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Determination of particle size distribution by gravitational liquid sedimentation methods — Part 1: General principles, requirements and guidance

Détermination de la distribution granulométrique par les méthodes de sédimentation par gravité dans un liquide — Partie 1: Principes généraux et lignes directrices

Second edition

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 24, Particle ~~characterisation~~ characterization including sieving, Subcommittee SC 4, Particle ~~characterisation~~ characterization.

This second edition cancels and replaces the first edition (ISO 13317-1:2001), which has been technically revised.

The main changes are as follows:

- ~~revision of~~ core terms and definitions have been revised;
- ~~revised and expanded~~ — the explanation of measurement principle and techniques has been revised and expanded;
- ~~inclusion of~~ sedimentation velocity as measurand has been included;
- a guide for the determination of measurement uncertainty has been included.

A list of all parts in the ISO 13317 series can be found on the ISO website.

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Introduction

Gravitational sedimentation has been an established principle of particle size analysis for several decades. It is employed in various academic and industrial fields of application. Numerous national and international standards address ~~the according~~ gravitational sedimentation techniques and analytical methods.

Although manifold new particle sizing techniques have emerged during the last two decades, sedimentation techniques have been recently rediscovered. This is due to substantial technical advancements and the fact that they are based on a first-principle measurement of the particles' directed motion (migration) under gravity.

The measurands of gravitational sedimentation techniques are the distributions of sedimentation velocity and corresponding particle size. They are derived from observations of phase separation – either by monitoring the deposition of particles or the depletion of dispersion. The physical principles employed to determine the quantity of particles differ widely, whereas sedimentation velocity is in each case computed from the vertically migrated distance and measurement time. This computation does not demand essential preconditions and theoretical assumptions. Yet, the transformation of velocity into particle size relies on the applicability of Stokes' law. As fractionating technique, sedimentation analysis facilitates to distinguish between particle fractions of close sedimentation velocity. Accordingly, particle size distributions can be very finely resolved, which is an advantage compared to spectroscopic ensemble techniques.

The ISO 13317 series covers ~~the methods for determining~~ to determine the distributions of sedimentation velocities and particle size of particulate materials by gravitation-induced particle migration in liquids. The direction of this motion depends on the density difference (density contrast) between dispersed and continuous phase. During the measurement, particles should not undergo any physical or chemical change in the continuous phase (liquid).

The primary measurand is the particle velocity distribution, which is converted into size distribution based on established sedimentation theory. The measurement techniques described in this ISO 13317 series are applicable to liquid dispersions, like suspensions and emulsions. The measurable particle size range depends on material properties and typically reaches from 200 nm to 100 µm for aqueous samples, whereas sedimentation velocity can be quantified in the range from 0,6 µm/s to 10 mm/s. Sedimentation analysis is conducted for low particle concentrations. The maximum permissible value depends on ~~the~~ measurement technique and theory of the analysis theory. In general, the volume fraction of particles is well below 1 %.

It is the responsibility of the user of this document to establish appropriate safety and health practices and to determine the applicability of the regulatory limitations prior to its use.

Determination of particle size distribution by gravitational liquid sedimentation methods — Part 1: General principles, requirements and guidance

1 Scope

This document specifies the principles of particle size analysis by gravitational sedimentation, the principal types of measurement techniques as well as the general rules for conducting measurements, method validation, determination of the uncertainty budget and representation of results.

This document covers neither particle migration by centrifugal, electric or magnetic forces nor sedimentation at high particle concentrations (e.g. zone sedimentation). Moreover, ~~this document~~ does not deal with the determination of properties other than sedimentation velocity and particle size (i.e. neither particle concentration, particle shape, particle density, zeta-potential nor apparent viscosity).

NOTE This document can involve hazardous materials, operations and equipment. This document does not purport to address all the safety problems associated with its use. Explosion proof analysers are required when examining volatile liquids with a low flash point.

