle either directly or via one another

3.1.4
closed pores
cavities that are closed off by surrounding solid and are inaccessible to the external surface

3.1.5
oversize
portion of the charge which has not passed through the apertures of a stated sieve

3.1.6
undersize
portion of the charge which has passed through the apertures of a stated sieve

3.1.7
effective particle density
particle mass divided by the volume of liquid it displaces

3.1.8
true particle density
particle mass divided by the volume it would occupy excluding all pores, closed or open, and surface fissures

NOTE True particle density is sometimes referred to as the absolute particle density.

3.2 Symbols

For the purposes of this part of ISO 13317, the following symbols apply.

Quantity	Symbol	Unit	Derivative unit
Effective particle density	ρ_s	kg·m^{-3}	g·cm^{-3}
Liquid density	ρ_l	kg·m^{-3}	g·cm^{-3}
True particle density (no porosity)	ρ_p	kg·m^{-3}	g·cm^{-3}
Liquid viscosity	η	Pa·s	mPa·s
Acceleration due to gravity	g	m·s^{-2}	—
Sedimentation distance	h	m	mm
Sedimentation time	t	s	—
Stokes diameter	x_St	m	µm
Upper Stokes diameter	$x_\mathrm{St,U}$	m	µm
Lower Stokes diameter	$x_\mathrm{St,L}$	m	µm
Particle diameter exiting measurement zone	$x_{\mathrm{St},h}$	m	µm
Particle diameter entering measurement zone	$x_{\mathrm{St},h\Delta h}$	m	µm
Terminal settling velocity	v	m·s^{-1}	µm·s^{-1}
Reynolds number	Re	dimensionless	—
Grouped parameter	K_1	m·s	—
Grouped parameter	K_2	m^3·s^{-1}	—
Hyperbolic scan constant	K_scan	m·s	—
Boltzmann constant	k	J·K^{-1}	—
Absolute temperature (Kelvin)	T	K	—
Particle porosity	ε	dimensionless	—
Fraction of open particle porosity filled with sedimentation liquid	f	dimensionless	—
Fractional uncertainty of particle position due to thermal diffusion	f_diff	dimensionless	—
Statistical average positional change in one direction for large number of particles due to thermal diffusion	Δh_diff	m	µm
Thickness of measurement zone	Δh_zone	m	µm
Resolution ratio	P	dimensionless	—
Minimum acceptable resolution	P_min	dimensionless	—
Zone-height-limited resolution	P_zone	dimensionless	—
Minimum settling distance for acceptable resolution, P_min	$h_{\mathrm{zone},P\mathrm{min}}$	m	µm

4 Principles

4.1 General

Gravitational sedimentation methods are based on the settling velocity, under a gravitational field, of particles in a liquid. The relationship between settling velocity and particle size reduces to the Stokes equation (1) at low Reynolds numbers. The Reynolds number should not exceed 0,25 if the inaccuracy in determining the value of Stokes diameter is not to exceed 3 %.

Stokesian sedimentation analyses depend on the applicability of Stokes law. This law defines the relationship between particle size and the change in height (within the suspending fluid) of the particle as a function of the time that the particle has fallen after reaching its terminal velocity.

$$h_{\mathrm{fall}} = \frac{(\rho_{\mathrm{s}} - \rho_1) g x_{\mathrm{St}}^2 t}{18\eta} \qquad (1)$$

Note that h_{fall} is defined so that it increases as the particle falls to lower positions in the sedimentation vessel. This equation may be expressed such that the Stokesian diameter of the particle may be inferred from the distance it has fallen in a given time, t.

$$x_{\mathrm{St}} = \sqrt{\frac{18\eta\, h_{\mathrm{fall}}}{(\rho_{\mathrm{s}} - \rho_1)\, g\, t}} \qquad (2)$$

Sedimentation techniques may be classified as either incremental or cumulative. Incremental methods are used to determine the solids concentration (or suspension density) of a thin layer at a known height and time. Cumulative methods are used to determine the rate at which solids settle from the suspension. In both methods, the powder may be introduced either as a thin layer on top of a column of liquid (the line-start technique), or uniformly dispersed at the start of the analysis (the homogeneous technique). The cumulative method is not part of this part of ISO 13317. The incremental homogeneous technique is more often used in gravitational sedimentation (Figure 1) and is described in this part of ISO 13317. The line-start technique is more applicable to centrifugal sedimentation and is part of ISO 13318-2.

