

Determination of particle size distribution by centrifugal liquid sedimentation methods —

Part 1: General principles and guidelines

ICS 19.120

National foreword

This British Standard reproduces verbatim ISO 13318-1:2001 and implements it as the UK national standard. Together with BS ISO 13318-2 and BS ISO 13318-3 (to be published), it supersedes BS 3406-6:1985, which will be withdrawn on publication of BS ISO 13318-3.

The UK participation in its preparation was entrusted by Technical Committee LBI/37, Sieves, screens and particle sizing, to Subcommittee LBI/37/4, 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.

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**Determination of particle size distribution
by centrifugal liquid sedimentation
methods —**

Part 1:
General principles and guidelines

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

Partie 1: Principes généraux et lignes directrices



Reference number
ISO 13318-1:2001(E)

Contents

Page

Foreword.....	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms definitions and symbols	2
4 Principles	4
5 Particle size, shape and porosity limitations	9
6 Test conditions	10
7 Sampling	11
8 Preparation for a sedimentation analysis	11
9 Tests in duplicate and validation	12
10 Reporting of results	12
Annex A (informative) The effect of measurement zone depth	14
Annex B (informative) Accuracy of Stokes law as a function of Reynolds number	15
Bibliography	16

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 13318 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 13318-1 was prepared by Technical Committee ISO/TC 24, *Sieves, sieving and other sizing methods*, Subcommittee SC 4, *Sizing by methods other than sieving*.

ISO 13318 consists of the following parts, under the general title *Determination of particle size distribution by centrifugal liquid sedimentation methods*:

- *Part 1: General principles and guidelines*
- *Part 2: Centrifugal photosedimentation method*
- *Part 3: Centrifugal X-ray method*

Annexes A and B of this part of ISO 13318 are for information only.

Introduction

Centrifugal sedimentation particle size analysis methods are among those in current use for determining size distribution of many powders. Typically, centrifugal methods apply to samples in the 0,1 μm to 5 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 to the majority of cases. The purpose of this part of ISO 13318 is to obtain uniformity in procedure of centrifugal methods in order to facilitate comparisons of size analysis made in different laboratories.

Centrifugal 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.

Gravitational sedimentation methods are discussed in ISO 13317-1, ISO 13317-2 and ISO 13317-3.

Determination of particle size distribution by centrifugal liquid sedimentation methods —

Part 1: General principles and guidelines

1 Scope

This part of ISO 13318 covers methods for determining the particle size distributions of particulate materials, typically in the size range 0,1 μm to 5 μm , by centrifugal sedimentation in a liquid.

NOTE This part of ISO 13318 may involve the use of hazardous materials operations and equipment. This part of ISO 13318 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 13318 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 13318 are applicable to slurries, particulate materials which can be dispersed in liquids and some emulsions. A positive density difference between the discrete and continuous phases is necessary, although centrifugal photosedimentation can be used for emulsions where the droplets are less dense than the liquid in which they are dispersed.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 13318. 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 13318 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 — Pycnometer 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 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 13318, 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 centrifugation 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 13318, the following symbols apply.