2 Normative references

~~The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.~~

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

~~There are no normative references in this document.~~

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1

sedimentation

directional motion of *particles* (3.7) in a viscous liquid under the action of gravity or centrifugal fields

Note 1 to entry: For a positive *density contrast* (3.17), sedimentation occurs in the direction of gravitational acceleration; it is counter directed to this acceleration for a negative density contrast.

Note 2 to entry: A downward motion under gravity is also called settling or falling.

Note 3 to entry: An upward motion under gravity is also called creaming (e.g. droplets) or more general rising and floating.

3.2

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migration

directional motion of *particles* (3.7) in a viscous liquid under the action of a force field

Note 1 to entry: Migration in gravitational or centrifugal fields is called *sedimentation* (3.1).

3.3

terminal sedimentation velocity

sedimentation (3.1) velocity in case that gravity or centrifugal force is completely balanced by buoyancy and drag force

3.4

Stokes diameter

equivalent diameter of a sphere that has the same *buoyant density* (3.16) and *terminal sedimentation velocity* (3.3) as the real *particle* (3.7) in the same liquid under *creeping flow* (3.19) conditions

Note 1 to entry: The general rule that the buoyant density (3.16) is used for calculating the Stokes diameter applies also to coated particles or multiconstituent particles (such as droplets in multiple emulsions). The buoyant density (3.16) can be approximated with the *skeleton density* (3.14) for monoconstituent particles.

Note 2 to entry: For porous particles, it is common use to compute particle size based on the *apparent particle density* (3.15). This approach considers the stagnant liquid in the *open pores* (3.9) as intrinsic constituent of the dispersed phase. Thus, the obtained size values are hydrodynamic equivalent diameters.

Note 3 to entry: For closed-packed *agglomerates* (3.8) or aggregates, the buoyant density (3.16) can be replaced by the apparent particle density (3.15) – with particle referring to the agglomerate/aggregate – in order to get the hydrodynamic equivalent diameter.

3.5

shape correction factor

ratio of the *sedimentation* (3.1) velocity of a non-spherical *particle* (3.7) to the one of a spherical particle of the same volume and *apparent density* (3.15)

3.6

hindrance function

ratio of the *terminal sedimentation velocity* (3.3) of a *particle* (3.7) placed in a well-mixed dispersion divided by its sedimentation velocity in an infinite vessel for the absence of other particles

3.7

particle

minute piece of matter with defined physical boundaries

[SOURCE: ISO 26824:2013, 3.1.1, modified — Notes 1, 2 and 3 to entry have been deleted.]

3.8

agglomerate

cluster of *particles* (3.7) held together by weak or medium strong forces with an external surface area, which is similar to the sum of the surface areas of the individual particles

Note 1 to entry: The forces acting between the constituent particles of an agglomerate are relatively weak. They result, for example, from van der Waals attraction or simple physical entanglement.

Note 2 to entry: Agglomerates are also termed secondary particles and the original source particles are termed primary particles.

[ISO 26824:2013, 1.2, modified — the grammatical structure has been changed.]

3.9**open pore**

pore not totally enclosed by its walls and open to the surface either directly or by interconnecting with other pores and therefore accessible to liquid

[SOURCE: ISO 15901-1:2016, 3.11, modified — "fluid" has been replaced ~~by~~ with "liquid" in the definition.]

3.10**closed pore**

pore totally enclosed by its walls and hence not interconnecting with other pores and not accessible to liquids

[SOURCE: ISO 15901-1:2016, 3.10, modified — "fluids" has been replaced ~~by~~ with "liquids" in the definition.]

