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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

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Foreword

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

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

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

Attention is drawn to the possibility that some of the elements of this 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 — 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 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 Liquid density	 ρ_s ρ_l	 kg·m^{-3} kg·m^{-3}	 g·cm^{-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_{minp}	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	$x_{St,U}$	m	µm
Lower Stokes diameter	$x_{St,L}$	m	µm
Particle diameter exiting measurement zone	$x_{St,p}$	m	µm
Particle diameter entering measurement zone	$x_{St,p-\Delta p}$	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	$\Delta r_{TB,t}$	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_{minp}	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 ($\frac{2\pi N}{60}$)	ϖ	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:

$$h_{\text{fall}} = \frac{(\rho_{\text{s}} - \rho_{\text{l}})gx_{\text{St}}^2 t}{18\eta} \tag{1}$$

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

$$x_{\text{St}} = \sqrt{\frac{18\eta h_{\text{fall}}}{(\rho_{\text{s}} - \rho_{\text{l}})\, gt}} \tag{2}$$

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 ϖ^2, where ϖ is the centrifugal angular velocity. The settling distance, h_{fall}, in time, t, is replaced by $\ln(M/S)$ for time, t.

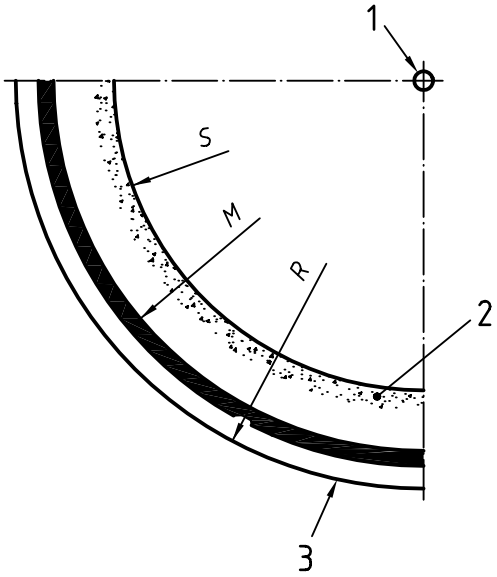
The Stokes diameter, x_{St}, at measurement radius time, t, is given by

$$x_{\text{St}} = \sqrt{\frac{18\ \eta \ln(M/S)}{(\rho_{\text{s}} - \rho_{\text{l}})\varpi^2 t}} \tag{3}$$

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.

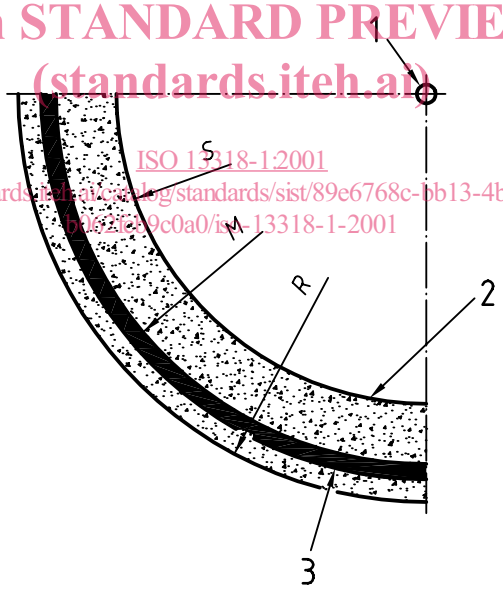
The sample may be introduced either as a thin layer of dispersion on top of the spinning liquid medium, the line-start incremental technique, or uniformly dispersed throughout the sedimentation medium, the homogeneous incremental technique (Figures 1 and 2).



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.