Quantity	Symbol	Unit	Derivative Unit
Density			
Effective particle density	ρ_s	kg·m ⁻³	g·cm ⁻³
Liquid density	ρ_l	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	—
Time detector scan passes $M_{\min p}$	t_{limit}	s	—
Stokes diameter	x_{St}	m	μm
Stokes diameter of particles, in measurement zone, commencing from position r_i	x_i	m	μm
Upper Stokes diameter	$x_{\text{St,U}}$	m	μm
Lower Stokes diameter	$x_{\text{St,L}}$	m	μm
Particle diameter exiting measurement zone	$x_{\text{St,p}}$	m	μm
Particle diameter entering measurement zone	$x_{\text{St,p-}\Delta p}$	m	μm
Terminal settling velocity	v	m·s ⁻¹	μm·s ⁻¹
Reynolds number	Re	dimensionless	—
Combined parameter	K_1	m ² ·s	—
Combined parameter	K_2	m ³ ·s ⁻¹	—
Hyperbolic scan constant	K_{scan}	m·s	—
Boltzmann constant	k	J·K ⁻¹	—
Absolute temperature	T	K	—
Fractional uncertainty of particle position due to thermal diffusion	f_{diff}	dimensionless	—
Extinction coefficient for particle of size x_i	E_i	dimensionless	—
Resolution ratio	P	dimensionless	—
Zone-height-limited resolution	P_{zone}	dimensionless	—
Distance of particle from axis of rotation	r	m	mm
Distance of particle from axis of rotation at time t	r_t	m	mm
Starting position of particles diameter x_i	r_i	m	μm
Statistical average positional change in one direction for large number of particles: root mean square distance	$\Delta r_{\text{TB},t}$	m	μm
Distance from rotation axis to liquid-air interface of sample (vortex radius)	S	m	mm
Distance from rotation axis to centrifuge wall (inner disc radius)	R	m	mm
Centrifuge speed (rev/min)	N	r·min ⁻¹	—
Distance from rotation axis to measurement zone (measurement radius)	M	m	mm
Thickness of measurement zone	ΔM	m	μm
Distance for minimum acceptable resolution relative to measurement zone (ΔM)	$M_{\min p}$	m	—
Mass fraction of particles less than diameter x_i	F_i	dimensionless	—
Measured concentration (relative)	C	dimensionless	—
Ratio $(M/S)^2$	y	dimensionless	—
$y_i(x_i/x_j)^2$ (Kamack's)	y_{ij}	dimensionless	—
Centrifugal angular velocity ($\frac{2\pi N}{60}$)	ω	rad·s ⁻¹	—

4 Principles

4.1 General

Centrifugal sedimentation methods are based on the rate of settling, under a centrifugal 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 settling distance (within the suspending fluid) of the particle as a function of the time that the particle has fallen after reaching its terminal settling velocity, and in a gravitational field. This relationship may be expressed as:

$$h_{\text{fall}} = \frac{(\rho_s - \rho_l) g x_{\text{St}}^2 t}{18\eta} \quad (1)$$

The Stokesian diameter of the particle may then be inferred from the distance it has fallen in a given time, t .

$$x_{\text{St}} = \sqrt{\frac{18\eta h_{\text{fall}}}{(\rho_s - \rho_l) g t}} \quad (2)$$

The basic principles of gravitational sedimentation are unaffected in a centrifugal analysis, except that the factor g in Stokes law [see equation (2)] now becomes a variable according to the speed and radius of rotation of the particle. In centrifugal sedimentation, the velocity of a given size of particle is not constant but increases as the radius of rotation increases with particle settling distance. The force due to gravitational acceleration, g , is replaced by $\omega^2 r$, where ω is the centrifugal angular velocity. The settling distance, h_{fall} , in time, t , is replaced by $\ln(M/S)$ for time, t .

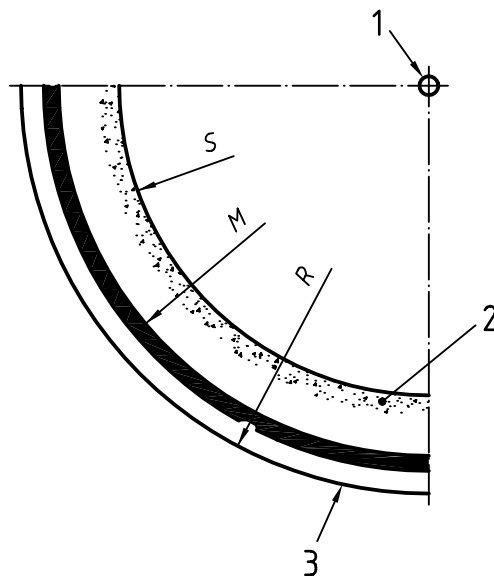
The Stokes diameter, x_{St} , at measurement radius time, t , is given by

$$x_{\text{St}} = \sqrt{\frac{18 \eta \ln(M/S)}{(\rho_s - \rho_l) \omega^2 t}} \quad (3)$$