4.2 Calculation of particle size

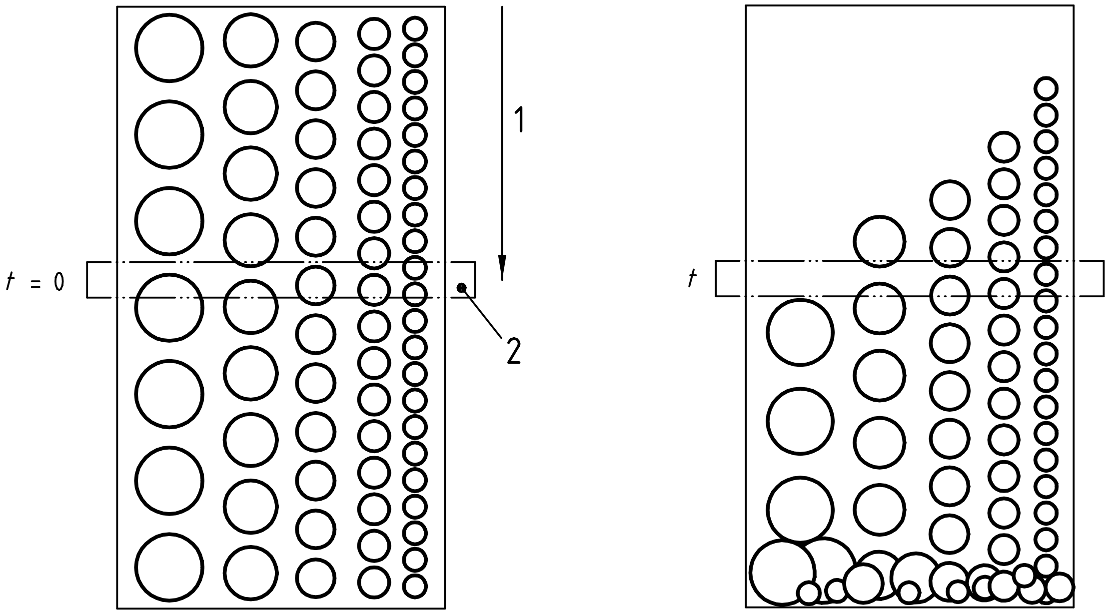
Stokes diameters are calculated according to equation (2).

4.3 Calculation of cumulative mass percentage

The cumulative mass percentage according to the particle concentration gradient in the gravitational pipette method and in the gravitational X-ray method shall be determined according to ISO 13317-2 and ISO 13317-3 respectively.

4.4 Effect of measurement zone height on resolution

Information on the effect of measurement zone height on resolution is given in annex A.



Key

t Time

1 Settling height

2 Measurement zone

Figure 1 — Homogeneous, incremental, gravitational sedimentation

5 Particle size, shape and porosity limitations

5.1 Upper size limit

Stokes equation predicts that the terminal settling velocity that a particle will reach in a gravitational field is

$$v = \frac{x_{\mathrm{St}}^2}{K_1} \qquad (3)$$

where

$$K_1 = \frac{18\eta}{(\rho_{\mathrm{s}} - \rho_1)\, g} \qquad (4)$$

is expressed to solve the Stokesian diameter of the particle

$$x_{\mathrm{St}} = \sqrt{K_1\, v} \qquad (5)$$

Since the terminal settling velocity is constant and attained quickly, $h_{\mathrm{fall}} = v \cdot t_{\mathrm{fall}}$, the particle diameter can be estimated from the distance the particle falls during a given time:

$$x_{\mathrm{St}} = \sqrt{\frac{K_1 h_{\mathrm{fall}}}{t_{\mathrm{fall}}}} \qquad (6)$$

Upper size limit is defined by the largest particle having the terminal settling velocity which satisfies the condition $Re < 0{,}25$. The Reynolds number is the ratio of inertial to viscous forces on the settling particle and is defined by the following equation:

$$Re = \frac{\rho_\mathrm{l}\, v\, x_\mathrm{St}}{\eta} \tag{7}$$

Stokes equation is valid only under conditions of creeping (laminar) flow, for which the Reynolds number is less than 0,1 (see annex B). Its predictions are increasingly inaccurate at higher Reynolds numbers. The inaccuracy in determining x_St from v is 3 % at a Reynolds number of 0,25; beyond this, Stokes law does not provide a good estimate of particle size based on sedimentation velocity. Substituting 0,25 for Re in equation (7), solving for v, and substituting in equation (5) yields the recommended upper size limit of validity for the gravitational sedimentation method as:

$$x_\mathrm{St,U} = \sqrt[3]{\frac{0{,}25\, K_1\, \eta}{\rho_\mathrm{l}}} \tag{8}$$

EXAMPLE A gravitational sedimentation measurement is carried out at 293,15 K using solid quartz spheres ($\rho_\mathrm{s} = 2\,650$ kg·m^{-3}) in 1-propanol ($\rho_\mathrm{l} = 804$ kg·m^{-3} and $\eta = 2{,}256$ mPa·s). From equation (4), $K_1 = 2{,}24 \times 10^{-6}$ m·s, and from equation (8), the maximum particle size for which Stokes law may be used (with an error of less than 3 %) is $x_\mathrm{St,U} = 116$ µm, for which $v = 6{,}03$ mm·s^{-1}.

5.2 Lower size limit

The lower size limit to which gravitational sedimentation methods can be applied is controlled by temperature variation, causing circulatory currents in the suspension, by flocculation of particles during the progress of sedimentation and by diffusion or Brownian motion of the very small particles.

