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
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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 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	$\text{kg}\cdot\text{m}^{-3}$	$\text{g}\cdot\text{cm}^{-3}$
Liquid density	ρ_l	$\text{kg}\cdot\text{m}^{-3}$	$\text{g}\cdot\text{cm}^{-3}$
True particle density (no porosity)	ρ_p	$\text{kg}\cdot\text{m}^{-3}$	$\text{g}\cdot\text{cm}^{-3}$
Liquid viscosity	η	Pa·s	mPa·s
Acceleration due to gravity	g	$\text{m}\cdot\text{s}^{-2}$	—
Sedimentation distance	h	m	mm
Sedimentation time	t	s	—
Stokes diameter	x_St	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	$x_{\text{St},h}$	m	µm
Particle diameter entering measurement zone	$x_{\text{St},h\Delta h}$	m	µm
Terminal settling velocity	v	$\text{m}\cdot\text{s}^{-1}$	$\mu\text{m}\cdot\text{s}^{-1}$
Reynolds number	Re	dimensionless	—
Grouped parameter	K_1	m·s	—
Grouped parameter	K_2	$\text{m}^3\cdot\text{s}^{-1}$	—
Hyperbolic scan constant	K_scan	m·s	—
Boltzmann constant	k	$\text{J}\cdot\text{K}^{-1}$	—
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	$h_{\text{zone},P\text{min}}$	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.

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

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.

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

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.