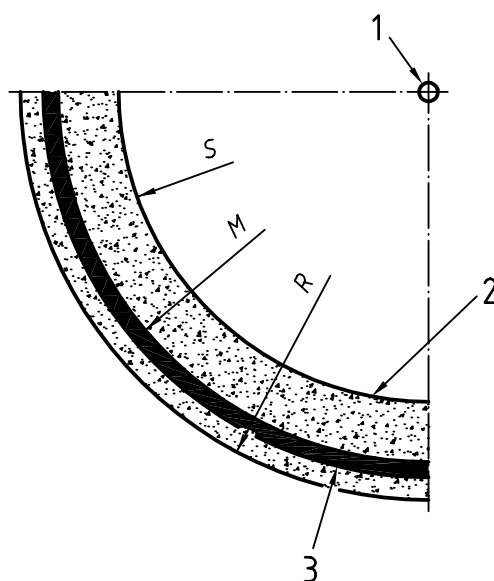
Particle concentrations are determined by the attenuation of a white light or an X-ray beam.

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 settling distance and time. Cumulative methods are used to determine the rate at which solids settle from the suspension. The cumulative method is not included in this part of ISO 13318.

The sample may be introduced either as a thin layer of dispersion on top of the spinning liquid medium, the line-start incremental technique, or uniformly dispersed throughout the sedimentation medium, the homogeneous incremental technique (Figures 1 and 2).

**Key**

- 1 Centre of rotation
- 2 Sample
- 3 Centrifuge disc wall

Figure 1 — Line-start incremental**Key**

- 1 Centre of rotation
- 2 Liquid/air interface
- 3 Measurement zone

Figure 2 — Homogeneous incremental**4.1.1 Line-start incremental method**

All particles are initially in a thin band at the vortex radius, S , of the sedimentation zone. As centrifugation proceeds, larger particles fall outward faster than the small ones of the same particle density. At time, t , the only particles in the detection zone are those with a Stokes diameter close to that given by equation (7). In practice, there will be a range of sizes in the measurement zone due in part to the finite radial width of that zone and in part due to thermal broadening.

4.1.2 Homogeneous suspension incremental method

The particles are initially distributed evenly throughout the fluid. As centrifugation proceeds, larger particles fall outward faster than the smaller ones of the same particle density so that at time, t , no particles larger than x_{St} are present inside of radius M .

4.2 Calculation of particle size

The calculation of particle size is dependent on Stokes law, which may conveniently be stated in the following form for particles sedimenting in a centrifugal field:

$$v = \frac{dr}{dt} = \frac{rx_{St}^2}{K_1} \quad (4)$$

where

r is the distance of the particle from the axis of rotation.

For convenience, some parameters have been grouped so that

$$K_1 = \frac{18\eta}{(\rho_s - \rho_l)\omega^2} \quad (5)$$

4.2.1 Line-start method

The position of the particle as a function of time, t , as it moves from radius S at time 0, may be determined by integrating equation (4) to obtain

$$r_t = S \exp\left(\frac{x_{St}^2 t}{K_1}\right) \quad (6)$$

where

r_t is the distance of the particle from the axis of rotation at the end of time, t .

If all particles start from the same radial distance, S , then only particles for which $r_t = M$ will be at the measurement zone at time, t .

$$x_{St,t} = \sqrt{\frac{K_1 \ln\left(\frac{M}{S}\right)}{t}} \quad (7)$$

4.2.2 Homogeneous suspension method

The relationship is similar for the homogeneous technique, except that particles begin sedimenting from different radii. After time, t , the diameters of particles in the measurement zone (distance M from centre of rotation), are related to their starting positions, r_i , by

$$x_{i,t} = \sqrt{\frac{K_1 \ln\left(\frac{M}{S_i}\right)}{t}} \quad (8)$$

Hence, there will be a range of particle sizes present in the measurement zone. Since the smallest starting radius is S , the coarsest particle will have a diameter x_{St} .