3.11**dynamic viscosity**

measure of flow resistance for Newtonian liquids calculated as the ratio of the shear stress to the rate of shear for laminar flow exposed to a pre-set shear stress or strain

3.12**apparent viscosity**

measure of flow resistance for non-Newtonian liquids at a defined shear stress or strain calculated as the ratio of the shear stress to the shear rate

3.13**true density of the dispersed phase**

ratio of mass to volume for a body solely consisting of the dispersed phase without pores, voids, inclusions or surface fissures

3.14**skeleton density**

ratio between sample mass and the volume of the sample including the volume of closed pores (if present) but excluding the volumes of *open pores* (3.9)

Note 1 to entry: The skeleton density refers to solid *particles* (3.7) and is determined for samples of dry powder.

[SOURCE: ISO 12154:2014, 3.3, modified — ~~the volume~~ "as well as that" of void spaces between particles within the bulk sample ~~is not mentioned~~ has been deleted from the definition and Note 1 ~~wasto entry~~ has been added.]

3.15**apparent particle density****effective particle density**

ratio of mass to volume for a *particle* (3.7) including particulate inclusions, entrapped stagnant liquid and gas in pores, voids and surface fissures as well as surface layers and coatings

Note 1 to entry: The apparent particle density is the density of a migrating entity and is calculated as the weighted average of its constituents.

Note 2 to entry: The apparent particle density depends on wettability of *open pores* (3.9) and the kinetics of wetting or replacement of pore liquid. Therefore, it is affected by sample preparation.

Note 3 to entry: The apparent particle density is not identical with the *buoyant density* (3.16). They deviate from each other in particular for porous particles and particle *agglomerates* (3.8).

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3.16

buoyant density

ratio of mass to volume for a *particle* (3.7) including particulate inclusions, liquid and gas in closed pores and voids as well as surface layers and coatings but excluding the liquid continuous phase that penetrates *open pores* (3.9)

Note 1 to entry: The buoyant density equals the (hypothetical) density of the continuous phase for which the gravitational force acting on the immersed particle is counterbalanced by buoyancy.

Note 2 to entry: The buoyant density of a particle can be experimentally determined (see ISO 18747-1, and ISO 18747-2).

Note 3 to entry: The buoyant density of monoconstituent particles can be approximated with their *skeleton density* (3.14).

Note 4 to entry: The buoyant density of multiconstituent particles (e.g. coated pigments, droplets of multiple emulsions) can be approximated with the averaged densities of the single constituents.

Note 5 to entry: The buoyant density is affected by the adsorption of dissolved species at the particle surface and therefore depends on the solvent and its composition.

Note 6 to entry: The buoyant density is not identical with the *apparent particle density* (3.15). This holds particularly true for porous particles and particle *agglomerates* (3.8).

3.17

density contrast

difference between the *particle* (3.7) density and the density of the continuous phase

Note 1 to entry: For quantifying the density contrast, one typically refers to the *buoyant (particle) density* (3.16), but for porous particles the *apparent particle density* (3.15) is more appropriate.

3.18

particle Reynolds number

dimensionless parameter expressing the ratio of inertial to viscous forces within a fluid flowing past a *particle* (3.7)

Note 1 to entry: The particle Reynolds number is based on the volume equivalent diameter.

Note 2 to entry: In other contexts, the definition of the particle Reynolds number can refer to different equivalent diameters or to the equivalent radii.

Note 3 to entry: The particle Reynolds number is a characteristic of the flow field and mobility of the particle.

3.19

creeping flow

type of flow that is solely governed by viscous forces and not affected by inertial effects

Note 1 to entry: For moving *particles* (3.7) or for the flow past a particle, the creeping flow condition holds approximately true if the *particle Reynolds number* (3.18) is well below 0,25.

3.20

Brownian motion

random motion of *particles* (3.7) caused by collisions with the molecules or atoms of the surrounding continuous phase

Note 1 to entry: The trajectory of Brownian motion is not differentiable.

Note 2 to entry: Brownian motion results on a macroscopic level in mass transport of the dispersed phase, e.g. in case of diffusion, thermophoresis or photophoresis.

3.21

lower size limit

size of the smallest particles that are detectable and for that, the diffusional particle flux is negligible compared to the sedimentational particle flux

Note 1 to entry: The ratio of sedimentational flux to diffusional flux (also called Péclet number, Pe) should be > 1 .