Note that charged particles in weak electrolytes have associated with them an electrical double layer. When these particles settle, the double layer is distorted with the result that an electrical field is set up which opposes motion. These electro-viscous effects can be reduced by the use of non-ionic liquids, where possible.

Information on the accuracy of Stokes law as a function of Reynolds number is given in annex B.

5.2.1 Thermal diffusion (Brownian motion)

The random collisions of the molecules making up the liquid with a particle cause differences in the pressure on the particle from one part of the surface to another such that the particle is displaced (Brownian motion). The equation which represents the statistical average change in position for a particle of diameter x along any one direction of motion in the absence of other forces (such as gravity) is:

$$\Delta h_\mathrm{diff} = \sqrt{\frac{K_2\, t_\mathrm{fall}}{x_\mathrm{St}^{5/2}}} \tag{9}$$

where

$$K_2 = \frac{2\, k\, T}{3\, \pi\, \eta} \tag{10}$$

Note that this is the statistical average of the changes in position in one direction for a large number of particles; some of the particles will travel farther from the starting point than this and some will travel a shorter distance than the average. If both gravity and thermal diffusion are considered, then a spherical particle that travels a distance, h_fall, downward in time, t_fall, could be:

a) a particle whose vertical thermal motion averaged out to zero and whose diameter is correctly determined from equation (6);

b) a particle whose vertical thermal motion increased the vertical distance travelled and whose diameter is smaller than the value computed from equation (6);

c) a particle whose vertical thermal motion decreased the vertical distance travelled and whose diameter is larger than the value computed from equation (6).

The ratio of distances travelled due to thermal motion and sedimentation is, from (6) and (9):

$$f_{\mathrm{diff}} = \frac{\Delta h_{\mathrm{diff}}}{h_{\mathrm{fall}}} = K_1 \left[\frac{K_2}{x_{\mathrm{St}}^5 t_{\mathrm{fall}}} \right]^{1/2} \tag{11}$$

The lower size limit Stokesian sedimentation analysis is generally taken as the size for which $f_{\mathrm{diff}} = 0{,}1$. Using this value and solving equation (11) for x gives

$$x_{\mathrm{St,L}} = \sqrt[5]{\frac{100\, K_1^2 K_2}{t_{\mathrm{fall}}}} \tag{12}$$

EXAMPLE Using the same materials and temperature as the example given in 5.1, gravitational sedimentation occurs over a period of t_{fall} 1800 s (30 min). From equation (10) K2 = $3{,}81 \times 10^{-19}$ $\mathrm{m^3 \cdot s^{-1}}$ and from equation (12), the smallest particle size for which the thermal broadening is less than 10 % of the sedimentation distance is $x_{\mathrm{St,L}} = 0{,}64$ µm.

5.3 Particle shape

At a low Reynolds number, the orientation of non-spherical particles is random, so a single particle will have a low range of settling velocities. As the Reynolds number increases, particles tend to align to give maximum drag and thus will settle at the slowest of the range of velocities possible with random orientation, so a particular particle may have a low or high velocity depending on its orientation.

5.4 Particle porosity

It is recommended that the effective particle density be determined where possible, i.e. the particle density be determined in the suspending liquid, plus dispersant that will be used in the sizing measurement. This will compensate for the presence of any closed porosity and also for open porosity to the extent that the chosen liquid penetrates the open porosity. For particles that are non-porous and of known composition, a density value may be taken from a handbook or determined experimentally. Information on the effect of open pores on the terminal velocity of spherical particles is given in annex D.

6 Test conditions

6.1 Temperature

The analysis temperature affects the liquid density and viscosity and, less significantly, the solid density values in the Stokes equation. Consequently, it is also important that the sample temperature be maintained within narrow limits for the duration of the analysis. It is recommended that the temperature of the sedimentation vessel be kept constant to within ± 1 K, since the viscosity of some liquids can change significantly with temperature. If the temperature varies more than ± 1 K, then it is recommended that the temperature be noted at the beginning and at the end of the analysis and that the average value be used for viscosity calculation. To minimize convection currents, it is recommended that the rate of change of temperature be maintained at less than $\pm 0{,}05\ \mathrm{K \cdot min^{-1}}$.

The temperature of the suspension may be controlled or the agitated suspension may be left standing until temperature equilibrium is attained. Temperature control requirements increase with increasing fineness of the powder. The lower size limit is due, in part, to the longer sedimentation times required for fine particles and the requirement to maintain stable conditions in the sedimenting suspension.

The sedimentation vessel should provide for a covered system to minimize evaporation of the upper layer of the sedimentation liquid which would give rise to convection currents.