4.2.3 Homogeneous suspension method with scanning

For a scanning system, the measurement radius is at radius M_0 at time $t = 0$, and at time, t , the measurement radius will be at M_t . The largest particle present in the measurement zone at time, t , is determined using equation (8) with M_t in place of M .

4.3 Calculation of cumulative mass percentage

4.3.1 General

The method of calculation depends both on the method and on the type of operation.

4.3.2 Line-start

The line-start method is applicable to photosedimentation. It is not used in the X-ray centrifugal method due to the requirement for higher sample concentrations.

In a line-start, the particles are injected into the centrifuge to form a thin layer so it may be assumed that all particles start sedimenting from the same radius. Therefore, the concentration of particles, $C(M,t)$, in the measurement zone at radius, M , and time, t , is directly proportional to the fractional mass of particles of size x_{St} , i.e. the mean size of particles in the measurement zone at time, t , calculated using equation (7).

4.3.2.1 Photosedimentation method

In the photosedimentation method, the attenuation of light is proportional to the cross-sectional area of the particles in the beam for dilute systems of particles larger than the diffraction limit. A plot of attenuation against x_t represents area against diameter (Figure 3). The integration of this curve gives the cumulative undersize by mass.

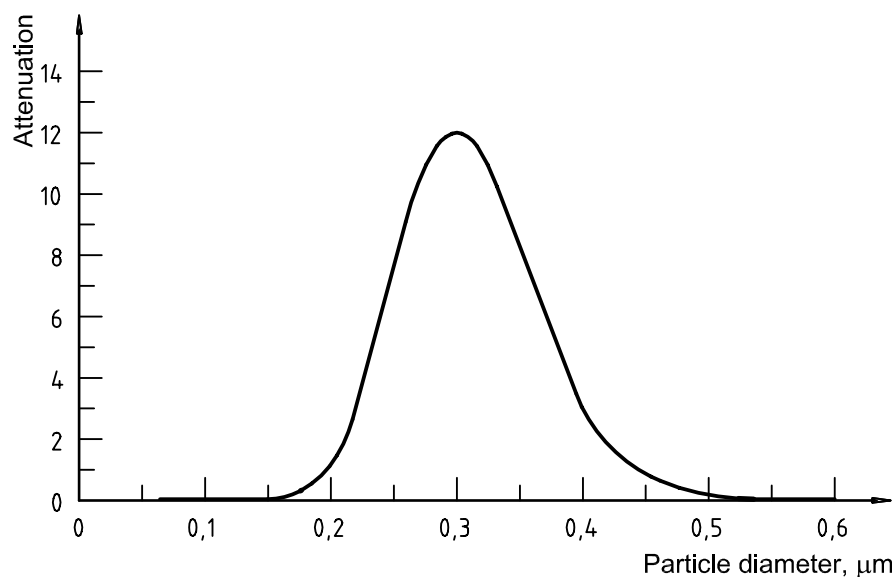


Figure 3 — Attenuation versus Stokes diameter for the line-start photocentrifugal method

A correction is required for the breakdown in the laws of geometric optics which occurs when the size of the particles approaches the wavelength of light. This correction is applied in the form of an extinction coefficient E_i where:

$$E_i = \frac{\text{amount of light cut off by a particle of size } x_i}{\text{amount of light which would be cut off if the laws of geometric optics held}} \quad (9)$$

This correction is usually applied in the equipment software. The value of E is unity for large particles, rising to 2 as the particles pass through the Mie scattering region (about 0,5 μm), and rapidly reducing as the particle size decreases into the Rayleigh scattering region (see reference [1] in the bibliography).