3.22

upper size limit

size of the largest *particle* (3.7) that satisfies the condition of *creeping flow* (3.19) and of which the *terminal sedimentation velocity* (3.3) is detectable

3.23

type of quantity

specification of the physical property employed to quantify the individual *particle* (3.7) fractions

Note 1 to entry: The type of quantity is a cumulable property of single particles or disperse systems, such as number, mass, intensity of scattered light (within the single scattering limit), light extinction (within Lambert-Beer-limit), refractive index increment or X-ray attenuation.

Note 2 to entry: The type of quantity is indicated by a numerical or character subscript when symbolising the density and cumulative function of a size distribution. Moreover, the subscript also specifies distribution parameters, such as median, mean and modal values or any quantiles.

Note 3 to entry: The following conventions apply for the subscript of geometric or gravimetric properties:

- number: subscript $r = 0$
- length: subscript $r = 1$
- area: subscript $r = 2$
- volume or mass: subscript $r = 3$

Note 4 to entry: The following conventions apply for the subscript of physical properties:

- light extinction: subscript toq = "ext"
- light intensity: subscript toq = "int"

~~[SOURCE: ISO 9276-1:1998, 4.3, modified — the definition and notes have been expanded to include frequently used non-geometrical quantities.]~~

3.24

sensitivity

change of instrument response with respect to changes in concentration or absolute quantity of *particles* (3.7) in a specified size class

Note 1 to entry: A concentration or quantity can be given in relative or absolute values in dependence on the detection aim.

Note 2 to entry: Sensitivity depends on the *type of quantity* (3.23).

Note 3 to entry: Sensitivity is a function of size.

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3.25

limit of quantity detection

smallest quantity of specified *particle* (3.7) size class, for which the instrument response can be distinguished from the background

Note 1 to entry: It depends on, for example, the size range, on precision, noise level, smoothing algorithms.

Note 2 to entry: It affects the lower and upper size limit.

3.26

measurement uncertainty

uncertainty of measurement

parameter associated with the result of a measurement that characterises the dispersion of the values that can reasonably be attributed to the measurand

[SOURCE: ISO/IEC Guide 98-3:2008, 2.2.3], modified — the term "measurement uncertainty" has been added.]

4 Symbols and abbreviated terms

For the purposes of this document, the following symbols given in Table 1 apply.

Table 1 — Symbols and descriptions

Symbol	Description	Unit
Ar	Archimedes number	dimensionless
$bias$	systematic deviation of measured value from true value	varying
C	transformation coefficient, see Formula (32) Formula (34)	$m^{0.5} \times s^{0.5}$
C_D	drag coefficient	dimensionless
$C(x)$	concentration density	varying
c_M	concentration with respect to extensive property M	varying
D	diameter of a spherical particle	m
d	diameter	m
D_p	particle diffusion coefficient	$m^2 \times s^{-1}$
F_D	drag force (also: hydrodynamic resistance)	N
g	gravitational acceleration	$m \times s^{-2}$
h_{sed}	sedimentation distance	m
k	coverage factor	dimensionless
k_B	Boltzmann constant	$J \times K^{-1}$
L	length	m
L_j	Ljaščenko number	dimensionless
M	extensive property indicating the amount of dispersed phase	varying
m	number of bias determinations	dimensionless
N	number of replicate analyses	dimensionless
P	cumulative function	%
Pe	Péclet number	dimensionless