6.2 Concentration of suspension

Stokes equation applies to the sedimentation of a single spherical particle settling relatively slowly in a liquid of infinite extent. These requirements are never fulfilled in a sedimentation analysis where particles are separated from each other and the vessel walls by finite distances. This provides for a mutual affect of the particles on each other and also by neighbouring surfaces. In order to minimize these effects, low concentrations are recommended, for example 0,2 % volume concentration. If the recommended maximum concentration has to be exceeded, then analyses should be carried out at two or more concentrations in order to determine if the concentration effects are negligible. Wall-to-wall distances should be at least 5 mm to reduce wall effects to an acceptable level.

6.3 Sedimentation vessel

The vessel shall be vertical to avoid convection and be free from vibration to avoid disturbances on particle settling. Unstable conditions have been attributed to the walls of the sedimentation vessel, or elements (e.g. stirrer) within the walls, being slightly out of vertical. Even for vertical elements there is an inherent instability because the return flow of liquid displaced by the sedimenting particles tends to be along vertically inclined surfaces. The vessel shall be vertical, otherwise convection currents are set up, flowing up the walls that are sheltered from sedimenting particles. These currents produce errors in the estimated size distribution.

6.4 Transient flow

The time required for a particle to reach its steady (terminal) velocity is negligible, but excessively short sedimentation times should be avoided. Prior to particles attaining their terminal velocity, there are other local velocity fluctuations due to the sudden cessation of the initial agitation. These transient eddies and flows shall be allowed to subside.

7 Sampling

Controlled sampling is a necessary condition for obtaining representative sample results for sedimentation tests. The sample shall be taken according to ISO 8213. The sample division shall be according to the future ISO 14488, *Sample preparation — Sample splitting*.

8 Preparation for a sedimentation analysis

8.1 Density of liquid and particles

The density of the liquid at the measuring temperature shall be determined in accordance with ISO 7580 and the density of the particles in accordance with ISO 787-10.

8.2 Removal of oversize particles

As indicated in 5.1, for a given liquid the largest particle of the analysis sample should not exceed a certain value. The size distribution of the oversize fraction can be found by repeating the analysis using a more viscous liquid such that the coarsest particle present in the sample is less than that given by the limiting Reynolds number.

Oversize particles can be removed, by wet or dry sieving (see ISO 2591-1). Wet sieving should be carried out with the sedimentation liquid. Record the percentage residue on the sieve or sieves. The data from the sieve analysis, carried out on a separate portion of the laboratory sample, should be combined with the sedimentation data.

8.3 Selection of suspending liquid

Many powders may not always form a good dispersion in the suspending liquid alone. A suitable dispersing agent may be necessary to inhibit the formation of flocs or agglomerates during sedimentation. This agent may be incorporated in the suspending liquid or added directly to the powder. The suspending liquid shall have negligible reactivity with the sample and shall satisfy the following criteria:

a) the liquid shall have a sufficiently different density from that of the test powder to permit use of the method;

b) the liquid shall have a viscosity to accomplish the analysis within an acceptable analysis time. It should not be too long for the finer particles and not too short for the coarsest particles;

c) the liquid shall not swell or shrink the particles. If such an effect occurs it should be determined that the effect does not exceed 5 % diameter;

d) the liquid shall not cause the sample to go into solution. If such an effect occurs it shall not exceed 5 g powder per 1 kg of liquid.

8.4 Dispersion of sample

If the particles do not readily wet in the liquid or if they form flocs under quiescent conditions, then a dispersing agent should be added to the system. See ISO 14887 for assistance in identifying and applying a suitable dispersing agent.

9 Tests in duplicate and validation

9.1 Tests in duplicate

Perform tests in duplicate on representative analysis samples taken from the same laboratory sample. The results for duplicate analyses should normally differ less than 2 % for the proportions of mass at the same Stokes diameter. This figure would be expected to hold for the Certified Reference Materials proposed in 9.2. A wider tolerance may be required for other sample types with narrower distributions.

9.2 Validation

The checking at regular intervals of both operator procedure and instrument performance is essential to validate the test results. The frequency of checking is a matter for each laboratory to determine. Primary validation can be made with any suitable certified reference material. The total measurement procedure is examined when the reference material is analysed, including the sampling, the sample dispersion, the measurement and the subsequent calculation. The validation procedure will meet the standard if the mean value of the x_{10}, the x_{50} and the x_{90} obtained from three independent measurements lies within the certified range of values of the reference material.

It is recommended that validation be carried out where possible using Certified Reference Materials from the Bureau communautaire de référence, Brussels, or the US National Institute of Science and Technology (NIST), Gaithersburg MD. A record of all validation activities shall be maintained1).

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1) Information on the types and sources of certified material standards available for calibrating or checking the operation of particle sizing instruments is available on the website of the Particle Technology Forum – a division of the American Institute of Chemical Engineers – at http://www.erc.ufl.edu/ptf/partstds.htm

10 Reporting of results

The data shall be presented in graphical, or graphical and tabular form. Results will typically be presented as Stokes diameter versus cumulative distribution by mass reported to the nearest 0,1 %. In the case of a plot, the diameters will be placed on the abscissa and the cumulative mass percentage on the ordinate. The representation of results should conform to ISO 9276-1.