If E is assumed constant, then the method may be used as a comparison method only as there may be substantial errors in the 1 μm size range. The value of E may be evaluated from theory or determined experimentally. Note that the value of the extinction coefficient depends on the wavelength of the light and can also depend on the detector configuration. Consequently, exact determinations of the extinction coefficient are not readily determined. For practical purposes, a value of unity might be assumed and should be stated. If an assumed correction is made by the manufacturer of the apparatus, then this should be stated.

The requirement for extinction coefficient corrections also applies to photosedimentation used in the homogeneous method.

4.3.3 Homogeneous suspension

The homogeneous technique is limited because of approximations which need to be made in the calculation of the results (see reference [3] in the bibliography).

4.3.3.1 Photosedimentation method

In the photosedimentation method, the extinction coefficient corrections detailed in 4.3.2.1 are also required in the homogeneous method.

4.3.3.2 Centrifugal X-ray method

In the centrifugal X-ray method, the attenuation of the X-ray beam is directly proportional to the mass of sample present in the beam and the result is presented automatically. The centrifugal X-ray method is used only in the homogeneous mode due to the requirement for a higher sample concentration than would be feasible using a line-start method.

4.3.3.3 Radial dilution correction

If two monosized spheres are released twenty radii apart at the surface of a gravity sedimentation apparatus, they will remain evenly spaced as they settle. If the same spheres are placed a similar distance apart on the inner surface of a centrifugal sedimentation apparatus, they will become more widely separated as they settle, since they sediment along radial lines. This effect leads to a dilution of the particles as they settle outwards in the centrifuge. The dilution is of concern in the homogeneous mode, since particles of the same diameter commence the analysis at different originating radii; it is of negligible significance in a line-start, since all particle sizes have effectively the same originating radius.

Particles originating at radius S_i in an annular element of thickness ΔS_i will occupy an annulus of thickness ΔM at a fixed measurement radius M . The relationship between F_i (the mass fraction of particles smaller than diameter x_i) and the measured concentration, C , at radius M is:

$$F_i = \int_0^C \left(\frac{M}{S} \right)^2 dC \quad (10)$$

The general solution (Kamack's equation, see reference [3] in the bibliography) is:

$$F_i = \frac{1}{2} (y_i + y_{i-1,i}) C_i + \sum_{j=1}^{i-1} \left(\frac{y_i + y_{i-1,i}}{y_{j+1,i} + y_{ji}} - \frac{y_i + y_{i-1,i}}{y_{ji} + y_{j-1,i}} \right) F_j \quad (11)$$

where

$$y_i = \left(\frac{M}{S} \right)^2 \quad (12)$$

$$y_{ij} = y_i \left(\frac{x_i}{x_j} \right)^2 \quad (13)$$

$$i = 1, 2, 3, 4, \dots, m$$

$$y_{0,i} = 1$$

$$y_{ii} = y_i$$

Equation (11) is a set of linear equations which express the desired values of F_i explicitly in terms of the measured values of C_i . The coefficients in the equation depend on the ratios of the values of x_i corresponding to the measurement time. Consequently, if these are chosen in a geometric sequence, the coefficients are considerably easier to calculate and the equations themselves are simplified. The coefficients depend also on the geometry of the centrifuge bowl and the amount of suspension (see reference [1] in the bibliography).

4.3.4 Homogeneous suspension with scanning

When scanning is employed with a homogeneous suspension, then M changes continuously during the course of the scan (see reference [1] in the bibliography). Radial dilution correction requirements still apply.

5 Particle size, shape and porosity limitations

5.1 Upper size limit

The upper size limit, $x_{St,U}$, by centrifugal sedimentation is largely dependent on the reliability of measurements taken in the first minute of the analysis. As in gravitational settling, the restriction on the Reynolds number limits the upper size. An additional complication is that the particles are no longer settling at a constant terminal velocity but are continually accelerating. For a reliable analysis, the accelerating force must be small in comparison to the centrifugal force, as this restricts the upper particle size which can be measured. Centrifugal sedimentation is typically used for sizing particles up to about 5 μm , but this can be extended by the use of a more viscous liquid and/or slower centrifuge speeds.