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Q_{toq}	cumulative function of a distributed quantity, index “toq” indicates the type of quantity, in which the fractions are weighted	dimensionless
q_{toq}	density function of a distributed quantity, index “toq” indicates the type of quantity, in which the fractions are weighted	varying
Re_p	particle Reynolds number	dimensionless
S	saturation of open pores with continuous phase	dimensionless
s	standard deviation	varying
T	absolute temperature	K
t_{sed}	sedimentation time	s
U	expanded uncertainty	varying
u	uncertainty	varying
V_{sed}	terminal sedimentation velocity	$\text{m} \times \text{s}^{-1}$
x	particle size (equivalent diameter)	m
x_{Stokes}	Stokes diameter	m
x_V	volume equivalent diameter	m
z	Cartesian coordinate in vertical direction, vertical position	m
$\Delta\rho$	density contrast	$\text{kg} \times \text{m}^{-3}$
$\dot{\gamma}$	shear rate	s^{-1}
$\delta\hat{d}_{\text{layer}}$	layer thickness	m
$\epsilon_{\hat{d}_x}$	porosity relative error of the calculated particle size	dimensionless
η_{eE}	viscosity of the continuous phase	$\text{Pa} \times \text{s}$
$\rho_p \rho_c$	particle density	$\text{kg} \times \text{m}^{-3}$
$\rho_c \rho_d$	particle density of the continuous phase	$\text{kg} \times \text{m}^{-3}$
$\rho_a \rho_c$	true density of the dispersed continuous phase	$\text{kg} \times \text{m}^{-3}$
$\rho_p \rho_d$	particle true density (generic) of the dispersed phase	$\text{kg} \times \text{m}^{-3}$
φ_V	volume fraction	dimensionless

In addition, for the purposes of this document, the following subindices are frequently employed: subscripts apply.

aggl	agglomerate, also aggregate	-
app	apparent	-
bouy	buoyant	-
c	combined	-
incl	inclusion	-
lab	laboratory	-
max	maximum	-
occl	occluded pores/voids	-
open	open pores	-
ref	Reference	-

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rel	Relative	-
rep	repeatability	-
Rw	reproducibility	-
sk	skeleton	-
sus	suspension	-
toq	type of quantity	-
tot	total	-

Moreover, for the purposes of this document-uses, the following abbreviated terms apply.

ALS	angular light scattering
DLS	dynamic light scattering
CRM	certified reference material
FBRM	focussed beam reflectance method
HSM	homogeneous-start method
LSM	line-start method
PTA	particle tracking analysis
QCM	quality control material
RM	reference material
SOP	standard operating procedure

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5 Measurement principle and technical realisations

5.1 General measurement principle

Gravitational liquid sedimentation techniques are established tools for the characterisation of suspensions and emulsions. They quantify the separation of particles (the dispersed phase) from the continuous phase (also called: dispersion medium or dispersion liquid) under the presence of gravity. This phase separation relies on the directional, migratory motion of each particle, called sedimentation. Its rate, the sedimentation velocity, depends on the particle size and thus offers a chance for the granulometric characterisation of particle systems.

NOTE 1 Gravitational liquid sedimentation techniques are generally called "sedimentation techniques" in this document.

Sedimentation occurs for any particle dispersed in a quiescent viscous liquid, as long as a density contrast exists. It is driven by gravity, which acts on the particle (weight) and the displaced liquid (buoyancy). The resulting net force (excess force) causes a migratory particle motion, which is retarded by a frictional force (also called the drag force), which increases linearly with the sedimentation velocity and eventually leads to a steady-state of zero net force, at which particles move with a terminal sedimentation velocity (see [Reference \[39\]](#), chapter 6.4). Theoretically, terminal velocity is never reached. Sedimentation time to reach 99 % of the terminal velocity is generally very fast and depends on the ratio of particle density and liquid viscosity and the particle size squared for particle systems and operational conditions considered within this [standard document](#) (i.e. for creeping flow). It amounts to 0,2 μ s and 49 ms for spherical gold particles ($r_p = 19\ 300\ \text{kg/m}^3$) of 0,3 μ m and 100 μ m, respectively, settling in water. If the excess force is positive (i.e. weight **>is greater than** buoyancy), the particle motion is called falling or settling. In the opposite case, it is called rising, creaming or floating.