The report should include:

- a reference to this part of ISO 13317;
- the name of the testing establishment;
- the date of the test;
- a unique report identification;
- operator identification;
- instrument type used;
- test sample identification;
- the powder, its density and mass, where applicable;
- the suspending liquid, its temperature, density, viscosity and volume, where applicable;
- the dispersing agent and its concentration;
- the method of dispersion of the suspension, including the time of dispersion;
- the method of treatment of the test sample (drying, de-agglomeration), if any;
- any other operations not specified in this part of ISO 13317 which might have influenced the results.

Annex A
(informative)

The effect of measurement zone height

The detector in a sedimentation analyser measures the particle concentration in a thin horizontal slice of a column of sedimenting particles. This concentration does not distinguish between the large particles and the smaller ones, so the thicker the measurement window the lower the resolution. The zone-height-limited resolution (P_{zone}) can be defined as the ratio of the diameters for particles just exiting the bottom of the measurement zone ($x_{\text{St},h}$) to the difference between that diameter and that for particles just entering the top ($x_{\text{St},h\text{-}\Delta h}$) of the measurement zone.

$$P_{\text{zone}} = \frac{x_{\text{St},h}}{x_{\text{St},h} - x_{\text{St},h-\Delta h}} \tag{A.1}$$

where

h is the height (to the bottom of the measurement zone);

Δh is the thickness of the measurement zone [1, 2].

Substituting for x_{St} from equation (6) and noting that the time at which the concentration measurement is made is the same at the top and bottom of the slice, gives:

$$P_{\text{zone}} = \frac{\sqrt{h}}{\sqrt{h} - \sqrt{h - \Delta h_{\text{zone}}}} \tag{A.2}$$

The resolution ratio acceptable for Stokesian analysis is generally taken as 14 [2]. Equation (A.2) can be rearranged to determine the distance to the bottom of the measurement zone as a function of the measurement slice thickness and the resolution:

$$h_{\text{zone},P} = \frac{\Delta h_{\text{zone}} P^2}{2P - 1} \tag{A.3}$$

The minimum distance for acceptable resolution ($P_{\text{min}} = 14$) in the gravitational sedimentation method is the $h_{\text{zone},P\text{min}} = 7{,}26\ \Delta h_{\text{zone}}$. This equation is valid whether the method uses a fixed position for the measurement zone or scans the zone upward at an ever-decreasing rate (a "hyperbolic scan") so that the position of the detector zone varies with time as:

$$h_{\text{zone}} = \frac{K_{\text{scan}}}{t} \tag{A.4}$$

The time at which the detector passes $h_{\text{zone},P\text{min}}$ is the limit of scan time for which resolution is acceptable, so:

$$t_{\text{limit}} = \frac{K_{\text{scan}}}{K_{\text{zone},P\,\text{min}}} = \frac{K_{\text{scan}}}{7{,}26 \Delta h_{\text{zone}}} \tag{A.5}$$

Substituting this as t_{fall} in equation (12) yields the lower limit of particle size for which gravitational measurement is acceptable for instruments using the hyperbolic scan method:

$$x_{\text{St,L},P\,\text{min}} = \sqrt[5]{\frac{726 \quad K_1^2 K_2 \Delta h_{\text{zone}}}{K_{\text{scan}}}} \tag{A.6}$$

EXAMPLE A gravitational sedimentation measurement is carried out at 293,15 K using solid quartz spheres (ρ_s = 2 650 kg·m^{-3}) in 1-propanol (ρ_l = 804 kg·m^{-3} and η = 2,256 mPa·s). Measurement zone of height Δh_{zone} = 100 µm is scanned upward at an ever-decreasing rate with a hyperbolic scan constant K_{scan} = 2 m·s. From equation (A.3) and (A.5), the height at which the resolution drops below 14 is $h_{\text{zone},P\text{min}}$ = 726 µm and the time the detector reaches this point is t_{limit} = 2755 s (49,3 min). From equation (A.6), the minimum particle size for which resolution and thermal broadening are acceptable is $x_{\text{St,L},P\text{min}}$ = 0,58 µm.

Annex B
(informative)

Accuracy of Stokes law as a function of Reynolds number

In Figure B.1, the calculated particle size, making the assumption that Stokes law applies, is ratioed to the measured particle size for a range of Reynolds numbers. At a low Reynolds number, the particles settle in random orientation. At a higher Reynolds number, there is an increasing tendency for the particles to orientate to give maximum resistance to motion. Stokes law underestimates the drag coefficient as the Reynolds number increases. Thus the measured size will be larger than that estimated by using Stokes law. It would be necessary to apply diameter corrections at a higher Reynolds number, otherwise the determined diameter will vary with fluid density or viscosity [1]. The gravitational methods are recommended to apply to samples where the sedimentation condition for a Reynolds number < 0,25 is satisfied for the largest size of particle in the test portion.