5.2 Lower size limit

The lower limit of size to which the centrifugal 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. 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), see 5.2.1. Density currents are much more liable to occur in a centrifugal device than in a gravity system. All these factors render it advisable to shorten the sedimentation process as much as possible.

Note that charged particles in weak electrolytes have an electrical double layer associated with them. 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.

Annex A gives additional information concerning lower size limit.

5.2.1 Effect of thermal diffusion (Brownian motion)

A lower size limit, $x_{St,L}$, under ideal vibration-free constant temperature conditions, can be estimated as follows.

The root mean square distance that a particle of diameter x_{St} will diffuse due to thermal broadening (Brownian motion) during time, t , is:

$$\Delta r_{TB,t} = \sqrt{\frac{K_2 t}{x_{St}}} \quad (14)$$

where parameters have been grouped so that:

$$K_2 = \frac{2kT}{3\pi\eta} \quad (15)$$

For accurate results, $\Delta r_{TB,t}$ should be less than 10 % of the distance through which the particle settles to the measurement zone ($M-S$), so solving for t in equation (14), substituting 0,1 ($M-S$) for $\Delta r_{TB,t}$, and substituting for t in equation (6) gives:

$$x_{St,L,TB} = \sqrt[3]{\frac{100K_1K_2 \ln\left(\frac{M}{S}\right)}{(M-S)^2}} \quad (16)$$

EXAMPLE For a measurement made at 293,15 K using solid quartz spheres ($\rho_s = 2\,650\text{ kg}\cdot\text{m}^{-3}$) settling in 1-propanol ($\rho_l = 804\text{ kg}\cdot\text{m}^{-3}$, $\eta = 2,256\text{ mPa}\cdot\text{s}$) in a centrifuge rotating at 750 r/min ($78,54\text{ rad}\cdot\text{s}^{-1}$), $K_1 = 3,57 \times 10^{-9}\text{ m}^2\cdot\text{s}$ and $K_2 = 3,81 \times 10^{-19}\text{ m}^3\cdot\text{s}^{-1}$. If $S = 40\text{ mm}$ and $M = 50\text{ mm}$, the lower limit for reliable size analysis is $x_{St,L,TB} = 0,067\text{ }\mu\text{m}$. Doubling the rotational speed reduces $x_{St,L,TB}$ to $0,042\text{ }\mu\text{m}$.

5.3 Particle shape

At a low Reynolds number, the orientation of non-spherical particles is random so a single particle will have a range of settling velocities. As the Reynolds number increases, particles tend to orientate themselves to give maximum drag and will settle at the slowest of the range of velocities possible with random orientation (see reference [2] in the bibliography).

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.

6 Test conditions

6.1 Temperature

The analysis temperature determines the liquid density and viscosity values for the Stokes equation. Consequently, it is 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 $\pm 1\text{ K}$, since the viscosity of some liquids can change significantly with temperature. If the temperature varies more than $\pm 1\text{ K}$, then it is recommended that the temperature be noted at the beginning and at the end of the analysis and the average value

used for viscosity calculation. To minimize convection currents, it is recommended that the rate of change of temperature be maintained at less than $\pm 0,05 \text{ K min}^{-1}$.

6.2 Concentration of suspension

Stokes equation applies to the sedimentation of a single spherical particle settling infinitely slowly in a liquid of infinite extent. These requirements are never fulfilled in sedimentation analysis where particles are separated by finite distances and mutually affect each other and are also affected by neighbouring surfaces. In order to minimize these effects as far as possible, low concentrations should be used. A recommended maximum volume concentration is 0,2 %; wall to wall distances for the sedimentation vessel should be of at least 5 mm to reduce wall effects to an acceptable level. Sample volume concentrations for the X-ray centrifuge are expected to exceed 0,2 % in order to achieve adequate signal attenuation. 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.