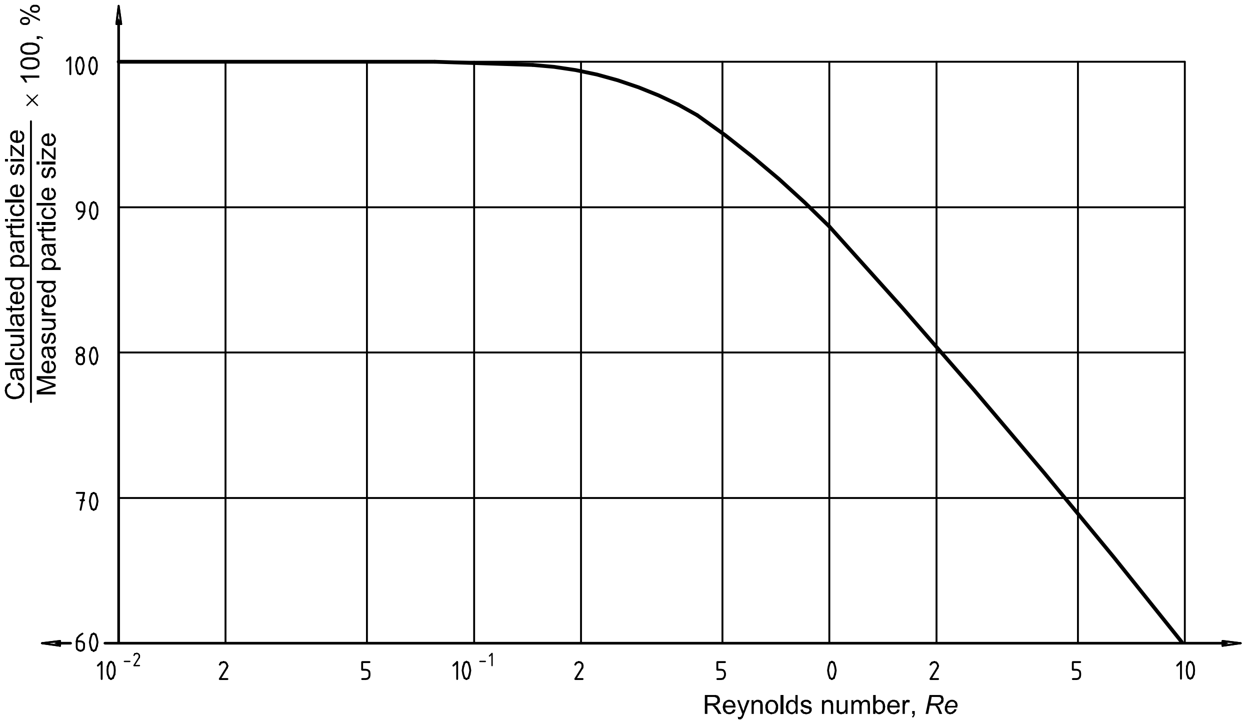


Figure B.1 — Accuracy of Stokes law as a function of Reynolds number

Annex C
(informative)

Particle displacement due to Brownian motion

Figure C.1 shows particle size (on the *x*-axis) versus the ratio of Brownian motion displacement to total particle displacement (on the *y*-axis). This is given for a range of particle-liquid density differences, at one minute after sedimentation has commenced, and enables the potential error due to Brownian motion for a given particle size to be estimated for a given sedimentation time. The particle size of interest is selected and the time axis extended to the desired time of analysis. The vertical intercept from this point to the appropriate density difference curve permits the potential error due to Brownian motion to be determined on the *y*-axis [1].

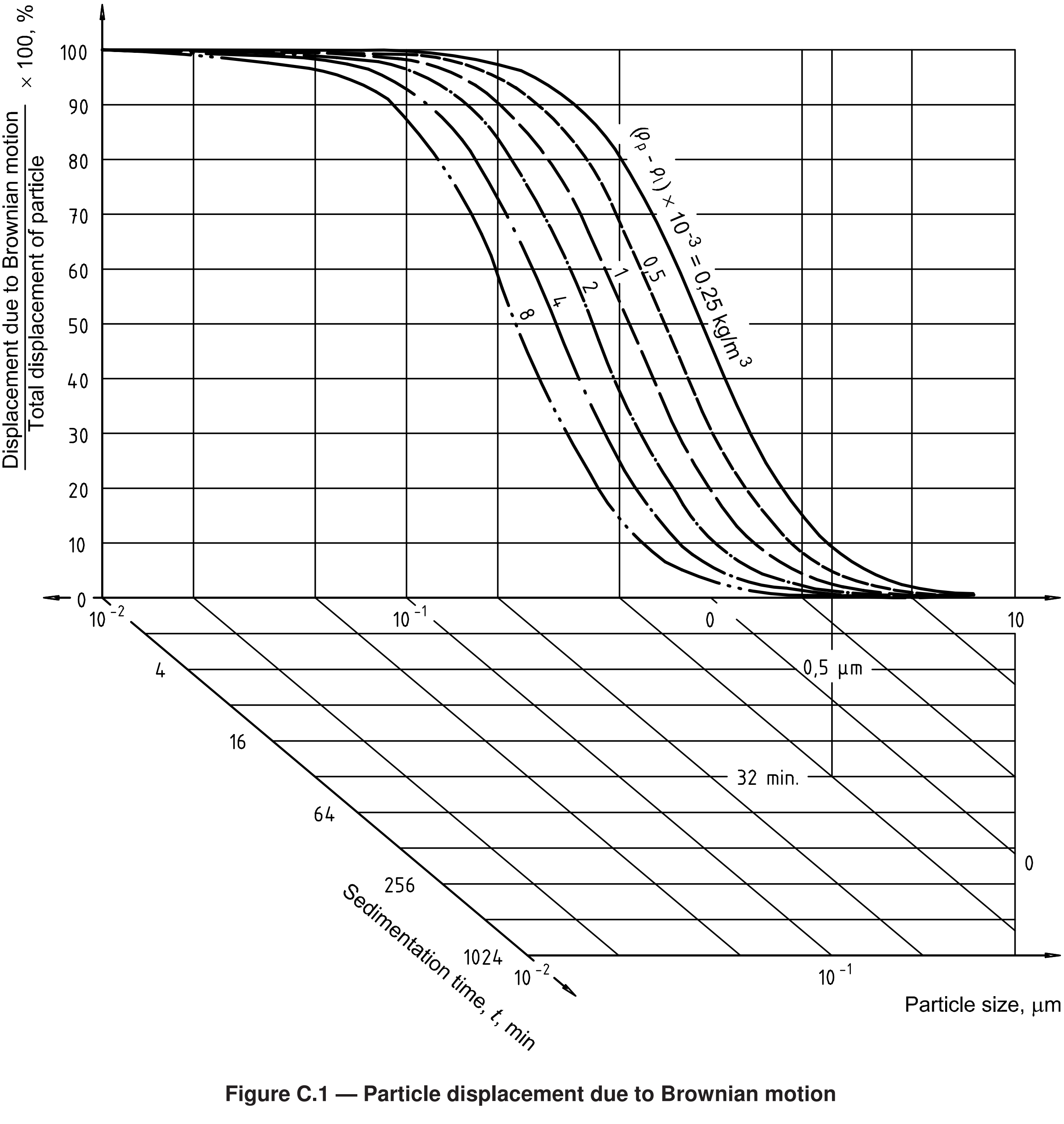


Figure C.1 — Particle displacement due to Brownian motion

Annex D
(informative)

Effect of open pores on the terminal velocity of spherical particles

Particles may include porosity. This porosity can consist of open pores, where cavities are connected to the external surface of the particle, or closed pores, where cavities remain inaccessible to the external surface, or a combination of both porosity types. If all the pores are open, then it is possible to evaluate the particle size measurement error due to the presence of this open porosity.

When a spherical particle of diameter, x, true density, ρ_p, and porosity, ε, is filled with liquid of density, ρ_l, to the fraction of f and sediments in liquid of density, ρ_l, then its terminal velocity, v, can be determined as follows.

Volume of the particle is given by

$$\left(\frac{\pi}{6}\right)x^3 \tag{D.1}$$

the result being that volume of the particle corresponding to the "envelope particle volume" i.e. including the volume contributed by the open porosity.

The mass of particle is given by

$$\left(\frac{\pi}{6}\right)x^3\left[\left(1-\varepsilon\right)\rho_\mathrm{p}+\rho_\mathrm{l}\varepsilon f\right] \tag{D.2}$$

the result being the "effective mass" of the particle, i.e. allowing for that mass of liquid inside the pores of the particle which during sedimentation moves wholly as if part of the particle itself.

Gravitational force acting on particle is given by

$$\left(\frac{\pi}{6}\right)x^3\left\{\left[\left(1-\varepsilon\right)\rho_\mathrm{p}+\rho_\mathrm{l}\varepsilon f\right]-\rho_\mathrm{l}\right\}g \tag{D.3}$$

and the drag force acting on the particle is given by

$$3\,\pi\,\eta\,x\,v \tag{D.4}$$

then terminal velocity is

$$v=\left(\frac{x^2\left\{\left[\left(1-\varepsilon\right)\rho_\mathrm{p}+\rho_\mathrm{l}\varepsilon f\right]-\rho_\mathrm{l}\right\}g}{18\eta}\right) \tag{D.5}$$

In a measurement, however, this velocity is regarded as corresponding to a particle of density, ρ_p, with no porosity. This measured diameter, x', which makes no allowance for the presence of fluid-filled open porosity, can be expressed by

$$v=\frac{x'^2\left\langle\rho_\mathrm{p}-\rho_\mathrm{l}\right\rangle g}{18\eta} \tag{D.6}$$