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 and 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

If not already known, determine the density of the liquid at the measuring temperature in accordance with ISO 7580:1976 and the density of the particles in accordance with ISO 787-10:1981.

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.

8.3 Selection of suspending liquid

The powder may not always form a good dispersion in the suspending liquid alone, in which case a suitable dispersing agent is necessary. This agent may be incorporated in the suspending liquid or added directly to the powder. The liquid suspending medium shall satisfy the following criteria:

- a) the viscosity of the liquid shall be selected such that the analysis can be carried out in an acceptable period of time. It should not be too long for the fine particles or too short for the coarser particles;
- b) the liquid should inhibit the formation of flocs or agglomerates during sedimentation. This may often only be achieved by the addition of wetting or dispersing agents;
- c) the solid should not dissolve or change chemically in the liquid and should also not swell or contract.

8.4 Dispersion of sample

If the particles do not wet into the liquid readily 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.

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 requirements if the mean value of the x_{10} , x_{50} and the x_{90} obtained from three independent measurements lie 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 Centre du Reference, Brussels or U.S. National Institute of Science and Technology (NIST), Gaithersburg MD. A record of all validation activities shall be maintained¹⁾.

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:

- reference to this part of ISO 13318;
- the name of the testing establishment;
- the date of the test;
- a unique report identification;
- operator identification;
- instrument type used;
- mode of operation (e.g. line-start, homogeneous);
- 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;

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>

- the method of dispersion of the suspension, including the dispersion time;
- buffer layer and, where applicable, the type and volume;
- centrifuge speed;
- Mie correction, applied or not applied (photocentrifuge);
- any other operations not specified in this part of ISO 13318 and susceptible to have had an influence on the results;

NOTE The following instrument characteristics, typically determined by the manufacturer and pre-programmed into the equipment software, may be optionally reported if available:

- measurement radius, M , (not fixed in scanning mode);
- inner disc radius, R ;
- inner disc thickness, used to calculate vortex radius, S .

Annex A (informative)

The effect of measurement zone depth

The detector in a centrifugal incremental sedimentation analyser measures the particle concentration in a thin measurement zone 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. Where M is the distance to the outer edge of the measurement zone and ΔM is the thickness of the measurement zone (see reference [1] in the bibliography), the zone-height-limited resolution (P_{zone}) can be defined as the ratio of the diameters for particles just exiting the measurement zone ($x_{\text{St},M}$) to the difference between that diameter and that for particles just entering the measurement zone ($x_{\text{St},M-\Delta M}$). Since the particles that have just settled to the inner limit of the measurement zone $M-\Delta M$ are smaller than those that have just settled to the outer limit M , the resolution of the scan will be affected by how wide the measurement zone is. The zone-height-limited resolution can be defined as:

$$P_{\text{zone}} = \frac{x_{\text{St},M}}{x_{\text{St},M} - x_{\text{St},M-\Delta M}} \quad (17)$$

so at a given time, t , using equation (7) and substituting for x_{St} ,

$$P_{\text{zone}} = \frac{1}{1 - \sqrt{\frac{\ln(M - \Delta M) - \ln S}{\ln M - \ln S}}} \quad (18)$$

Equation (18) can be solved by successive approximation to compute M as a function of S , ΔM , and P_{zone} . The minimum acceptable resolution is 30. For a typical laboratory instrument, this occurs at the distance $M_{\text{minp}} = S + 15 \Delta M$ (see reference [2] in the bibliography). The detector scan function for the particular instrument will determine the time (t_{limit}) at which the detector passes M_{minp} . The size of particle that has just settled from S to M_{minp} at this time is the lower limit of particle size for which centrifugal measurement is acceptable for that S , ΔM and detector scan function.

$$x_{\text{St,L,p}} = \sqrt{\frac{K_1 \ln\left(\frac{M_{\text{minp}}}{S}\right)}{t_{\text{limit}}}} \quad (19)$$