The ratio of the measured particle diameter, x', to the true particle diameter, x, may be expressed as

$$\frac{x'}{x} = \sqrt{\frac{\left\langle (1-\varepsilon)\rho_\mathrm{p} + \rho_\mathrm{l}\varepsilon f \right\rangle - \rho_\mathrm{l}}{\rho_\mathrm{p} - \rho_\mathrm{l}}} \tag{D.7}$$

When the particle porosity is completely filled by sedimentation liquid (“wet” in Figure D.1) then the following relative particle size relationship holds:

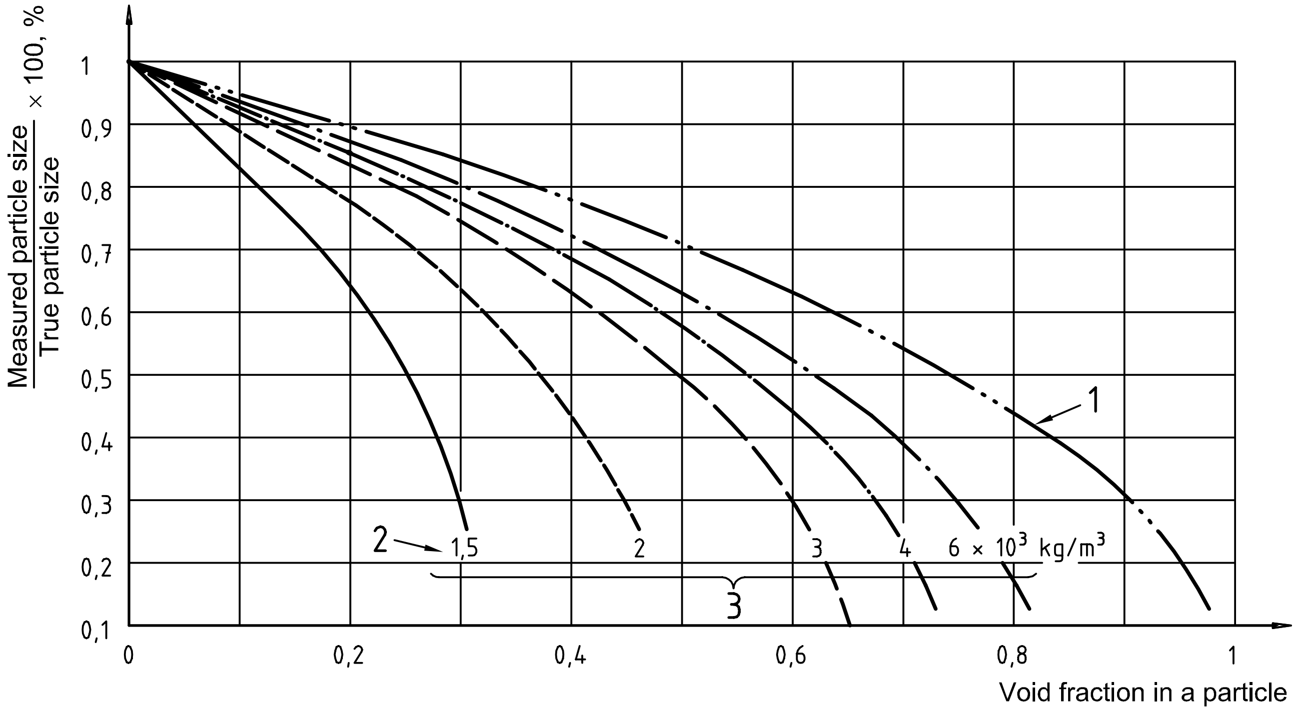
$$\frac{x'}{x} = \sqrt{1-\varepsilon} \tag{D.8}$$

When no liquid enters the open porosity (“repel” in Figure D.1) then the relative particle size relationship is given by

$$\frac{x'}{x} = \sqrt{1-\frac{\rho_\mathrm{p}\varepsilon}{\rho_\mathrm{p}-\rho_\mathrm{l}}} \tag{D.9}$$

The effect on the measured diameter of filling the particle void fraction, with water as the sedimentation liquid ($\rho_\mathrm{l} = 1 \times 10^3$ kg·m^3), is assessed in Figure D.1 for a range of particle densities.

Note that the particle density, ρ_p, in the diagram is the “true” density when there is no particle porosity. The curves in Figure D.1 consider the case only for the complete absence of filling, $f = 0$ (repel), and for the complete filling of open porosity, $f = 1$ (wet). Where f has some value between 0 and 1, then intermediate curves, corresponding to partial pore filling, would obtain between the “wet” and “repel” curves for a given particle density.



Key

1 Wet
2 Particle density
3 Repel water

Figure D.1 — Effect of open pores on the terminal velocity of spherical particles

Bibliography

[1] ALLEN, T. (1997). *Particle Size Measurement*, Fifth edition, Chapman and Hall.

[2] HEYWOOD, H. (1947). *Symposium on Particle Size Analysis*, Trans. Inst. Chem. Engrs. **25**, pp. 14-24.

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