EXAMPLE For a measurement made at 293,15 K using solid quartz spheres ($\rho_s = 2\,650 \text{ kg}\cdot\text{m}^{-3}$) settling in 1-propanol ($\rho_l = 804 \text{ kg}\cdot\text{m}^{-3}$, $\eta = 2,256 \text{ mPa}\cdot\text{s}$) in a centrifuge rotating at 750 r/min ($78,54 \text{ rad}\cdot\text{s}^{-1}$), $K_1 = 3,57 \times 10^{-9} \text{ m}^2\cdot\text{s}$ and $K_2 = 3,81 \times 10^{-19} \text{ m}^3\cdot\text{s}^{-1}$. If $S = 40 \text{ mm}$ and $M = 50 \text{ mm}$ and given $\Delta M = 50 \mu\text{m}$, equation (18) can be solved to give $M_{\text{minp}} = 40,75 \text{ mm}$. For $t_{\text{limit}} = 1\,800 \text{ s}$ (i.e. 30 min), equation (19) gives $x_{\text{St,L,p}} = 0,192 \mu\text{m}$.

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 (see reference [1] in the bibliography). 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.

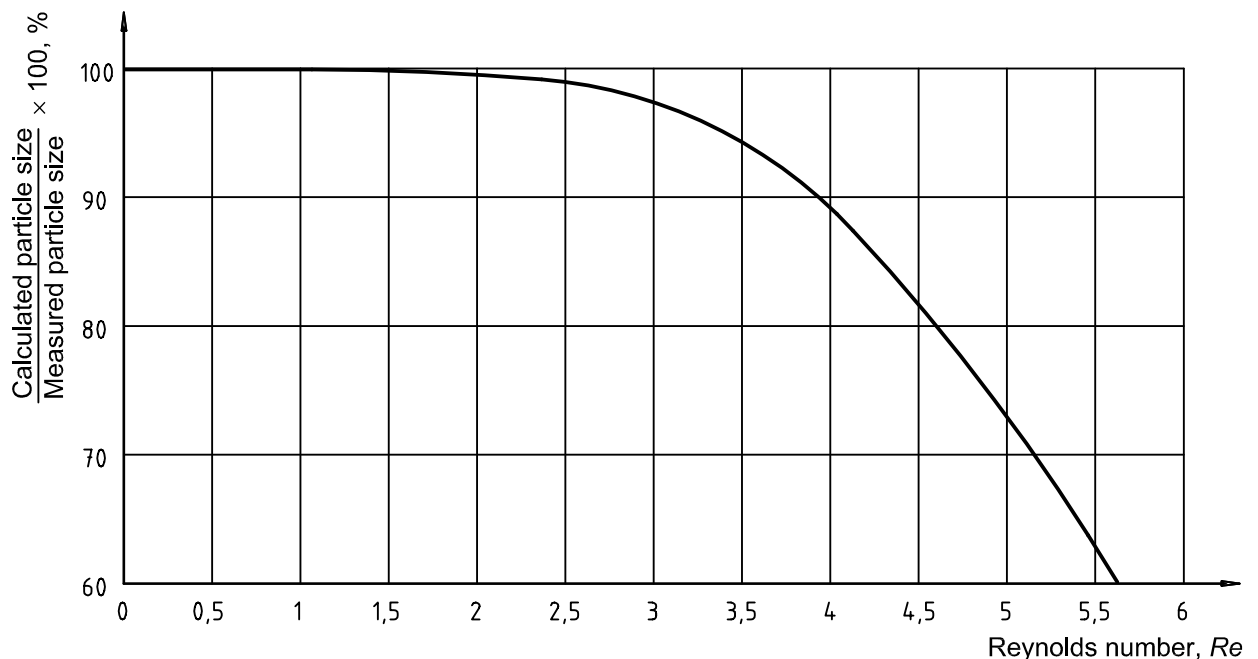


Figure B.1 — Accuracy of Stokes law as a function of Reynolds number

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